Vigor Tech USA

Glovebox & Gas Purification Systems

General Operation Manual

Operation Manual Index

Section 1 Preparation

Warnings symbols – Meanings and locations

 Hazardous voltages are present. Access by untrained individuals is prohibited. Found on the lockable electrical compartment of the equipment and in this manual. Keys will be found inside. After locking, the keys should be held by the maintenance supervisor.

 Warning: Failure to properly adhere to the manufacturer's instructions may result in damage to the equipment. Found within this manual.

 Caution: Failure to follow the manufacturer's usage or maintenance suggestions may result in degraded performance or waste of resources, either time or tangible goods. Found within this manual.

1.1 Location Preparation

1.1.1 Room

Temperature Control	Minimum 5°C (41°F), Maximum 40°C (104°F)
Humidity	20% to 70%
Condition	Good Ventilation
Altitude	Up to 2000 meters
Additional	Access to exhaust system for glovebox vent tubes

1.1.2 Floor

Condition	Flat and level, Able to support equipment weight at least 700 Kg (1600 lbs.)

1.2 Gas Supply

1.2.1 Working Gas

Use	Required for operating certain system valves and to create an inert atmosphere inside the glovebox
Gas Types	Helium (He), Nitrogen (N_2), or Argon (Ar), gas cylinders (or boil-off from cryogenic liquid)
Quality	99.995% to ultra-high purity, 99.999%
Quantity	A type K or T* cylinder at approximate 2,400 ~ 3,000 psi (165 ~ 205 bar). K and T are supplier-dependent designations, but both are 250 cubic feet of gas.
Regulators	CGA350, Two-stage, capable of a delivery pressure of at least 80psi (~5 bar)
Connection	8 mm compression fitting (Provided by Vigor) to 1/4" NPT

1.2.2 Regeneration Gas

Use	Regenerates the purifier by reducing copper oxide back to copper metal.
Gas Types	Inert gas to match your working gas plus 5% Hydrogen, for example; 5% Hydrogen (H_2) in Helium (He); 5% Hydrogen (H_2) in Nitrogen (N_2) 5% Hydrogen (H_2) in Argon (Ar)
Qualities	99.995% to ultra-high purity, 99.999%
Quantity	Requires approximately ½ to 1 full type K or T cylinder at 2,400 ~ 3,000 psi (165 ~ 205 bar) for each regeneration cycle
Regulators	CGA580 or CGA350, Depending on the gas supplier, two-stage, capable of a delivery pressure of at least 40 psi (~3 bar)
Connection	8 mm compression fitting (Provided by Vigor) to 1/4 “or 3/8 “NPT

1.3 Power Supply (Standard USA Configuration, 220VAC units are also available)

Main Power	Dedicated 110V – 120VAC, 60Hz, 15A Circuit, Grounded (USA) Connection to the supply is via a standard grounded AC power plug.
Heated Antechamber (option)	~120VAC, 60Hz Units with flat metal heaters are 300W/plate. Units have two (FLC 5A) or three plates (FLC 7.5A). Units with IR heating tubes require a maximum FLC of 8.4A. The heater unit connects to the supply via a standard grounded AC power plug.
Freezer (option)	~120VAC, 60Hz, 300W (Maximum FLC 2.5 A) The freezer unit connects to the supply via a standard grounded AC power plug.

Section 2 Installation

Note: See warning on p. 14 regarding sensor lifetimes

2.1 Equipment Placement

Four casters are installed under the purifier compartment. The equipment can be easily moved on them and positioned, where desired, in the room. After positioning the glovebox at the intended location, please use a wrench to adjust the levelers in each of the four legs of the stand so that the casters are raised are off the ground and the glovebox is level in both planes.

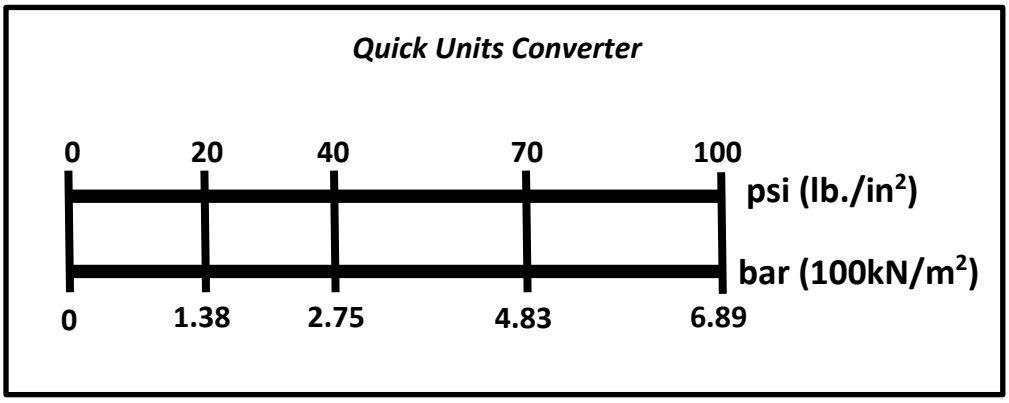
Caution! (Model dependent note)

To install a double length glovebox comprising two separable sub-units, first adjust the height of, then level, one glovebox section. Then adjust the height of the other glovebox section to match the first one and apply vacuum grease to the rubber seals. All the bolts and nuts are inserted and slowly tightened in an alternating diagonal sequence. Don't over-tighten as you may actually crush the rubber seal.

Inside the glovebox, a vacuum line connects the two glovebox sections together at the seals. This is a small diameter SS tube (4 mm). Tighten the fittings firmly.

2.2 Gas Connections

Warning!
Setting the working gas pressure too high (> 100 psi) will damage the equipment. However, the best value to use is usually 80 psi.



2.2.1 Working Gas

Attach a gas regulator to a cylinder of your working gas, which may be nitrogen, helium or argon. Use the included length of nylon tubing (8 mm OD, maximum pressure 120 psi) or copper tubing to connect the gas regulator to the working gas port of the glovebox. Adjust the regulator to deliver the working gas at 80 psi.

2.2.2 Regeneration Gas

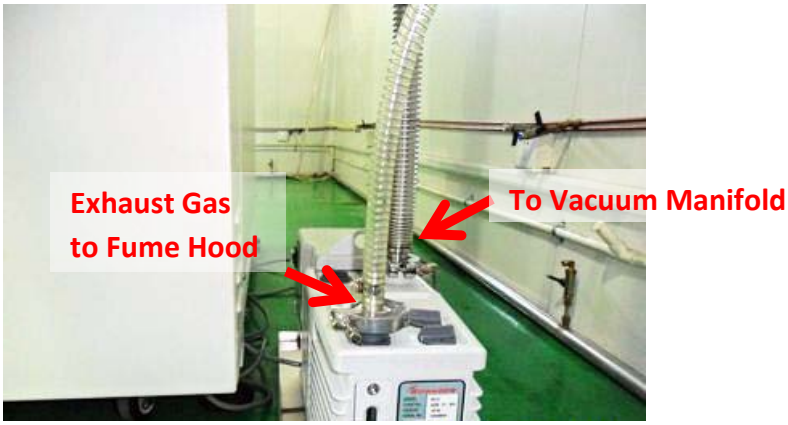
Attach a gas regulator to the cylinder containing the regeneration gas. Use the other included length of nylon tubing (8 mm OD, maximum pressure 120 psi) or copper tubing to connect the gas regulator to the regeneration gas port of the glovebox. Adjust the regulator to deliver the regeneration gas at 15 to 20 psi.

2.2.3 Vent

During the regeneration of the purifier, regeneration gas flows through the heated purifier to the vent port. Because the purifier has adsorbed some chemicals, and because the regeneration gas also contains hydrogen, it should be vented to the outdoors or to a fume hood using the provided vinyl tubing.

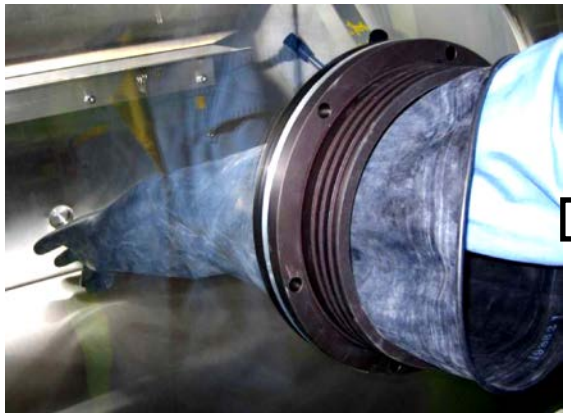
Warning!
If harmful materials are used in the glovebox, all exhaust gases from the glovebox system, whether resulting from regeneration, purging or from the vacuum pump, should be vented directly to the outdoors or to a functioning fume hood.

2.3 Vacuum Pump and Exhausted Gas



Vent the exhausted gas from the vacuum pump to the outdoors or to a functioning fume hood using the included plastic tubing, secured to the pump with a hose clamp.

2.4 Installing the Gloves



Insert your hand and arm into the glove to be installed and slide the glove into the box through the glove port. Rotate the glove so that the thumb is pointed up. Ambidextrous gloves are the most common, but some gloves may have left and right-handed versions. Check before installation.

Slide your hand out and turn the cuff inside out and fold it back over the glove port. Rotate the cuff of the glove to further adjust the thumb's position if needed.

There are three grooves on the glove port. Adjust the glove cuff so that the bead of the glove is positioned in the first groove from the window. Place two O-rings over the gloves and seat them into the other two grooves.

2.5 Feedthrough installation for power strip (Optional)

Remove the blanking flange on a feedthrough port of your choice.

Feed the plug through the KF-40 O-ring and through the port.

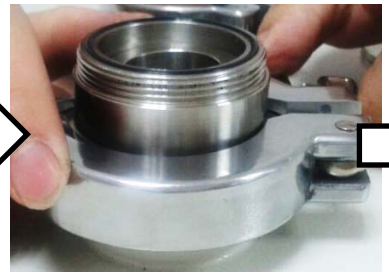
Slide the cable into the box until the flange on the cable mates with the KF-40 O-ring on the flange of the glovebox. Use a KF40 clamp to attach the two flanges firmly together.

2.6 Installation of oxygen and moisture analyzers (purge first, see first page following section 2.6.2)

2.6.1 Oxygen analyzer (GE oxy.IQ)

Set the box pressure lower limit to +5 mbar. Remove the blank cap, and install the KF-40/KF-25 adapter.

Tighten the KF-40 clamp to firmly secure the adapter.



Connect the power cable to the rear of the transmitter.

After connecting the power, a display will appear on the LCD screen.

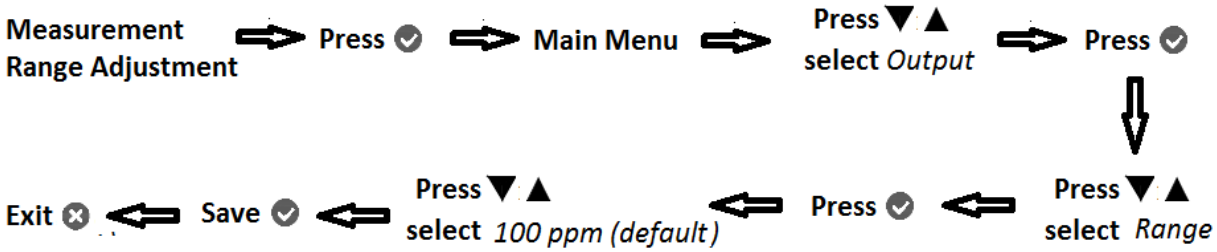
Open the sealed bag containing the OX1 sensor cell and remove the red tape tabs.

Orient the sensor so that its gold-plated electrodes are facing the spring-loaded contact pins of the transmitter base. Firmly press the analyzer into the base.

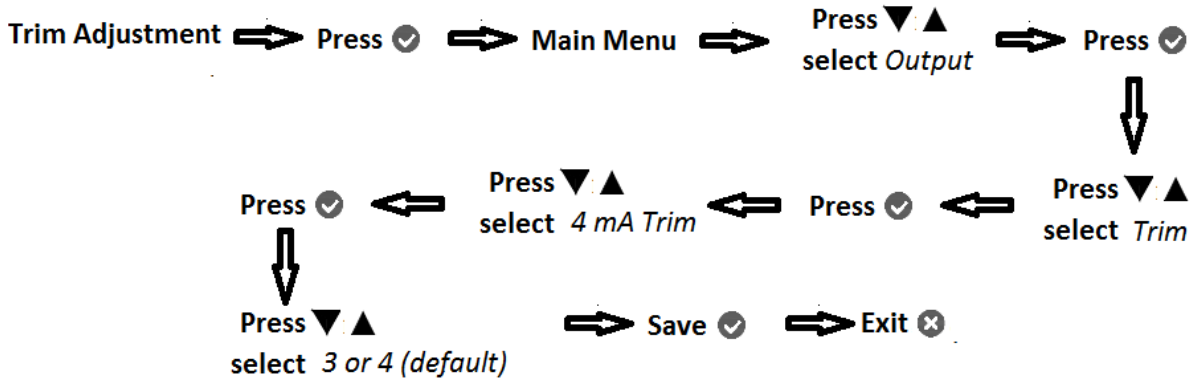
You should now notice a non-zero value on the display. Attach the transmitter to the adapter and firmly tighten.

The measured oxygen level should now start to display on the LCD screen.

Oxy.IQ - Measurement Mode



Note: If O_2 value on the small display is different from touch screen display (> 0.1 ppm), please adjust the **Trim.**

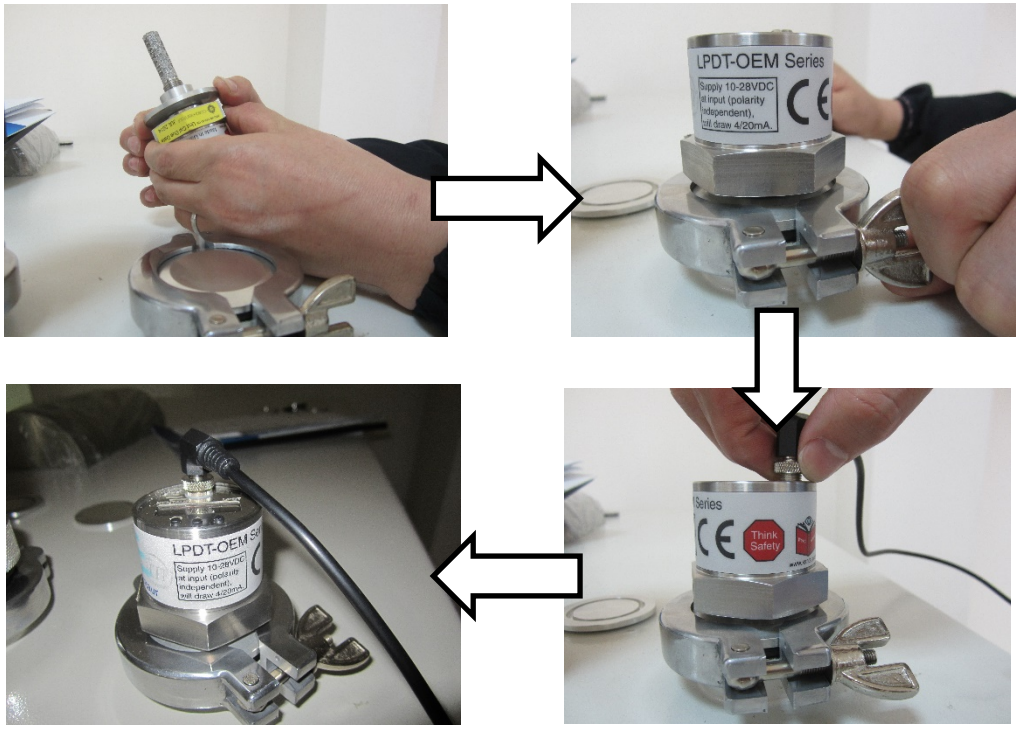


*All the instructions and illustrations are from the *GE Oxygen Transmitter Model Oxy.IQ User's manual, pages 4, 15, 16 & 46.*

2.6.2 Moisture Analyzer (Xentaur)

Install the moisture analyzer and rubber washer into the special KF-40 flange. Open the KF-40 clamp, remove the blanking cap, and quickly put the analyzer flange into place and tighten the KF-40 clamp.

Gently hand-tighten the analyzer until the washer is fully compressed to form a gastight seal. Don't use excessive force since the seal is rubber.



The analyzer becomes active as soon as the cable is attached.

