GeoSoilEnviroCARS
The University of Chicago

Argonne National Laboratory • Advanced Photon Source

GAS LOADING SYSTEM

Instructions for standard operation
&
Operation log

Introduction

In the gas loading system, the DAC is inserted in a holder which acts as a clamp: the DAC is closed by pushing the top and bottom surfaces against each other. The closed holder is then taken off the system and the DAC screws have to be closed through holes in the holder.

The pressure is checked using the ruby fluorescence from the side of the DAC with the screws, so place the ruby chip on the proper side for opaque samples.

Take into account the compressibility of the pressure medium when preparing your sample chamber. To ensure quasi-hydrostatic conditions the sample (particularly single crystals) should be nicely surrounded by the medium. The sample chamber shrinkage with pressure depends on the diamonds size and shape and on the type of gasket used. He is the most compressible among the gases we are loading, we observed a reduction of more than 50% of the gaskethole diameter at 10 GPa.

GAS LOADING SYSTEM

Short instructions

Plese remember to **fill the operation log** for every loading.

First of all Safety !

- you have to be an **Authorized Users for gas loading and laser operation**
- **wear safety goggles!**
- **delimit the working area with proper signs** on the floor

DAC & cell holder

- **DAC's screws should not stick out** of the cell surfaces
- make sure you have **no set screws**
- the total height of the cell holder should not be greater than **88 mm**,
- leave ~**1 mm** gap between the cell holder piston and the body
- make sure that you have **access to the DAC's screws**

Loading Gas

make sure both large and lecture **gas bottles are open** before start the compressor

1) Fill the lecture bottle

a) Fill the lecture bottle directly from the large gas cylinder (you can skip this operation if the pressure on the gas bottle is already quite high, around 1600PSI):

- **close V4**, **close V3, close V5**, **open V2**, **open V6**, and finally **open V1**
- When the lecture bottle has finish filling (low pressure gauge has stopped increasing) then **close V1**

b) Fill the lecture bottle from the large gas cylinder with the compressor

- Ensure that the regulator on the large gas cylinder is set to 1500PSI or less; **close V4, close V5, close V2, open V3, open V6,** and **open V1**. **Turn on the compressor**
- When the lecture bottle has finished filling (low pressure gauge has stopped increasing) **turn off the compressor**, **close V1**, and **close V3**

2) Pressurize the pressure vessel

- equalize the pressure at the lecture bottle and at the pressure vessel: **close V6, closeV7, Open V3** and **V5**
- when the pressure is equalized, **Close V3** and **Open V2**
- **Turn on the compressor**. Take note of the starting time of pressurizing. When the pressure reaches the target value (5 to 15 minutes) **turn off the compressor, close V5** and wait for one minute, checking for leaks (high pressure gauge decreasing). Contact GSECARS staff in case of leaking. Fill out in the operation log the pressurizing time and the pressure after compression in the lecture bottle.

Close the cell

- Rotate **the motors together** in little steps (increase)
- Use the video system and ruby fluorescence system to determine when the cell is sealed and the desired pressure is reached.

Recapture gas and Vent the pressure vessel

- **Open V3**, wait for the pressure of the low pressure system to equalize with that on the high pressure system (flat graphs)
- **Close V2**, **Close V3**, **Close V5**, **Open V6**.
- **Wait for the vessel to be fully vented**, high pressure meter value is below 190 PSI, then you can open the door and remove the cell

Close the DAC

- The DAC is now kept closed by the cylinder, **close the DAC screws**.
- You can now remove the DAC from the holder if you are confident of having closed the DAC with the screws. Otherwise
- reload the cylinder into the pressure vessel. Tip: if you can't put the cell smoothly back, turn the hex screws back by about 0.05.
- move the together motor back to the original position while checking eventual P variations. If the P drops too much, you may have to close the DAC's screws more

Please acknowledge GSECARS and COMPRES for the use of the system.

Close the Gas bottles, turn off laser and the microscope light when you are done. Thank you!

GAS LOADING SYSTEM

Instruction manual

These notes are meant to be useful for the standard gas loading procedure we are carrying out, which involves a symmetrical DAC and He or Ne gases. Please add your notes when you'll find anything incorrect or unclear, it will help us keeping instructions updated.

Please contact GSECARS people anytime before operating the system. Please check with GSECARS staff if your DAC is of different type and if you want to use a different gas than the one installed (among those allowed: He, Ne, Ar, Kr or Xe).

Please don't forget to fill out the **operation log** for our record and statistics.

Please acknowledge GSECARS and COMPRES for the use of the system.

a) Before starting

General Safety Rules

1- Only persons who have been trained in the use of the gas loading system, and whose names are on the list of **Authorized Users** may use the gas loading system. Users must agree that they understand this document, and will following these standard operating procedures. In order to use the laser on the ruby fluorescence system users must also read the standard operating procedures for the laser, and have their names on the separate list of authorized users for the laser system. You will be allowed to operate the laser in the user mode only.

2- ***Personnel Protective Equipment:*** Users must wear **safety goggles** when any component of the gas loading system is pressurized above 1500 PSI.

