UNIVERSITY OF WISCONSIN–MADISON

Operating Procedures for the Pulsed Oxidation Chamber

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Introduction

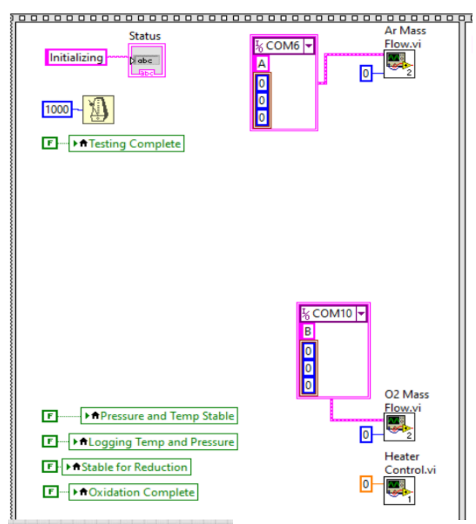
The pulsed oxidation chamber (POC) is a unique testing apparatus that allows for sample exposures at high temperatures and short exposure times, with optional reduction prior to exposure. The POC provides precise control of temperature, process gas, partial pressure, and exposure time. This manual will give an overview of the mechanical components and LabVIEW involved in POC operation and troubleshooting for common errors. Please review the entirety of this document, especially the LabVIEW Guide, before operating the POC, and refer back to it as you continue experiments.

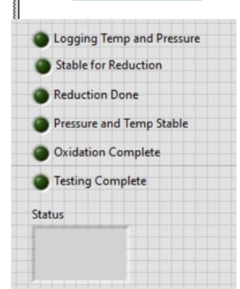
Components

Manuals for all physical equipment, as well as extra parts and assorted documentation, are kept in Rm 1136 along with the POC. If not physically available, digital copies of manuals and documentation may be accessed within the POC folder on the computer in 1136 (username: *admin*; password: *whypass9*). A short overview/refresher on LabVIEW by NI Instruments can be found at the following address: https://www.ni.com/pdf/manuals/373427j.pdf.

- **Chamber**
  - **Fused Quartz Chamber:** Custom made; contact information for replacements is listed in the manual binder
  - **Crucible/Sample Holder:** Custom made; Alumina and BN available, replacements available through consultation with most engineering ceramics suppliers
- **Pressure Control**
  - **Roughing Pump**: Edwards nXDS Scroll Pump
  - **Turbo Pump**: Pfeiffer HiPace 80 w/ DCU Controller
  - **Gas Mass Flow Controller (MFC)**: Alicat MC-Series (1 per process gas)
  - **Gas Supply**: UHP Argon and Simulated Air (20% O_2, 80% Ar)
  - **Vacuum Gauges**
    - Kurt J Lesker 354 Series Ion Gauge – Used for pressure monitoring during the experiment, automatically turns off for chamber pressures >5.00×10^{-2} Torr. Automatically turned on when running the VI.
    - MKS 925 Micro-Pirani Gauge – Used for leak checking and pressure monitoring outside of LabVIEW. Must be turned on separately from the VI. Operational from atmospheric pressure to 1×10^{-5} Torr.
  - **Gate Valve**: VAT Series 64 w/ PM 5 Controller
- **Temperature Control**
  - **Thermometer**: Photrix OEM Optical Thermometer w/ attached fiber optic cable & sapphire light pipe
  - **Heater & Induction Coil:** Magneforce Heat Station 5000 R2
- **LabVIEW**

- LabVIEW is a visual programming language in which you can build virtual instruments (VIs) to gather, display, and process data. The POC VI consists of many sub-VIs, all of which are located in the same POC folder, and can be accessed and viewed separate from the main VI. The **Visual Guide** at the end of this manual contains a detailed description of each stage of the LabVIEW script and annotated figures of the Block Diagram.
- The most important things to look at it LabVIEW are the ***Front Panel*** and the ***Block Diagram***. The Front Panel is the data display portion of your VI, and allows you to control external equipment, view outputs, and access any other functions you might normally see on a physical instrument. The Block Diagram represents all the wiring and logic behind the Front Panel. This is where you'll find most of the key settings, all of the sub-VIs, and a whole host of logical and data structures. For many experiments, only the Front Panel need be used. However, if you wish to change any parameters of the experiment not listed in the Front Panel (e.g. cooling rate, flow rates, experiment structure, etc.), there are corresponding input parameters in the Block Diagram that must be changed.
- The POC VI Block Diagram reads left to right, using a flat sequence structure. This allows each step to be laid out ordinally, ensuring that oxidation will not occur prior to the set number of pumpdowns, which will not occur prior to user input, etc.. Each main step has a status indicator, which can be seen on the right side of the





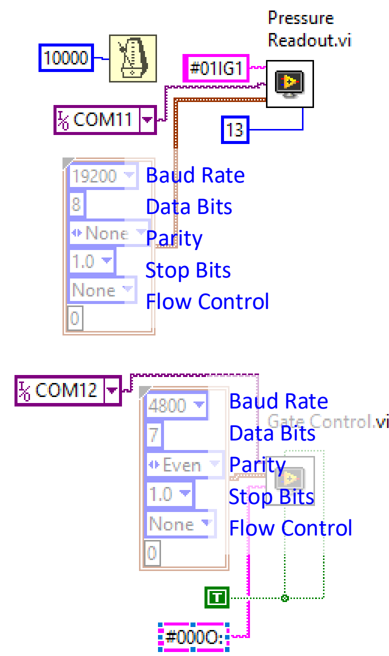
Top: ***Initialization*** *phase; the grey box surrounding each step is the flat sequence structure*

Left: *Indicators & status box from the front panel*

Front Panel. When a step begins, the indicator will light, and the *Status* box will provide additional information

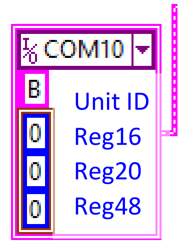
- LabVIEW communicates with physical equipment using instrument drivers and National Instruments Data Acquisition packages (NI-DAQs). Information from each piece of equipment is relayed to the computer and LabVIEW through a serial port. Each pin on the serial port is assigned a number, and these numbered pins are referred to as COMs within LabVIEW. If you look at the Block Diagram (Ctrl-E or Window > Show Block Diagram), you can see the different COMs connected as inputs to sub-VIs which control gas flow, pumping/backfilling, heating, gate valve position, and data acquisition.
- **COMs List**
  - *COM(1-5) – Unused*
  - **COM6** – Alicat Mass Flow Controller (Ar)
  - **COM7** – Photrix Optical Pyrometer
  - *COM8 – Unused*
  - **COM9** – Alicat Mass Flow Controller (Reducing Gas)
  - **COM10** – Alicat Mass Flow Controller (O2)
  - **COM11** – Vacuum Gauge (KJL 354)
  - **COM12** – Gate Control