Connect the BNC signal cable to the moisture analyzer. It will only attach in the proper orientation.

Warning!
New glovebox installations require purging with at least 1 cylinder of working gas per glovebox section prior to installation of the analyzers. Exposure of the oxygen sensor to high oxygen levels will shorten its lifespan.

The life span of the oxygen sensor cell (GE OX-1) can be one - two years. If you need a replacement, please contact Vigor Customer Service or GE. As the sensor fails, it normally begins to display artificially high values for oxygen.

The time between calibrations for the moisture sensors is 1 year. If you need a recalibration or a replacement, please contact Vigor Customer Service or the sensor manufacturer. A sign of a failing sensor is a low and unchanging moisture reading.

To efficiently bring the glovebox online

To maximize your efficiency in removing atmospheric gasses, the following steps should be taken when bringing your glovebox online.

When a new glovebox leaves the factory, it has already been put through a series of tests to assure that everything is performing as it should be. Since recirculation is tested, the purification train has already been exposed to some amount of air and moisture. So, when the glovebox arrives at your facility you start out with 210,000 ppm oxygen inside and a partially depleted zeolite/Cu bed. We still want to make efficient use of the residual purifier capacity.

Once you have completed the mechanical steps in setting up, take a moment to look things over once more. Check the little things, like making sure there are no creases in the gloves under the O-rings, that all clamps are tight (specifically, we suggest checking that every joint that is held together with an O-ring and clamp be examined and determined to be fully finger-tight), and that your supply lines are well sealed, etc. Before starting the actual purge, you can run a pressure-based leakage test (as shown in touch screen document) for a test period of 5 minutes. When the test completes, see the settings page to view the leakage rate. If the leakage rate exceeds 0.5 %vol/hr., turn off the working gas supply, find and fix any leaks, re-test and then turn the working gas supply on again in preparation for the purge.

i. The glovebox needs to be purged at about 1- 2 bars of working gas pressure if your purge exhaust valve (on the RHS of the glovebox, typically) is electrically actuated. If it has a plastic gas supply line running into it, it is a pneumatic valve and purging is performed at 3-5 bars. The actual purging pressure is facility-dependent and depends on the partial vacuum level of the exhaust. **The correct purging pressure for your particular glovebox is determined by starting at low pressure and increasing the**

pressure, slowly, until all valve noises stop. Purging removes the vast majority of the oxygen and nitrogen, and a good part of the moisture. Moisture is more slowly removed since it exists not only in free vapor form, but also as physically adsorbed moisture on the interior surfaces of the glovebox. To achieve a thorough purge, approximately one to one and a half K or T cylinders of working gas are needed per single glovebox unit or two to three cylinders for a double box. The sensors are installed after completion of this step. The residual oxygen level is usually between 100 and 200 ppm at this point and so the touch screen typically shows an overflow value near 100 ppm and is apparently unchanging.

ii. Since the bellows tubing still contains some atmospheric gasses, a recirculation is performed to sop up the residual impurities in the box and the bellows, including water which is slowly desorbing from surfaces. **After purging, you must set the working gas delivery pressure back to 5 bars for normal operation (to recirculate).** After a short time (an hour is common) the displayed oxygen value drops below 100 ppm. Recirculation is continued for as long as the sensors indicate that oxygen and moisture levels are significantly dropping. If you don't have sensors, consider running this step for a minimum of 4 to 6 hours and overnight if time allows. Oxygen typically drops to below 20 ppm by the time of completion of this step and frequently reaches single digit ppm levels.

iii. At this point the purification beds have expended much of their capacity and a regeneration cycle should be run. Once you start the regeneration process we suggest that you let it run to completion, typically almost a full day, and although stopping in the middle is possible, this is not a good idea.

iv. If the previous circulation didn't get the box to very low impurity levels, a short recirculation with the fully activated purifier bed will quickly accomplish this. Note that water may take a couple days to stabilize with an empty glovebox, and longer if you have already brought items into the glovebox.

We will now proceed to examine the actual control systems needed to accomplish the startup in section 3.

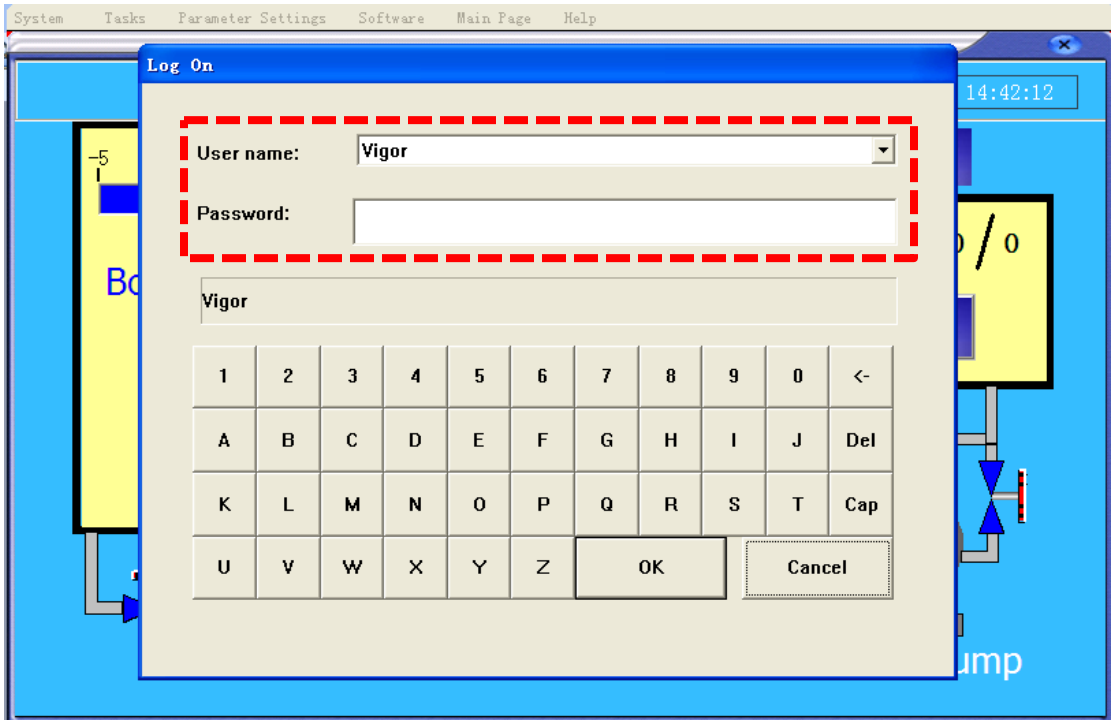
Section 3 System Startup

3.1 System Startup

The main electric power supply switch for the glovebox is located on the side of the electrical control box.

Main Power	0 = OFF
	1 = ON

3.2 System Login



There are three types of users set up in glovebox system. In order to fully use glovebox system functions, please login using VIGOR as the user name and no password. The three user accounts are:

User name	*Password*
ADMIN	(Blank)
VIGOR	(Blank)
USER	(Blank)

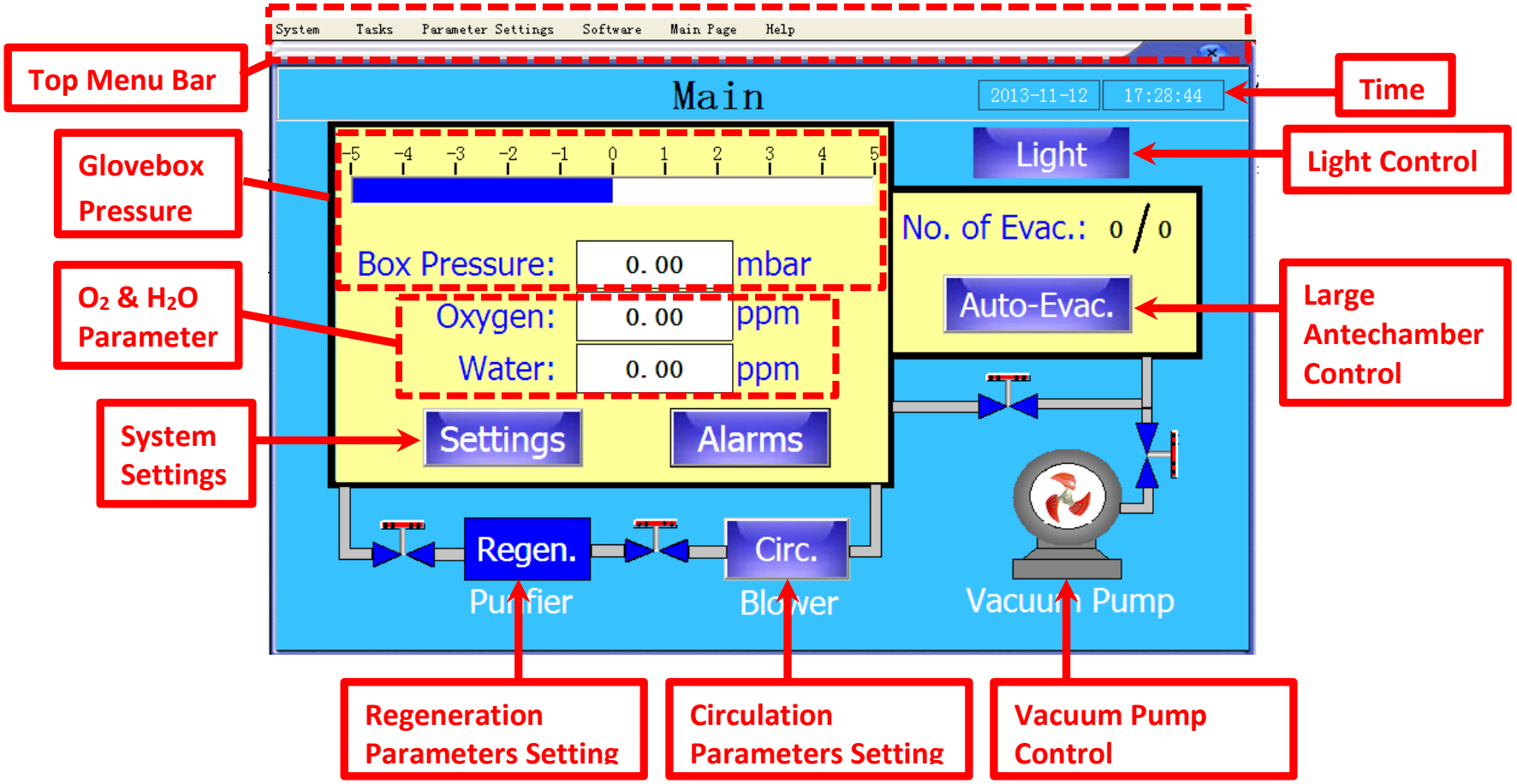
You can select passwords and save them as needed.

To Log in, please look to the menu bar, press “System”, “User Account”, “Log in” and then choose account name, enter password (or leave blank if you don’t set a password), and press “OK” to log in.

Caution!
If you log into the main menu of the glovebox system, but you are still unable to control glovebox functions or change parameters on the main menu or sub menus, please contact Vigor customer service to request further log in instructions.

3.3 Main Menu

About ~30 seconds after logging in to glovebox system, the touch screen will display the main page.



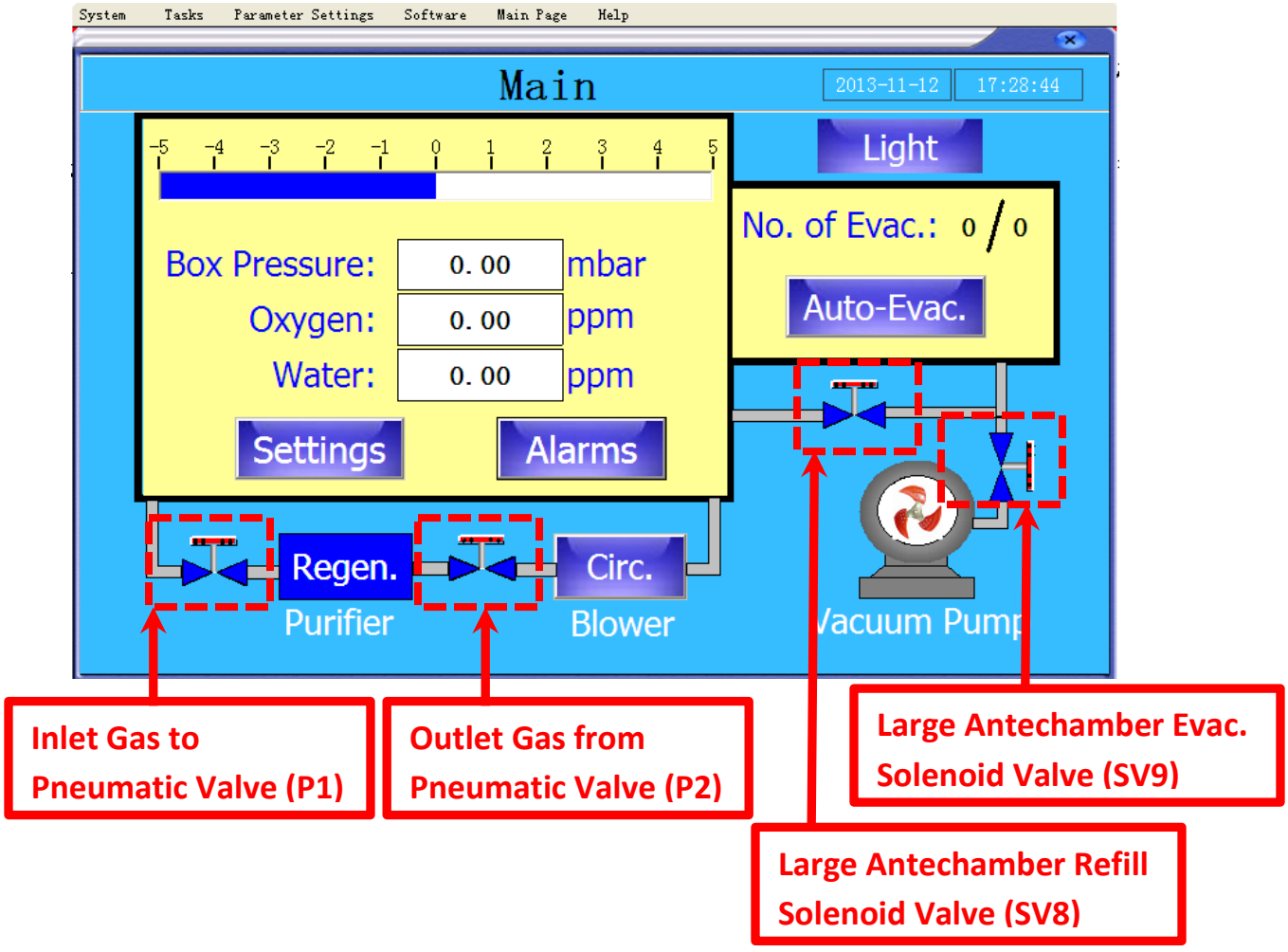
3.4 Functions

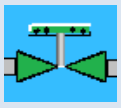
There are several accessible functions on the main menu page.

	Alarm Setting	When the system encounters problems, this button will flash. Press the button to enter the Alarm page to get specific information.
	Large Antechamber Control	Touch this button to enter the Auto-Evacuation page to set the auto-evacuation parameters and to start auto-evacuation of the large antechamber.
	Circulation	Touch this button to enter the Circulation page to control the purifier circulation.
	Light	Touch this button to turn the glovebox light on or off.
	Regeneration	Touch this button to enter the purifier Regeneration page.
	Setting	Touch this button to enter the System Settings page where the system parameters are entered and displayed. The system parameters entered here include the Box Pressure limits and the impurity levels necessary to cause an alarm.
	Vacuum Pump Control	This touchscreen icon represents the vacuum pump. Touching the icon once will turn the vacuum pump on and touching it again will turn it back off.

3.5 Valves Controls

On the main menu page, there are four user-controlled valves (subject to the limitations by active processes in play at the time). The valves are opened and closed by touch. Green valves are open and blue valves are closed.

