

Trough Set Up Checklist

TROUGH

1. Transport trough from chemistry lab to c-station on a cart.
2. Turn on table lock.
3. Loosen clamps, and slide spectrometer breadboard downstream by ½". Use spacers provided, according to the figure below.

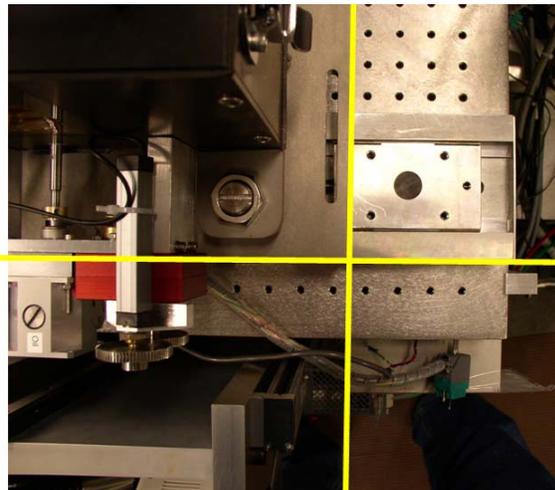
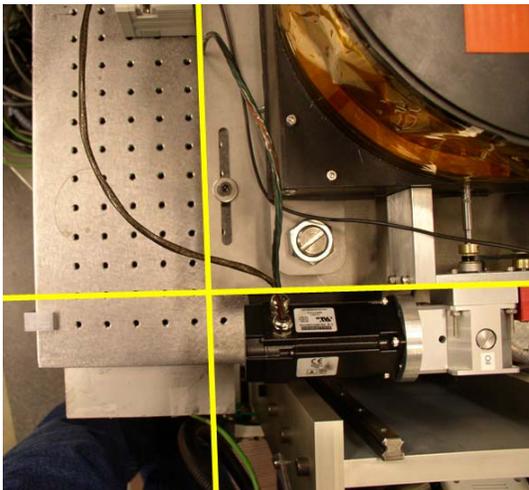


Downstream side



Upstream side

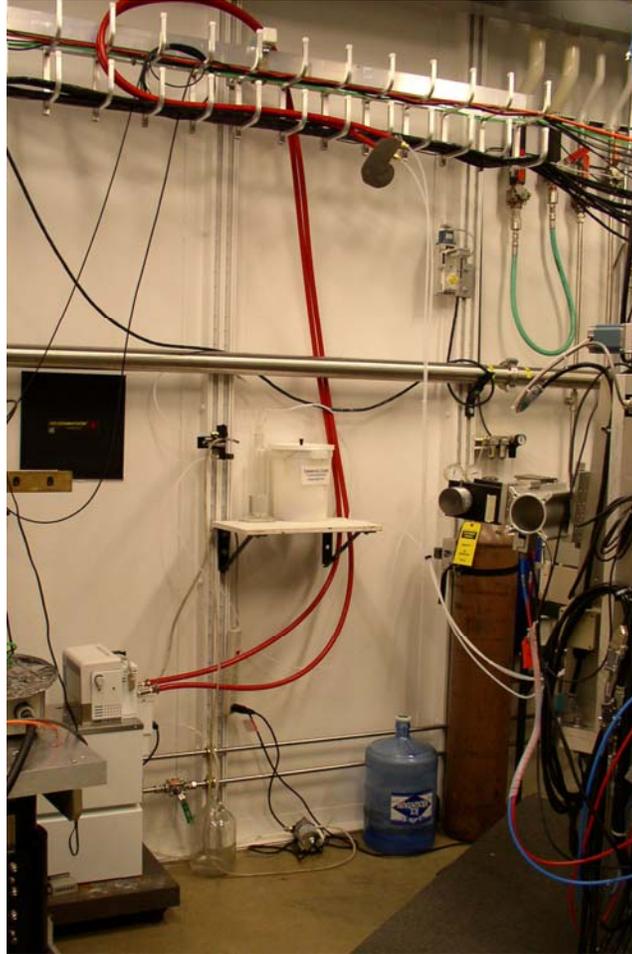
4. In c-station, use hoist to lift the black anodized trough box off slotted base plate, and remove spacer blocks and base plate from underneath.
5. Position metal spacer blocks on spectrometer breadboard.
6. Place rubber pads on each block.
7. Place slotted base plate on blocks and position according to the figure below. Front edge of base plate should align to second row of breadboard holes. Bolt to breadboard with long, black bolts.



8. Use hoist to lower trough onto baseplate, so that feet rest in kinematic mounts.
9. Adjust spring tension in Halcyonics table.
10. Using bubble level inside trough, adjust the three large leveling bolts that make up the kinematic mount of the trough.
11. Turn off table lock.

NESLAB

Heavy, red tubes from NESLAB can be looped around cable tray, as shown below. Connect swagelock fittings to back of trough. Check water level in NESLAB. If low, add clean water from Hinckley-Schmitt bottle.

