

SOP

**Hydrogen Uptake System &
Mini Reactor**

Schlaf Group 2011

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Introduction to the Hydrogen Uptake System, Mini Reactor and Control Tower

THIS SOP IS NOT MEANT TO REPLACE HANDS-ON TRAINING FROM QUALIFIED PERSONNEL!

This SOP describes the basic procedure for operating and maintaining the hydrogen uptake system as well as the mini reactor and URC tower associated with it. This SOP will also give a brief insight into a few of the common problems arising through use of the system as well as solutions to these problems.

1 General Comments and Introduction

Treat the pressure reactor and its components with respect at all times. If you are unsure, or feel uncomfortable, while operating the reactor system, STOP and ask for assistance from qualified personnel.

The volume of the reactor vessel is 50 mL. The maximum reaction volume is 25 mL, the minimum reaction volume to allow stirring is 15 mL.

The reactor has a burst disc rated for 1750 psi at room temperature. Maximum working pressure should be no more than 1400 psi.

The hydrogen reservoirs attached to the uptake system have a burst disc rated for 1900 psi at room temperature. Maximum working pressure should be no more than 1800 psi.

Familiarize yourself with the Pressure Reactor Checklist, posted in the hydrogenation lab and be sure that you understand all safety precautions when working with hydrogen gas and pressurized systems.

Familiarize yourself with the mini reactor documentation and explosion drawings of the reactors. This information can be found in the binders located in the hydrogenation lab and is essential for a full understanding of the reactors. These diagrams are also necessary for trouble shooting various problems and replacing damaged parts within the reactor.

Spare parts (ferrules, nuts, O-rings, bearings, etc) are located in a plastic storage unit. These parts should be routinely checked and restocked as necessary from Autoclave Engineers. The catalog numbers are labeled on the front of the container containing the spare part. Exception, Kalrez O-rings are ordered from Daemar Inc. (product number 029-K-4079). A sample order form for these O-rings can be found on the bench top in the hydrogenation lab (MACN 342).