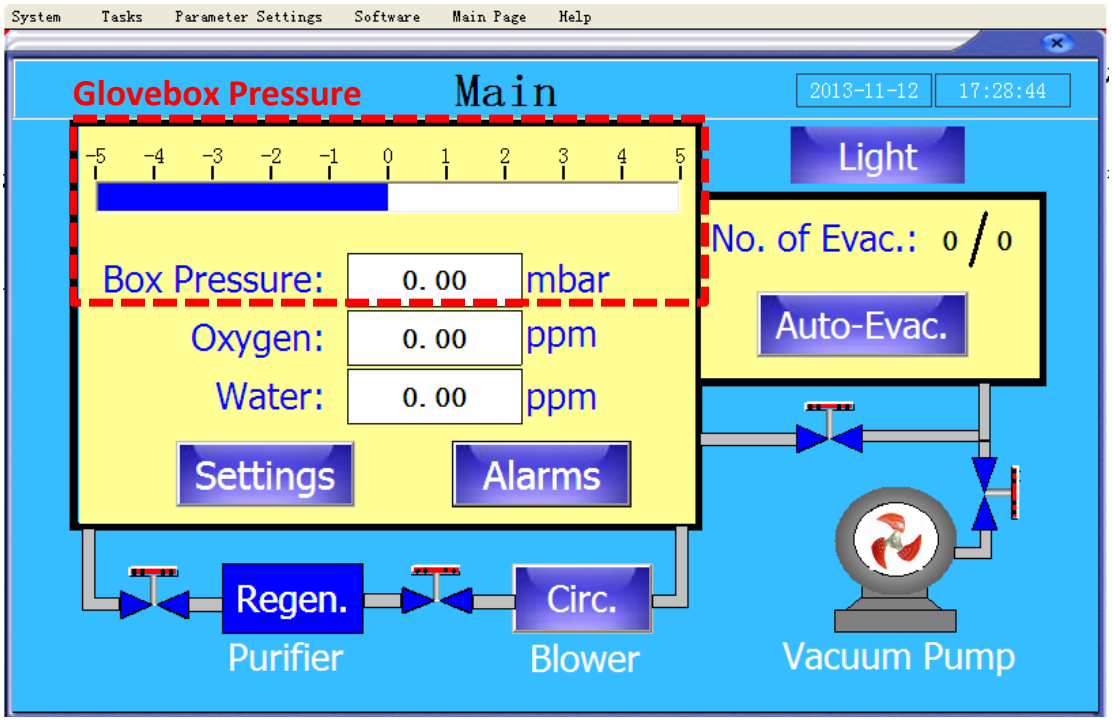


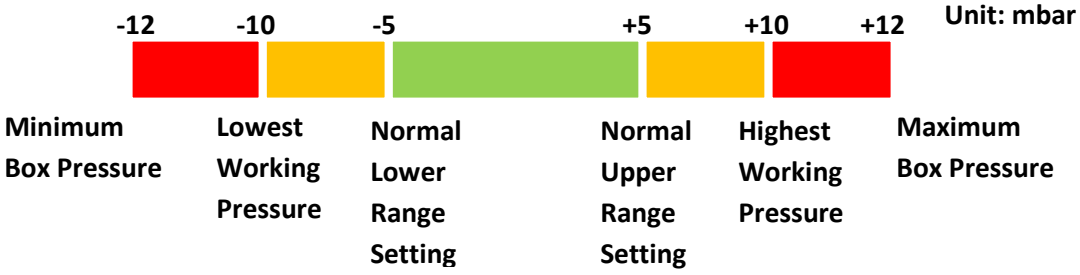
	Valve Opened
	Valve Closed

3.6 Glovebox Pressure

The pressure limits set for the glovebox interior are relative pressures, referenced to the surrounding atmospheric pressure. A positive pressure indicates the pressure inside the box is greater than the outside atmospheric pressure, while a negative pressure indicates a lower pressure than outside the glovebox. The unit of the pressure used here is the mbar, or one thousandth of a bar.

Section 4.4 has the necessary details for setting the glovebox pressure range. Generally, operating pressures limits between 0 and a few mbar are good for most users.





The working pressure in the glovebox can be set to a pressure ranging from **-10 mbar to +10 mbar**. Once the range is set by the user, the PLC will automatically maintain the working pressure within the specified range.

Maximum Box Pressure	The highest permissible glovebox pressure is 12 mbar. When the glovebox pressure exceeds this value, the safety valve on the glovebox is actuated and the excess pressure is vented to avoid damage to the equipment.
Highest Working Pressure	The highest working pressure is +10 mbar.
Normal Upper Range Setting Pressure	The box pressure is automatically maintained at or below the Upper Set Point. When the glovebox pressure is higher than the set point, the valve connecting the box to the vacuum system is opened and some of the gas inside of the glovebox is evacuated to bring the measured pressure below this set point.
Normal Lower Range Setting Pressure	The box pressure is automatically maintained at or above the Lower Set Point. When the glovebox pressure is lower than the set point, the valve connecting the box to the working gas is opened and gas is added to the glovebox to bring the glovebox pressure above this set point.
Lowest Working Pressure	The lowest working pressure is -10 mbar.
Minimum Box Pressure	The lowest permissible glovebox pressure is -12 mbar. When the glovebox pressure lower than -12mbar, the glovebox pump will automatically shut down.

Section 4 Functions and Operation

4.1 Intended Use

The intended use of this equipment is to provide an inert environment suitable for the performance of experiments and processes which would be harmed by exposure to atmospheric gasses and/or atmospheric moisture. The glovebox, as mentioned earlier, may use nitrogen, argon or helium, as appropriate for the processes. While a glovebox is sometimes configured to control one or the other of the primary atmospheric impurities, in the vast majority of cases, both oxygen and moisture are controlled. This glovebox was designed to typically keep both of these impurities below 1 ppm. If your glovebox is equipped with dual purifiers connected sequentially, the impurities are typically held below 0.1 ppm. If your glovebox is equipped with dual purifiers in parallel, it provides a way of instantly switching to a fresh purifier from a used one to avoid work interruption.

4.1.1 The effects of integrated equipment

The glovebox is often modified to integrate other devices. These can be standard glovebox add-ons such as refrigerators or heated antechambers or they can also be research devices such as deposition systems, spin-coaters, microscopes, etc. or devices to support them, such as air-conditioners. The effect of most integrated equipment on the normal operation of the glovebox is negligible. Special equipment, such as tube furnaces, or anything which can exert a substantial effect on the glovebox environment, must be discussed with our factory engineers.

4.2 Purge

When impurities are introduced into glovebox, we recommend turning off the circulation system and purging the impurities out of the glovebox (5 – 10 minutes). Doing so will avoid introducing the impurities into the gas purification column and prematurely consuming the purification capacity.

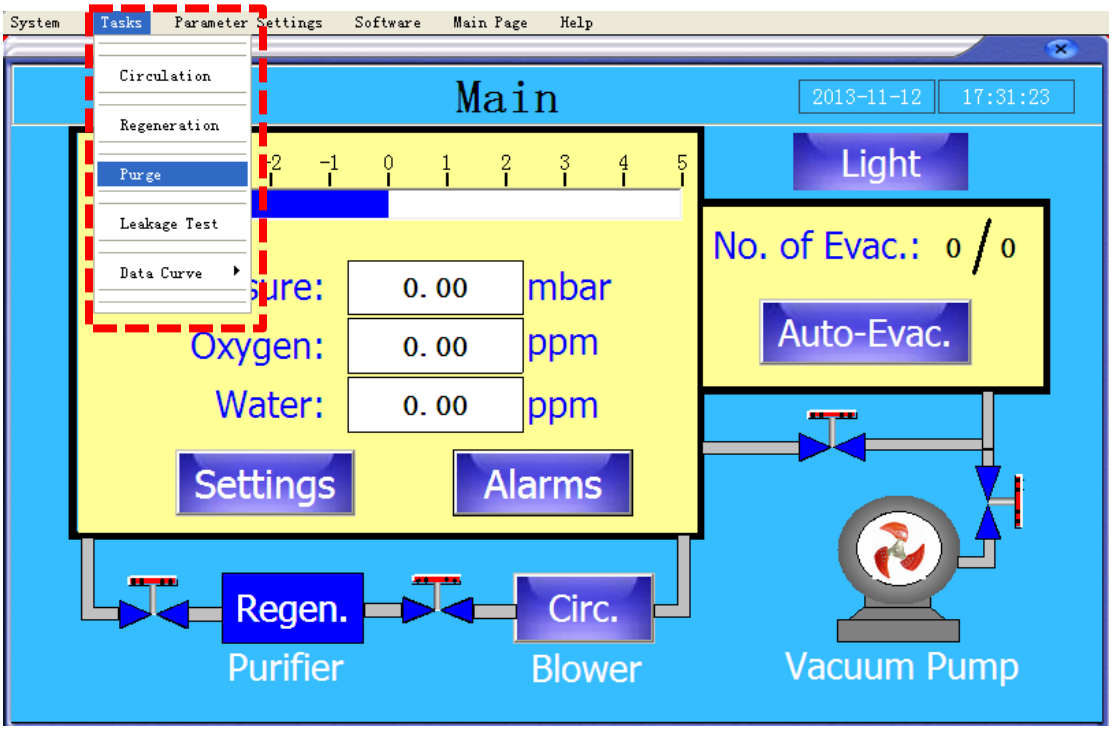
Caution!

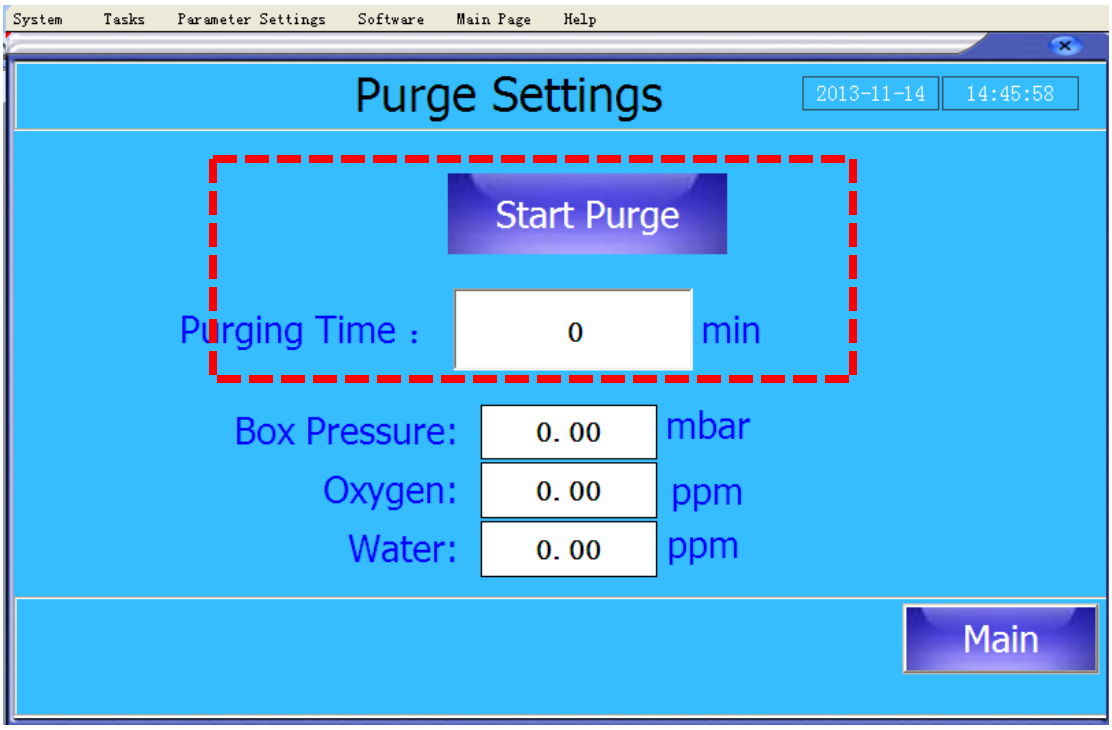
1. Set the Box Pressure upper limit to **8 mbar** and lower limit to **5 mbar** before activating the purge function.
2. Be sure that working gas is being delivered at a minimum ~**35 to 40 psi**. If not, the glovebox will be unable to open the pneumatic valve (P3) which vents purge gas.

Warning!

To activate the purge function, the Circulation and Regeneration need to be completely turned off. Circulation should be set to manual and then turned off.

Please enter the purge function page via the top menu bar and select Purge





Enter a value in the **Purging Time** box and press **Start Purge** to activate the purge function. Purging will stop after the specified time period. It can be stopped at any time by pressing the **Stop Purge** button.

Tips!

1. When a new glovebox is installed, the initial atmosphere is air which must be purged to bring impurities to 100 - 200 ppm before turning on the gas circulation system. This requires a minimum of 1 cylinder of working gas/glovebox section. Set the Purging Time to a large number (e.g. 500 = 500 minutes) to assure that it doesn't turn off during this long purge.

2. Some organic solvents or reagents may damage the adsorbents. The manufacturer of the O_2 adsorbent states: The **lifetime of the catalyst** depends on the application, the operating conditions, and the level of contaminants, such as arsine, sulfur, chlorine, silicon and mercury in the glovebox atmosphere. According to the manufacturer, typical lifetimes range from 5 to 10 years, unless poisoning occurs. Purging is a prudent practice whenever chemicals are released into the glovebox atmosphere, but particularly so if the above mentioned elements and/or compounds are present.

3. When gloves or seals are damaged, air may leak into the box and the oxygen level may quickly exceed 100 ppm. The purification system should be immediately turned off and purging performed until the oxygen level decreases to less than 100 ppm before investing time to find/fix the issue. This will protect your samples and the oxygen sensor.

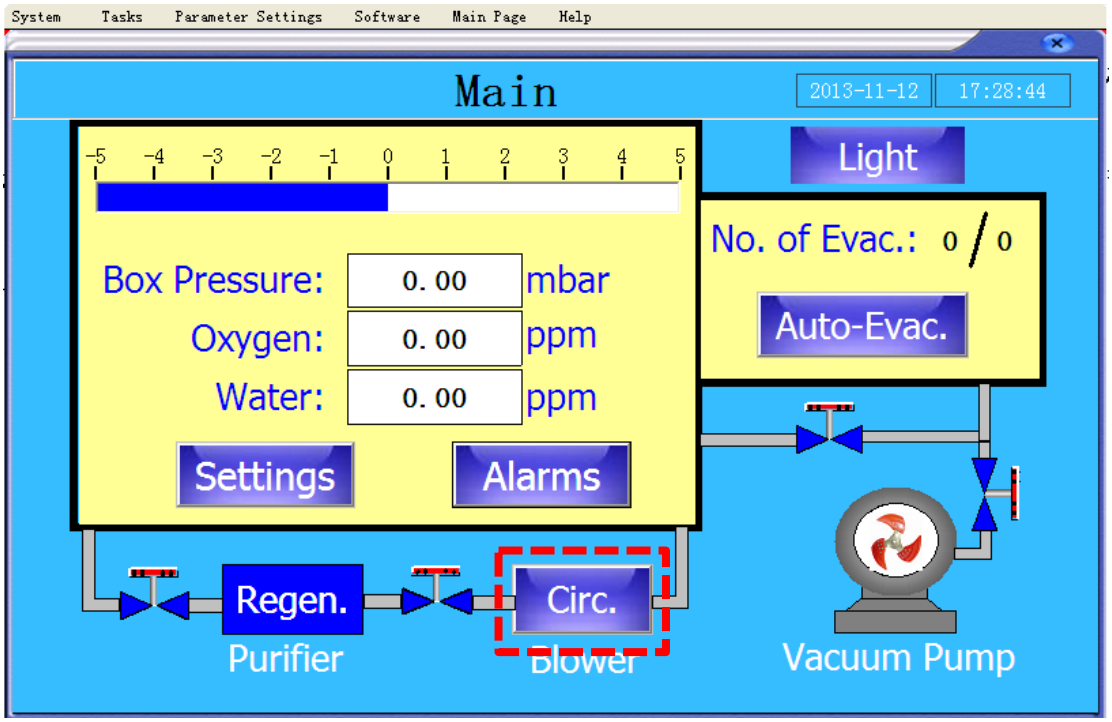
4.3 Circulation

After a purge or regeneration cycle completes, the circulation function removes residual impurities to < 1 ppm. Note: The very first circulation, performed during set up, may only bring impurities to single digits before regeneration is needed. Thereafter, it will achieve an impurity level of < 1 ppm until it is time to regenerate it again.

Warning!
Before starting the circulation, please check to assure that:

1. The purge function is not running.
2. Any running regeneration cycle has fully completed and that the purifier(s) is/are completely cooled down to room temperature. *Please DO NOT restart circulation before the regeneration fully completes (20 hours) as this may damage the glovebox permanently.*
3. At least **80 psi (~5 bar)** of working gas pressure is available to allow circulation to start. If the pressure is too low, circulation will not start.
4. The oxygen level is <100 ppm (or from 100 - 200 ppm during initial startup).