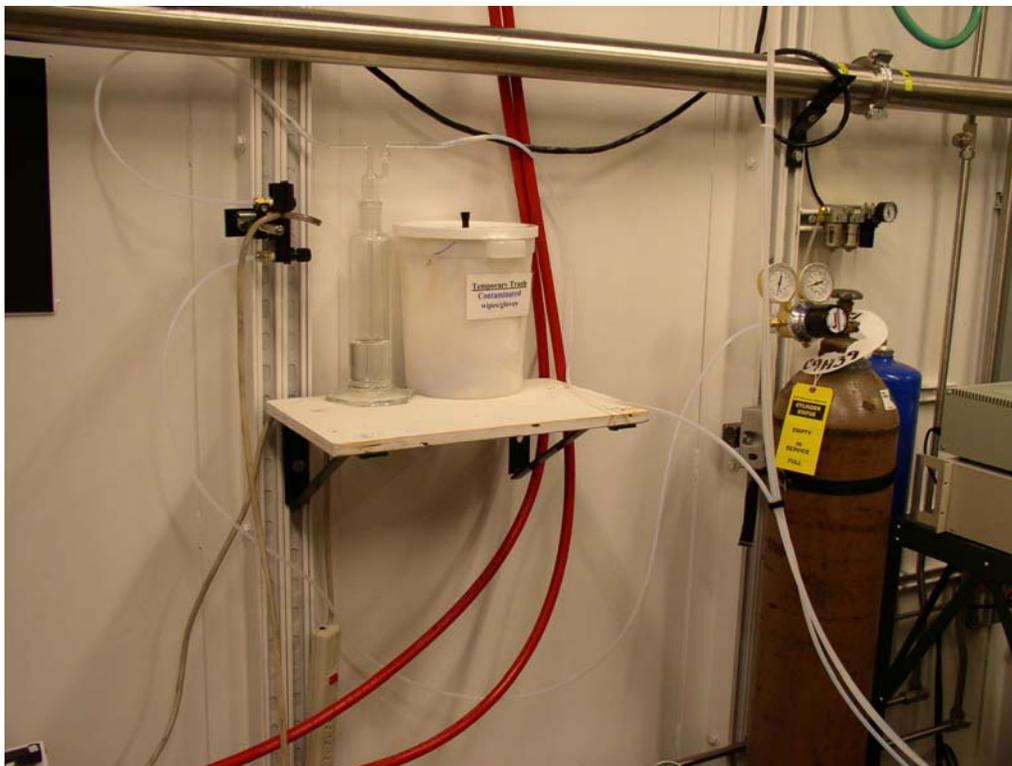


ASPIRATOR

Connect aspirator pump via the foot pedal switch. Plug these into the power strip shown in the figure above. Don't lay the power strip on the floor, since this presents a shock hazard in the event of flooding. Waste should be accumulated in 1 gallon glass jug on floor. Use Pasteur pipettes with rounded tips for use in trough.

BUBBLER

1. Set up tubing connections below according to scheme below:
Gas tank \Rightarrow Regulator \Rightarrow Tubing \Rightarrow Flow gauge \Rightarrow Tubing \Rightarrow Bubbler \Rightarrow Trough
2. Set regulator output pressure to 10 psi
3. Insure that air flows into the bubbler through the long slender inner tube; otherwise, water will be forced into the trough box!
4. Open the vent valve on the back of the trough
5. Open the flow gauge valve slowly to obtain moderate bubbling rate



Air may be purged from the inside of the trough in order to reduce the impact of ozone damage to films and secondarily, to reduce x-ray absorption. A sensor within the sealed box allows readout of oxygen levels. Typically, helium is used as the purge gas, with very slow flow rates sufficient to maintain <5% O₂ levels. A bubbler is typically used to humidify the helium prior to entry (thereby reducing evaporative liquid loss from sample) and to also remove dust from the gas.

An oxygen sensor is available for monitoring the oxygen level within the trough box. The output of this sensor is a (low) voltage. Read this voltage with the Keithley lab multimeter. Typical atmospheric concentration of oxygen (20%) generates a voltage of 9-13 mV. Note that the sensor loses sensitivity over the course of months/years, so the ambient voltage should be checked at the beginning of each experiment. The output of the Keithley meter can be viewed in EPICS. See Jeff Gebhardt for setup.

ELECTRICAL CONNECTIONS

Plugging Things In

Put the following modules on separate circuits, since they draw large currents.

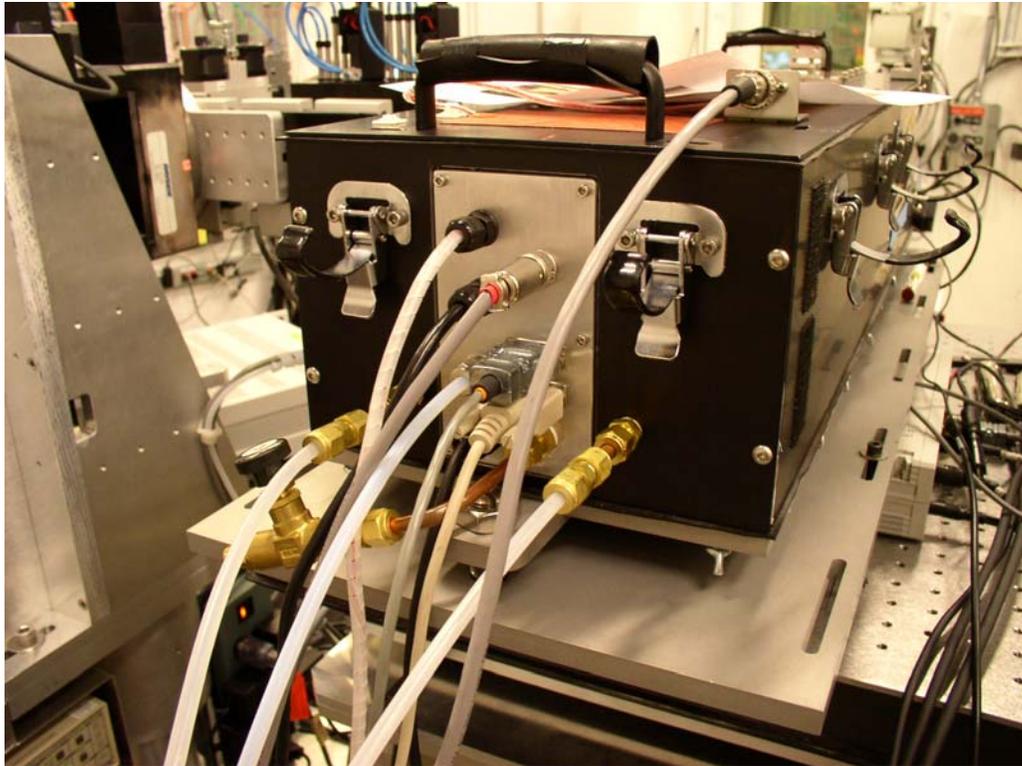
- IMAC control box
- NESLAB + subphase pump
- Lakeshore controller