2 The Uptake System

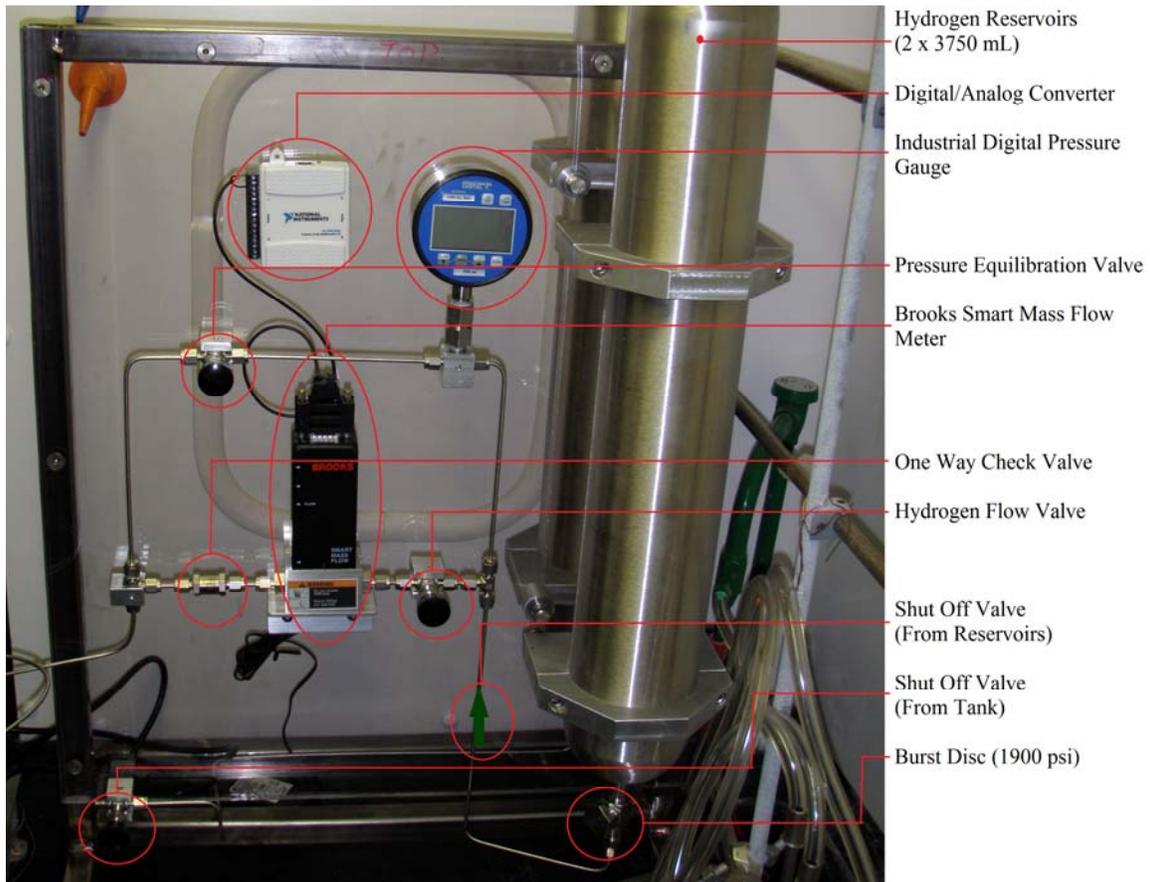


Figure 1: Hydrogen uptake system

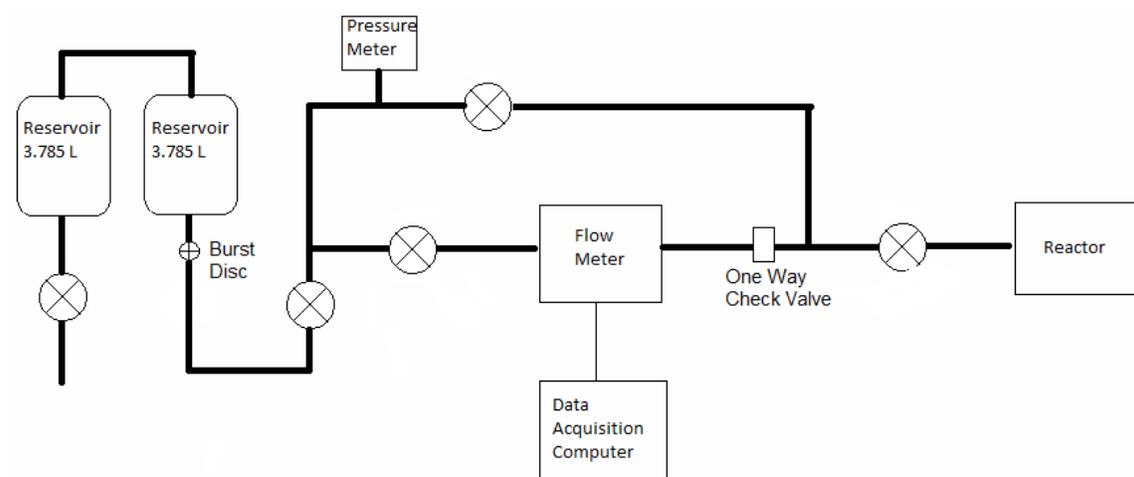


Figure 2: Schematic Diagram for the hydrogen uptake system

2.1 Set-Up

Note: The shut-off valve from the reservoirs (green valve) remains open at all times unless performing maintenance to the uptake system (changing batteries, changing mass flow meter, fixing leaks etc.). Pressurizing while this valve is closed can result in over pressurizing the reservoirs as the digital pressure gauge is at this time not reading the true pressure in the reservoirs !

1. Ensure all valves to and from the uptake system are CLOSED. Turn on the industrial digital pressure gauge found on the uptake system. Flip the main power switch, located on the rear of the control tower.
2. Assemble the reactor vessel containing substrate solution and catalyst by screwing the reactor vessel into the body.
3. Open the main valve to the hydrogen tank, listen for leaks before opening the valve from the regulator. Listen for leaks between the regulator and the shut-off valve to the hydrogen uptake system.
4. Open the shut-off valve from the tank into the uptake system. Monitor the pressure via the industrial digital pressure gauge. Fill approximately 15-20 psi above the desired set point of the reaction to be run.
5. Upon reaching desire pressure, close the shut-off valve from the tank. Close the regulator valve as well as the main valve to the hydrogen tank.
6. The pressure of the reservoir will fluctuate depending on how quickly the reservoir was filled, the pressure will drop slightly as the temperature returns to ambient. Allow the digital meter to equilibrate and ensure no leaks are present (a more rapid decrease in pressure indicates a leak in the system). LEAKS: Use snoop to find by checking all joints and valves.
 - a. If a leak is found; close the shut-off valve from the reservoirs and vent the system. Tighten the fitting(s) affected. Re-pressurize and check for leaks. Repeat until no more leaks are present. NEVER WORK ON A PRESSURIZED SYSTEM.
7. Open the pressure equilibration valve leading to the reactor.
8. Open the reactor valve connected to the uptake system and allow the reactor to reach pressure slowly. Ensure the reactor has no leaks by once again using snoop solution.

- a. If leak is found; close reactor valve leading from uptake system and vent the reactor. Tighten the necessary fitting(s). Re-pressurize as above and check for leaks. NEVER WORK ON A PRESSURIZED SYSTEM.
9. Vent the reactor to ~100-125 psi and evacuate the reactor using the water aspirator for approximately 2 minutes. Close the valve, remove the vacuum hose, and turn off the water to aspirator. (Turning off water before removing hose can result in water being sucked into the reactor!) DO NOT CARRY OUT THIS STEP IF YOUR SOLVENT OR SUBSTRATE ARE VOLATILE.
10. Repeat pressurization/evacuation cycle twice more.
11. During the last pressurization, at about 10 psi from the desired set point, slowly open the Hydrogen Flow Valve located beside the Mass Flow Meter in order to fill the line.
12. Record both the initial pressure of the reactor, the uptake system as well as the temperature from the reactor control tower.

2.2 Setting Temperature and Mixing Speed

1. Ensure the high temperature shutdown is set no higher than 350°C. Set this value according to the reaction being run (approximately 25% higher than the reaction temperature). To set, press and hold the red “set” button on the “Overtemperature” panel. Press the grey “loop” button twice until “TP-H” appears. Set the temperature using the blue arrows. Return the main screen by pressing “set” once.
2. Flip the two main power switches “heater” and “mixer” and ensure both values for the Process Temperature and Mixer Speed are set to “0”.
3. Set the temperature to the desired value using the blue arrows, the number will flash quickly until set. When the desired temperature is reached press “set”.
4. Set the mixer speed by setting the mixer speed to a minimum value of 500 rpm. Again, the value will flicker lightly until set. When the desired mixing speed is reached press “set”.
5. You should see the mixer speed quickly ramp to the desired rpm and fluctuate around the desired value. The Overtemperature panel should quickly begin to climb in temperature, the Process Temperature more slowly. With increasing Process Temperature the Process Pressure value should also increase.
6. Wait until the set temperature has been reached and ensure it goes no higher and that the pressure remains within a safe range (see introduction). Record the temperature and pressure from the control tower, turn off the digital pressure gauge on the uptake system.

BEFORE LEAVING THE HYDROGEN LAB: be sure the main valve on the hydrogen tank is close, the fumehood sash is lowered and the reactor is operating properly (no vibrations, noises etc.)