- **NOTE:** The turbo and roughing pumps are not controlled explicitly by a COM. They are turned on individually and separately from LabVIEW and will run for the entirety of the experiment. The pumping rate is controlled primarily by the gate valve, which separates the chambers from both pumps.
- For the **Gate Control** (GC) and **Pressure Readout** (PR) sub-VIs there are individual sets of serial commands that set fundamental parameters for data acquisition and equipment communication. Descriptions of the settings and standard settings follow:
  - **Baud Rate:** Speed of communication over a data channel (bits/second). Setting depends on the capability of the attached equipment. (GC: 4800 PR: 19200)
  - **Data Bits:** Sets the number of data bits in each character. The gate valve controller reads commands in ASCII, while Pressure Readout takes data from the ion gauge in 8-bit format (GC: 7 PR: 8)
  - **Parity:** Method of error detection in data transmission. (GC: "Even" PR: "None")
  - **Stop Bits:** Bits to signal the end of a character. (GC, PR: 1)
  - **Flow Control:** Regulates instances where data arrives too quickly to process. Not needed in this instance. (GC, PR: "None")



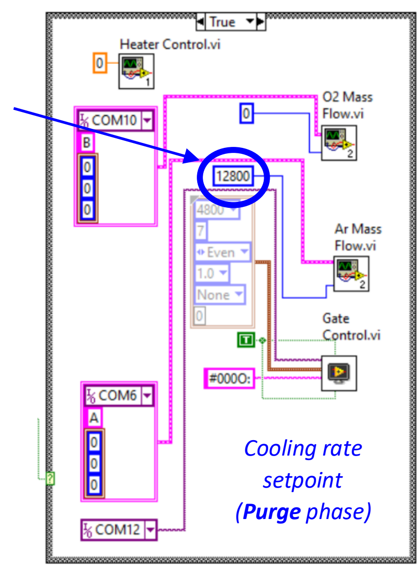
Top: *Pressure Readout serial inputs*
Bottom: *Gate Control serial inputs*

- The GC and PR require additional inputs for the COM (GC: 12 PR: 11) and for the command. GC commands set the gate position (000000 = closed 001000 = open). PR commands control the functions of the KJL354 ion gauge (a full description of serial commands for the ion gauge is in the KJL354 manual)



AliCat Control Inputs

- COM settings for the mass flow controllers and heater are different from the PR and GC serial communication setups. The MFCs use an AliCat control configuration and a setpoint for output (00000 = closed, 64000 = open). The heater simply takes an input from 0-10 which sets the power output to the coil
- During the oxidative exposure portion of the experiment, a PID loop for both pressure and temperature regulates chamber conditions to maintain experimental setpoints with reasonable stability. The proportional gain (K_c) and integral time (T_i) for Temperature and Pressure PID gains can be adjusted in a "guess and check" manner to adjust the time it takes to reach a stable pressure and temperature before exposure
- To adjust the cooling rate after exposure, the Ar Mass Flow setpoint must be adjusted within the **Purge** phase of the VI. Additionally, the glass gas supply tube may be adjusted or replaced such that the outlet is closer to or more focused on the sample



*Cooling rate setpoint (**Purge** phase)*

Operation

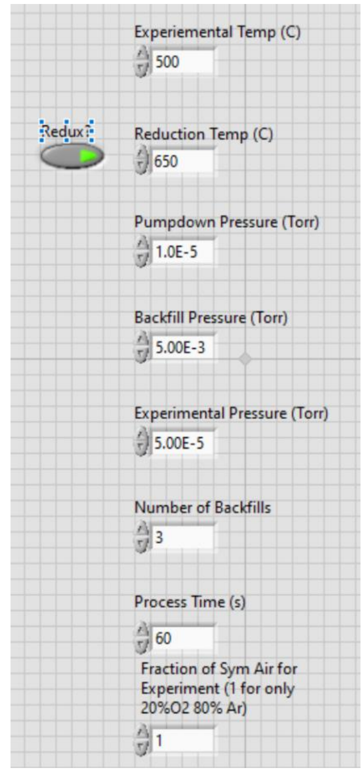
Pre-Experiment

- Open the chamber by removing all 8 bolts, nuts, and washers.
- Remove the dummy sample, and place your sample in the crucible, centered over the pyrometer
- Place a new copper gasket between the upper and lower portions of the chamber, replace the quartz dome and replace bolts & hardware, making sure to tighten bolts gradually and evenly around the base
- Place the induction coil around the quartz crucible. Align the coil at a level slightly above the sample, as the coil has a tendency to "fling" light samples off the crucible and into the bottom of the chamber
- Set the gate valve control to **CLOSED** to start

Experimental Procedure

- Open the POC VI: **Pulsed_Oxidation_Chamber_OLD.vi**
    - The file **Pulsed_Oxidation_Chamber.vi** is a version which accounts for the MKS 925 gauge within LabVIEW, and records pressure data once the VI starts. The connection from the 925 gauge to the COM port is functional, but the code itself has not been tested experimentally. Until further notice, it is best practice to use the established version in **Pulsed_Oxidation_Chamber_OLD.vi**

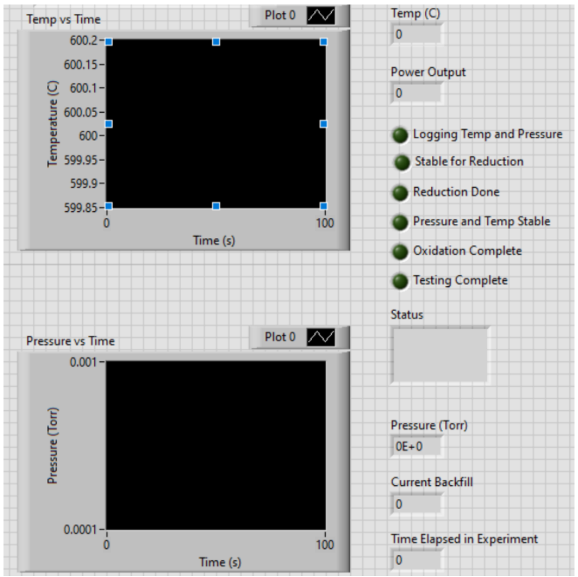
- Set all experimental parameters:



Input portion of front panel display for the POC VI

  - **Experimental Temp (C)**: Temperature at which exposure to simulated air will occur
  - **Reduction Temp (C)**: *Optional*. Temperature at which the sample will be reduced prior to oxidation. As of this manual's writing, the pulsed oxidation chamber is not set up for reduction. Nevertheless, **be sure to click the on/off switch for reduction!** If you need to perform a reduction, a supply of your reducing gas must be attached to a mass-flow controller that writes into COM9.
  - **Pumpdown Pressure (Torr)**: For each backfill, pumping will proceed to this pressure, followed by a backfill of UHP Ar. Standard is 0.01 mTorr.
  - **Backfill Pressure (Torr)**: Ar backfilling will proceed to this pressure for each backfill cycle. Generally 1-5 mTorr.
  - **Experimental Pressure (Torr)**: Oxidation will occur at this pressure.
  - **Number of Backfills**: The chamber will be pumped down and backfilled this amount of times prior to a final pumpdown and oxidation. Standard is 3.
  - **Process Time (s)**: This is the amount of time your sample will be exposed to simulated air at the experimental temp. Commonly 30-90 seconds.
  - **Fraction Sim Air**: Decimal input. This sets the relative amount of oxygen to argon in the simulated air during exposure. A value of 1 corresponds to 20% O_2 and 80% Ar. Fractions less than 1 decrease O_2, and fractions greater than 1 increase O_2 concentration. Standard is 1.
- Run the VI by clicking the "Run" arrow or using Ctrl-R

- Follow the on-screen prompt, replicated below:
  - Open gas tanks (Ar & Simulated Air)
  - Open cooling water flow to heater
  - Turn on heater power supply, check for any indicator lights
  - Turn on Pirani gauge
  - Turn on the roughing pump
    - If you hear hissing at this point, **DO NOT** continue until you are certain the chamber is sealed properly, lest you damage the turbo pump
    - At this stage you may see an error come up from LabVIEW relating to the **Pressure Readout VI**. Refer to the first entry of the **Troubleshooting** section to resolve
  - Pressure and temperature will be shown in real time on the front panel. The Photrix pyrometer doesn't read temperatures accurately below 265° C, so the temperature readout will be around 265° until the heater is turned on



Display portion of the front panel

  - After turning on the roughing pump, but before clicking **DONE** on the first "Initializing" prompt, make sure to move the gate control switch to the center position (Computer control). The chamber must be open to the roughing pump and pumped down before turning on the turbo. Otherwise the sudden load of atmospheric pressure on the turbo pump may damage the rotors
- Turn on the turbo pump

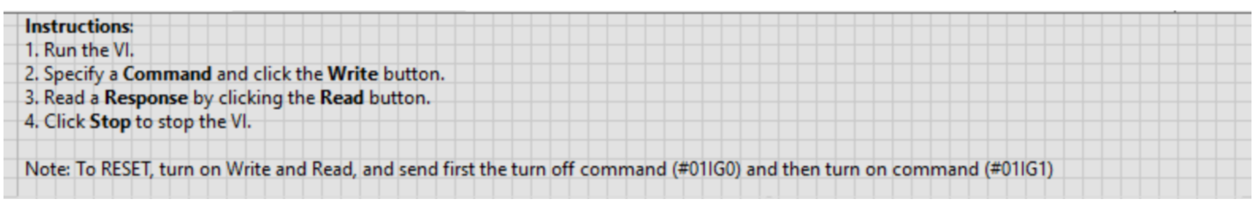
- The software will manage the pumpdowns to the specified pressure and backfill with UHP argon up the specified number of backfills, followed by a final pumpdown and backfill to the specified experimental pressure. Depending on the value used for pumpdown pressure, the initial pumpdown may take several hours. However, **it should not take more than two hours to pump down to $1x10^{-5}$ Torr with this system**. It helps to tighten the bolts slightly about halfway through the first pumpdown, ensuring a snug seal between the copper gasket and the plate. Be sure to tighten the bolts evenly across the plate, as before, but do not over-tighten, or it will be very difficult to retrieve your sample afterwards
- When prompted on-screen, turn on the heater using the switch on the side of the POC (below the gate valve switch)
- The temperature will rise and fluctuate about the experimental value, but should stabilize to experimental values within 60s or less. Pressure will also vary in this time, but should similarly stabilize at the experimental value. An on-screen prompt will ask you to confirm if Pressure and Temperature are stable. Confirming ***STABLE*** will begin the oxidation stage of the experiment
- During oxidation, you will hear the heater ramp up and down somewhat to maintain constant exposure temperature. The flow of simulated air will similarly fluctuate to maintain experimental pressure.
- Once the exposure is over, an on-screen prompt will direct you to turn off the heater and allow the sample to cool thoroughly before removal. It is best to let Ar flow through the chamber for at least five minutes before opening the chamber.
- When re-opening the chamber after an experiment, make sure the turbo pump is off, ensure the gate valve is closed, and use the vent valve at the bottom of the chamber to slowly equilibrate the chamber with atmospheric pressure

- Remove the 8 bolts and chamber once again
- Retrieve your sample and replace the dummy sample on the crucible ***OR*** place a new sample in the crucible and repeat the process.
- When finished for the day, make sure the dummy sample is in place to protect the pyrometer, close the chamber, and pump down for about a minute using the roughing pump to ensure minimal atmospheric damage to the chamber components

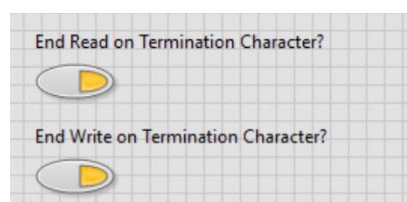
Troubleshooting

Most complications in the operation of the POC are related to the ion gauge and the turbo pump. Some of the most common problems and their solutions are listed here.

- **If LabVIEW is receiving no input from the ion gauge:**
  - Stop the POC software and open the "Pressure Readout" VI. Follow the instructions to reset the connection and restart the gauge:



  - Both switches relating to termination character must be lit:



  - In summary, to reset the gauge and LabVIEW connection, make sure both termination character switches are engaged as displayed above, send the "Turn Off" command by typing it into the input window and running the VI, then send the "Turn On" command similarly.
  - You should be able to run the POC VI and continue with your experiment
- **If the turbo pump controller shows an error:**