3- ***Allowed gases***

The only that may be used in the system are the following noble gases:

- He
- Ne
- Ar
- Kr
- Xe

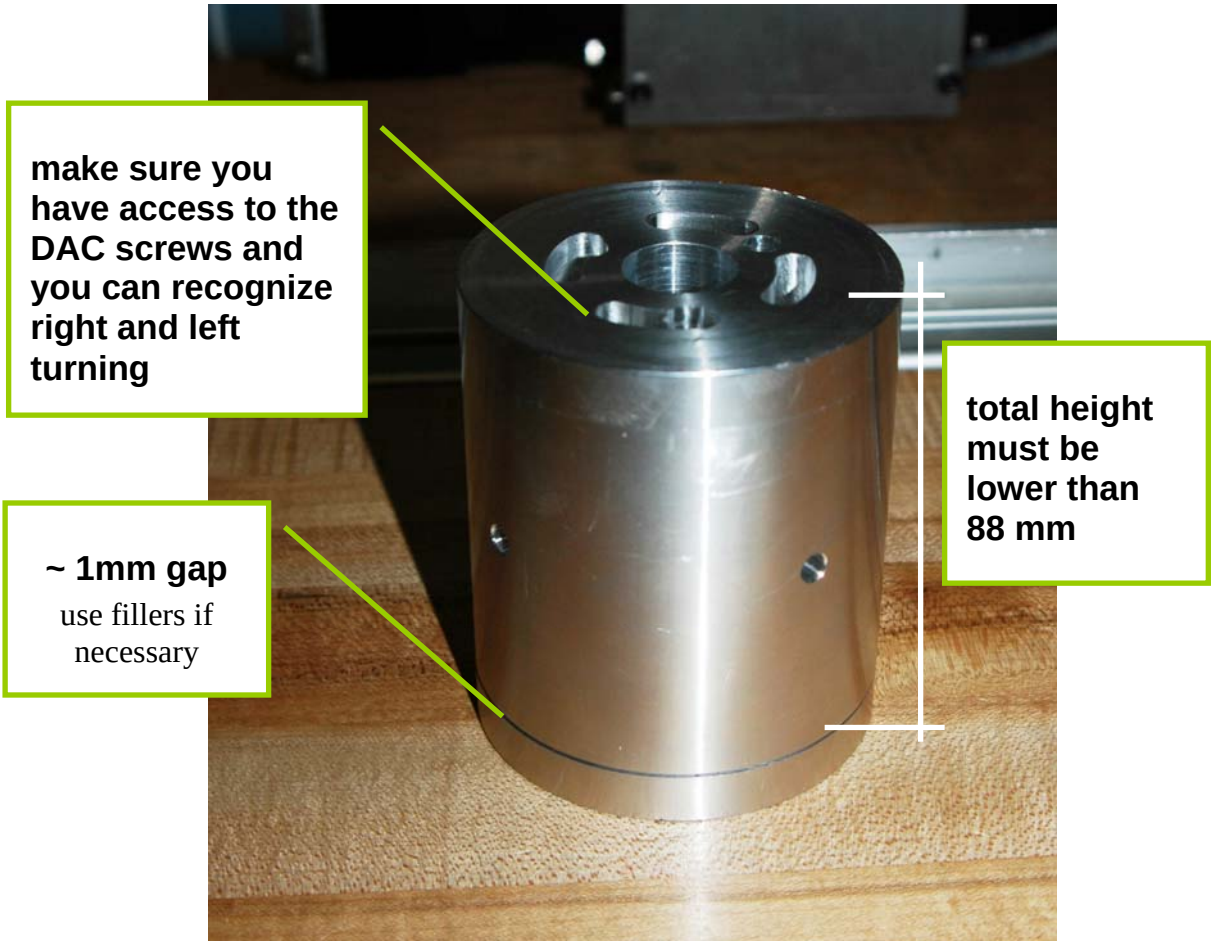
Only non-radioactive isotopes of these gases may be loaded.

Other gases may be allowed in the future, and these will be added to this list. It is forbidden to load any gas that is not listed in this section.

3- When pressurizing the system, only authorized users can stay close to the apparatus. Please place proper **signs** on the floor to delimit the working area.

DAC and cell holder

1- Currently, we can load symmetrical DACs, modified symmetrical and 4-pins. The last two DAC types needs adaptors to fit the cell holder, some adaptors are already available (please check with GSECARS staff).
2- **DAC's screws should not stick out** of the cell surfaces (or out of the adaptors), neither at the top nor at the bottom
3- make sure you have **no setting screws**
4- After having placed the DAC in the cylinder, the total height of the cell holder should not be greater than **88 mm**, and there should be some gap remaining (~**1 mm**) between the cell holder piston and the body to allow the DAC to be closed. Add some spacers between the DAC's bottom and the holder cylinder if necessary.
5- Place your DAC inside the cell holder, make sure that you have **access to the DAC's screws** from the top of the cylinder with allen wrenches.