3 The Multi-Range Hydrogen Flow & Mass Recorder Program

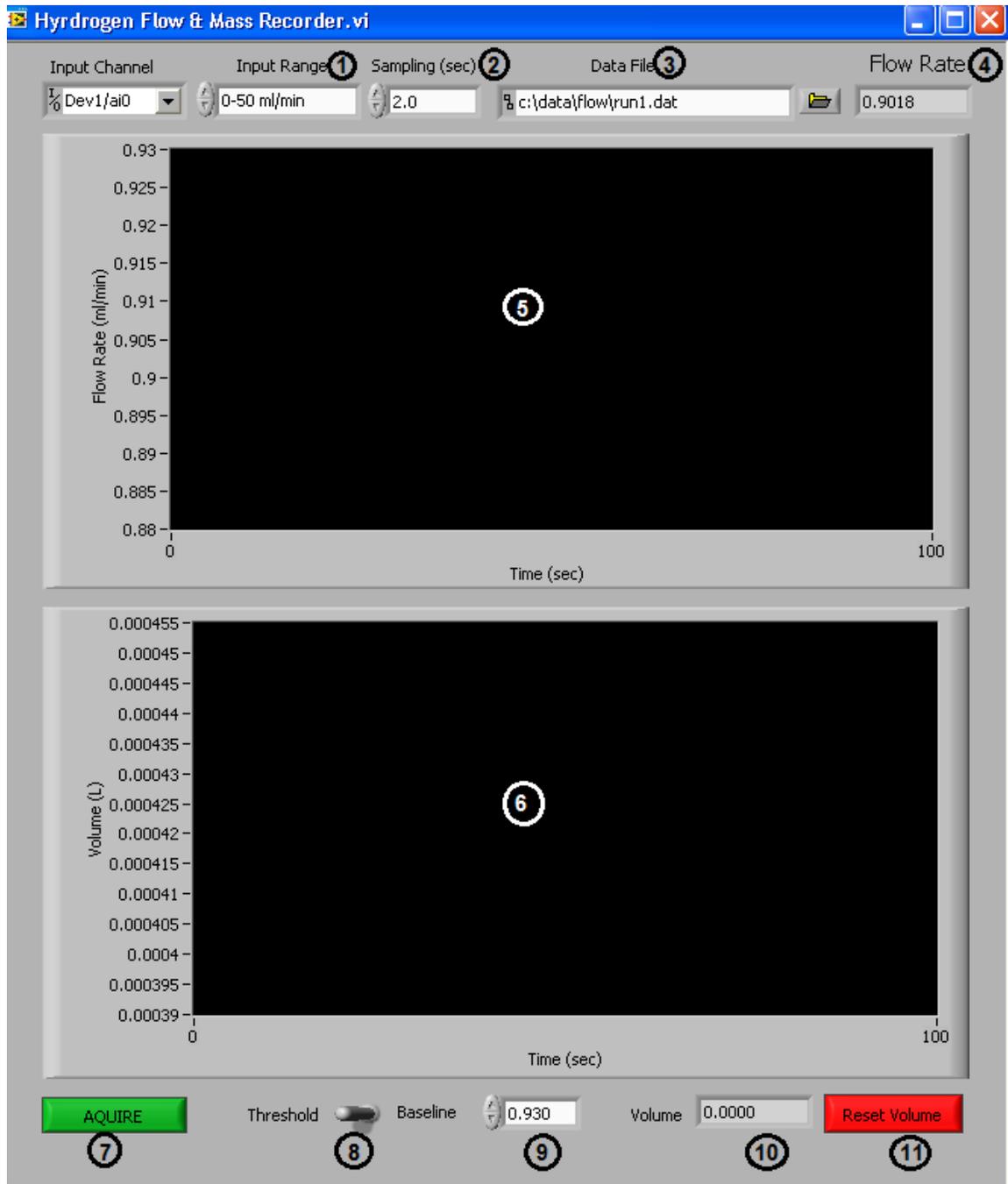


Figure 3: Multi-Range hydrogen flow and mass recording program.

Table 1: Components of the multi-range hydrogen flow & mass recorder program

	Name	Description
1	Input Range	The input range is selected based on the calibration level of the flow meter being used. In the figure illustrated above, the flow meter is calibrated for a flow of 0-50 mL/min.
2	Sampling Rate	The frequency at which flow measurements are taken during the experiment. Above, the rate is set for one measurement every 2 seconds.
3	Data File	Experiments are saved as data files in the location illustrated in the below window. Before beginning a new experiment replace “run1” with the name of your experiment.
4	Flow Rate	This value is a real time measurement of the current flow rate through the mass flow meter.
5	Flow Rate Plot	Plot of the Flow Rate (mL/min) vs Time (sec)
6	Volume Plot	Plot of Total Volume of Hydrogen (L) flowing through the mass flow meter over Time (sec)
7	Acquire	Acquisition command to commence mass flow rate measurements
8	Threshold/Baseline	Determines how the Flow Rate data is integrated into the Volume Plot
9	Threshold Value	Set the value for the threshold or baseline cut off
10	Volume	Total volume as measured over the course of the experiment
11	Reset Volume	Resets the volume plot and value to zero

Figure 3 and Table 1 give a general description of the mass & flow recorder program. The following illustrations describe the use of the Threshold/Baseline function. The Threshold/Baseline function is useful in cases when the Flow Rate is particularly low and accuracy begins to decrease. At low Flow Rates the program may read large variations in flow due to background noise or other factors. The sum of these inaccuracies can lead to large discrepancies between the actual and theoretical mass flow values for a given experiment.

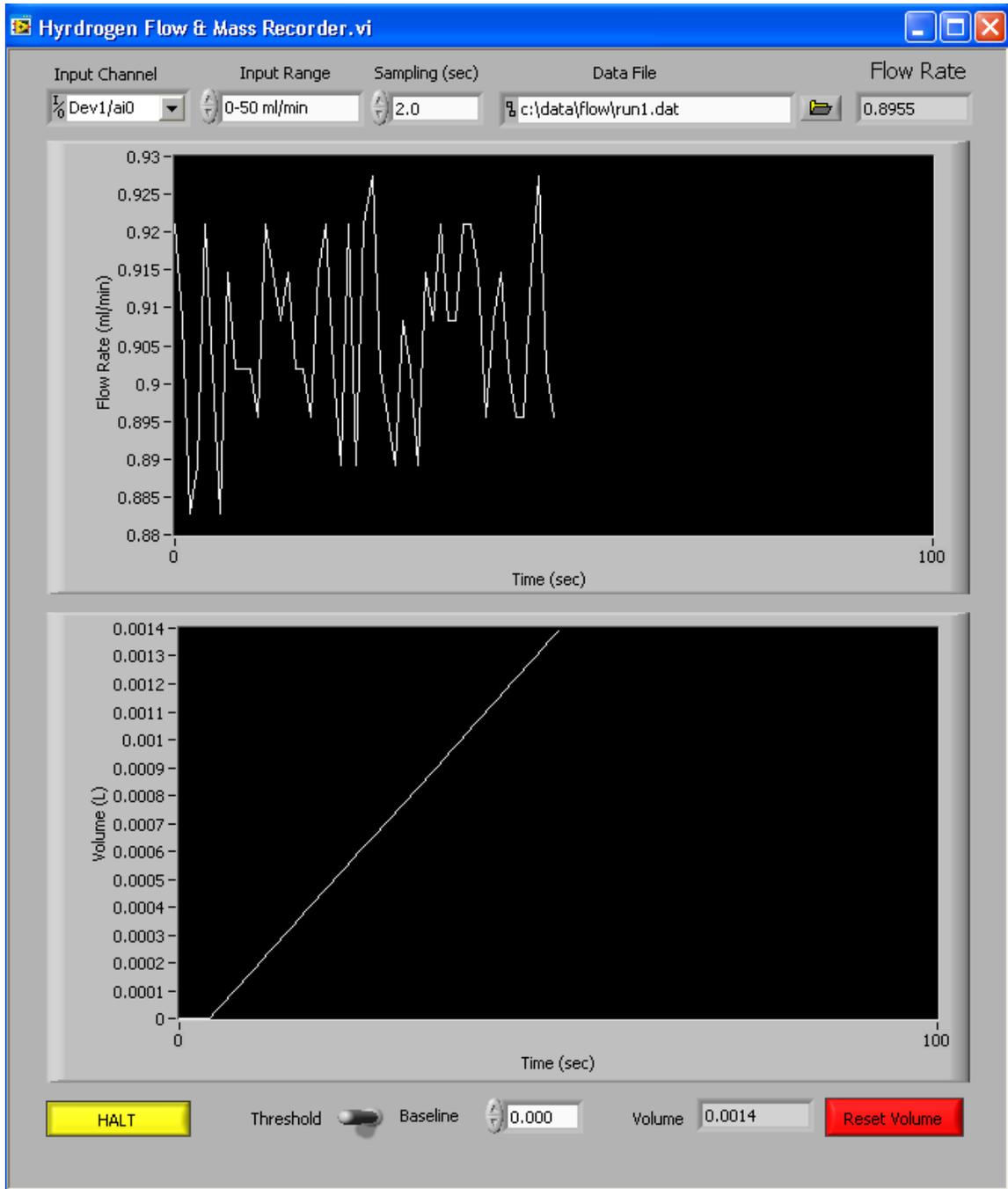
The flow rate may not read zero depending on the calibration of the Mass Flow Meter, but as long as the value is below 1 and constant you are able to proceed with the experiment. Adjust for a flow rate above zero by using either a Threshold or Baseline function. Pressing the Threshold/Baseline “switch” flips between the two options.