IMAC Control Box Connections

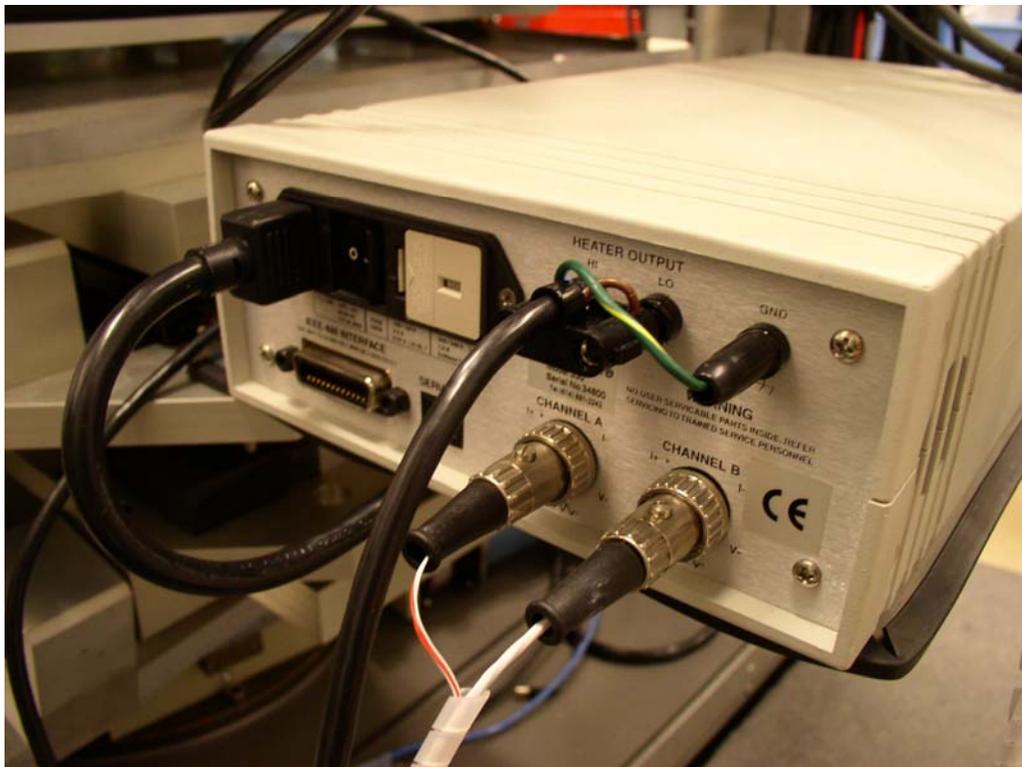
- Connect two heavy-duty chrome connectors from the IMAC control box to the motor.
- Connect the serial cable (directly beneath BNC jack) from the IMAC control box to the LOWER DB9 plug on the back of the trough. This connects the limit switches.
- Turn on main power toggle switch on the BACK of the IMAC control box.
- Flip up the toggle on the FRONT of the IMAC control box to activate the motor.
- Connect BNC cable from NIMA box to the BNC input plug.
- OPTIONAL: Connect second serial cable from IMAC control box to the controlling PC. This allows one to monitor and change motor parameters (e.g. tune the motor response).

NIMA Control Box Connections

- Connect 12V power supply to back of NIMA control box
- The cable exiting the NIMA “barrier A” receptacle splits into two sub-cables. One is a BNC connection that goes to the IMAC box. This subcable carries the control voltage from NIMA to IMAC. The second subcable runs to the potentiometer. It reads the position-dependent resistance from the 10 kohm variable resistor.
- Connect the pressure sensor cable to the “PA” plug on back of NIMA control box. Make sure it is connected to the pressure sensor inside the trough.
- Turn on power for the NIMA control box. The top LED should light red.