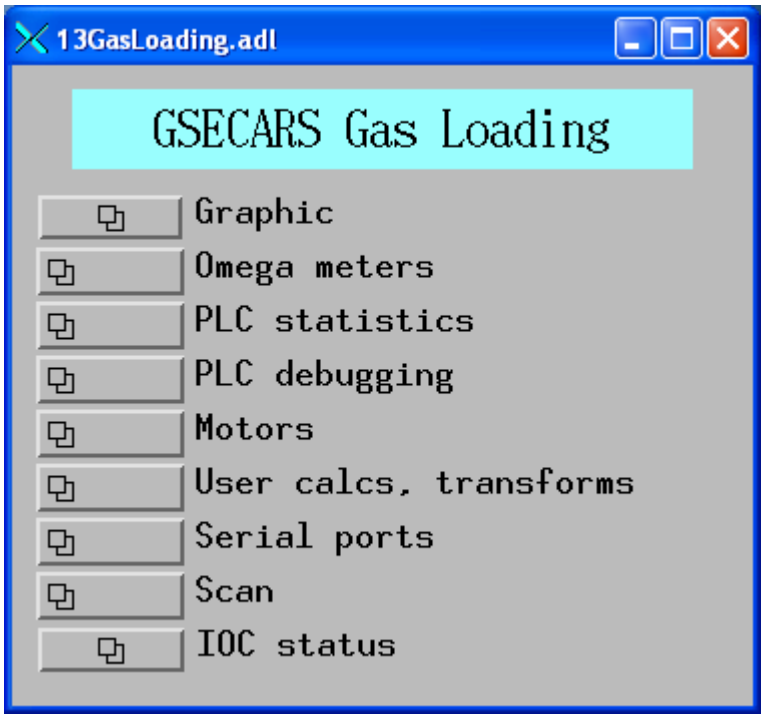


b) Overview of system controls

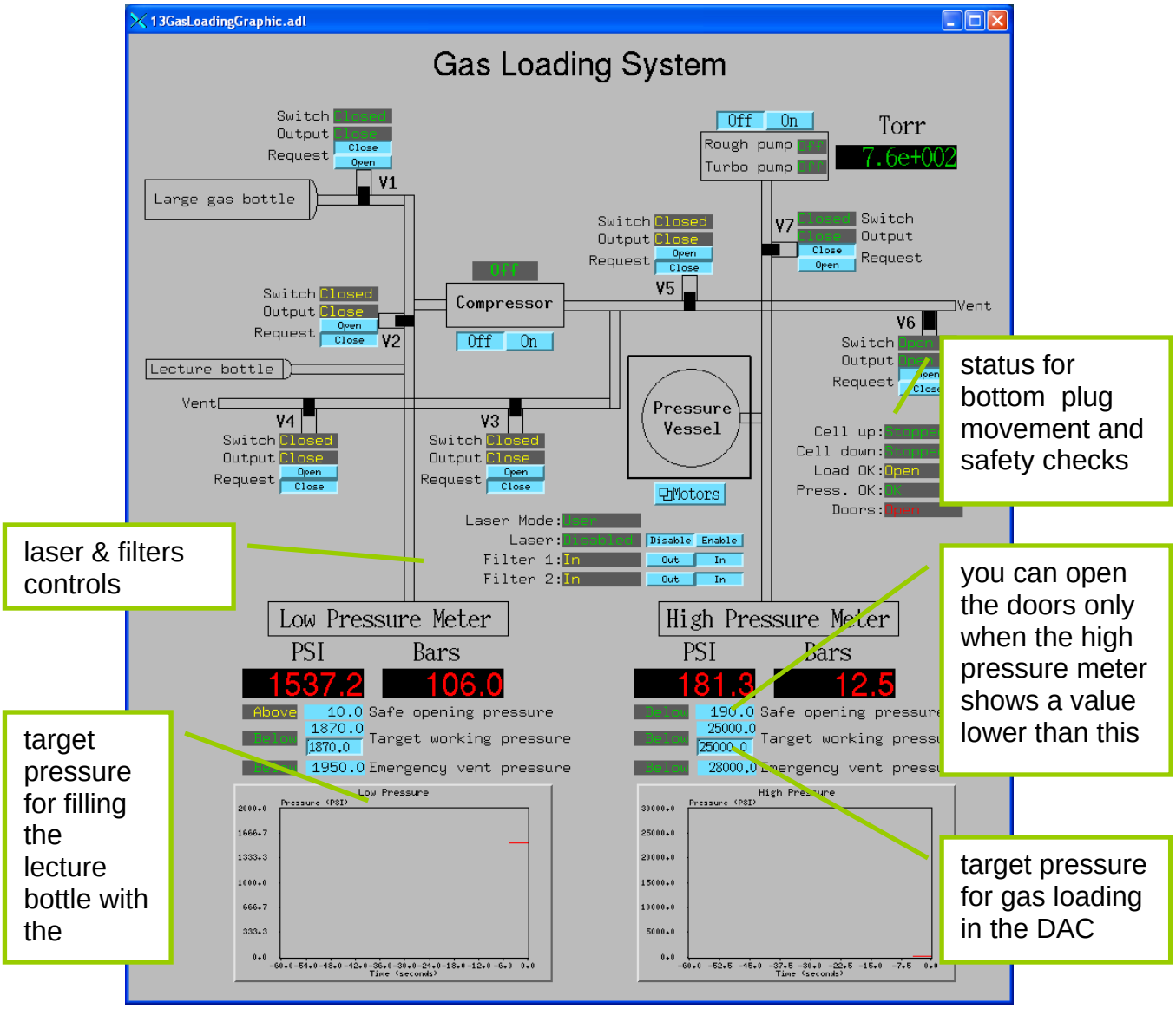
1) EPICS control

The MEDM Gas Loading control

Start the main control window double clicking on the “Epics Gas Loading” shortcut on the desktop



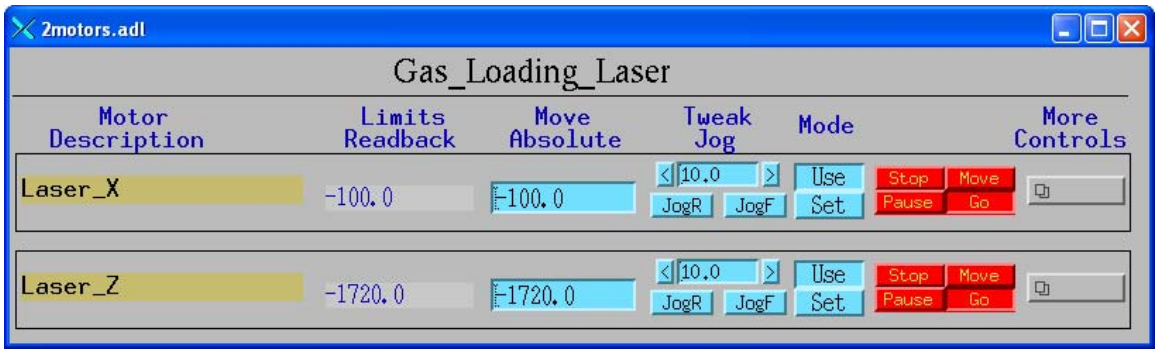
select “Graphic”, the following window will show up:



This window allows you to operate the valves, the compressor, and observe the pressure variations which are monitored in two distinct parts of the system. It is important to **observe how pressure changes** during the loading, particularly while operating the compressor.

