

CATALYTIC METHANATION START UP & SHUT DOWN PROCEDURES

Ch En 479

Startup

Three main ideas govern the startup procedures.

- A. The heater should never be turned on without gases flowing.
- B. The flow of H₂ should never be less than stoichiometric.
- C. The total flow of all gases during a run should sum to 200 SCCM.

Following the procedures below will ensure compliance with these principles.

1. Open the valve on the top of the CO₂ cylinder all the way and then close a quarter/half turn.
2. Open the valve on the top of the CO₂ regulator all the way and then close slightly.
3. Ensure the valves (top of cylinder and top of regulator) for He are opened properly.
4. Lift and hold open the window on the blue cabinet with the H₂ cylinder.
 - a. Open the valve on the top of the H₂ cylinder all the way and then close a quarter/half turn.
 - b. Open the valve on the top of the H₂ regulator all the way and then close slightly.
5. Open the LabView control panel for the unit using the Pale Moon browser. (Do not use Chrome or any other browser.)
6. Ensure the *line* pressures for all cylinders are about 40 psi.
7. Ensure the *tank* pressures for all cylinders are above 100 psi.
8. Turn on the black, 4-outlet, power strip on the back of unit to turn on the mass flow controllers.
9. Navigate to the appropriate Catalytic Methanation Control Page on the UO Lab webpage.
10. Right click on the control panel and select "Request Control."
11. Set the flowrates of H₂, CO₂, and He to the desired values.
 - a. The sum of the flowrates should equal 200 SCCM.
 - b. When changing flowrates, never do an order where there is less than stoichiometric H₂.
This may require temporarily setting the CO₂ flowrate to 0.
12. Ensure the Reactor Pressure is set to 18.4 psia (1.25 atm) and that the controller is in Auto mode.
13. Set the Temperature to the desired value.
14. Ensure the "Reactor Exhaust" is selected for the GC Analysis Stream.
15. Turn on the heater by clicking the appropriate switch.
16. Allow the system to reach steady state.
17. Take data as desired.

Data Collection

- The outlet concentrations are obtained from the GC as areas of peaks.
 - Refer to the video on how to run the GC.
 - Refer to the document "Gas Chromatography" to understand the peaks.
 - Obtain the calibrations for the GC for CO₂ and CH₄ from the website.
- Data from the GC must be recorded manually into a lab book.
- Data from the LabView may be recorded automatically and copied into the lab book.

Follow the steps below to record data from LabView at regular time intervals.

1. Click the "Replace File" button to clear the file saved from previous data recording sessions.
2. Set the time between points (in seconds)
 - a. The box is titled "Recording Increment (sec)"
 - b. Set this to approximately 1-2 seconds.
 - i. Shorter or longer intervals are possible.
 - ii. The data are written in chunks of approximately 63 lines. The file will be empty until at least 63 samples have been taken. If you set the recording increment to 1 second, you will need to wait about 1 minute before seeing any data.
3. Press the red "NOT RECORDING//Push to Record" button. The box will turn green, and its label will change to "Recording//(Push to Cancel)."
4. Obtain the data by scrolling to the bottom of the page and clicking "Get Data File (.csv)".
 - a. You will be prompted to open the file in Excel. Click "OK."
 - b. Data will be appended to the bottom of the file in chunks of 63 lines.
 - c. The file will continue to grow in length if you are recording data.
 - d. You can obtain the data while recording is occurring *without* stopping the collection.
 - e. Unless the "Replace File" button is clicked, newly recorded data will append to the bottom of the file.
 - f. You can start and stop recording data without affecting the data in the file.
5. Copy the data in the opened Excel file into your lab book or save the Excel file onto your J-drive with a new name.
 - a. The file obtained by clicking the link at the bottom of the page will be overwritten if the "Replace File" button is clicked by you or another group.
 - b. Your data will be lost unless you copy or move it to a new location.

Shutdown

Three main ideas govern the shutdown procedure.

- A. The heater must cool down with gases flowing.
- B. The flow of H_2 should never be less than stoichiometric.
- C. A small amount of helium should always flow to the GC.

Following the procedures below will ensure compliance with these principles.

1. Leave the flowrates at the last setpoint.
 - a. The flow of H_2 should be at least stoichiometric.
 - b. The total flow should be 200 SCCM.
2. Turn off the heater on LabView.
3. Set the reactor temperature to 0 °C.
4. Wait for the reactor to cool below 200 °C.
5. Set the flowrates of all gases to 0 SCCM.
6. Shut the cylinder and regulator valves on the H_2 and CO_2 .
7. Leave the cylinder and regulator valves on the He **open**.
8. Right click on LabView and select "Release Control."
9. Shut down Pale Moon and log off the computer.