The use of **Threshold** function allows the operator to set all Flow Rate integrations for values below a set limit to zero. This is valuable when considering the lower limit for the

accuracy of a given mass flow meter and setting a threshold for all values less than this accuracy setting.

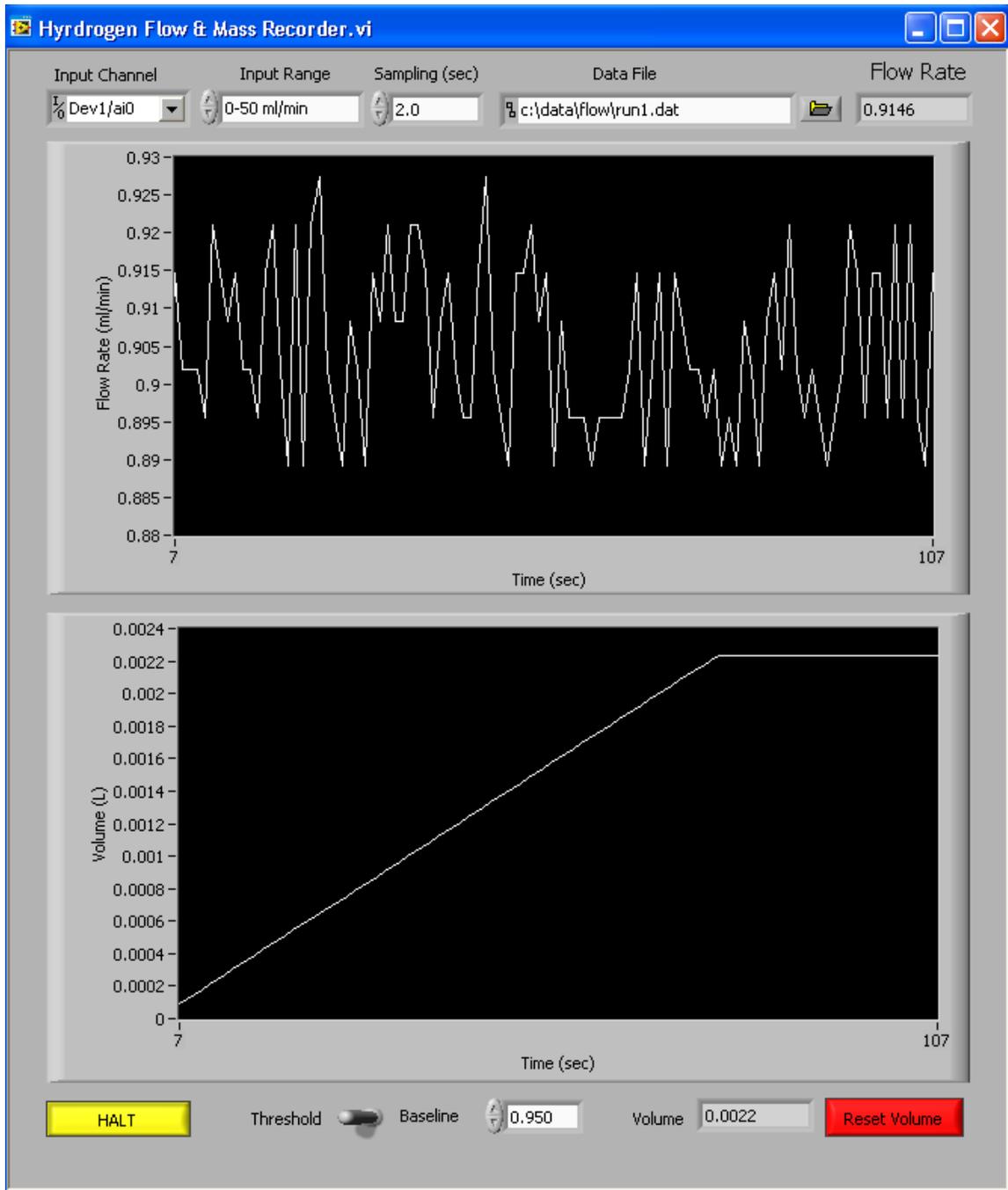
The **Baseline** function allows the operator to integrate all Flow Rate values as both positive and negative values in the Volume Plot such that inaccuracies due to fluctuations “balance out” over the course of an experiment.