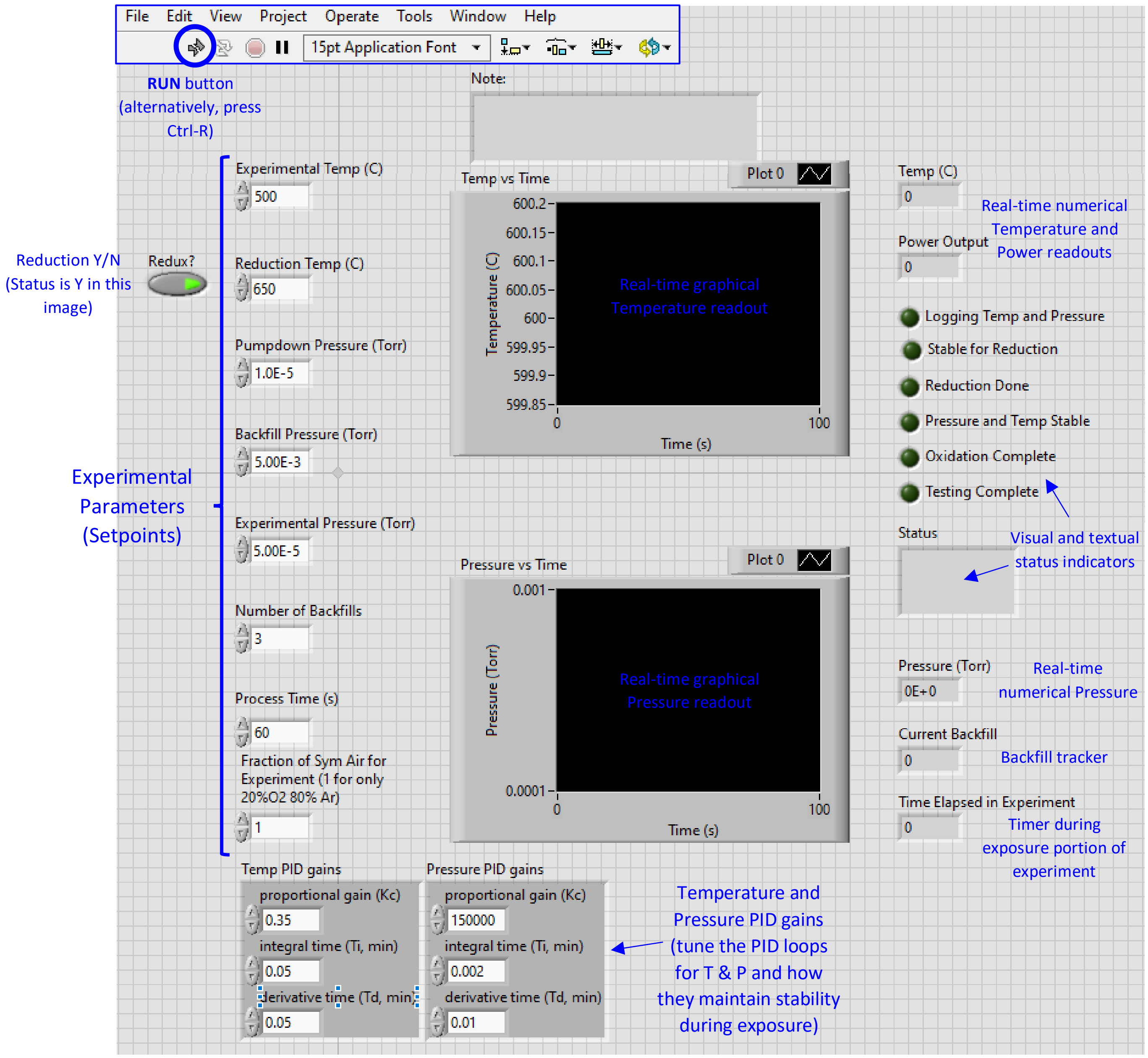
- Error code meanings can be found in the manual for the TC110 Electric Drive Unit. If the temperature is too high, there is usually either a leak in the system, or the turbo pump is experiencing a mechanical issue. Other error codes may show up, but they are often easily diagnosed with use of the manual

This is by no means a comprehensive list of errors you may encounter in using the POC. This is complicated piece of equipment. If you are unsure of what is going wrong or unsure if you are proceeding correctly, **STOP THE EXPERIMENT AND CONSULT SOMEONE WITH MORE EXPERIENCE.**

LabVIEW Visual Guide

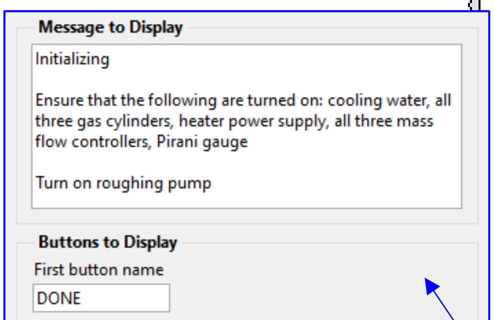
Front Panel

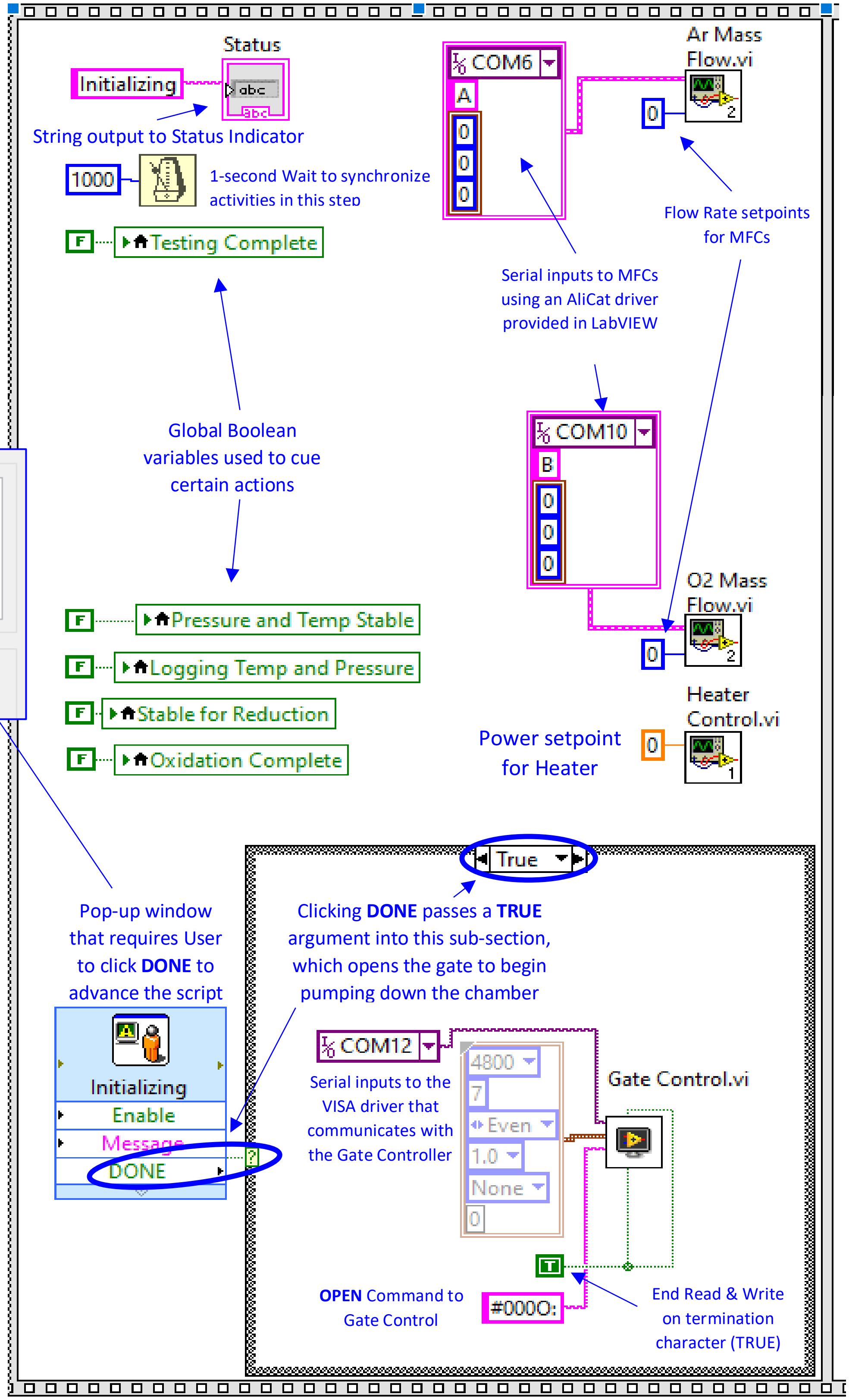
This window is all you really need for an experiment. If you want to make “big” changes to how the POC operates, you’ll have to access the Block Diagram (Ctrl-E).