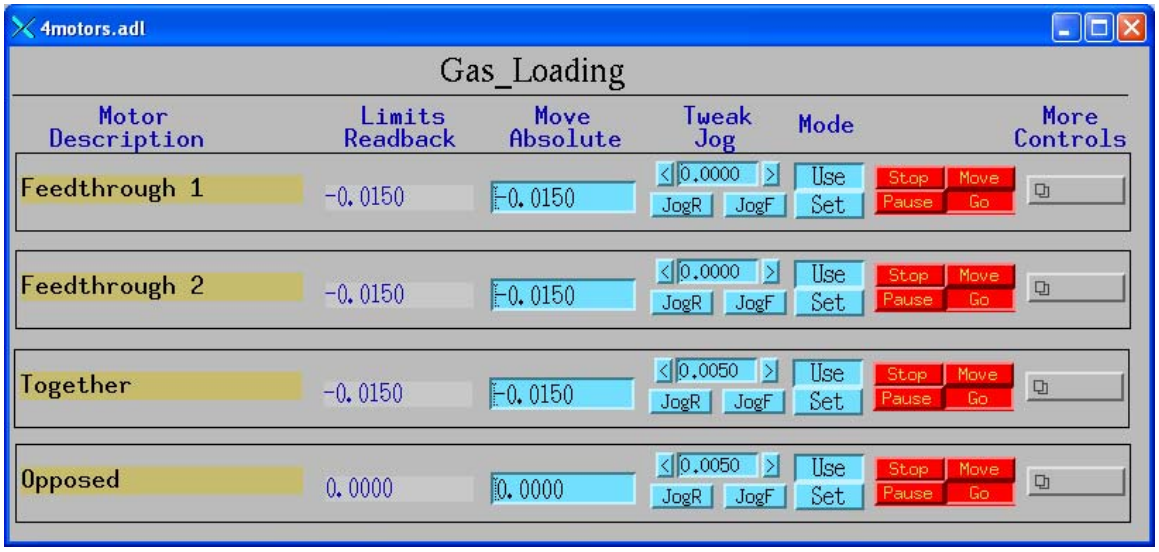
Control for the optical table motors

Click on Motors/Laser stage to open the dialog window below. These two motors allow shifting the optical table in the horizontal plane, so that you can align the optics with the gaskethole position: you can see the sample and collect spectra from ruby.



1) Control of the cell holder closure

Click on Motors/Sample stage to open the following window:

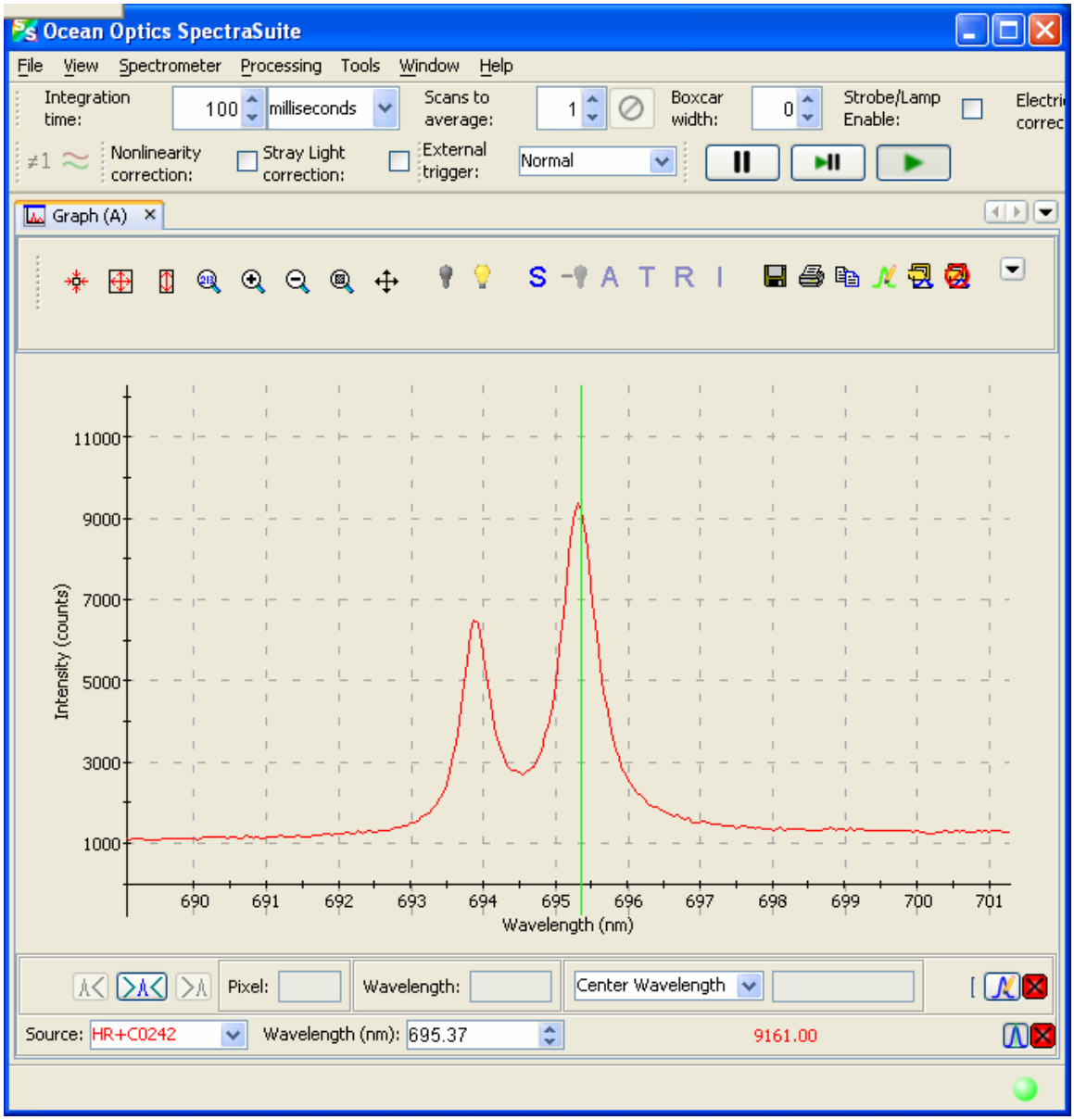


By operating the “Together” motors, both the feedthrough screws are rotated closing the cell holder. A rotation of 1.0 units corresponds to a full turn of the screws, it is convenient to operate in small steps (e.g. 0.005) to avoid overshooting the pressure. Most of cells are closed to ~ 2GPa with a turning from 0.2 to 0.6.

2) Collection of ruby fluorescence spectra

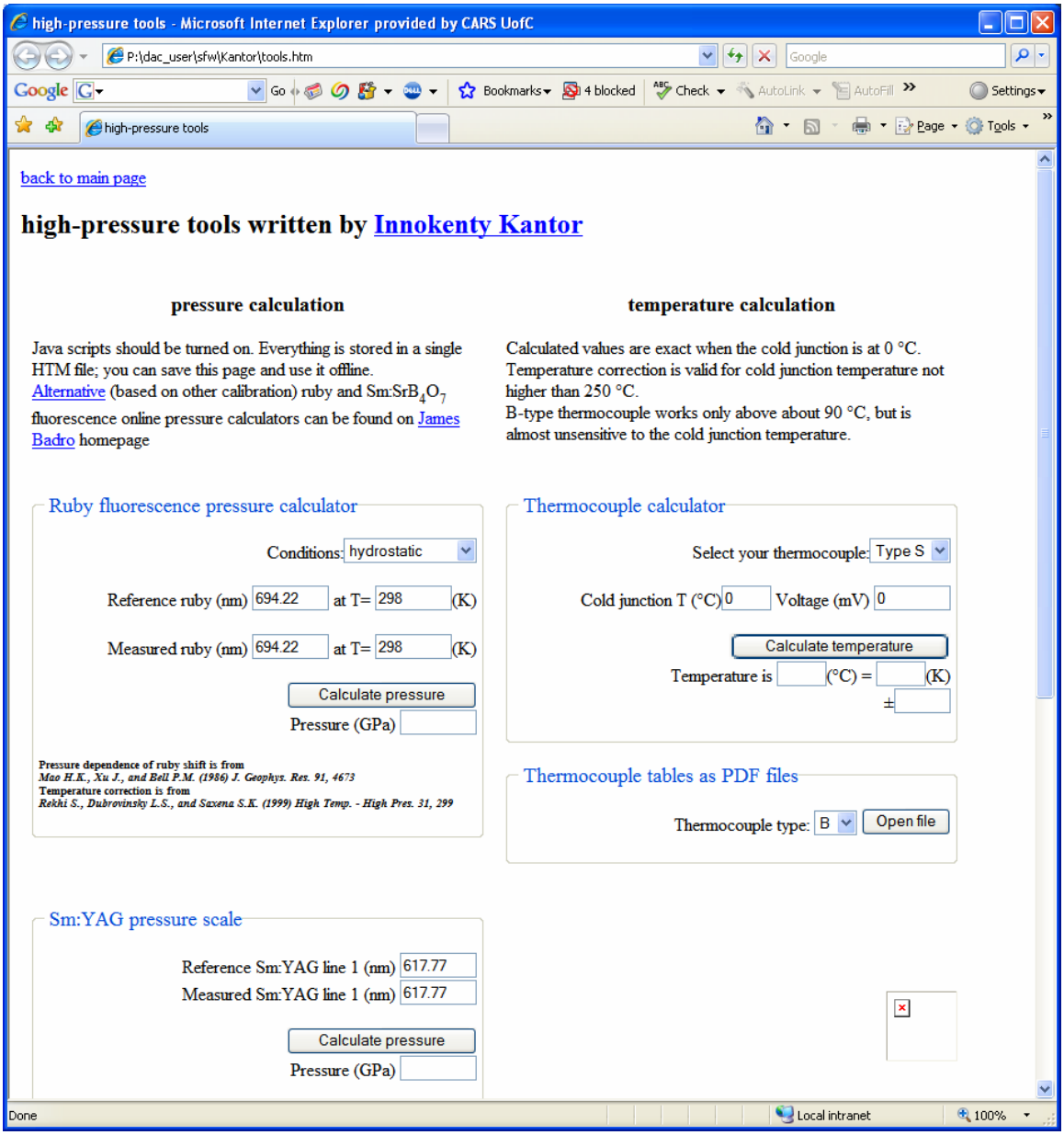
Click on the Ocean Optics icon on the desktop to start the software. The default mode is for continuous acquisition every 300msec. You can change exposure time if necessary in the menu bar.

- You can rescale the spectrum by clicking on this icon  , typically from 693 to 705nm for the abscissa is a good range to work with, but if you increase pressure too much you may need to increase the scale range
- click on  to rescale the vertical scale only



3) Pressure calibration spreadsheet

Double click on “tools.htm” on the desktop. Enter the reference (ambient P) wavelength in the left box, below enter the wavelength of the ruby line you are getting, and then click Calculate pressure.