Press Circ. on the main menu page to enter the circulation page. There you will specify whether you want the purifier to circulate in the manual or the automatic (on demand) mode.



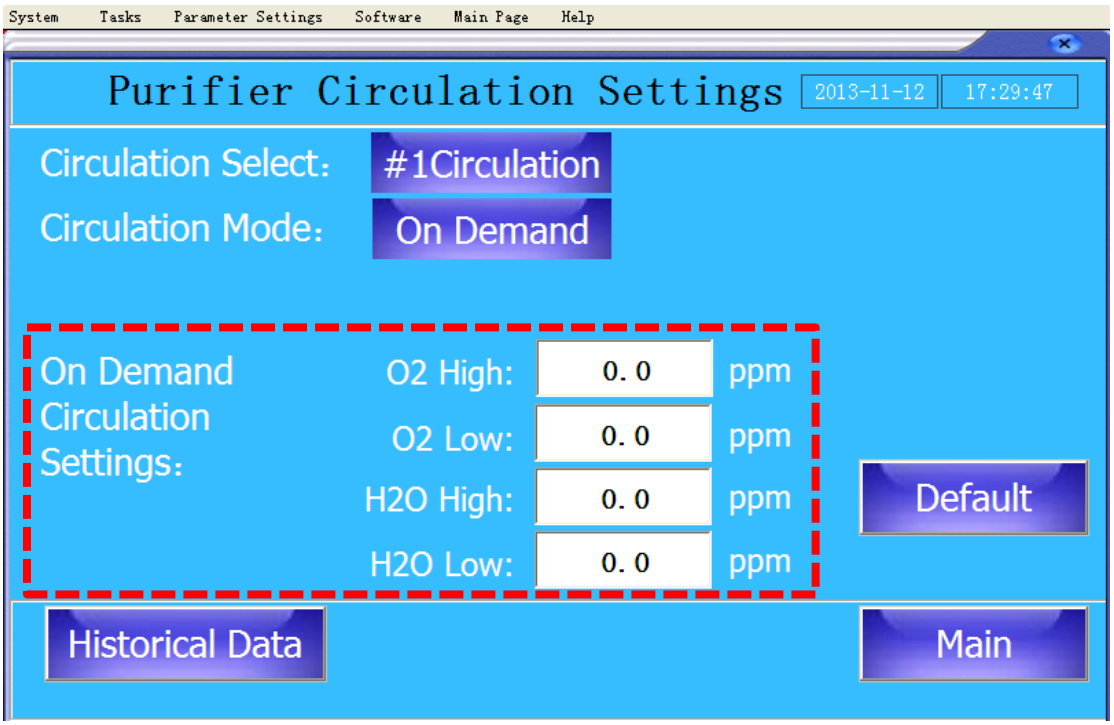
4.3.1 On Demand Circulation

On Demand circulation mode is the default mode for all Vigor gloveboxes. Under **On Demand** mode, circulation will start when the oxygen or moisture level exceeds the upper limit (which you have previously selected as suitable for your work, see settings window shown below). Circulation will continue until the impurity level reaches the lower limit that you've set, and then shut off.

Caution!
On Demand circulation mode requires that a minimum of one sensor unit be installed, either for oxygen or for moisture, to complete the control loop. Otherwise, the glovebox cannot activate the **On Demand** circulation mode and only manual control is possible.

On Demand Circulation - Default Settings

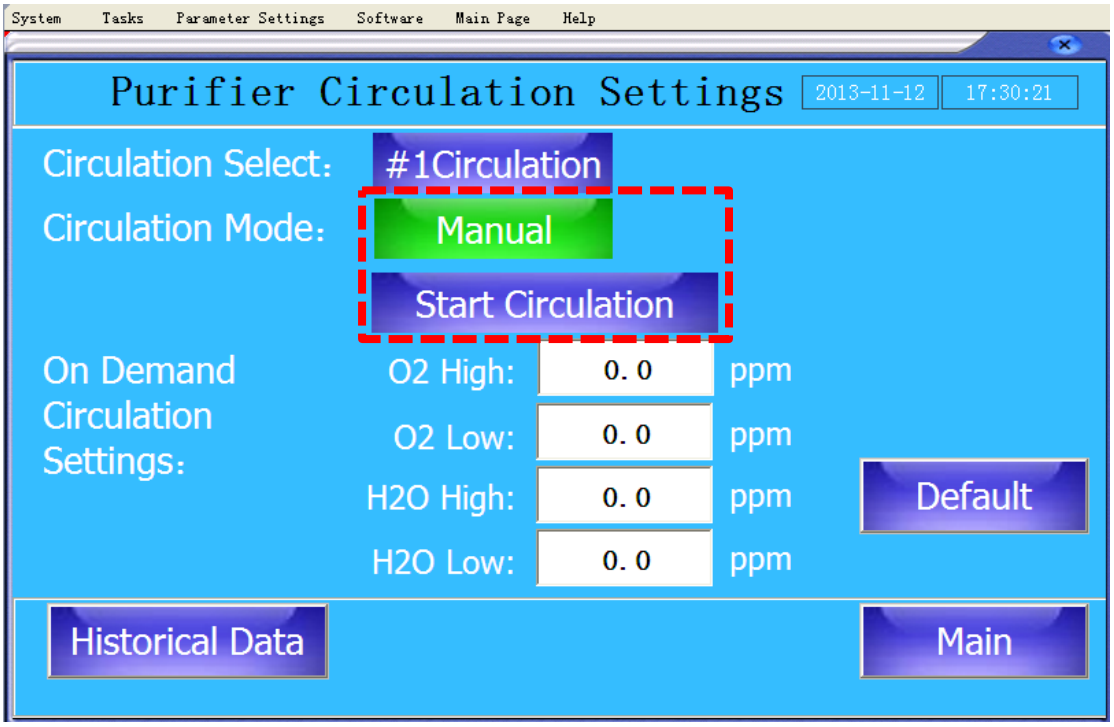
O_2 High	The circulation blower will start when oxygen exceeds this parameter setting.	Default: 1 ppm
O_2 Low	The circulation blower will stop when oxygen reaches this parameter setting.	Default: 0.2 ppm
H_2O High	The circulation blower will start when moisture exceeds this parameter setting.	Default: 1 ppm
H_2O Low	The circulation blower will stop when moisture reaches this parameter setting.	Default: 0.2 ppm



Caution!
Please do not set O_2 or H_2O high and low parameter too close to one another or on demand circulation will exhibit “hunting”. A minimum range of 2 mbar is suggested.

4.3.2 Manual Circulation

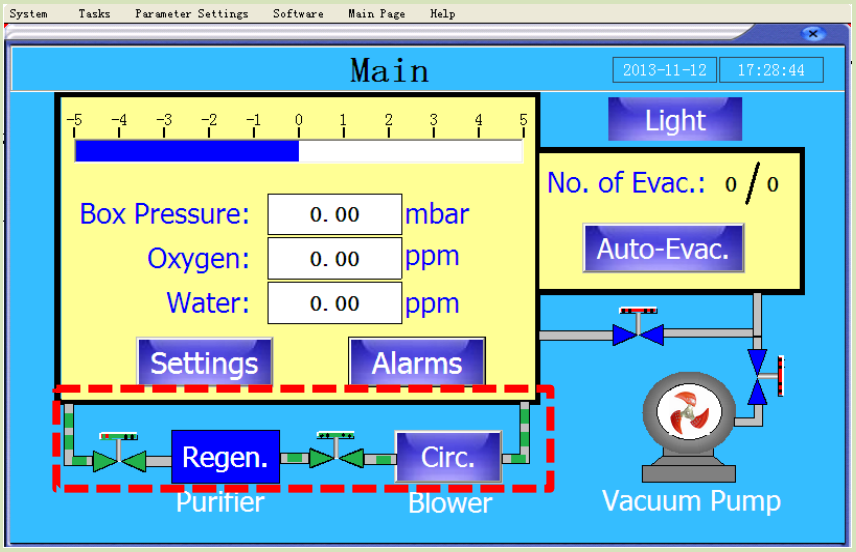
To control the circulation function manually, press the **On Demand** button once to transfer into the **Manual** control mode. A new button, **Start Circulation**, will appear. Press this once to start manual circulation. It will continue to run until you press the button, which now appears as a **Stop Circulation** button.



For a new installation, Vigor recommends setting the circulation mode to **Manual** and running the circulation continuously for at least one week, or setting it to **On Demand** mode with the upper limits for O_2 and H_2O set to 1 ppm and the lower limits for O_2 and H_2O to 0.2 ppm. During this startup period, moisture slowly (over days) desorbs from interior surfaces and the moisture level gradually drops. Oxygen will often drop much faster than moisture in the low ppm ranges.

Tips!

If circulation is running in either mode, the main menu will show green dashes moving through the pictorial representation of the purifier plumbing and both pneumatic valve icons (P1 and P2) will be green, signifying that they are open. If either valve, or both, fail to change from blue to green, circulation will not start.



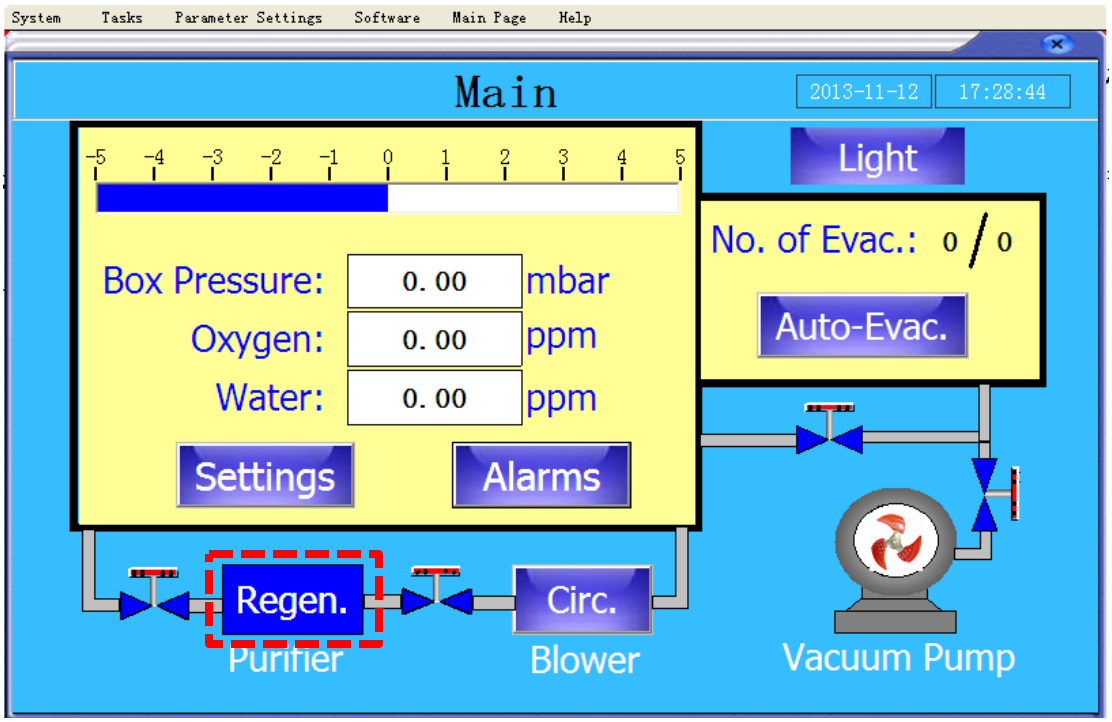
It typically takes **30 seconds** after pressing the circulation start button for the process to start and also display the indicators shown above.

4.4 Regeneration

4.4.1 Regular Regeneration

When the circulation process becomes unable to remove impurities from the glovebox sufficiently to reach the selected levels, or when this process greatly slows, regeneration is indicated. To enter the regeneration page, press the **Regen** button on the main menu.

Vigor highly suggests that following a new startup, after circulation has brought impurities to single digit ppm levels, a regeneration should be run since the purifier will be fairly saturated at that point.



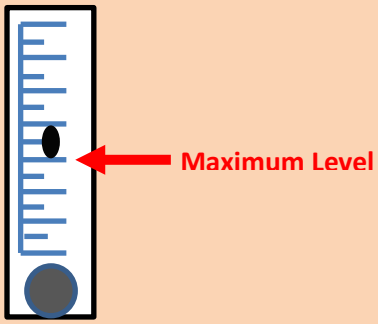
Warning!
Do not regenerate with pure hydrogen gas, or with forming gas with a hydrogen level exceeding 10% due to the possibility of ignition or explosion. Vigor suggests using an ultra-high purity grade mixture of 5% Hydrogen in 95% inert gas (to match your working gas), supplied at 15 psi, for regeneration.

Caution!

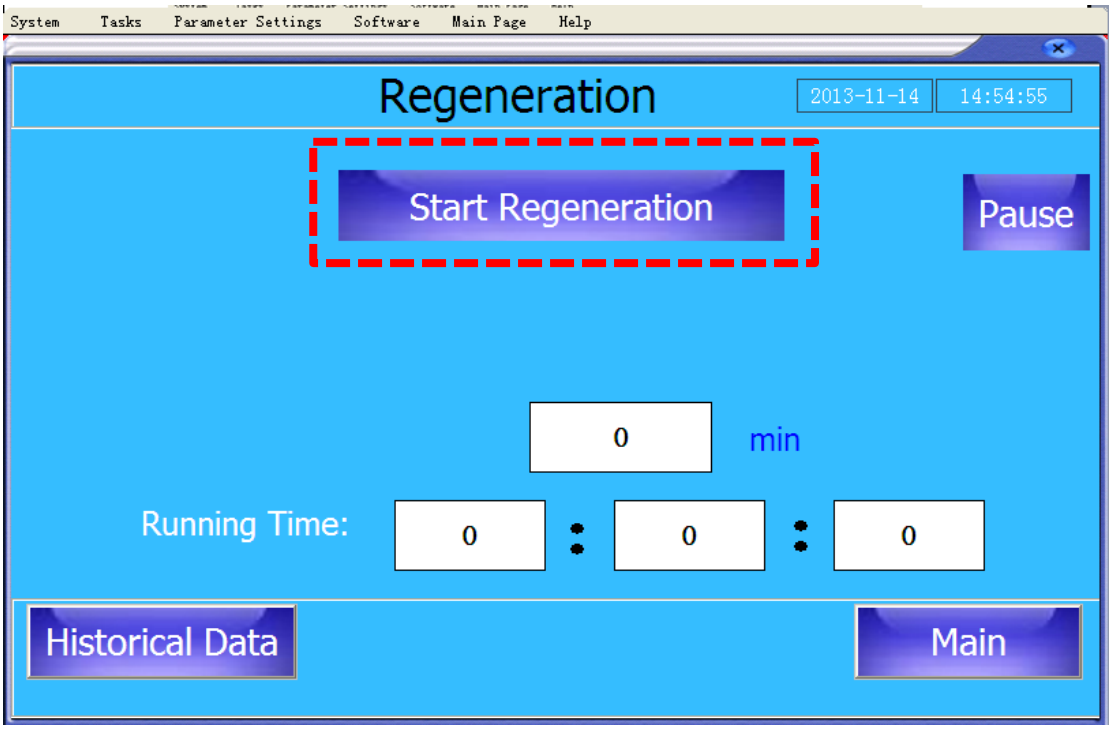
Gloveboxes manufactured during a small window starting at the end of 2016 and continuing into the start of 2017 don't require the user to adjust regeneration flow at the glovebox. These gloveboxes don't have a flowmeter located adjacent to the spot where gases are connected. Simply set the delivery pressure of the regeneration gas to 8 psi and you are ready to start regeneration.

If your glovebox was manufactured either earlier or later than this approximate time span, you'll find a flowmeter located in proximity to where the gas lines connect to the glovebox. Please use the following procedure:

To start regeneration, you must first adjust the rate at which regeneration gas will flow. Exactly when you adjust this depends upon the year the glovebox was manufactured, as screens have evolved over time. If you are immediately presented with an option to adjust the flow rate, do so as below. If you simply see a Start Regeneration button, press it, *a new screen comes up telling you to adjust the flow (within 3 minutes)* and then press Start Regeneration again to actually start the process. It ends 20 hours later on all newer units and 24 hours later on first generation gloveboxes. Please adjust the regeneration gas flow as follows; set the regeneration gas regulator pressure to about 15 - 20 psi (~ 1 – 1.3 bar) and then adjust the flow meter to **bring the bottom of the black ball even with the 5th to the 6th scribed line** from the bottom of the scale as shown below. Count each line, whether short or long.