Initialization

This stage ensures all the physical components of the POC are turned on and communicating properly with the VI. User input is required to advance, message shown below:

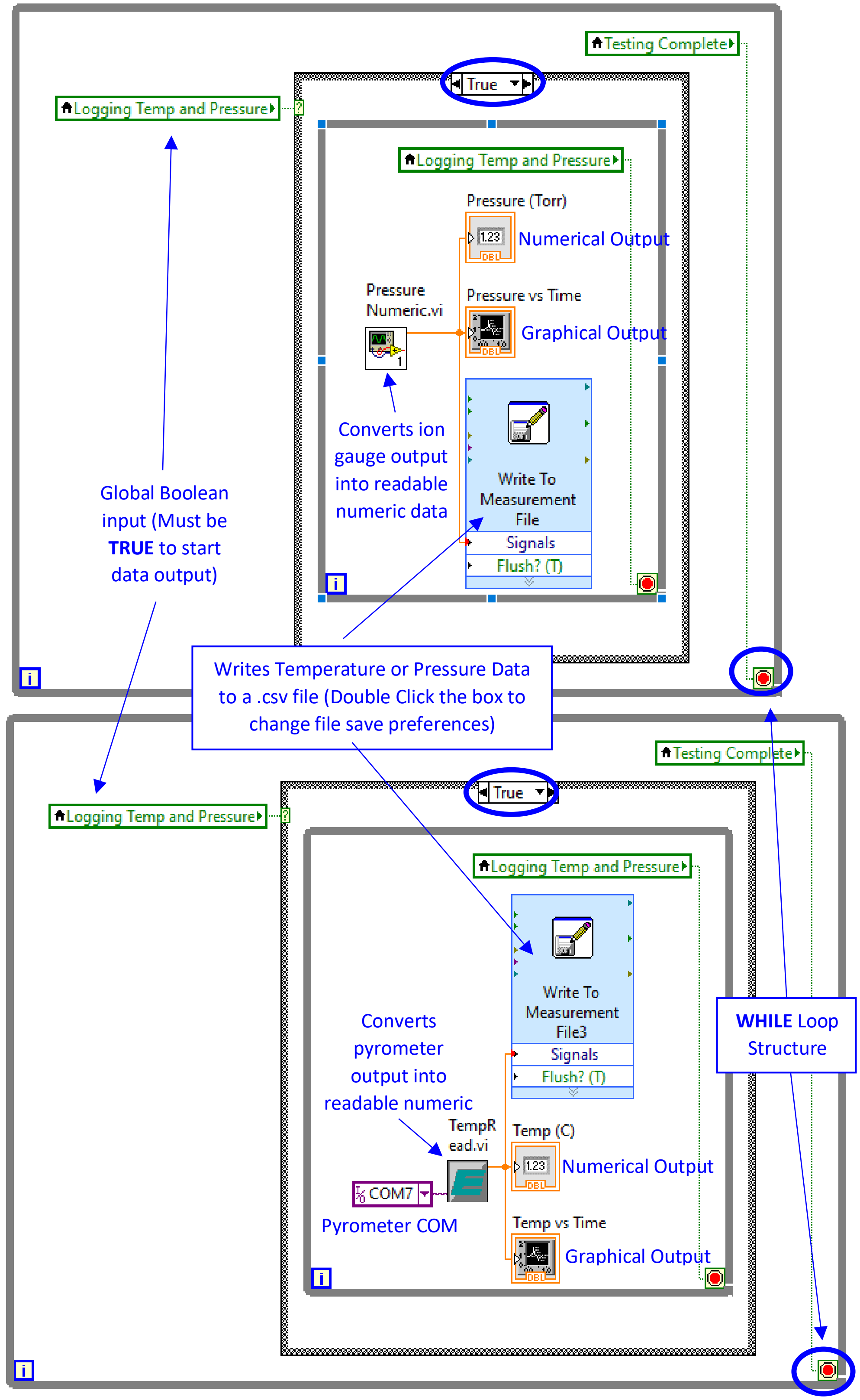




Logging Temperature and Pressure

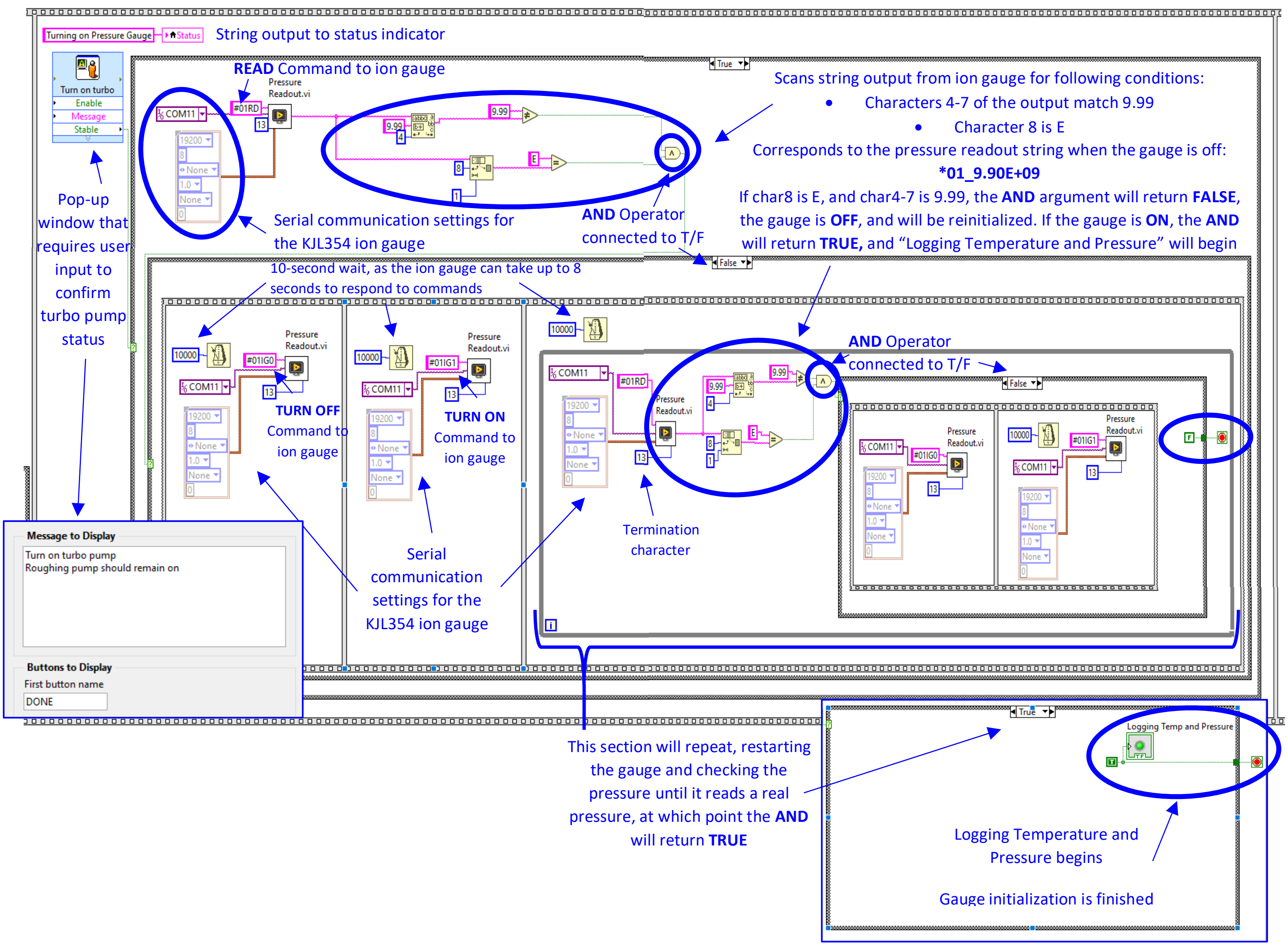
These are the only two modules outside the flat sequence. Both are *while* loops and will operate continuously until testing is complete.

These modules also control the graphical and numerical output of Temperature and Pressure to the Front Panel.



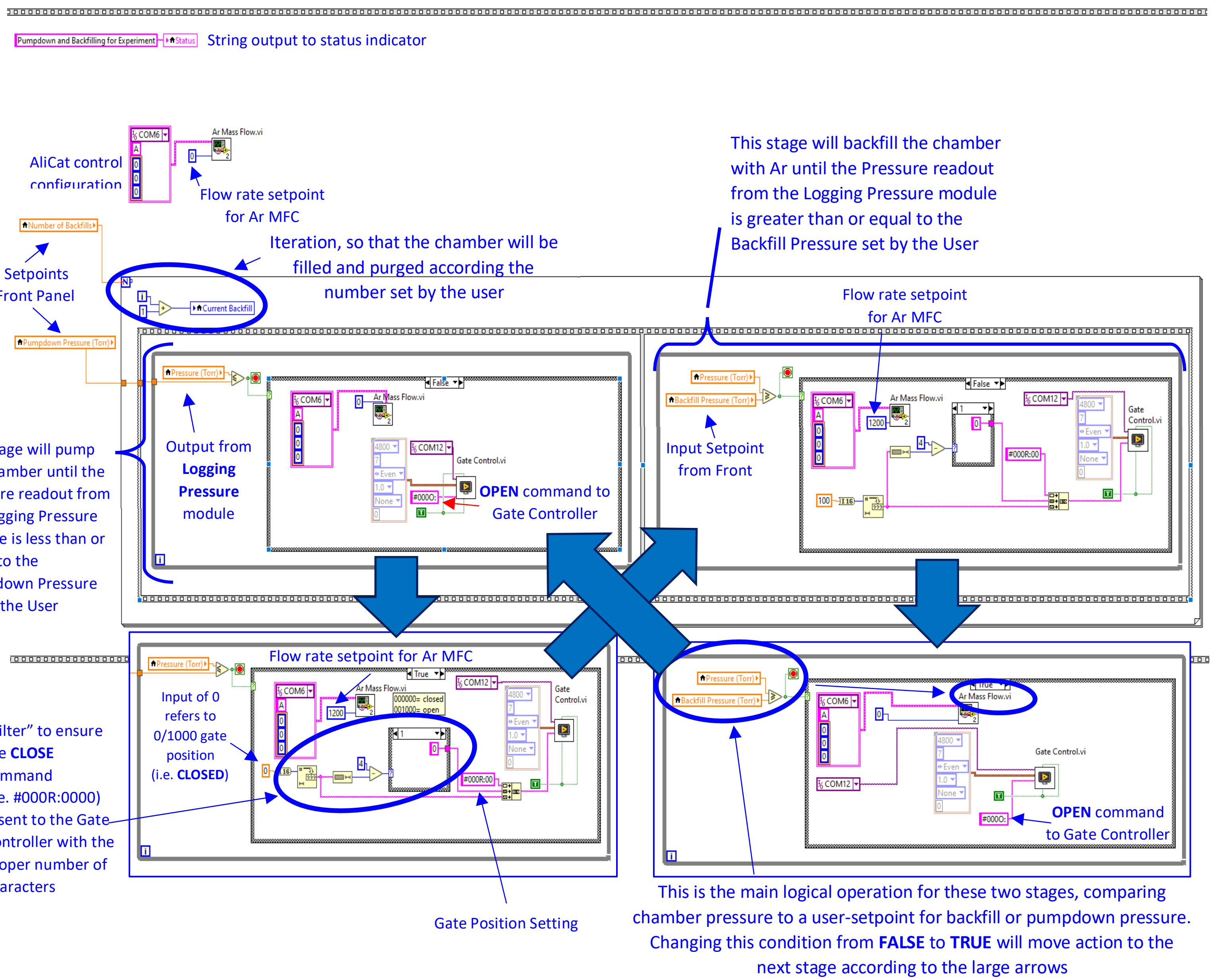
Turning on the Pressure Gauge

This stage initiates after input from the user confirms that the turbo pump is running. The ion gauge is only operational for pressures below 5.00x10^{-2} Torr. If the gauge is turned on above this pressure, it will automatically shut off to protect the sensitive components within. The purpose of this stage is thus to check the pressure of the chamber, and if the pressure is too high, re-initialize the gauge and check pressure again. Once the pressure is low enough, the "Logging Temperature and Pressure" Modules will begin to run.