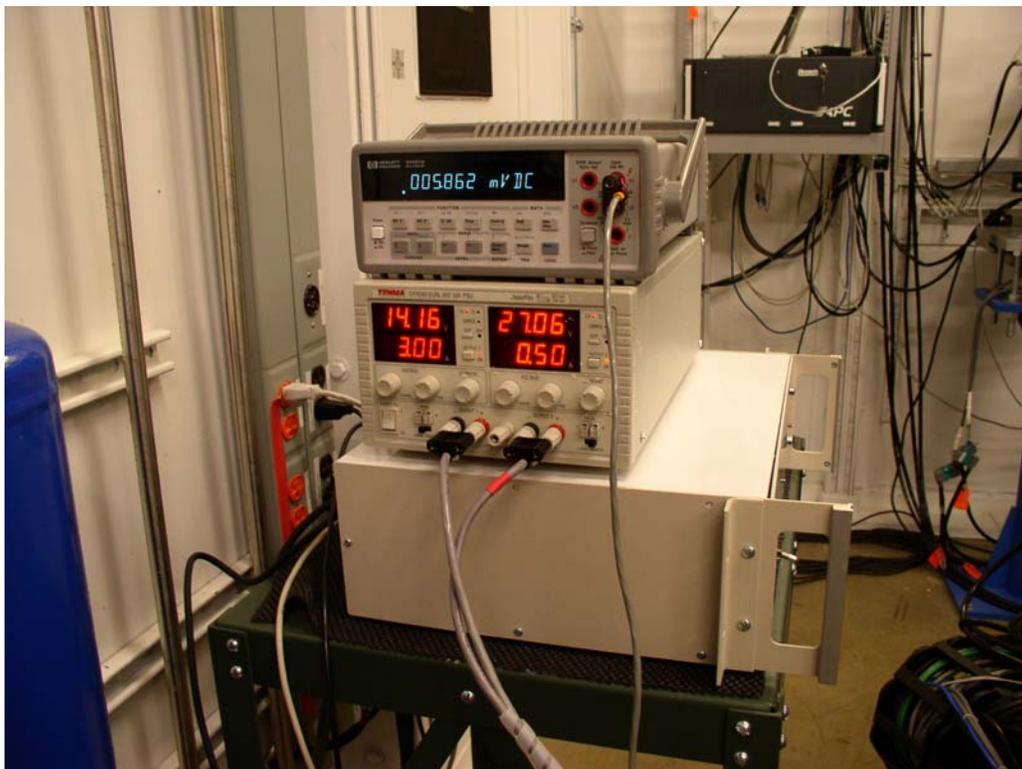
Note that top DB9 plug reads oxygen sensor voltage
Bottom DB9 plug reads limit switches for barrier



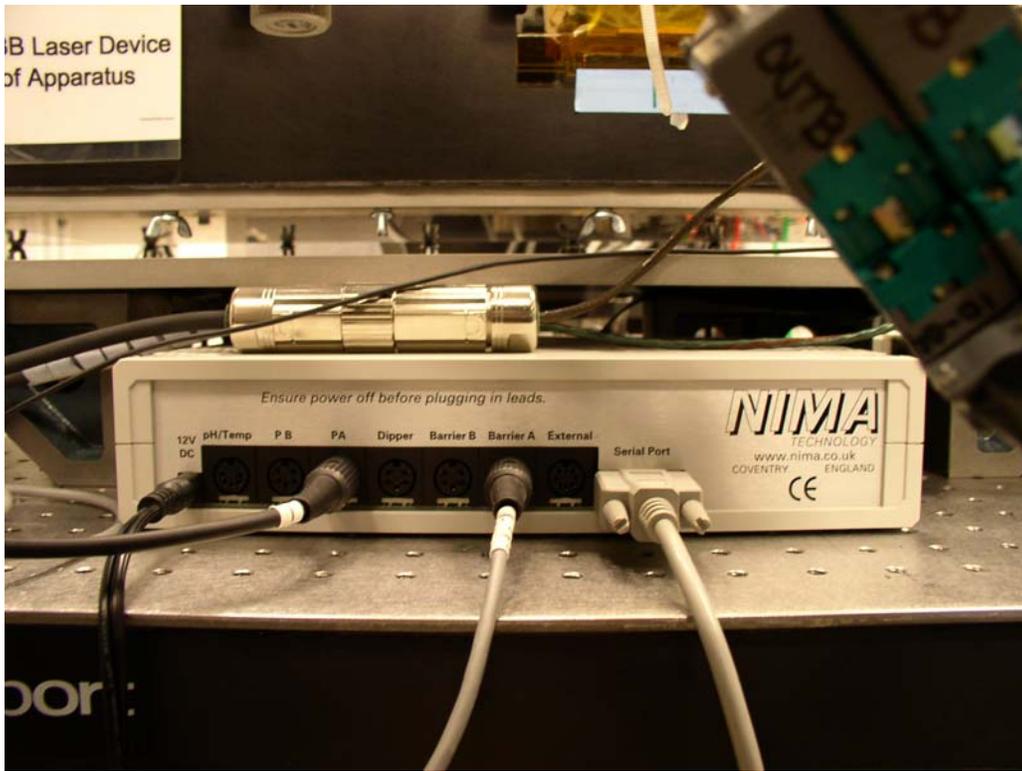
Lakeshore: Channel A=Silicon Diode Sensor, Channel B= Pt Wire RTD Sensor