If you find that regeneration used less than ½ tank of gas, you can turn the flow up a bit higher for the next regeneration.



Please press the **Start Regeneration** button to start the regeneration, which will proceed for the next 20 to 24 hours (manufacturing date dependent). To manually turn off the regeneration process, please click **Stop Regeneration** (Scram button on some models). Although, unless there is a compelling reason why this is necessary, we suggest that you don't interrupt the process.

Caution!

1. During regeneration, impurities levels will rise. This is primarily due to diffusion through the gloves. Starting the circulation after regeneration will quickly bring the levels of impurities back down again.
2. When you re-start the circulation, the gloves will suck into glovebox slightly due to the partial vacuum left in the purifier. This can be lessened by first adjusting the glovebox pressure to 4 – 5 mbar.

Here are some things to be aware of in the regeneration sequence:

i. Make sure the vacuum pump is on. It is essential to the process. Features are built into the software to prevent damage to the gloves and the system by shutting off the vacuum pump if the PLC thinks gas may be leaking out of the glovebox.

ii. After a power outage, the regeneration will continue from where it was interrupted as soon as the power is restored.

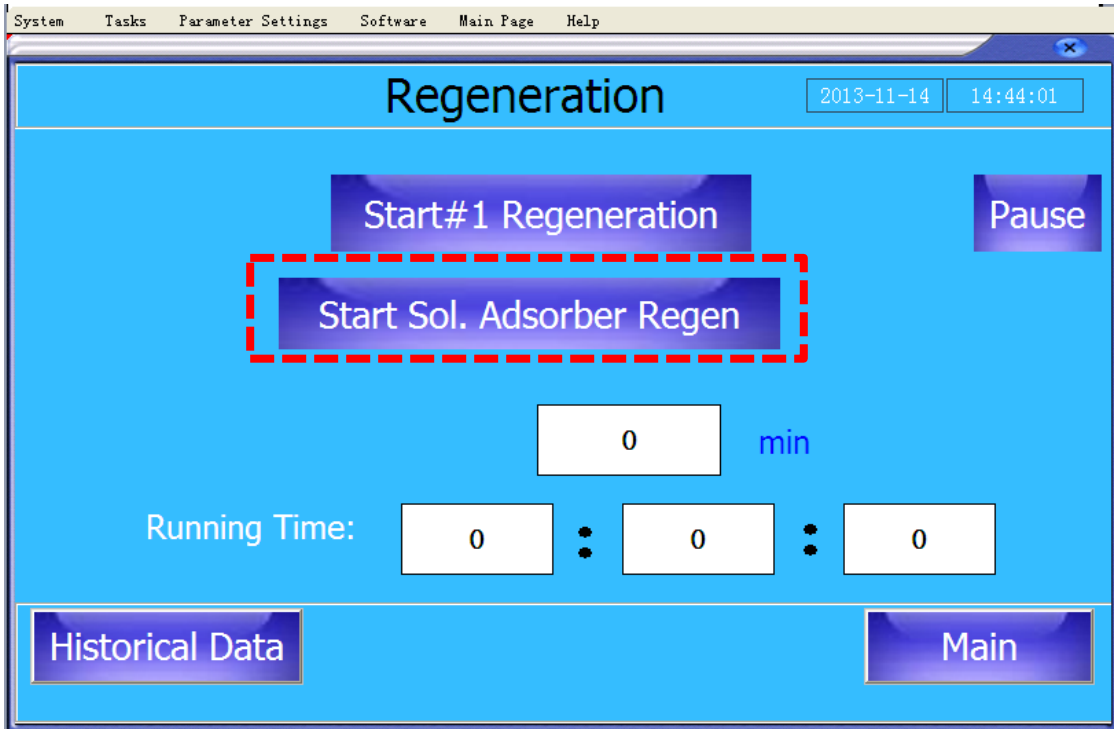
iii. Never restart circulation when the purifier column is still hot. If you must cancel a regeneration, please do not restart it immediately if it ran for more than 20 minutes. Otherwise, you may cause overheating of the purifier. You need to let the purifier cool down before restarting the regeneration.

iv. If you start a regeneration, after adjusting the regeneration gas flow, you must run the regeneration for at least 3 – 5 minutes. Adjusting the regeneration gas flow generates a positive pressure inside of the purifier because there is a check valve on the vent line. This pressurized gas may blow some adsorbent particles out of column unless it is removed by the evacuation which occurs when the regeneration cycle starts.

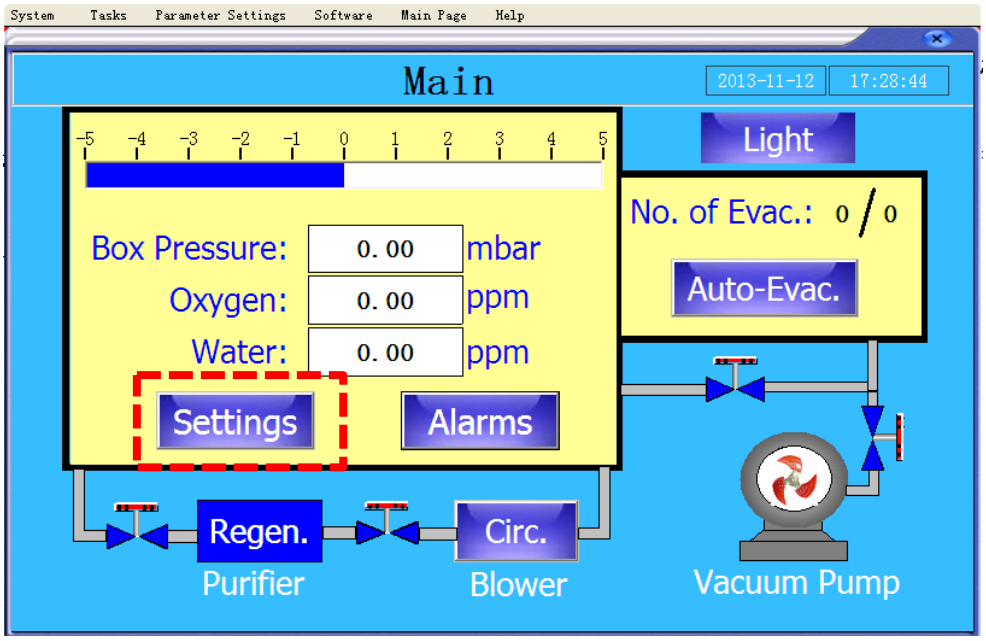
If your glovebox has a "Scram" button on the RHS of the "Start Regeneration" page, be advised that the effect of activating this button, either by design or inadvertently, is to pause a running regeneration. It will also prevent a regeneration cycle from starting if it hasn't already begun. For normal operation, this button will have a blue background. If the button has a green background, then it is active. Touching the button is all that is required to change states.

4.4.2 Solvent Regeneration (Optional Function)

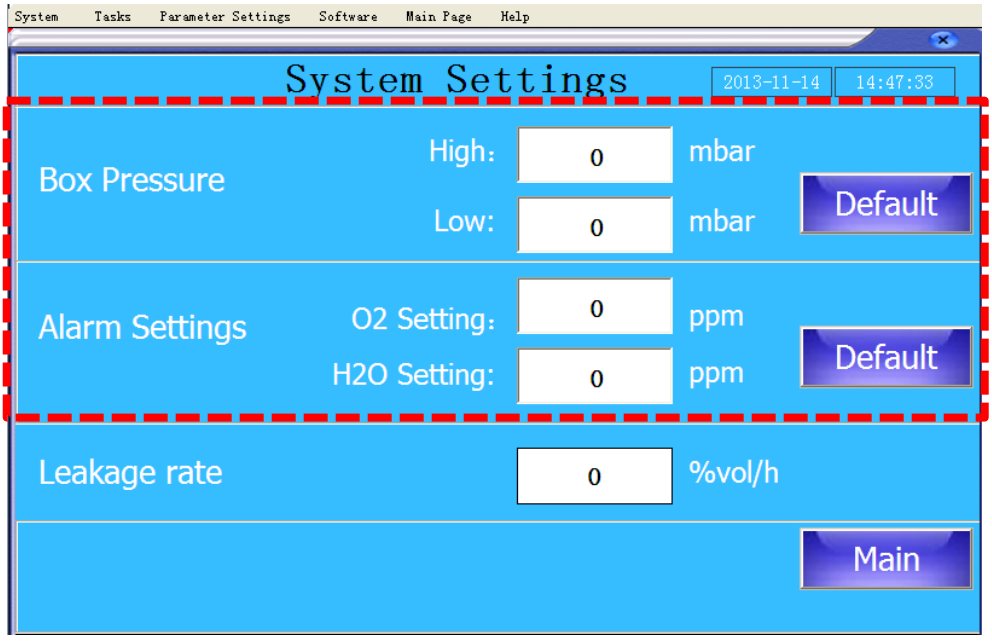
Solvent regeneration uses the same user interface as regular regeneration. In order to adjust solvent regeneration gas flow, users have to start a regular regeneration first, do the flow adjustment in the initial 3 minutes, stop the regeneration after 5 minutes, and then start the solvent adsorber regeneration. Then, click Start Sol. Adsorber Regen. Solvent adsorber regeneration will proceed over the next 20 hours. The regular regeneration process and solvent regeneration process are incapable of being executed at the same time. Saturation of the solvent absorbed bed can't be discerned by examining the displayed impurity levels. Vigor suggests users regenerate solvent absorber bed once a year, just before the regular regeneration process. If you are using solvents in the glovebox frequently, please do solvent adsorber regeneration once every three months.



4.5 System Settings

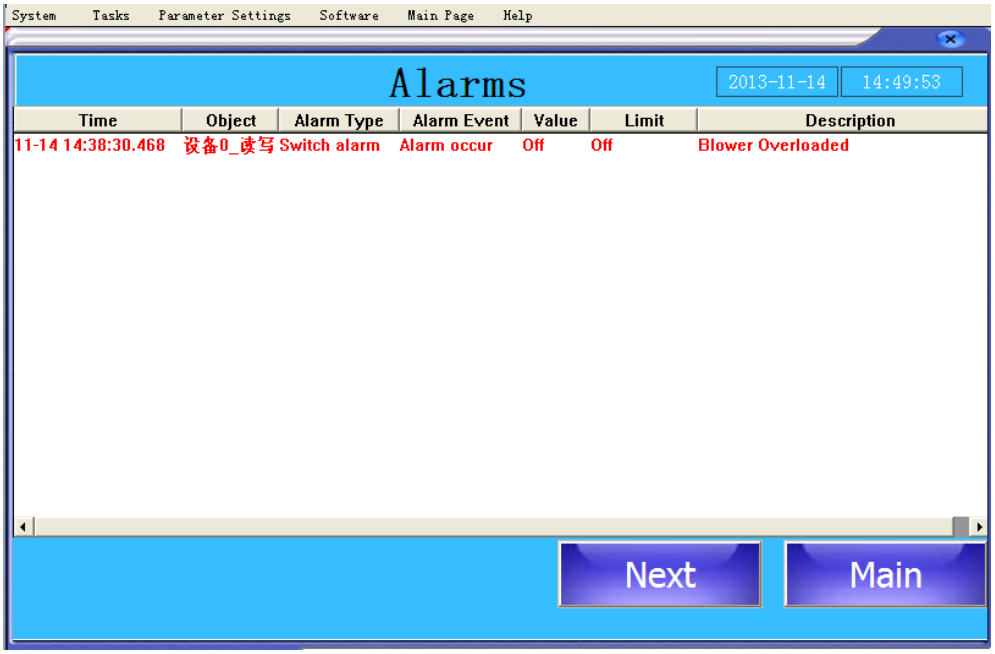


The glovebox working pressure range is regulated by the PLC unit and user-selected according to their preferences. To set the high and low limits for the glovebox pressure, press **Settings** on the main menu to enter the System Settings page. We recommend 0 – 3 mbar.



The oxygen and moisture levels which will trigger an alarm can also be set here. If the oxygen or moisture level reach the alarm setting, the Alarms icon will flash in red on the main menu page.

Pressing the **Alarms** button brings you to a page which lists and identifies errors. Section 5.6 of this manual gives descriptions of the error messages' meanings. If you need assistance with an error message displayed here, please contact Vigor customer service.



4.6 Antechambers

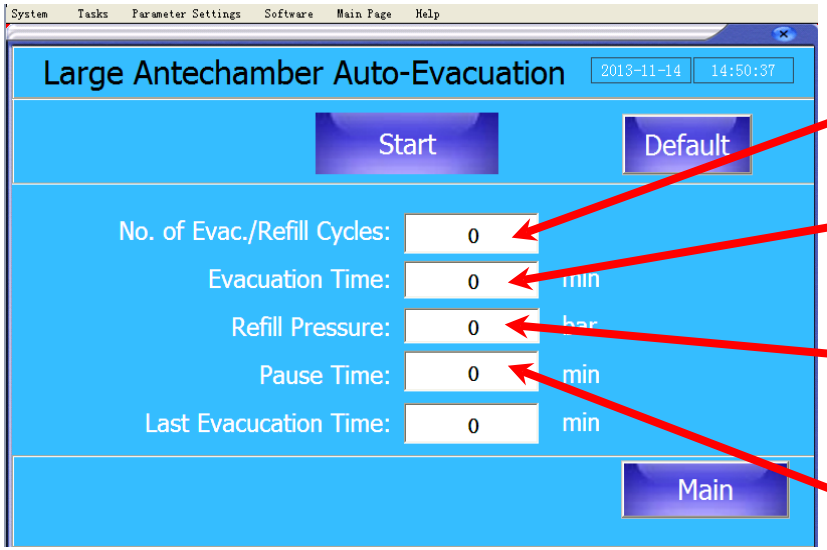
4.6.1 Transferring Materials via the Large Antechamber

Refill the large antechamber until the vacuum gauge reads 0 bars.

Turn the door handle counter-clockwise to open. Lift door upwards.

Place items on sliding tray. Empty vials and jars are not capped.

Close the door and turn the door handle clockwise to close. Don't use excessive force.



Number of Evac. / Refills cycles performed

The duration of the Evacuation cycle.

Level to which antechamber Is refilled between cycles

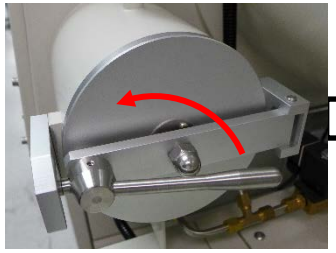
Pause time between Evacuation & refill

Evacuate to -1 bar for 30 min and then refill the antechamber (3 cycles) in order to completely remove impurities. Or, simply enter the default parameters and start the auto-evacuation cycle.

4.6.2 Transferring Materials via the Small Antechamber

Refill the small antechamber until the vacuum gauge reads 0 bars.

Turn the door handle counter-clockwise to release.

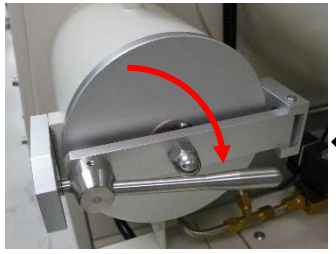


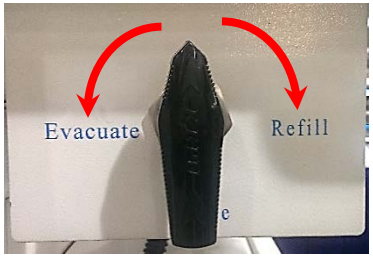
Open the antechamber door.

Place items on sliding tray (open the lids of empty containers).

Close the outside antechamber door.

Rotate the door handle clockwise to seal the antechamber door.