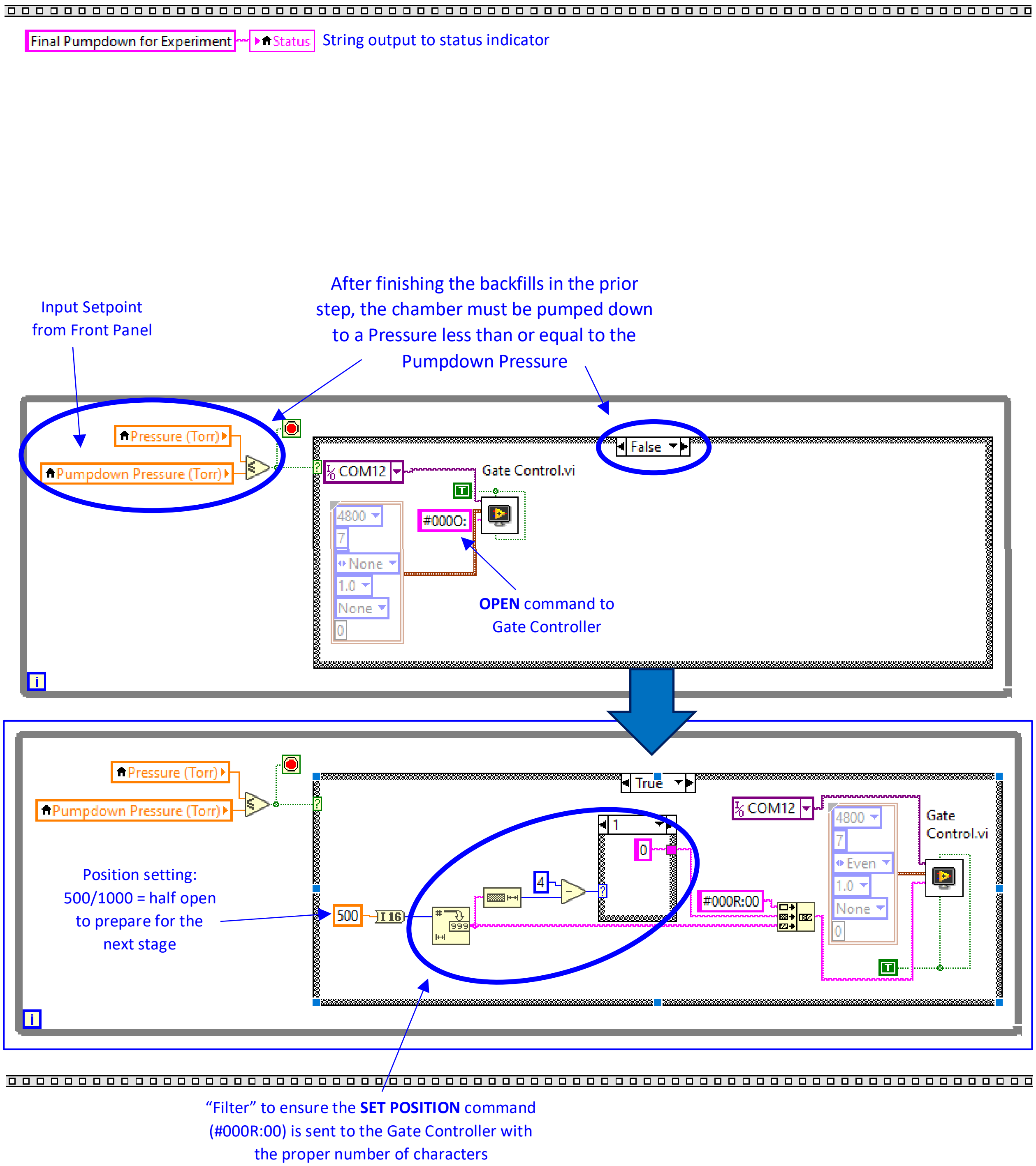
Pumpdown and Backfilling

Pumpdown and backfilling ensures the chamber is clear and the vacuum is sufficient for heating and exposure. However, looking at the Block Diagram, there is first a large section devoted to Reduction. There are sections for Pumpdown and Backfilling for Reduction, Final Pumpdown for Reduction, Heating for Reduction, Stabilizing Temperature, and Reduction. The oxidative exposure is often performed without prior reduction, so this glossary will focus on the oxidation phases, as they are very similar to the reduction steps, the main difference being the process gas during exposure.