3.1 Threshold Set to Zero



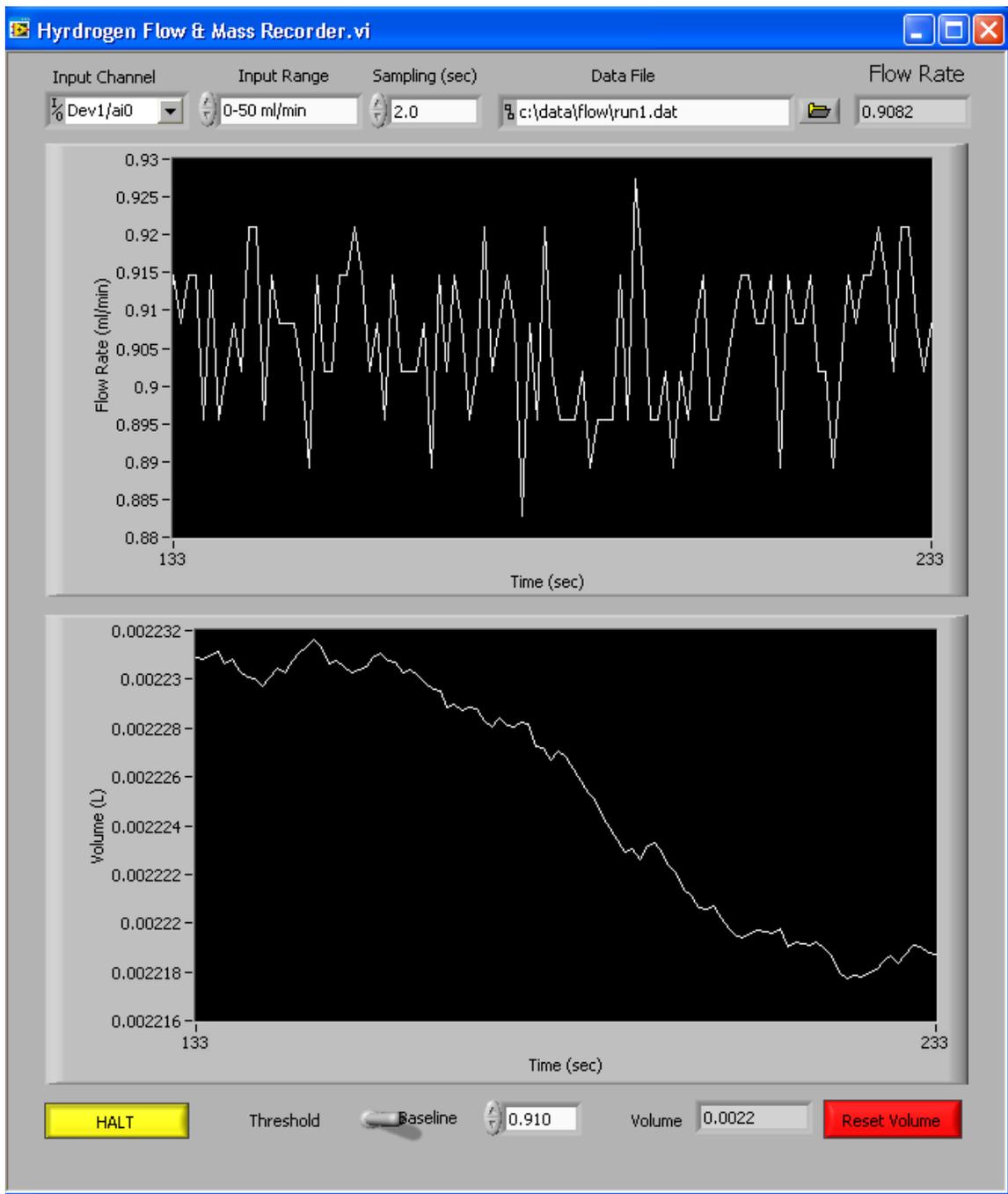
With the threshold value set to zero all Flow Rate values larger than zero will be integrated as a positive volume on the Volume Plot and all Flow Rate values less than and equal to zero are integrated as zero on the Volume Plot.

3.2 *Set Threshold Value*



When a threshold value is set (eg. 0.950 mL/min as above) all Flow rate Values above 0.950 mL/min are integrated as positive values on the Volume plot, while all values less than and equal to 0.950 mL/min are integrated as zero on the Volume Plot. In the above figure, the threshold was set to 0.950 mL/min in the middle of the experiment and as such the Volume Plot now shows a flat curve with no increase in volume despite the flow rate of 0.9146 mL/min.

3.3 Set Baseline Value



When the Baseline is set, Flow Rate values above the Baseline value are integrated as positive values on the Volume plot, while all Flow Rate values below the Baseline value are integrated as negative values on the Volume Plot. The figure above illustrates how the Volume Plot now illustrates increases and decreases in total hydrogen flowing through the system and into the reactor.

4 Operating the Uptake System

The hydrogen uptake system can be operated in two ways, Totalizer mode and Dynamic mode.

1. The Totalizer mode allows for accurate measurements of the total hydrogen taken up by the substrate over the course of the reaction as well as a measure of the total virial pressure drop from the digital pressure gauge. The Totalizer mode is suitable as a starting experiment for gaining a basic understanding of the uptake system while still gaining valuable information about the hydrogen uptake by the substrate.
2. The Dynamic mode allows not only for the accurate measurements of the total hydrogen uptake by the substrate over the course of the reaction as well as the virial pressure drop but also plots the real time rate of reaction during the hydrogenation. The Flow Rate plot illustrates an accurate representation of the rate of uptake by the substrate and also indicates when the reaction is complete when the flow rate drops to zero and the volume plot no longer increases. The Dynamic mode cannot be used if the experiment of interest experiences significant/substantial hydrogen uptake during the temperature climb to the set temperature and pressure.

The upcoming section gives a detailed description for the operation and understanding of the uptake system for both modes of operation. Preceding this section, the reactor should be pressurized and leak tested as illustrated in section **2.1 Set-Up**.

4.1 Totalizer Mode

1. With the reactor fully pressurized and leak tested, follow the instructions in section **2.2 Setting Temperature and Mixing Speed** and set the desired temperature and mixer speed on the control tower.
2. Record the initial temperature and pressures from both the control tower as well as the digital pressure gauge.
3. Allow the reactor to reach the set temperature, monitoring the pressure such that it remains within a safe range of the burst rating. Record the maximum temperature and pressure reached by the reactor.
4. Once the reaction has completed, turn off the mixer by first setting the mixer speed to “0” with the blue arrows and pressing the red “set” button. Allow the mixer speed to come down to zero and the mixer to stop before turning off the main power breaker. **DO NOT TURN OFF THE BREAKER BEFORE SETTING THE MIXER SPEED TO ZERO.**
5. Follow the same procedure for the temperature control, set the temperature to “0” with the blue arrows and press the red “set” button, once the reactor starts to cool it is safe to turn off the main power breaker.
6. Allow the reaction to cool to room temperature, this can be achieved faster by **CAREFULLY** removing the heating jacket and cooling with an ice bath.
7. Record the current temperature and pressures on both the control tower as well as the digital pressure gauge
8. Open the Hydrogen Flow & Mass Recorder Program, be sure that the Input range is set to the correct value for the flow meter being used and that the volume is zeroed.
9. Set the desired Threshold or Baseline as illustrated in section **3 The Multi-Range Hydrogen Flow & Mass Recorder Program**
10. Change the data file name to the name of your experiment by replacing “run1.dat” (eg., FW-II-55.dat)
11. Start the acquisition by hitting the green “ACQUIRE” button the Mass/Flow Program. The flow rate may not read zero depending upon the calibration of the Mass Flow Meter, take note of this and adjust using a Threshold or Baseline Function.