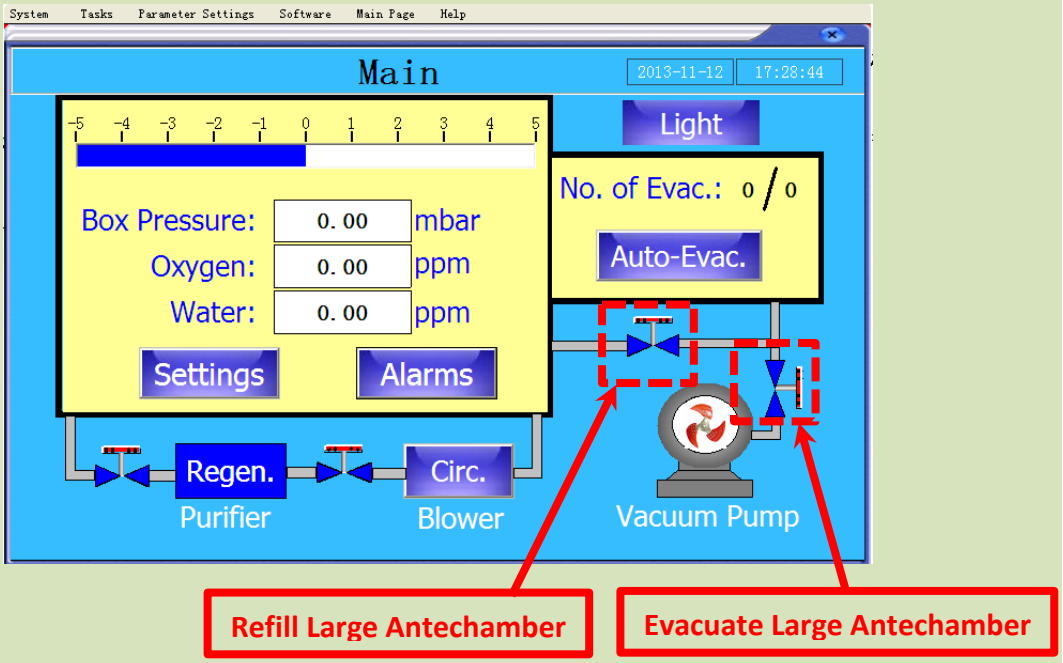


Evacuate the chamber to -0.8 bar and then refill 7 – 8 times (transit time <5 min) to completely remove impurities.

Tips!

I. Manually Refill and Evacuate the Large Antechamber

You can also manually evacuate and refill the large antechamber via the valve icons on the main page. Only one valve will open at a time.



II. Transfer Solvents and Chemicals Through the Antechambers

For commercial solvents, this means using sure/seal bottles. These are very heavy walled bottles with a tough rubber septum crimp sealed onto the bottle neck. Here is the procedure for use:

1. Put the bottle in a fume hood
2. Insert 2 syringe needles, one short one above the liquid, and one long one going to the bottom of the bottle.
3. Bubble dry N_2 or Ar (Ar box owners should use Ar) through the long needle for 30 min. to remove dissolved oxygen
4. Remove the needles and re-cap tightly.
5. Wrap the bottle cap and neck with plastic tape to seal it well.
6. Place into antechamber upright.
7. Don't use an extended pump down (like overnight), use the standard 3 x 30 min.

For reaction flasks, this means using flasks carefully inspected to exclude ones with weak spots like star cracks or chips.

1. Heavy walled flasks are best. Avoid cheap imports, some have very thin walls.
2. The stoppers must be greased for a good seal.
3. If the flask is under vacuum, things are relatively easy. As a precaution, use tape as described in 4 (below) to secure the stopper in place. Since the pressure inside the

flask is less than or, if a liquid phase is present, not hugely different from the vacuum drawn on it, the stopper is not subject to too high a force trying to pop it out. Obviously, volatile solvents like ether, methylene chloride or pentane present higher pressure differentials between the interior of the flask and the vacuum outside it. Secure these containers especially well.

4. If the flask is not evacuated, use vinyl tape under tension to secure stoppers in place. The holding pressure needs to be enough to resist the force of the stopper trying to push its way out due to the pressure differential of ~ 14.7 psi.
5. In either case, if liquid is present in the flask, it needs to stand upright. Use a cork (or better, rubber) ring or other means to support the flask.
6. Don't use an extended pump down (like overnight), use the standard 3 x 30 min.

For solid chemicals, the procedure is even simpler.
Make sure the material is sealed in a container under inert gas, or just loosen the bottle cap substantially so that the antechamber cycles can act on the contents of the container. Volatile solids are best transferred using the procedure described for reaction flasks.

Failure to observe suggested protocol in antechamber transfer operations can result in broken glassware, significant contamination of the pump oil and tedious antechamber clean-up operations.

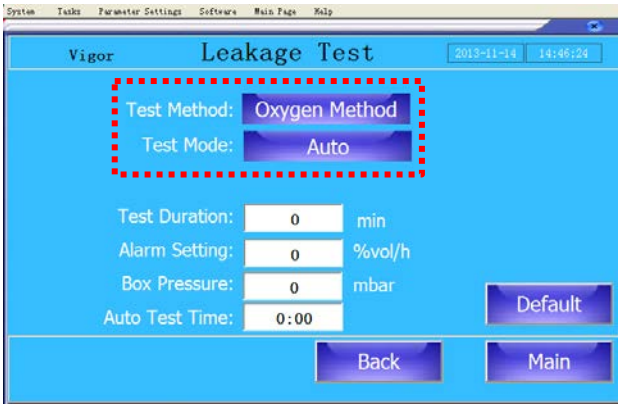
- Obviously, never take any (chemical containing) glassware through the antechamber that you wouldn't feel comfortable putting under a vacuum in normal service. This is especially true of glassware with flat sections, like Erlenmeyer flasks.

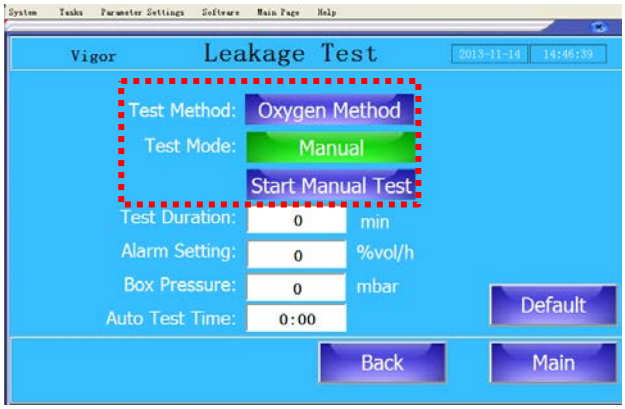
- It is useful to keep a container with a wide-mouth screw top inside the glovebox to dispose of solvent-wetted wipers, and other detritus, with the ability to release significant vapors into the glovebox atmosphere. Another container to receive glass waste like pipettes, melting point tubes and similar materials helps minimize the chances of glove punctures.

Caution!
After cycling through **Auto-Evac**, the antechamber will be left under vacuum. Before you try to open the antechamber doors, please refill the antechamber by clicking the **refill valve** button.

4.7 Leakage Tests

There are two types of leakage tests. The first is the **Oxygen Method** and the other is the **Pressure Method**. Enter the **Leakage Tests** page via the main page menu bar under **Tasks**.





Test can be started in an **Auto** or a **Manual** fashion. For the **Manual** mode, enter the desired test parameters and then press the **Start Manual Test** button to begin. For automated tests, enter the time the test should run as well as the other desired parameters. It will start automatically. When the test completes, the results are displayed on the Settings page.

Leakage rates (depends on glovebox model):

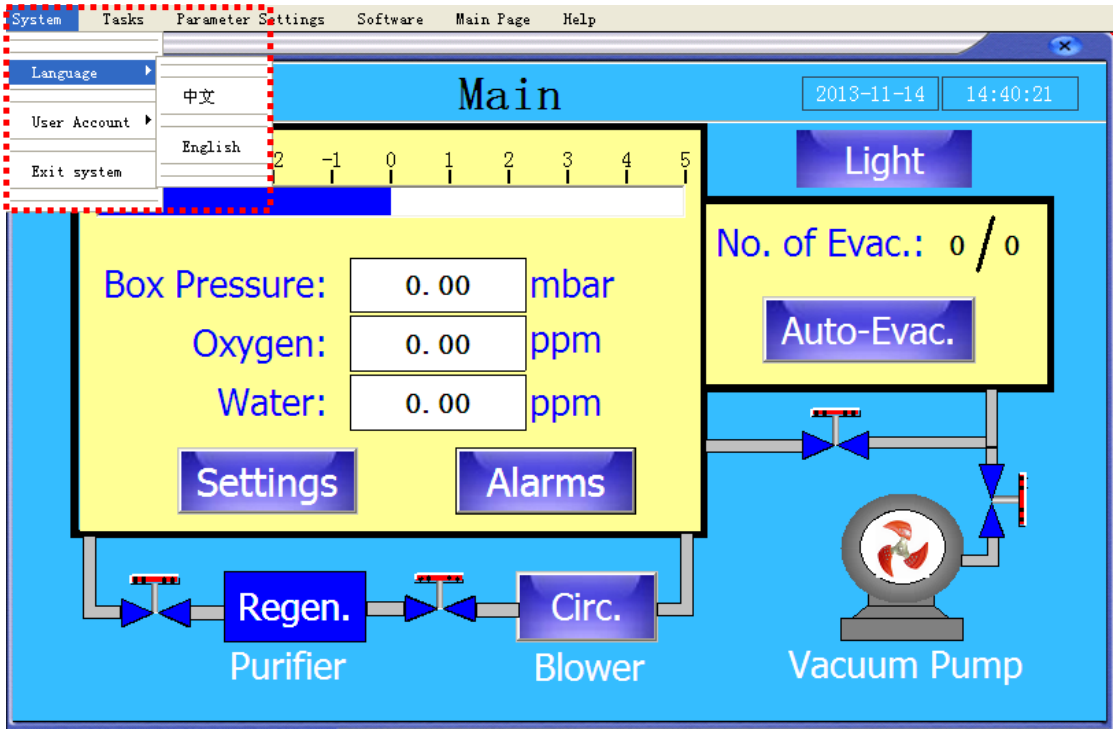
Pressure Method	0.5 %Vol/h
Oxygen Method	0.05 %Vol/h (30 minutes for SG1200/750TS glovebox)

The **Pressure Method** is only accurate when the glovebox temperature is stable. Its main usefulness is in searching for leaks. The **Oxygen Method** is recommended for accurate testing (the user must have an oxygen analyzer installed).

4.8 The Other Functions

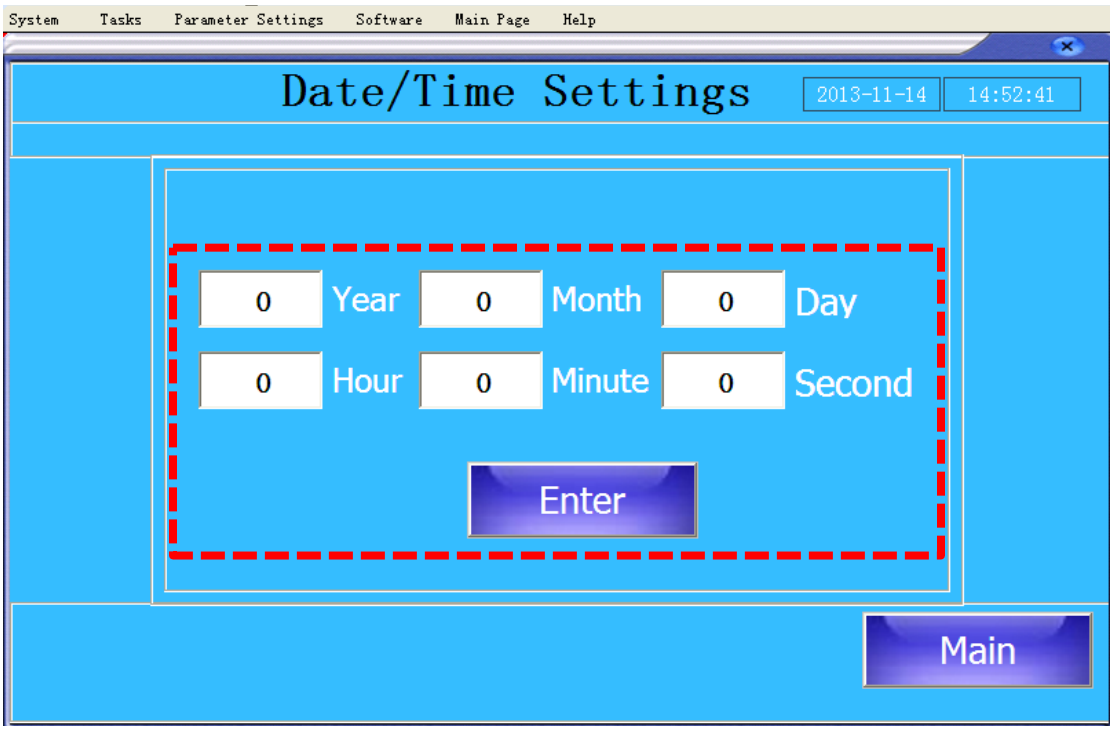
4.8.1 Language

There are two languages that can be selected for the glovebox system. One is English and the other is Chinese. To change this setting, please select **System/ Language** on the main page menu bar.



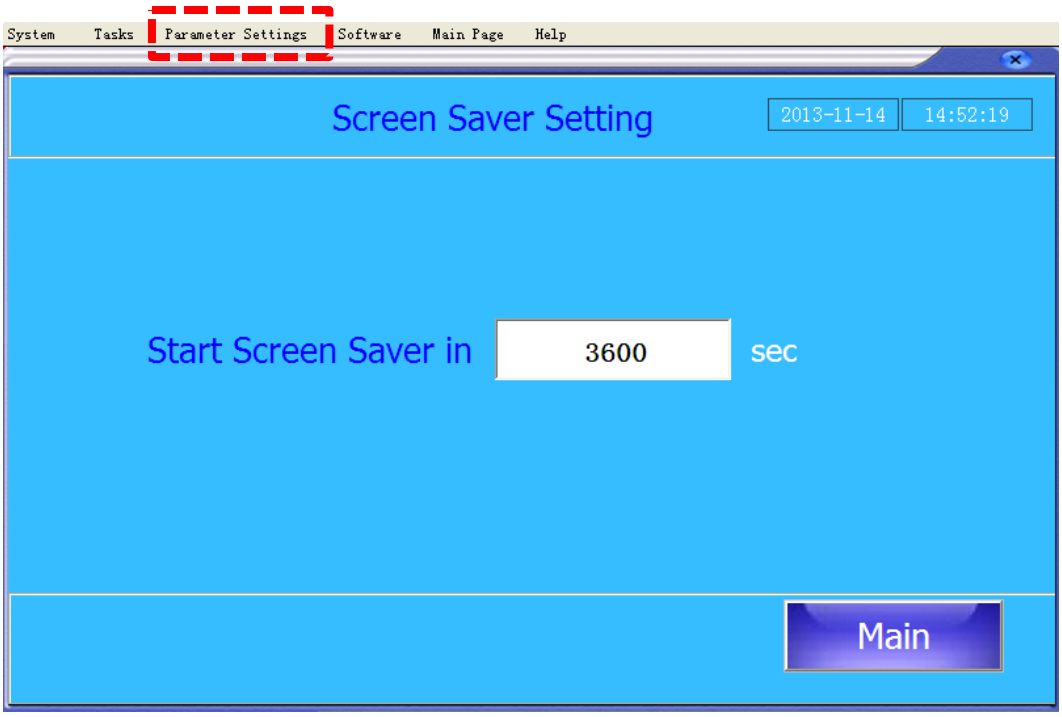
4.8.2 Time Setting

To set the displayed glovebox time, please select **Parameter Settings/ Date Time Settings** on the main page menu bar and then enter the correct time.