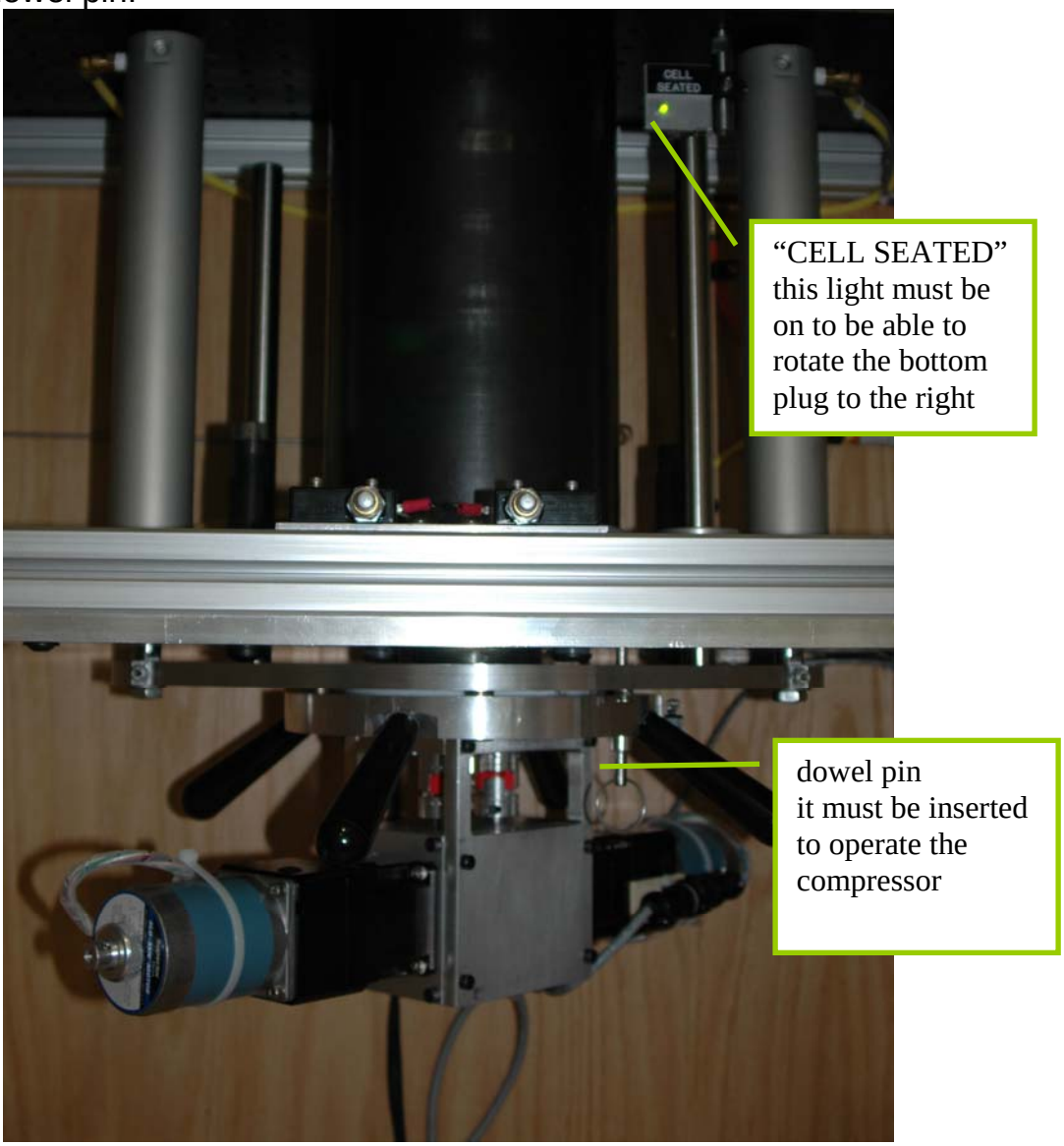
c) Loading the cell into the pressure vessel

To load a cell into the pressure vessel follow these steps.

• Lower the bottom plug of the pressure vessel if necessary. This is done by rotating the bottom plug to the left so that the “Load OK” switch indicates closed on the MEDM screen. Then press the Cell Down button on the Optimate panel, lowering the bottom plug with the air cylinders. **Only the person pressing the Cell Down button is allowed to be near the apparatus when lowering the plug, to avoid pinched fingers**.

• Place the cell into the cell holder. Be sure to install the springs and any filler pieces.
• Place the cell holder on top of the bottom plug, aligning the hex socket screws to the rotary feedthroughs.
• Make sure the bottom plug is still rotated to the left so that the “Load OK” switch indicates “OK” on the MEDM screen. Then press the Cell Up button on the Optimate panel, raising the bottom plug with the air cylinders. **Only the person pressing the Cell Up button is allowed to be near the apparatus when raising the plug, to avoid pinched fingers**.

• Once the bottom plug is fully up, release the Cell Up button on the Optimate, and quickly rotate the bottom plug to the right so that the Press OK switch indicates closed on the MEDM screen. This indicates that the plug is correctly installed, and allows the pressure vessel to be pressurized.
Insert the dowel pin.