12. Slowly open the Hydrogen Flow Valve (if this was not done during pressurization a large peak may result as the line is filled, the volume can be reset by pressing the red “Reset Volume” button on the Mass/Flow Program).
13. **SLOWLY** open the valve to the reactor, carefully monitoring the Mass/Flow Program for spikes in the Flow Rate plot. You must be careful not to exceed the flow rate limit of the Mass Flow Meter or inaccurate values will result.
14. Carefully maintain a flow rate between 40-90% of the upper limit of Mass Flow Meter in order to achieve maximum accuracy from the Mass Flow Meter.
15. Once the re-pressurization has completed close the valve to the reactor as well as the Hydrogen Flow Valve on the uptake system. Uptake is complete when the pressure of the reactor matches the pressure of the uptake system and the Flow Rate on the mass/flow program falls to and maintains a reading of “0” or the original flow rate observed when the acquisition began.
16. Stop the acquisition by pressing the yellow “HALT” button the mass/flow program
17. Record the final pressures from the control tower and the digital pressure gauge as well as the volume from the mass/flow program.
18. Using a balloon, obtain a gas sample of the head space. Vent the reactor into the fume hood.
19. Disassemble the reactor.
20. Plotting and acquiring data from the mass/flow program will be covered in section **4.3 Data Acquisition**.

