

BATCH DISTILLATION

version 97.5

I. STARTUP PROCEDURE

A. Fill Reboiler

1. Remove the thermometer.
2. Confirm that the reboiler clamp is positioned right side up. Turn the screw counter-clockwise to loosen, and squeeze the clamp to remove.
3. Holding the top of the reboiler, turn the jack screw counter-clockwise to lower the reboiler heater.
4. Continuing to support the reboiler, carefully wiggle it until it comes free from the column.
5. Fill the reboiler with the initial charge of feed (approx 1.5L is recommended).
6. Verify that a light film of vacuum grease is on the joint where the column connects to the reboiler.
7. Being careful that the reboiler goes on straight, that the thermometer opening is facing out of the way, and that the sampling valve is easy to reach, turn the jack screw clockwise to raise the reboiler until it is just snug with the column.
8. Reinstall the reboiler clamp right side up. Turn the screw clockwise until it is snug.
9. Reinsert the thermometer, **DO NOT OVERTIGHTEN**.
10. Confirm that the reboiler sampling valve is closed.

B. Checklist

1. Confirm that water is flowing through the condenser.
2. Confirm that the pressure relief cork is lightly inserted in position.
3. Notice the direction in which the thermometer sticks out of the reboiler to avoid breaking it during the experiment.
4. Verify that there is a distillation containment vessel (of sufficient size to contain your expected product) in position under the product cooler.

C. Reflux Mode Selection

Total Reflux mode

1. Verify that the distillate valve is CLOSED

2. Verify that the power to the Reflux Controller is OFF

Normal Reflux mode

1. Verify that the distillate valve is OPEN
2. Verify that the power to the Reflux Controller is ON. There is a switch for this on the power switch and also one on the left side of the controller.
3. Adjust the Reflux Controller to the desired setting.
 - (a) Open the glass cover
 - (b) Set the amount of time for Reflux by positioning the left arm. The farther to the left from the zero position (see the inside scale) the more time for reflux.
 - (c) Set the amount of time for Product by positioning the right arm. The farther to the right from the zero position (see the outside scale) the more time for product.

It is desirable for the column to switch frequently between the reflux and product modes in order to more closely approximate a steady state condition.

D. Powering Up

1. Verify that all the switches on the power strip are off.
2. Plug in the black cord labeled "Batch".
3. Turn ON the MAIN POWER switch.
4. Turn ON the REFRACTOMETER switch.
5. Turn ON the COLUMN LIGHT switch.
This allows you to see the boiling behavior on the trays through the slit running down the front of the column.
6. Turn ON the HEATER POWER switch.
7. ADJUST the power output (on the reboiler power control knob) between 0 and 100.