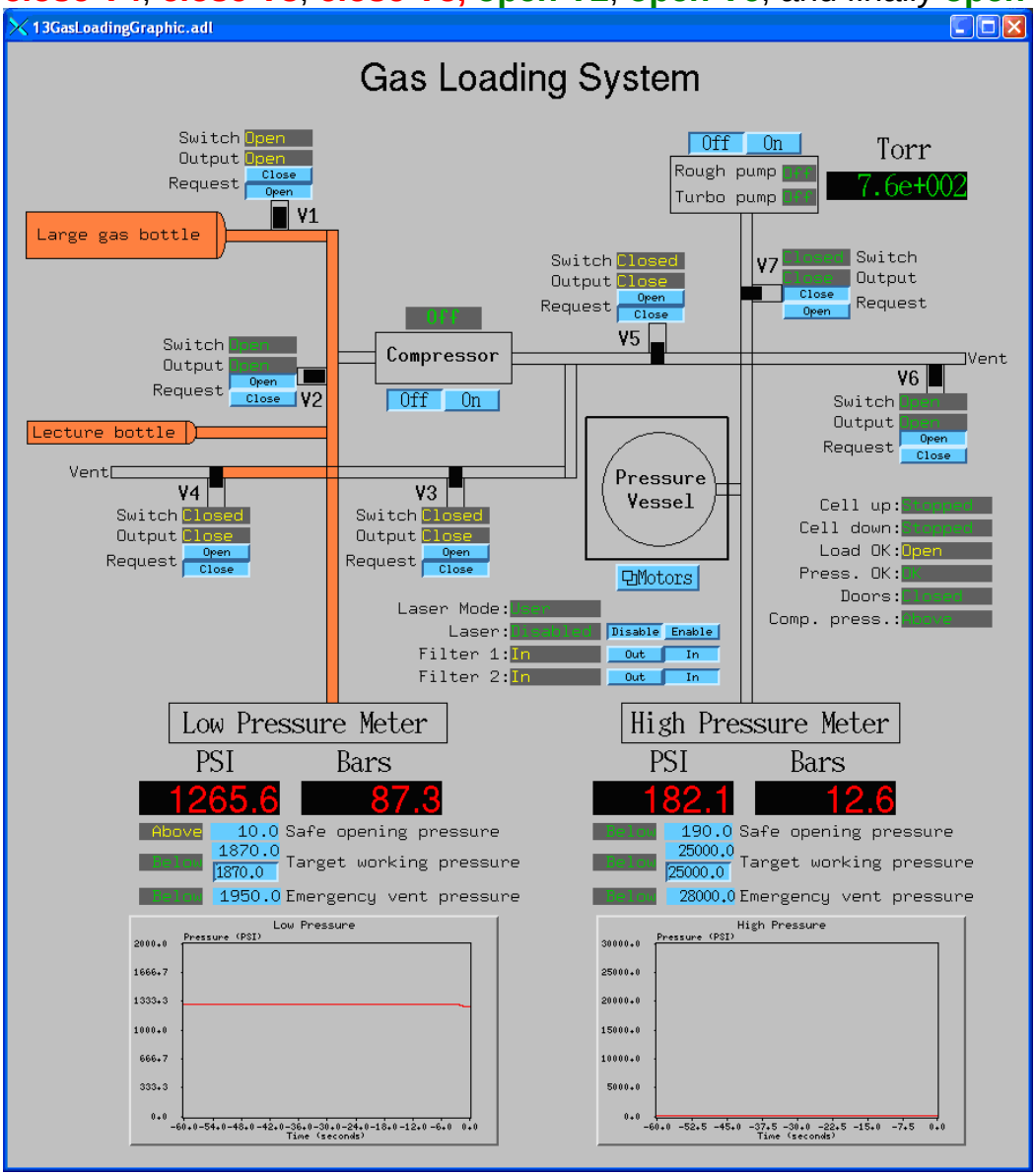


• Close the enclosure doors and verify that the Red Doors Open light on the Optimate panel is off and MEDM window shows Doors Closed status
• press the laser enable button on the front panel
• turn on the key of the laser control, turn it on
• turn on the microscope light
• turn on the controller for the table lift
• by using the motors the laser X and laser Z motors, and the further & closer buttons of the controller shift the optics and focus on the sample

• collect the spectrum from ruby, input the wavelength value as 0 pressure reference in the html document for pressure calculation

d) Pressurizing a lecture bottle

1) **Fill the lecture bottle directly from the large gas cylinder**.
close V4, **close V3**, **close V5, open V2**, **open V6**, and finally **open V1**.



This will fill the lecture bottle to the pressure controlled by the regulator on the large gas cylinder. When the lecture bottle has finish filling (low pressure gauge has stopped increasing) then **close V1**.

2) **Fill the lecture bottle from the large gas cylinder with the compressor**.

If the large gas cylinder does not contain sufficient pressure to fill the lecture bottle directly (typically less than **1500 PSI**) then the lecture bottle can be filled using the compressor as follows. Ensure that the regulator on the large gas cylinder is set to 1500PSI or less; **close V4**, **close V5**, **close V2**, **open V3**, **open V6**, and **open V1**.
Turn on the compressor. This will fill the lecture bottle to the pressure controlled by the target working pressure.