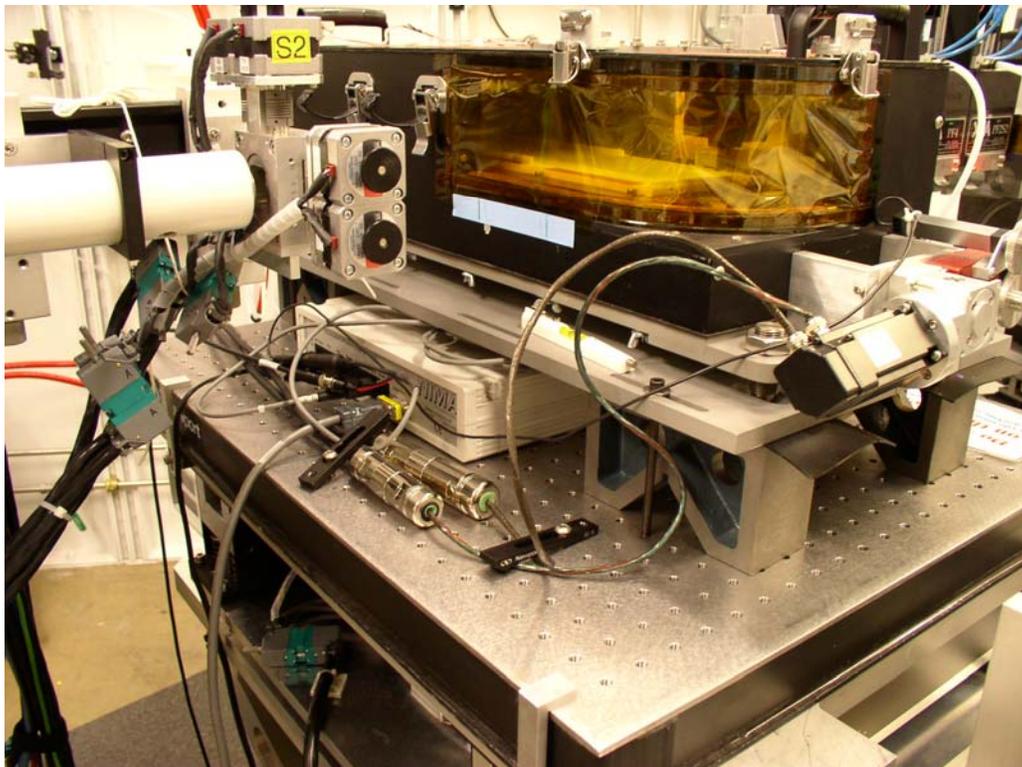
Heater and IMAC power supplies

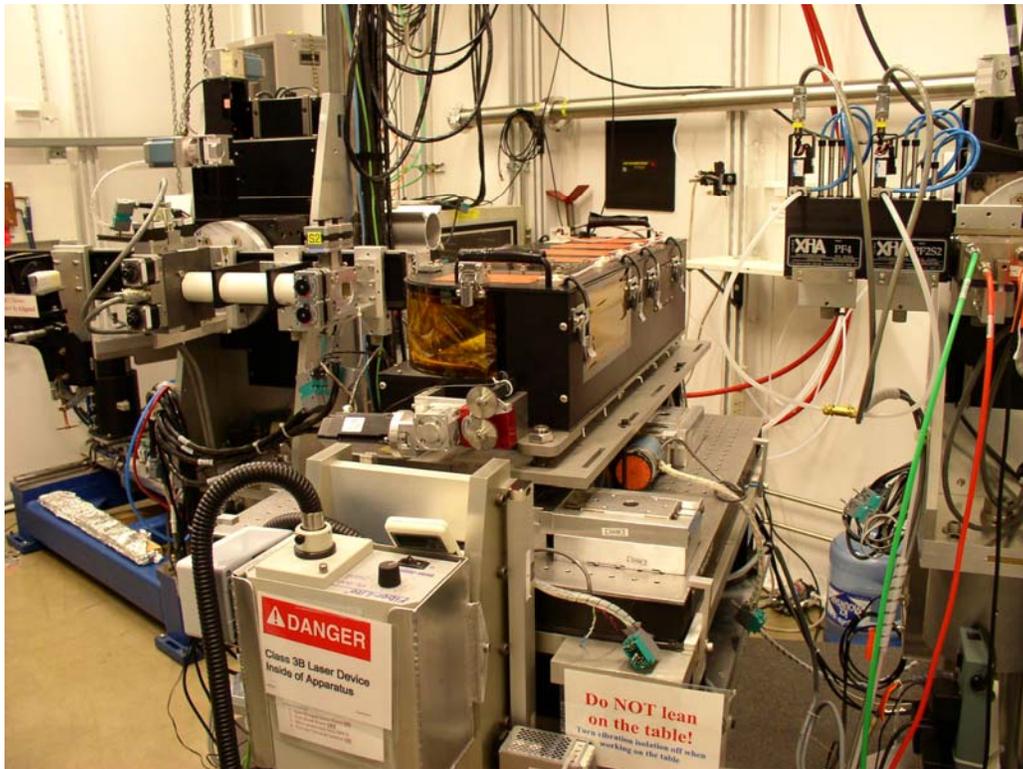
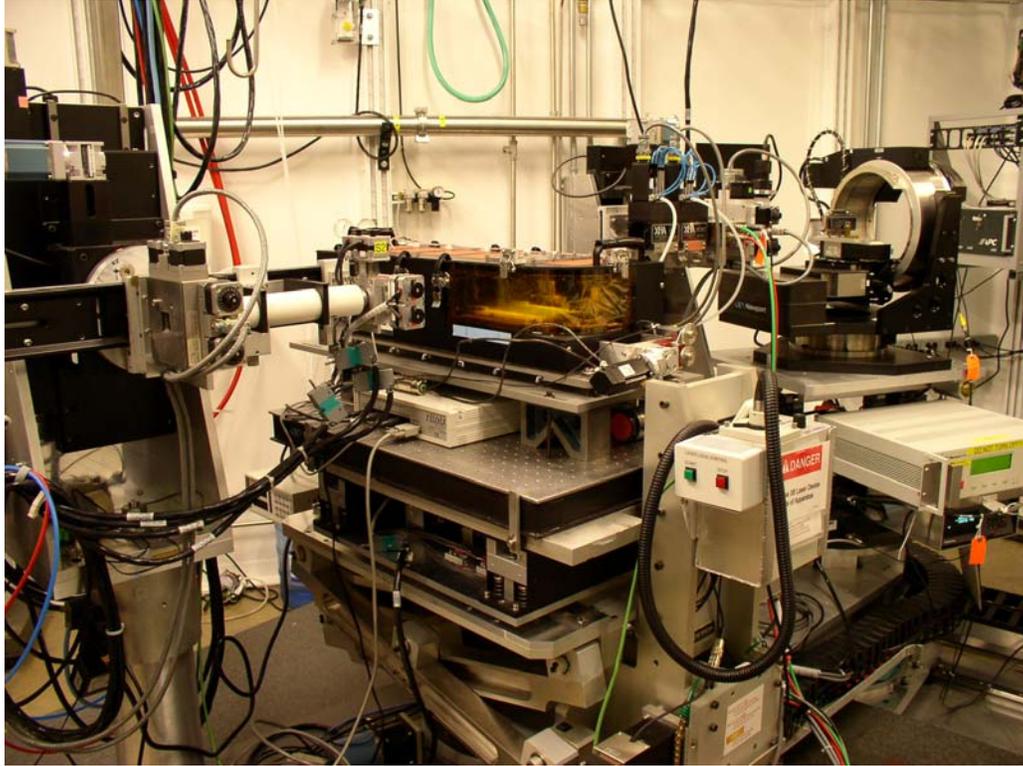