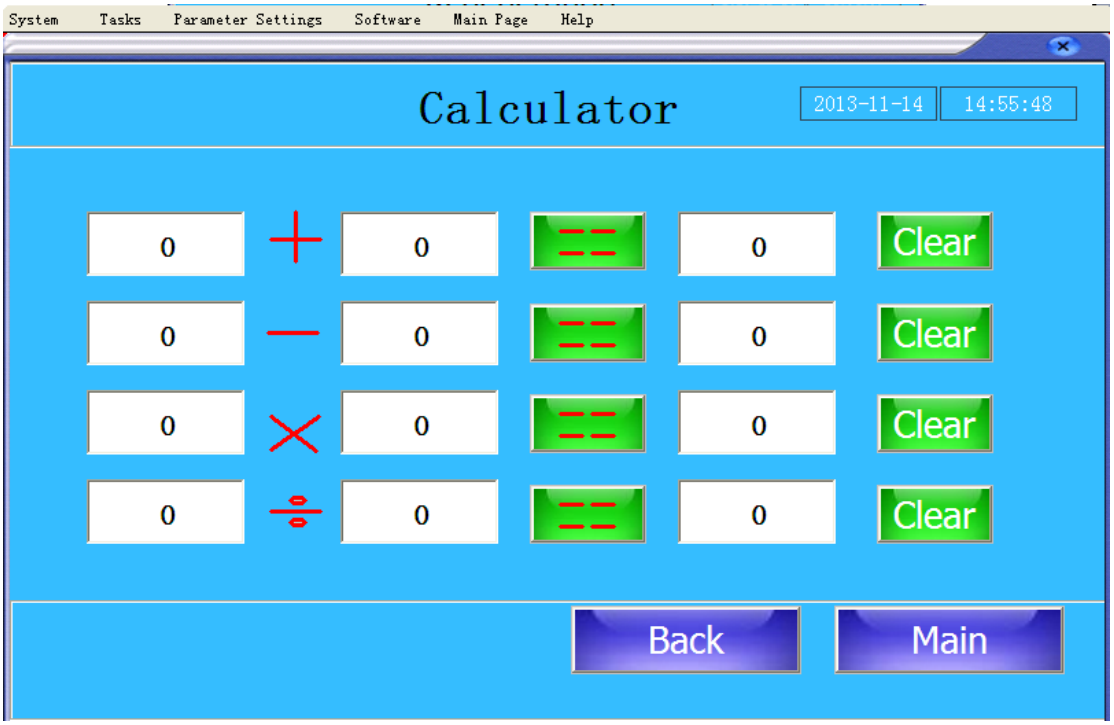
4.8.3 Screen Saver Setting

To set when to turn on the touch screen saver, please select **Parameter Settings/ Screen Saver Setting** on the main page menu bar and then enter the desired delay time.



4.8.4 Calculator

The glovebox system has built in a calculator which is accessed on the main page menu bar by selecting **Software/ Calculator**. The simple calculator function is shown below.

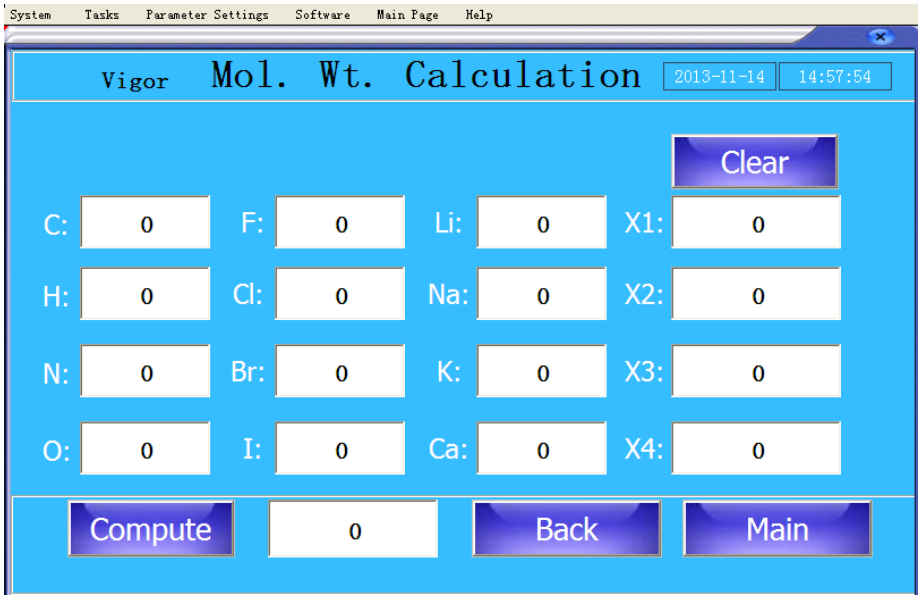


4.8.5 Reactant Requirements

The Vigor glovebox system has a built in chemistry reaction software package which will help users calculate amounts, yields, etc. To use this software, please go **Software / Reactants** Requirements via the menu bar on the main page.

The calculation can be based on **Weight (Wt)** or **mMoles (mMol)**. In the **Wt** mode, the software will perform calculations of the amounts of other reactants and products to be expected after the user simply enters the **weight** of reactant A into this box and the stoichiometric coefficients. In the **mMol** mode, the user simply enters the number of millimoles of reactant A into the **mMol** box as well as the stoichiometric coefficients and the amounts in millimoles of the other reactants and products are calculated automatically.

First, the user enters the stoichiometric coefficients for both reactants and products in the first row and the amount of one reactant. For example, if reaction were $Mg(OH)_2 + 2HCl \rightarrow MgCl_2 + 2H_2O$, the user would enter 1, 2, 1 and 2 in the "Stoi." boxes and the amount of one starting material. Then, when molecular weights of the reactants and products are to be entered on the second row, the user is brought to the Molecular Weight Calculation page. For example, for $Al(CH_3)_3$ you would enter a 3 in the C box, a 9 in the H box and then manually enter 26.98 at X1 for the Al. Press compute and the value will be automatically calculated and entered in the appropriate box on the previous page.



4.9 Cleaning and Decontamination

Use a dry cloth to gently wipe the surface of the device to remove dust. To remove dirt or grime, use a cloth moistened with warm, soapy water. The glass front may be cleaned with a standard household glass cleaning solution.

4.10 Disconnection from Supply

Disconnection of the glovebox from its supply sources is straightforward. The glovebox is turned off at the main power switch (0 = off, 1 = on) and the glovebox is simply unplugged from the wall outlet. All supporting devices (heated antechamber and/or refrigerator, if so equipped) are also disconnected by switching off and unplugging. The glovebox has an internal power strip which also should be unplugged. The gas supply lines are disconnected from their supplies by disconnection of the compression fittings at the supply regulators.

Section 5 Maintenance and Troubleshooting

5.1 General Maintenance

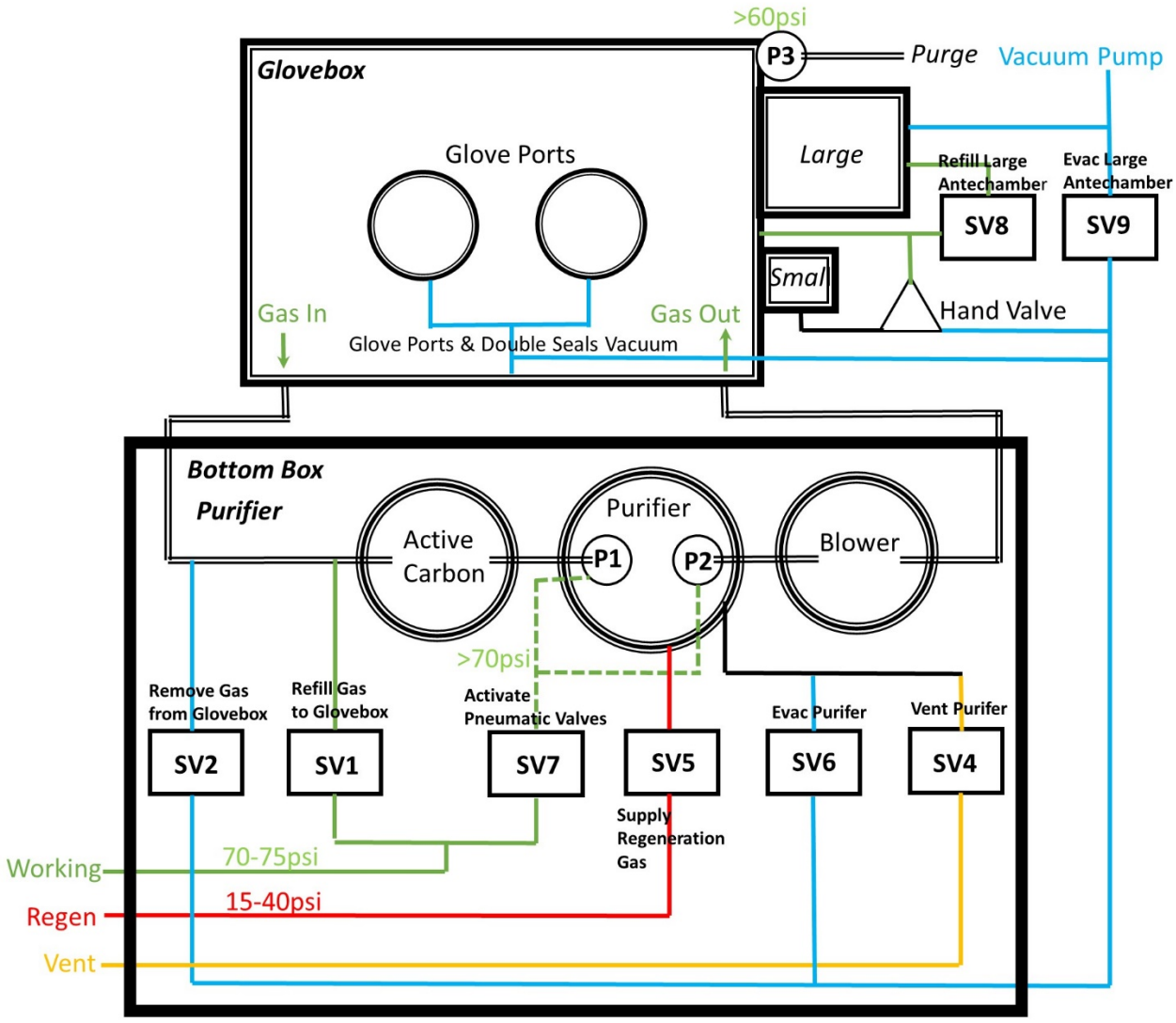
Vigor gloveboxes are designed to require minimum attention, but some simple checks and maintenance are still required. See the following table for details. Disassembly and/or maintenance of this equipment, beyond the scope of the operations described in this manual, can only be done by users who have received complete operational and maintenance training from the manufacturer. Disassembly and/or maintenance performed by untrained users may result in damage to this equipment, and will void the manufacturer's full warranty.

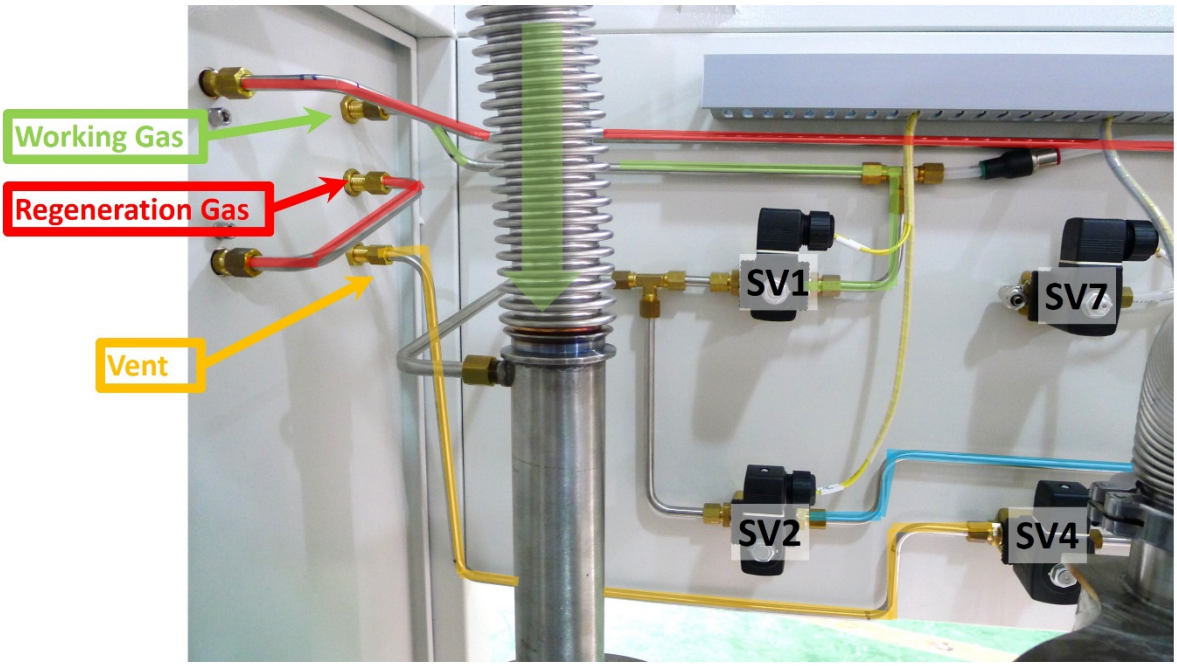
Warning: non-professional maintenance of this equipment carries the risk of electric shock.

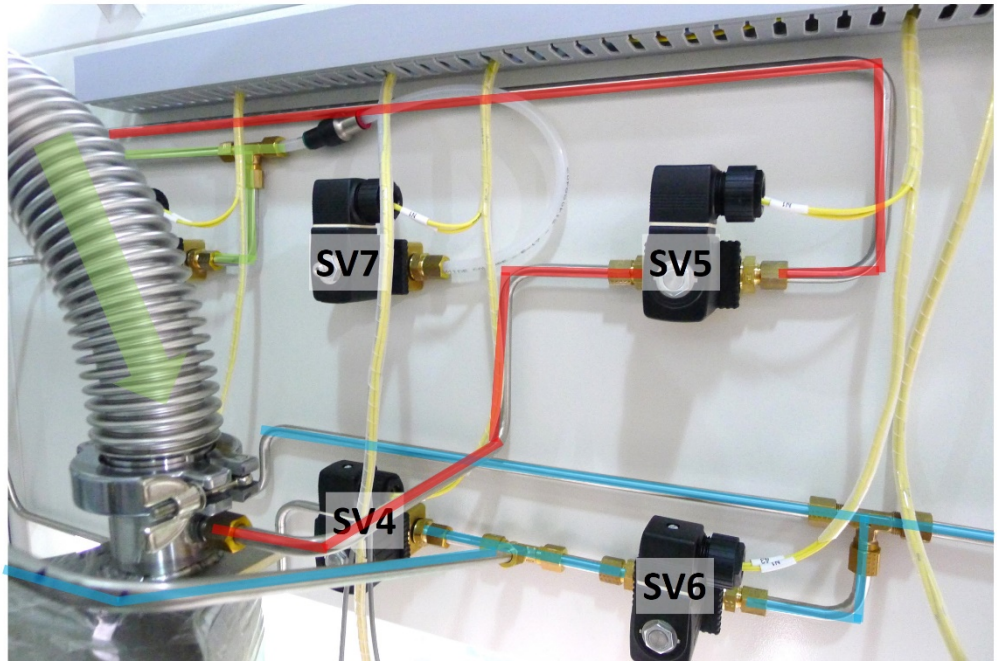
Item	***Frequency***	***Actions***
Working Gas	Daily	Check working gas cylinder to make there is enough gas to perform the task. Stock one extra cylinder to avoid an interruption in usage of the glovebox
Gloves	Daily	Check to make sure the gloves are in good condition and that there are no holes in them. To check, set the box pressure to > 5 mbar and wait for a couple of minutes for the pressure to stabilize. If the pressure drops, and the rate is greater than 0.02 mbar/min, the glovebox may be leaking and the gloves are the most likely cause.
Solvent Adsorber	As needed	When the solvent adsorber is saturated, organic vapor will start to accumulate inside the box. If strong odors come from the antechamber after exiting the glovebox, it is time to change the adsorbents in the solvent adsorber.
Vacuum Pump Oil	Monthly	Visually check the pump oil color and level. New pump oil is an almost clear liquid. If it turns brown, the oil should be changed. If the oil level is approaching the minimum level, add additional oil.
	Annually	Change oil after each regeneration cycle, or once a year, whichever comes first.

Filter	Quarterly	Check the filters at the inlet and outlet of the purification system. If they have visible dust accumulations, please change them to avoid damaging the blower.
Antechamber O-rings	Annually	Check antechamber O-rings annually. If there are any signs of deterioration, replace it (or them).
Glovebox Regeneration	Annually, Sooner if needed	If sufficiently low oxygen and moisture levels are unable to be achieved after running circulation for a long time, please regenerate the glovebox. If a freshly regenerated purifier train fails to correct the situation, please contact Vigor Customer Service about purifier catalyst replacement.