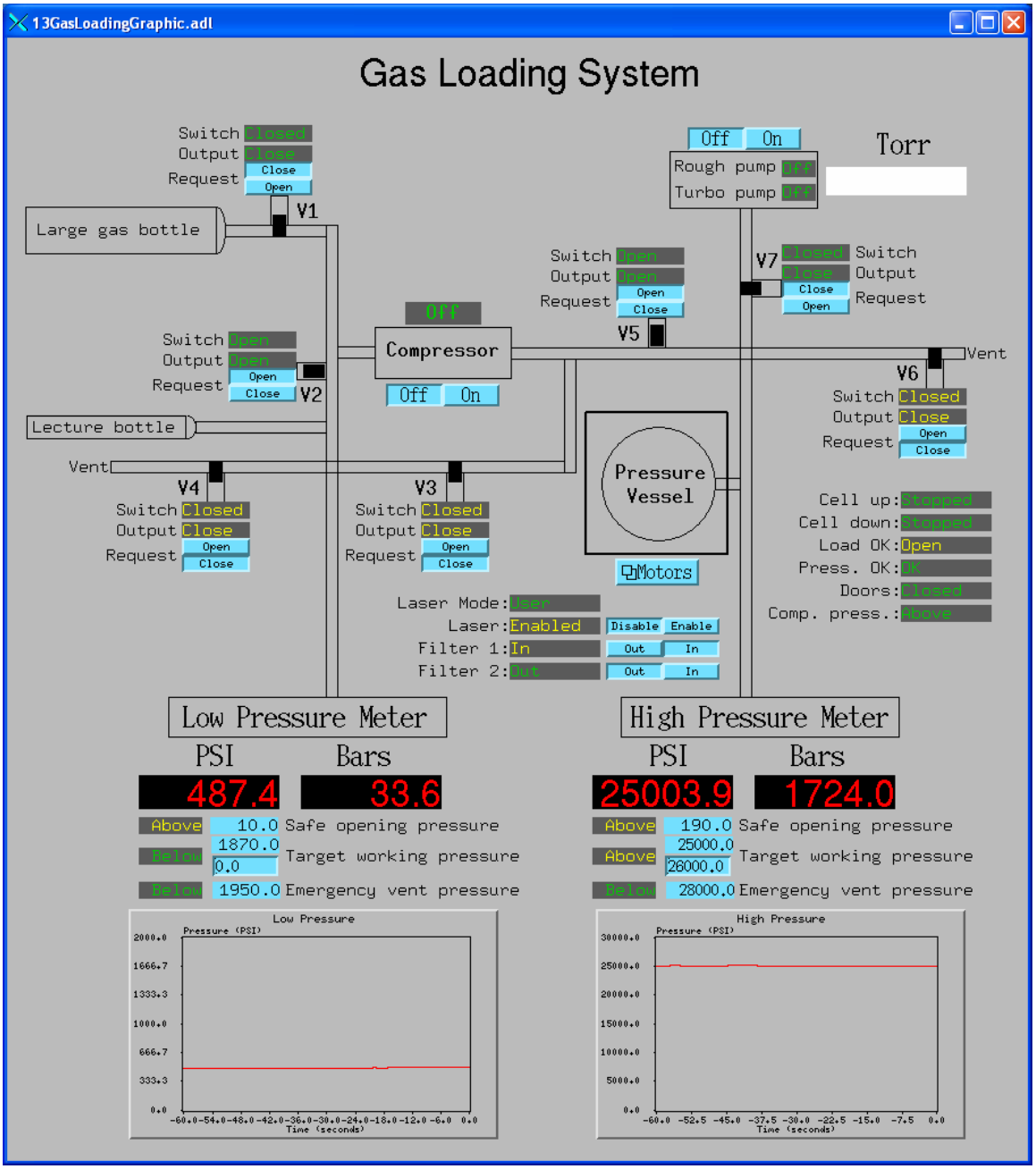
When the lecture bottle has finished filling **turn off the compressor**, **close V1**, and **close V3**.

e) Pressurizing the pressure vessel

Once the cell and bottom plug are correctly installed the pressure vessel can be pressurized as follows:

- Close the enclosure doors and verify that the Doors Closed light on the Optimate panel is illuminated.
- Put on safety goggles
- equalize the pressure at the lecture bottle and at the pressure vessel: **close V6, closeV7, Open V3** and **V5**
- when the pressure is equalized, **Close V3** and **Open V2**
- **Turn on the compressor**. Take note of the starting time of the pressurization.
- If **leaks** are found vent the system (**open V6**) and contact GSECARS staff.
- When the target pressure (usually set to 25000 PSI) is reached, **turn the compressor off** and **close V5**. If there are small leaks leave the compressor on and V5 open so that it keeps the pressure at the desired value.

Take note in the operation log of the pressurization time and of the low pressure meter.



The screenshot was taken after pressurizing the vessel. Please take note of the value of the low pressure meter in the operation log.

f) Closing the cell

Once the cell is pressurized it can be closed and sealed as follows:

- Open the motor control screen.
- Rotate **the motors together** (Together virtual motor) in the positive direction in small increments. 1 unit is 1 full revolution of the hex socket screws

• Use the video system and ruby fluorescence system to determine when the cell is sealed and the desired pressure is reached.

g) Recapturing gas and Venting the pressure vessel

Once the cell has been closed and sealed the pressure vessel can be vented as follows:

• **Turn off the compressor**

Open V3, ***open*** **V5** if closed

- Wait for the pressure of the low pressure system to equalize with that on the high pressure system
- Take note in the operation log of the value of the low pressure meter.

• **Close V2**
• **Close V5**
• **Close V3**
• **Open V6**

h) Removing the cell

- **Wait for the vessel to be fully vented**: the high pressure meter value is below the safe opening value, typically 190 PSI

- **Rotate the bottom plug** to the left so that the "**Load OK**" switch indicates closed on the MEDM screen. Then press the **Cell Down button** on the Optimate panel, lowering the bottom plug with the air cylinders. **Only the person pressing the Cell Down button is allowed to be near the apparatus when lowering the plug, to avoid pinched fingers**.

- Close the DAC with the DAC's screws
- In order to make sure you really closed the DAC with its screws, you may want to replace the DAC holder on the gas loading system and follow the variations of the ruby marker while turning back the together motors to the original position. Some P decrease is normal, if the P drops too much you may have to close the DAC screws more (repeat the removinc cell procedure"

i) Emergency Procedures

The Emergency Stop button can be pressed at any time. This will fully vent the system (opening V4 and V6), turn off the compressor, and disable the laser. If there is a problem then contact Mark Rivers or the APS Floor Coordinator.