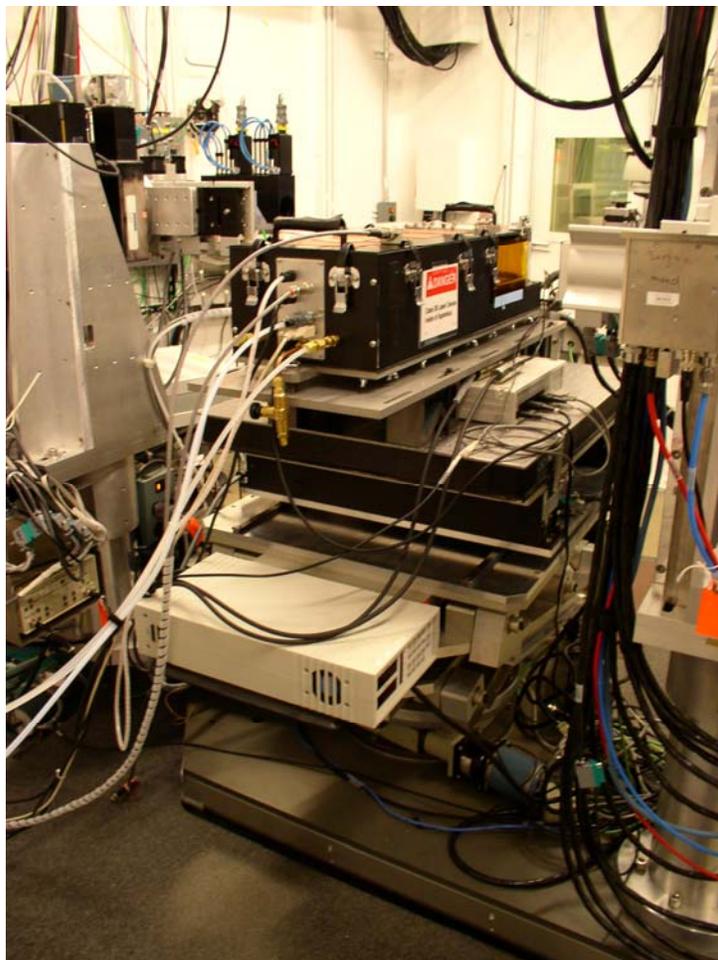
Top Unit (Keithley) = reads oxygen sensor voltage (5-10 mV DC)
Middle unit powers lid and window heaters: Left output=Lid (3 A) Right output=Windows (0.5 A)
Lower unit is IMAC motor control box



NIMA interface box, rear panel







SET UP NIMA SOFTWARE

- Start NIMA 5.16 software. Click on “OK” to rid pop-up panel. **Both red LEDs on the NIMA control box should light.** If this is not the case, there is a communication problem between the computer and the NIMA control box.
- From main menu, go to “setup” and click on “accept calibrations.”
- From main menu, go to “interface test.” Make sure that “Barrier A” and “Pressure Sensor A” have non-zero decimal readouts. Click on bottom of new panel to enable interface and motors. Test motor operation by sliding bar.
- When running in the “Isotherm” mode, the SIGN of the nominal speed should be positive for compression and negative for expansion. This sign must be entered manually by user.
- In “Monolayer Menu” you must set the maximum and minimum areas allowable under different types of scans.
- **If user wishes to hold the barrier in one position, then he must use the ‘Area control’ feature of the NIMA software. Otherwise, the barrier will slowly creep in one direction or the other until it hits a limit switch.**

CALIBRATE PRESSURE SENSOR

Only needs to be done occasionally. Note that filter papers must be hung directly from Pt wire that extends from sensor head. DO NOT use an ‘S’ hook with our sensor, since the balance assembly will then be too long for lid to close. Balance pan, weight and filter papers are in the NIMA supply kit. To calibrate, follow instructions in NIMA software, under heading ‘Calibrate Pressure Sensor.’ Note that the pressure sensor head can be raised and lowered with respect to the subphase by turning the knob on its support.