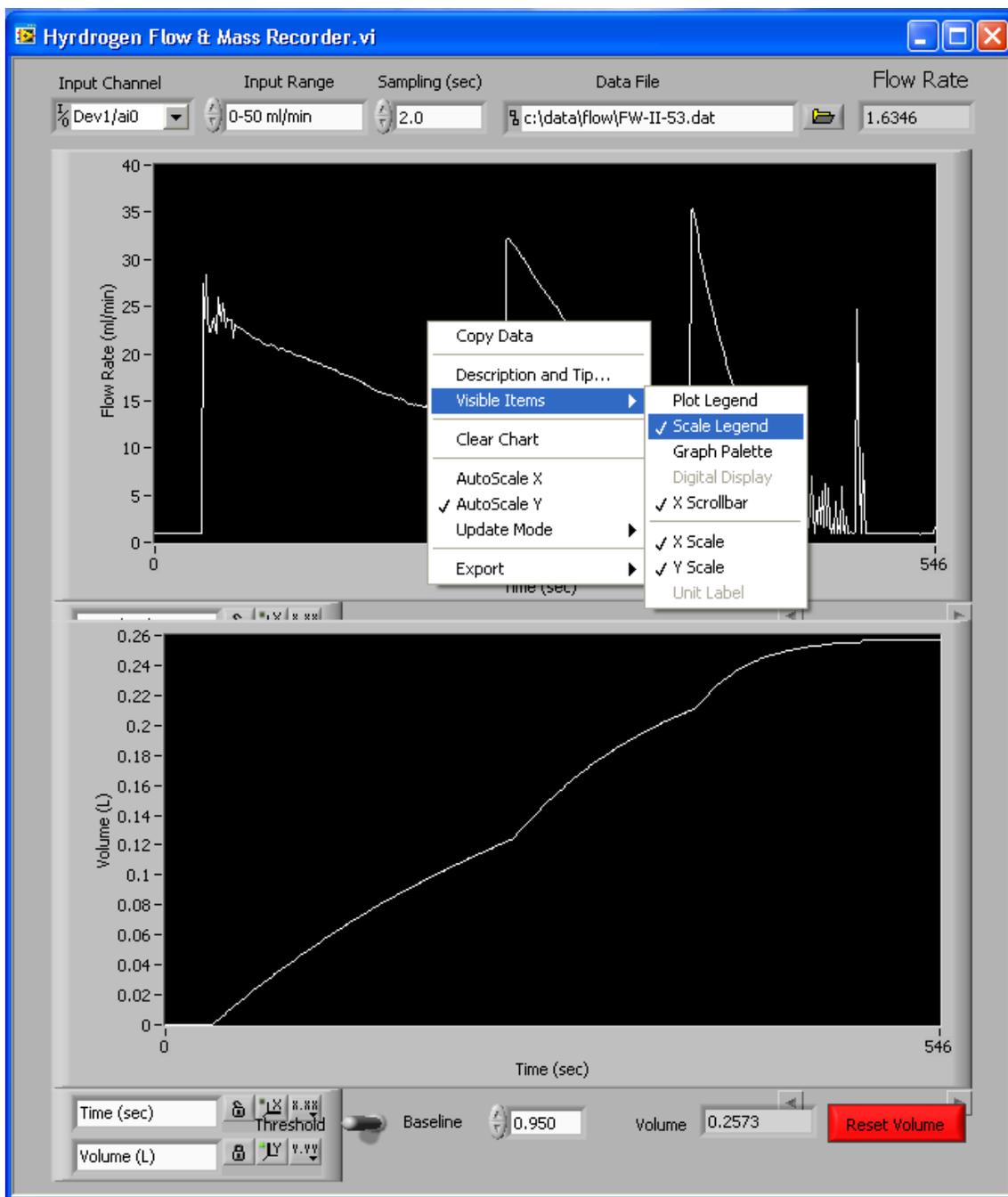
4.2 *Dynamic Mode*

1. Open the Hydrogen Flow & Mass Recorder Program, be sure that the Input range is set to the correct value for the flow meter being used and that the volume is zeroed.
2. Set the desired Threshold or Baseline as illustrated in section **3 The Multi-Range Hydrogen Flow & Mass Recorder Program**.
3. Change the data file name to the name of your experiment by replacing “run1.dat” (e.g., FW-II-55.dat).
4. With the reactor fully pressurized and leak tested, follow the instructions in section **2.2 Setting Temperature and Mixing Speed** and set the desired temperature and mixer speed on the control tower.
5. Record the initial temperature and pressures from both the control tower as well as the digital pressure gauge.
6. Allow the reactor to reach the set temperature, monitoring the pressure such that it remains within a safe range of the burst rating. Record the maximum temperature and pressure reached by the reactor.
7. Quickly, but carefully, fill the reservoirs of the uptake system to the same pressure as the reactor as indicated on the control tower. Record the pressure on the digital pressure gauge once it stops fluctuating.
8. Start the acquisition by hitting the green “ACQUIRE” button the Mass/Flow Program. The flow rate may not read zero depending upon the calibration of the Mass Flow Meter, take note of this and adjust using a Threshold or Baseline Function.
9. Slowly open the Hydrogen Flow Valve (if this was not done during pressurization a large peak may result as the line is filled, the volume can be reset by pressing the red “Reset Volume” button on the Mass/Flow Program).
10. **QUICKLY**, but carefully, open the valve to the reactor, carefully monitoring the Mass/Flow Program for spikes in the Flow Rate plot. The Flow Rate will quickly spike before re-equilibrating and returning to the true rate of uptake.
10. Allow the reaction to run until the Flow Rate on the mass/flow program falls to and maintains a reading of “0” or the original flow rate observed when the acquisition began.

11. Close the valve to the reactor as well as the Hydrogen Flow Valve on the uptake system.
12. Stop the acquisition by pressing the yellow “HALT” button the mass/flow program
13. Record the final pressures from the control tower and the digital pressure gauge as well as the volume from the mass/flow program.
14. Turn off the mixer by first setting the mixer speed to “0” with the blue arrows and pressing the red “set” button. Allow the mixer speed to come down to zero and the mixer to stop before turning off the main power breaker. **DO NOT TURN OFF THE BREAKER BEFORE SETTING THE MIXER SPEED TO ZERO.**
15. Follow the same procedure for the temperature control, set the temperature to “0” with the blue arrows and press the red “set” button, once the reactor starts to cool it is safe to turn off the main power breaker.
16. Allow the reaction to cool to room temperature, this can be achieved faster by **CAREFULLY** removing the heating jacket and cooling with an ice bath.
17. Using a balloon, obtain a gas sample of the head space. Vent the reactor into the fume hood.
18. Disassemble the reactor.
19. Plotting and acquiring data from the mass/flow program will be covered in section **4.3 Data Acquisition.**

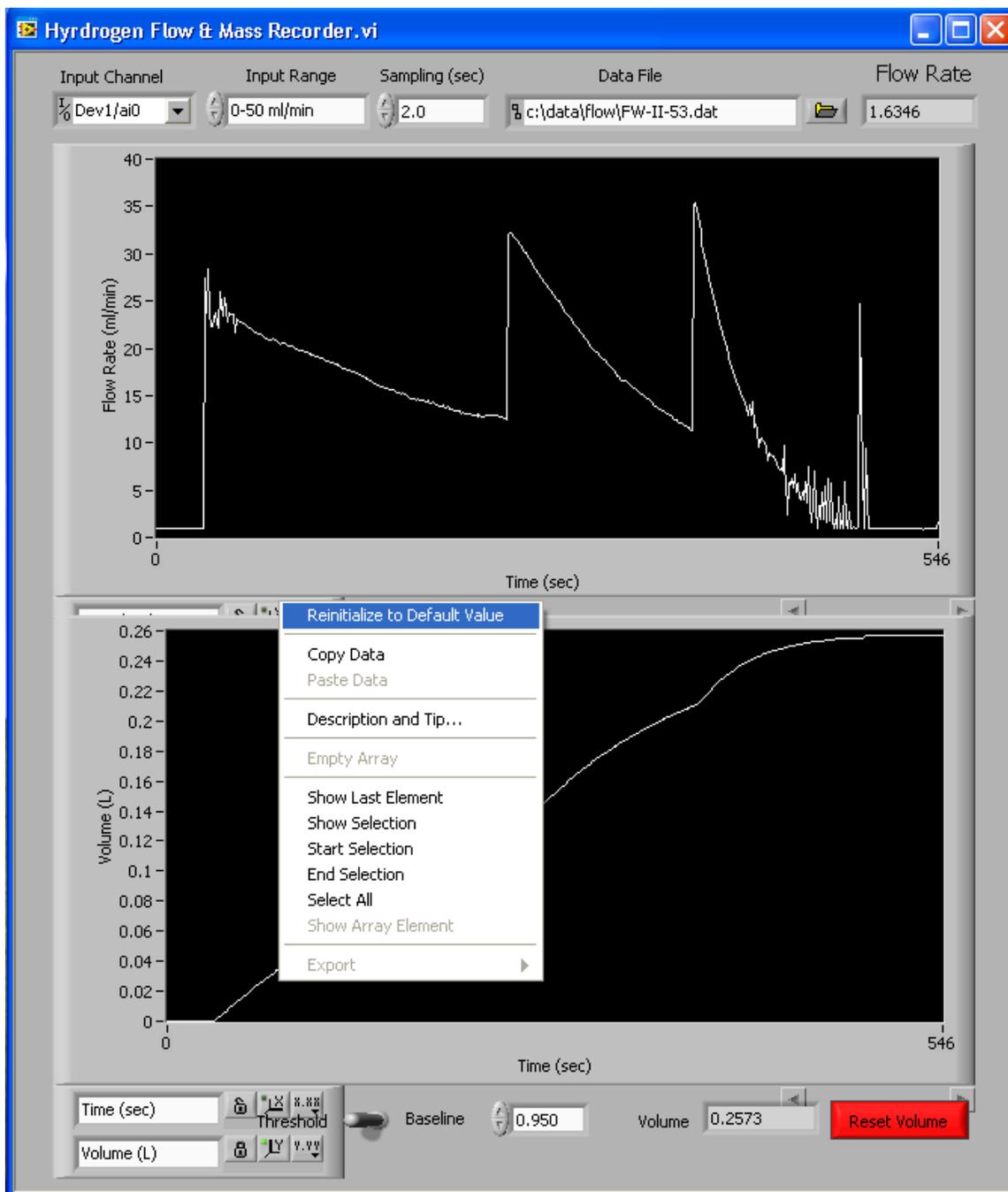
4.3 Data Acquisition

Following a reaction using the hydrogen uptake system two plots will be available for analysis, the Flow Rate plot and the Volume plot. Depending the type of reaction run, the length of the reaction may extend beyond the visible area of the x-axis which is set for 100 seconds. In order to re-scale the x-axis, right click the plot (example below is for the Flow Rate Plot), select “Visible Items” and “Scale Legend”.