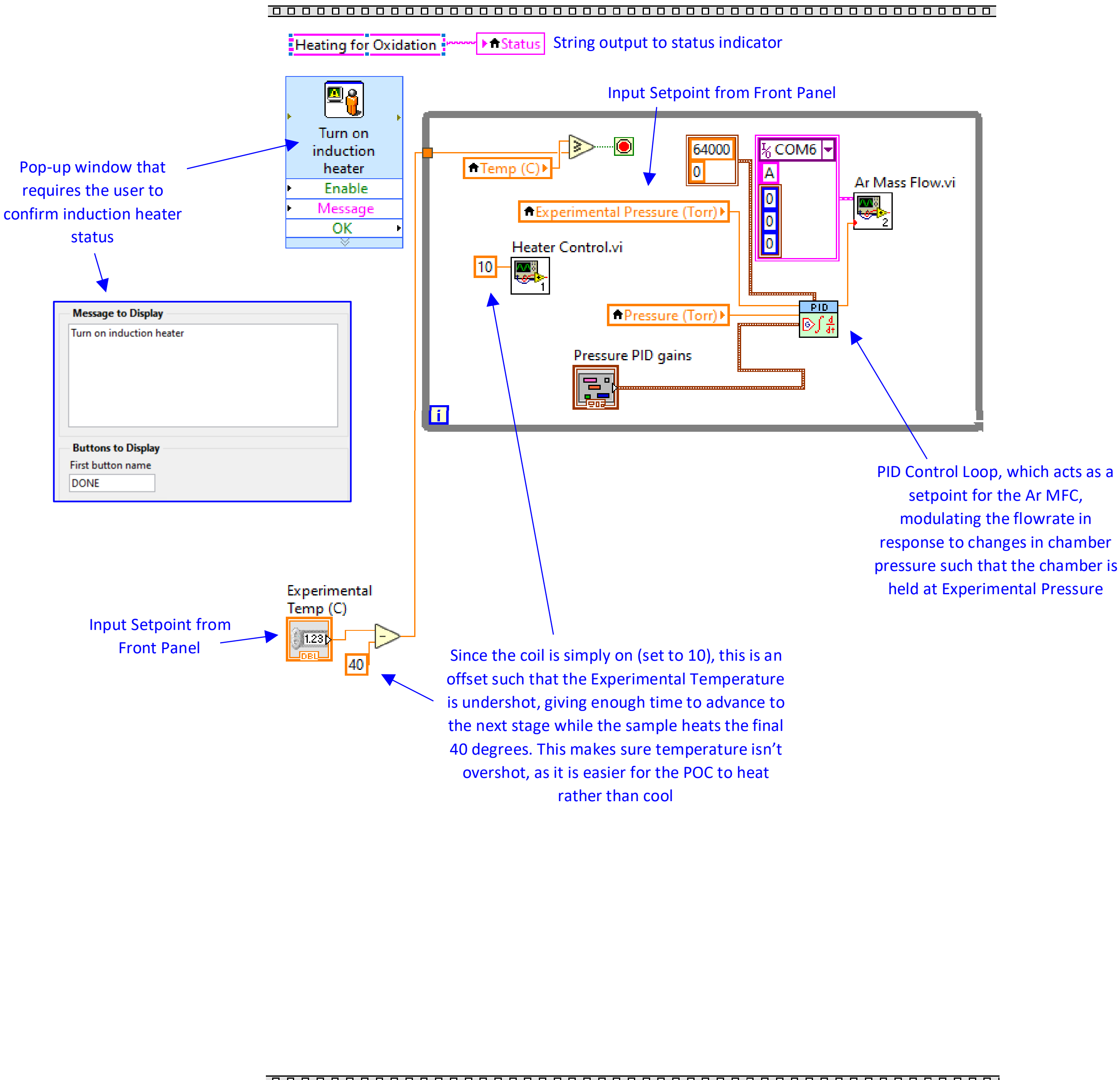
Final Pumpdown for Experiment

This phase follows the Pumpdown and Backfilling with a final Pumpdown to bring the chamber back down to the User-set Pumpdown Pressure. Note that this stage does not yet establish the Experimental Pressure, which begins in the next phase.



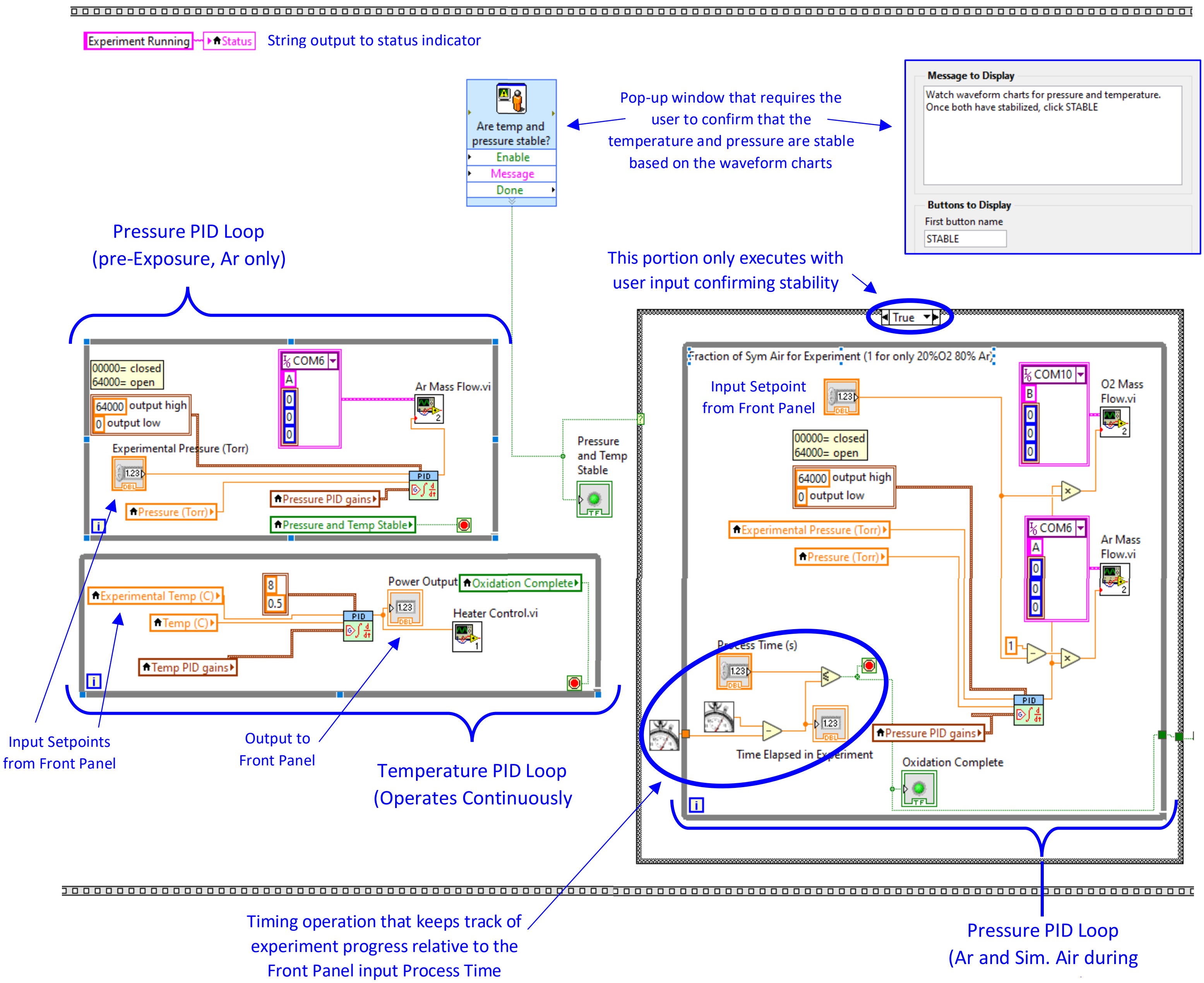
Heating for Oxidation

In this stage, the user is prompted to turn on the heater using the switch on the side of the POC. Doing so (and clicking **OK**) begins the ramp up to Experimental Temperature. Meanwhile, the gate is half open, which allows enough flow that the Ar MFC can reach and maintain the Experimental Pressure



Experiment Running

This stage is the actual experiment, where your sample will be exposed to simulated air at the Experimental Pressure for the Process Time. Maintaining stability in both Temperature and Pressure is tricky, and it is best to keep a keen eye on the Front Panel during this stage to ensure there are no unnoticed Experimental Temperature or Pressure deviations.



Purge & Cooling

This phase controls the effective cooling rate of the sample. Once the Process Time has elapsed, the chamber will be purged with argon for 10 seconds. Then the Logging Temperature and Pressure modules will be stopped, the gauge will be turned off, and the user will be prompted to turn off the heater, allowing the sample to cool under flowing argon.