TEACH BARRIER POSITIONS

Do this at the beginning of each trough run, or after changing the barrier from flat to notched (or vice versa). A ‘canned’ procedure can be found in NIMA drag-down menu, under the heading, ‘Teach Barrier Positions.’ Will need to move barrier to fully closed and fully open positions. Use a ruler to measure distance from leading edge of barrier to front edge of trough. Multiply this distance by 8.9 cm (width) to obtain the trough area.

TEACH BARRIER SPEED

After barrier positions have been taught, you can then use another ‘canned’ routine for teaching barrier speed. The can be found in NIMA drag-down menu, under the heading ‘Teach Barrier Speed.’ Follow the instructions. Note that the nominal speed is only used by NIMA when performing a scan (e.g. isotherm, constant pressure, constant area, etc) **Simply pushing the ‘open’ or ‘close’ buttons on the NIMA panel results in a maximum speed of about 1 mm/s.**

LIGHT CLEANING OF TROUGH AND BARRIER

For light cleaning of trough, wipe down with acetone first, then several times with chloroform. **Chloroform is a known carcinogen**, so should be used in small amounts and handled with nitrile gloves. Best to fill a small (10 mL) volumetric flask with chloroform and pour small amount onto kimwipe for wipedown. Place wet wipes in the white waste bucket and close its lid to reduce vapors.

When replacing the barrier, **make sure that the thumbscrews are tightened down fully**. These screws maintain the contact pressure between the barrier and trough edges. The wing nuts in the barrier bridge should also be tight, but these serve a different purpose: to secure the barrier firmly to the bridge.

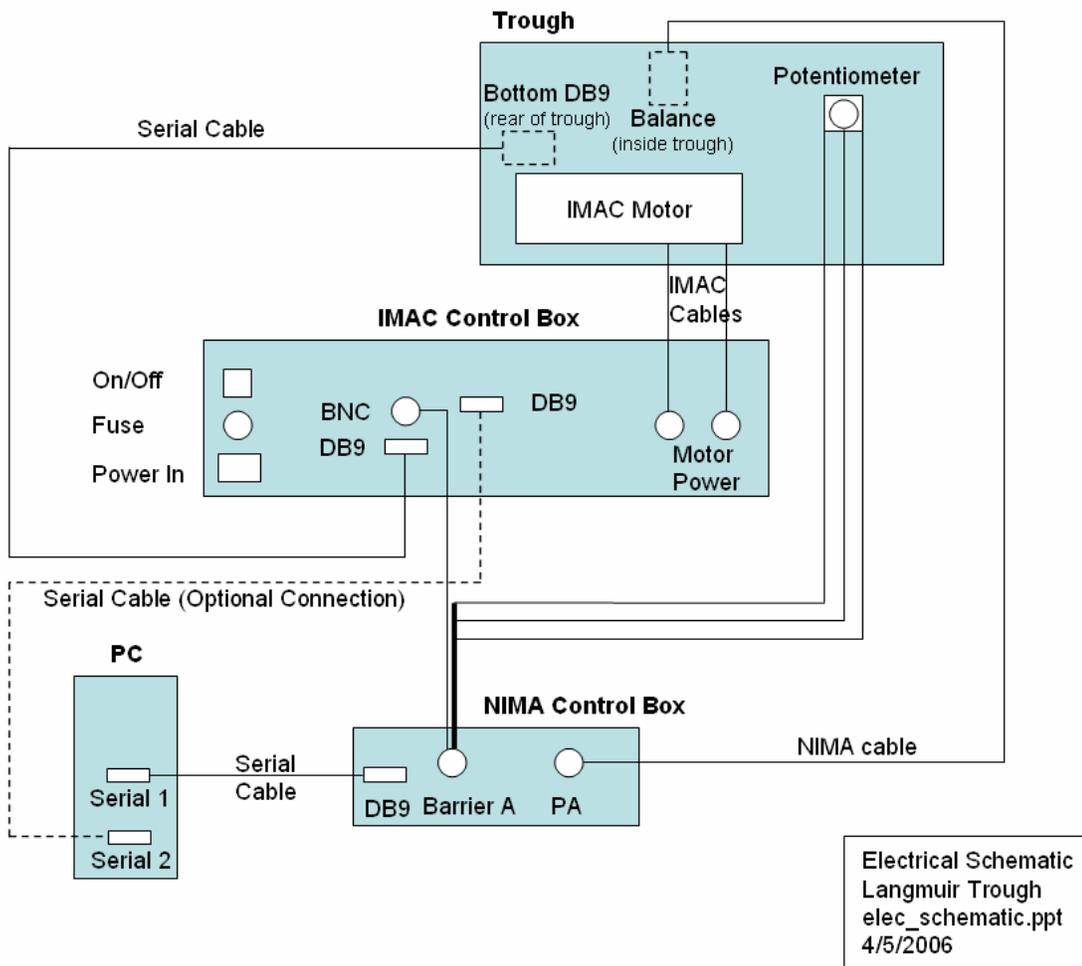
FINAL SUBPHASE PREPARATION

With barrier fully open, fill trough with clean water. Let stand for 10 minutes. Compress barrier to fully closed position and aspirate the compressed surface lightly. Expand barrier to open position and measure the surface pressure. If the system is clean, then you should measure a surface pressure of about 71 mN/m. (See table below, source is CRC handbook).