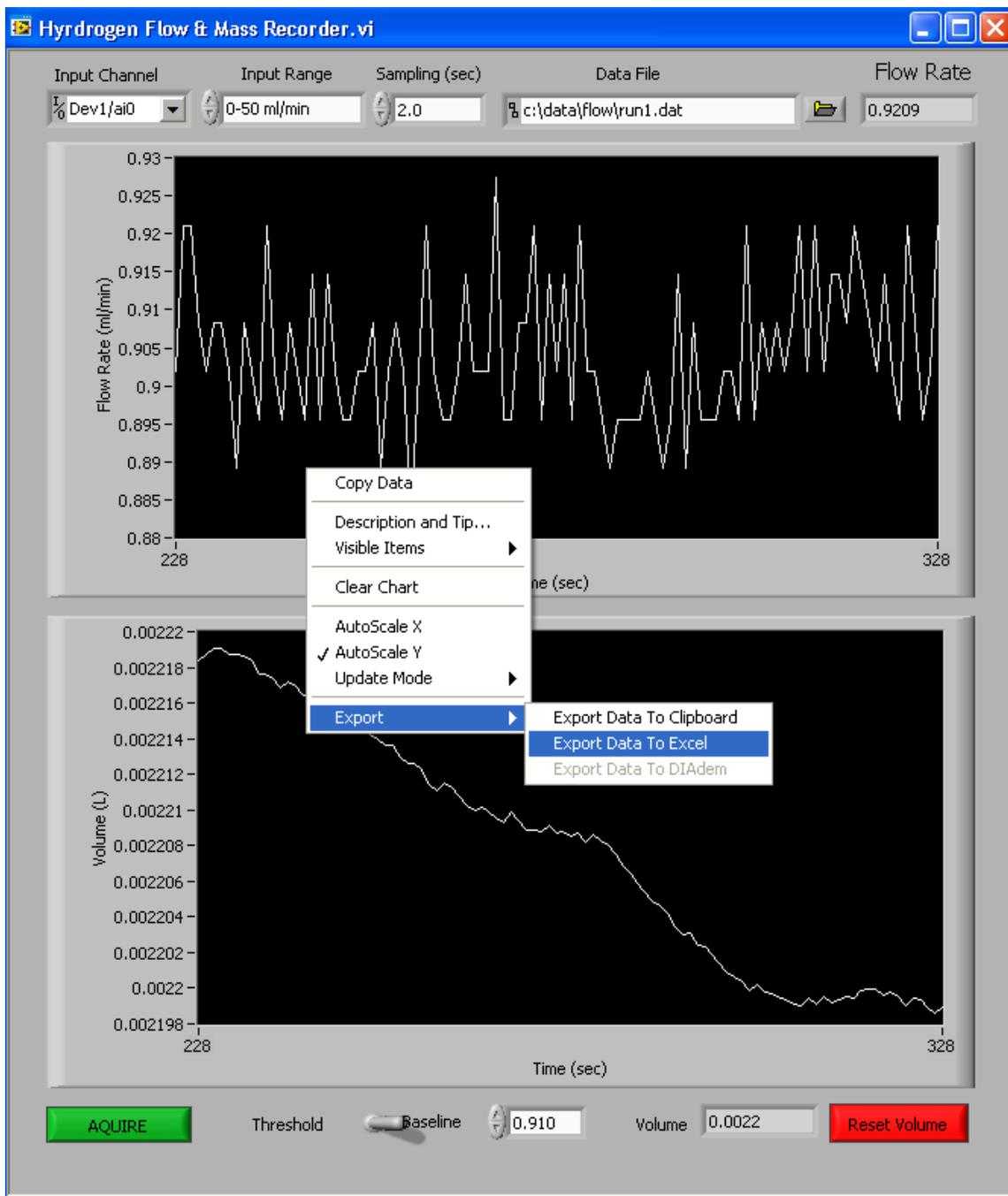
A new legend should appear for the plot, the one for the Flow Rate Plot may be difficult to see.

Click the “X” button, located beside the “Time (sec)” section of the legend. The Plot will then fit to screen but may be too large to fit in the visible area of the plot. To correct this, right click on the “X” button, and select “Reinitialize to Default Value”



The entire experiment should now be visible on a single plot as illustrated above. This procedure must be repeated for each plot individually.

Once the entire experiment is visible on both plots, the data can be exported directly to Microsoft Excel. This is done by right clicking the plot, selecting “Export” followed by “Export Data To Clipboard”. With Excel open, paste the data for both plots.



The original plot itself can also be exported by selecting the “Copy Data” option. Pasting into Excel or Word will reveal the black and white plot as seen on the Mass/Flow Program.

5 Maintenance of the Uptake System

Routinely the uptake system may require minor maintenance such as replacing batteries for the digital pressure gauge, replacing the mass flow meter, or sealing leaks in one of the various lines. Regardless of the maintenance to be done, begin by closing the green Shut-Off Valve from the reservoirs and venting the line, both through the Equilibration Valve as well as the Hydrogen Flow Valve.

LEAKS: Routinely check for leaks using Snoop, once the line has been vented tighten the necessary fittings and re-pressurize by opening the green Shut-Off Valve.

BATTERIES: The batteries for the digital pressure gauge will die over time and require replacing. Use the appropriate Allen key, found in the tool box in the reactor room, to loosen the tubing from the plexi-glass backboard and proceed to remove the pressure gauge from the line. Replace the batteries located at the back of the gauge and replace the gauge to the line. **BE SURE TO PRESSURE TEST THE LINE AFTER REPLACING THIS GAUGE.**

FLOW METER: First loosen the Hydrogen Flow valve from the plexi-glass backboard using an Allen key. With the line loose, unscrew the mass flow meter from the line as well as from the metal baseboard it is resting on (these screws are located underneath the flow meter). Reassemble the new flow meter and tighten the Hydrogen Flow Valve back onto the plexi-glass backboard. **BE SURE TO PRESSURE TEST THE LINE AFTER REPLACING THIS METER.**

BURST DISC: Have the machine shop replace the burst disc if it blows.

All enquiries involving the general operation and maintenance of the reactor or control towers should be directed to either the “Reactor SOP’s” or the Autoclave Engineers CD manual located in the hydrogenation lab.