As a further test, perform an isotherm compression on pure water surface. Net rise in pressure should not exceed a 0.2 mN/m for compression ratios > 8:1. **Activate the vibration-isolation unit before measuring isotherms.**

Temp (C)	Pressure (mN/m)
10	74.23
25	71.99
50	67.94

**Langmuir Trough Setup and Usage
Supplementary Information**



Temperature Control of Sample

Control of subphase temperature is achieved in two stages. A constant ‘sink’ temperature is maintained in the bottom-most block, using liquid circulant from a standard NESLAB module. The temperature difference from the sink temperature is achieved with a set of peltier chips which serve to directly heat or cool the bottom of the Teflon. **Set the NESLAB temperature 3-5 degrees below the desired subphase temperature.** The current supplied to these chips is controlled by a Lakeshore temperature controller, which reads the feedback temperature either directly from the subphase, or from the copper block directly beneath the Teflon trough. Currently, the Lakeshore is configured for sample heating only, although in principle the peltiers can also be used to cool. Typical temperature range = 25 - 40 ± 0.1 Celsius

Lakeshore Electrical Connections

The subphase temperature (measured with Pt-wire RTD sensor) is used as the readback temperature in the control loop. The Silicon diode sensor is simply used to monitor the rough temperature of the top copper block.

- Silicon Diode sensor -> Lakeshore channel “A” (control)
- Pt wire RTD sensor -> Lakeshore channel “B” (sample)

To change the readback sensor from Pt to Si, use the Lakeshore keypad. Use “Input” with “^” or “v” arrow key.

Setting the subphase temperature on the Lakeshore controller

Note that the top readout is for copper plate and the bottom readout is for subphase and target temperature setting. On lakeshore keypad (LK), hit ‘control’ to read the current subphase temperature. Hit ‘setpoint’ twice to make a change to the target temperature setting. Notice that the last digit of the readout will flash. Enter the new temperature using the numbers on the LK. When finished, hit ‘enter’ on the LK. Note that it will take a second or two for the unit to update the new control temperature. Hit ‘control’ to monitor the subphase temperature again. To turn on the heater used in the control loop, hit ‘heater’ button 3x so that heater readout switches from ‘off’ to ‘high.’ After a few seconds, you should see the heater power bars light up on the display. To turn off the heater, simply hit ‘heater’ again so that you see ‘off’ in the display. Lakeshore internal jumper set to 25 ohm load.

Peltier Circuit

- | | |
|------------------------|---------------------------|
| • Number of chips | 7 chips wired in series |
| • Maximum power | 50 W per chip |
| • Maximum current | 1.5 A |
| • Recommended settings | 25 V net, 1.0 A current |
| • Polarity | reversible (heat or cool) |

Lid and Window Heating

Condensation on windows and the inside of the lid of the trough is eliminated with a series of resistive heating elements. The power delivered to these elements is controlled by the user.

Lid Heaters

- Five, wired in parallel, 25 ohms each
- Maximum ratings: 450 W per pad
- Connect cable from *Tenma* output (left side) to the specialized (3-prong) receptacle on trough lid.
- Set the Tenma current limit to 3.0 Amps.
- Push “C.L.” mode button to apply power.
- Turn up voltage so that the current increases to this level.

Window heaters

- Two, wired in parallel, 100 ohms each
- Maximum ratings: 25 W per strip
- Connect cable from Tenma output (right side) to the specialized (2-prong) receptacle on back panel of the trough.
- Set the Tenma current limit to 0.5 Amps.
- Push “C.L.” mode button to apply power.
- Turn up voltage so that the current increases to this level.

Readback of Barrier Position with 100-Turn potentiometer

Align the leading edge of the barrier to the elevated Teflon guard shoulder. The decimal readout of the barrier position should be $\sim 12,000 \pm 500$. Read this in the “interface test” window. If not, then the potentiometer gear must be disengaged and rotated manually until this value results. This ensures that the potentiometer is close to midrange (5 kOhm) when the barrier is in the center of its range of motion, and hence won’t hit a hard limit when barrier is at either endpoint of its motion. In principle, this only needs to be done once, but it’s good to check at the beginning of each run.

Barrier Position	Area (cm ²)	Distance to front (cm)	Decimal Readout (Unitless)	Potentiometer Resistance (kOhm)
Closed	35.6	4.0	7,500	
Shoulder	120	13.5	12,100	
Mid-Range	174	19.5	15,000	5.0
Open	333	37.4	23,690	

Trough Test Data Sheet

Operator: _____

Date: _____

Temperature Control

	Temperature (Celsius)
Ambient room	
Circulant	
Control (copper plate)	
Sample (liquid)	
Lid	

Lid and Window Heater Control

	Current	Voltage
Lid		
Window		

Oxygen Purge

Purge gas type	
Ambient air sensor reading (mV)	
Operating base level (mV)	
<i>Stop gas flow and close outlet valve</i>	
Final level (mV)	
Elapsed time (minutes)	
Slope (mV/min)	

To calculate sensor reading after some elapsed time (with valves closed) use:

$$\begin{array}{l} \text{Final Sensor Reading} = (\text{Slope} \times \text{Time}) + \text{Initial Sensor Reading} \\ \text{[mV]} \qquad \qquad \qquad \text{[mV/min]} \qquad \qquad \qquad \text{[mV]} \end{array}$$

Barrier Control

	Area (cm ²)	Decimal
Minimum		
Maximum		