5.1.1 Glovebox Plumbing Diagram

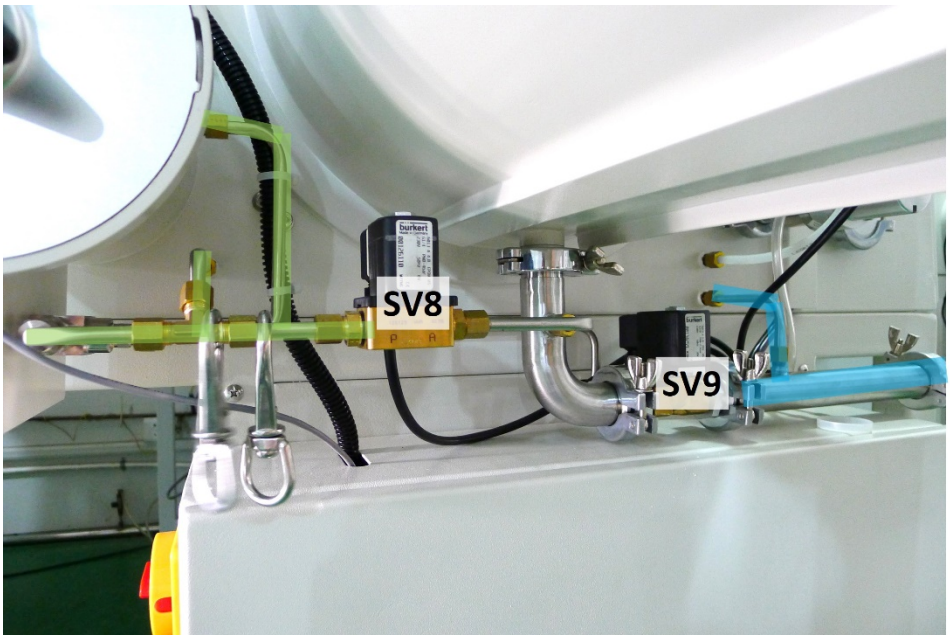


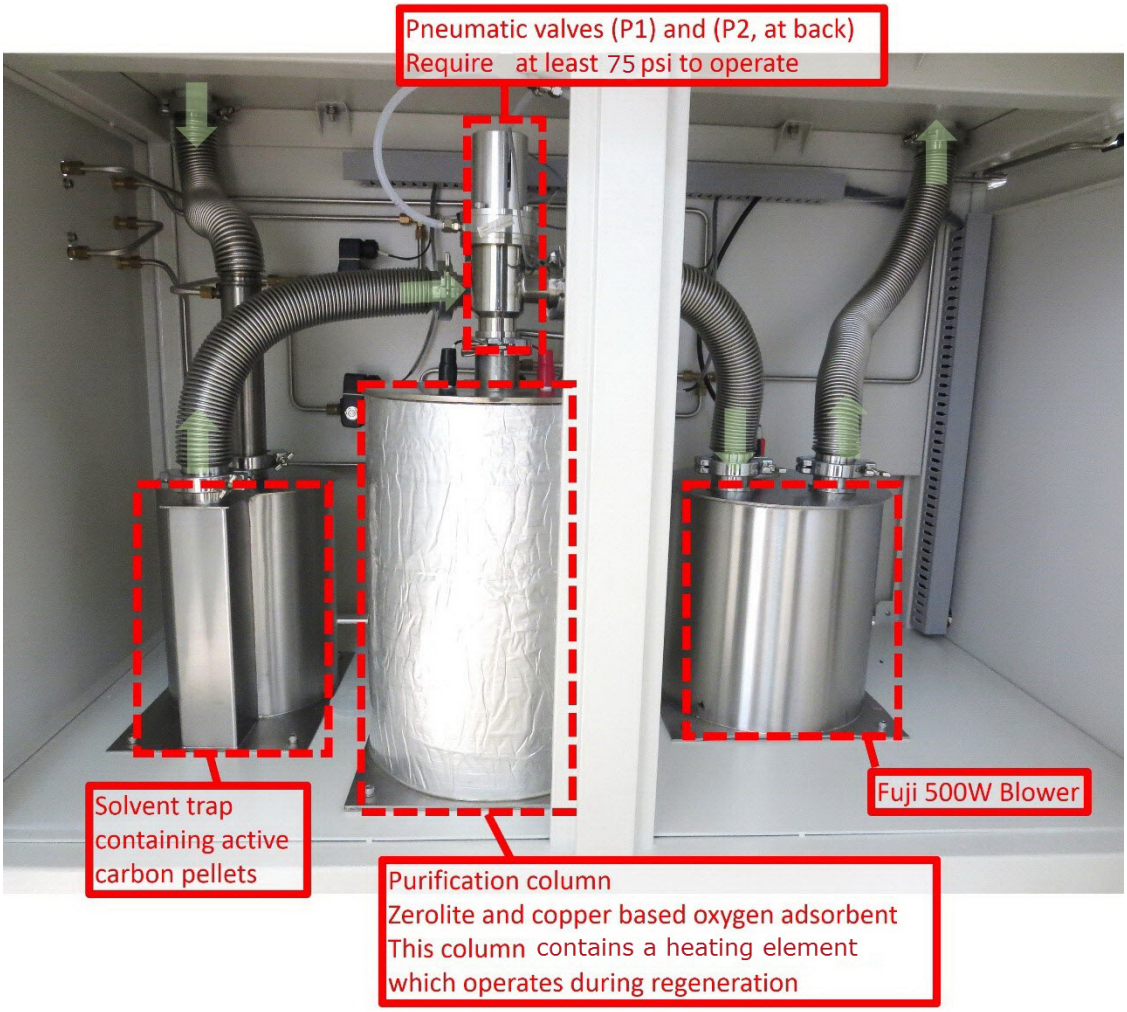




Vacuum lines Regeneration Gas

Working Gas Vent

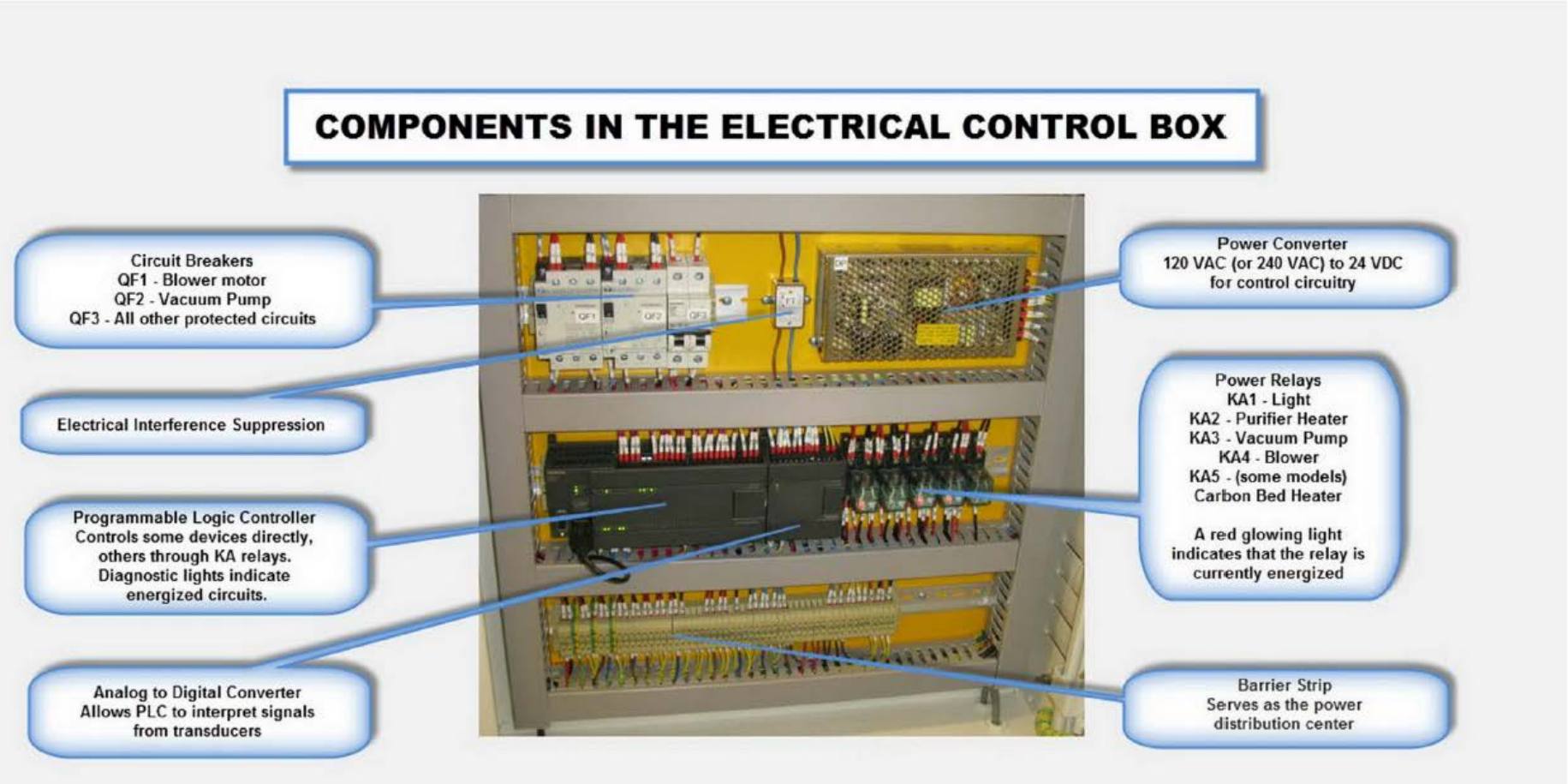


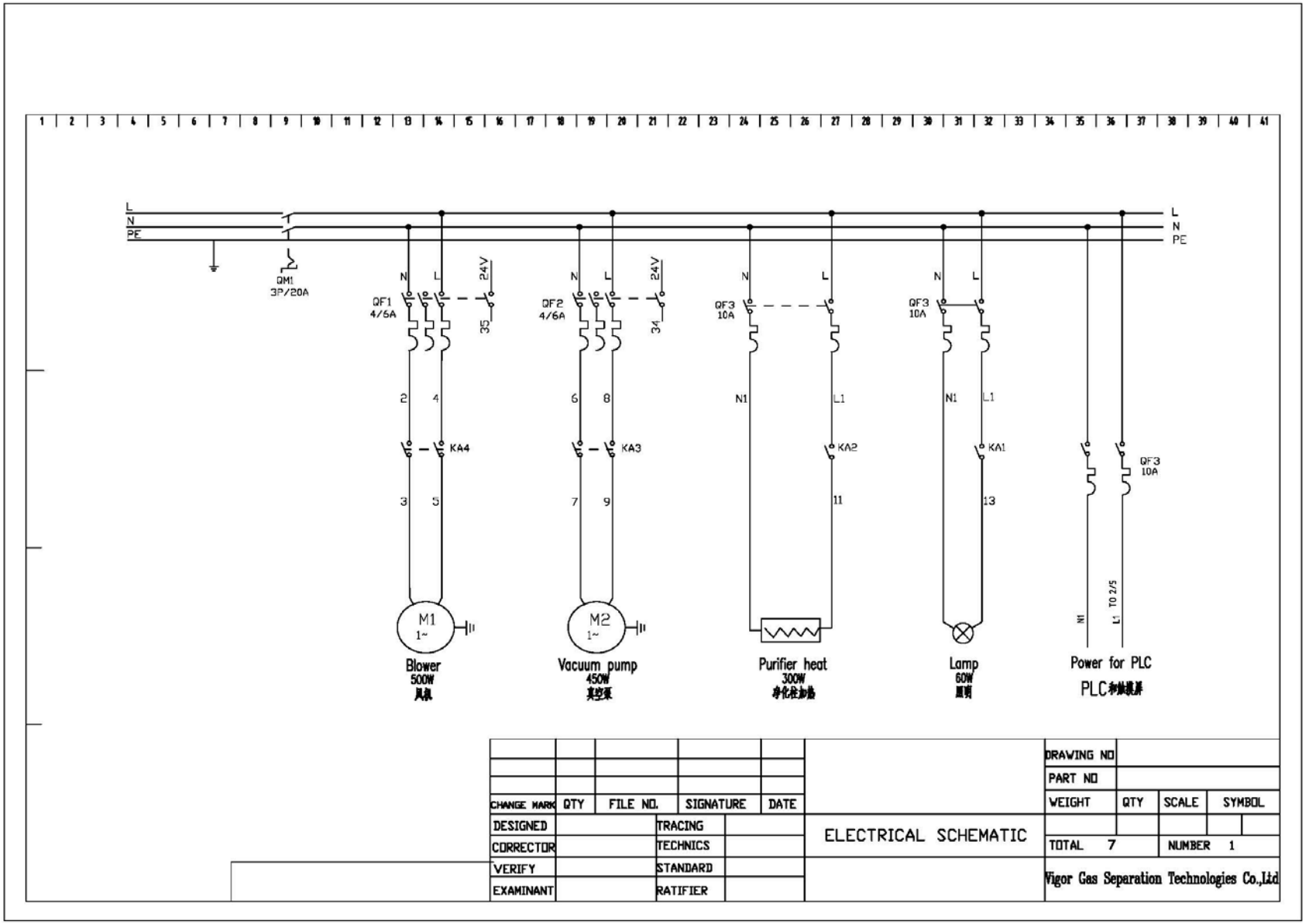


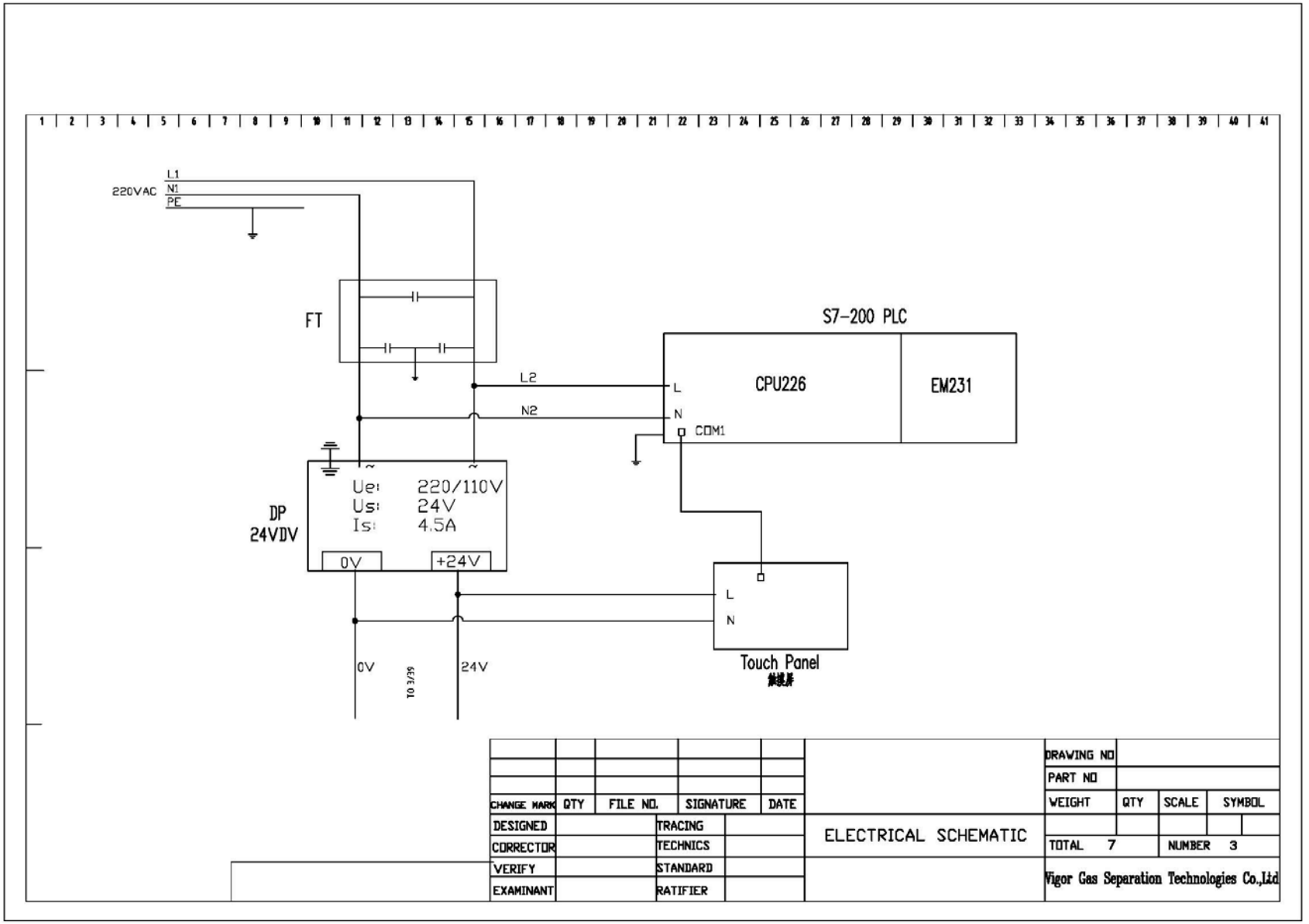
5.1.2 Electrical Box

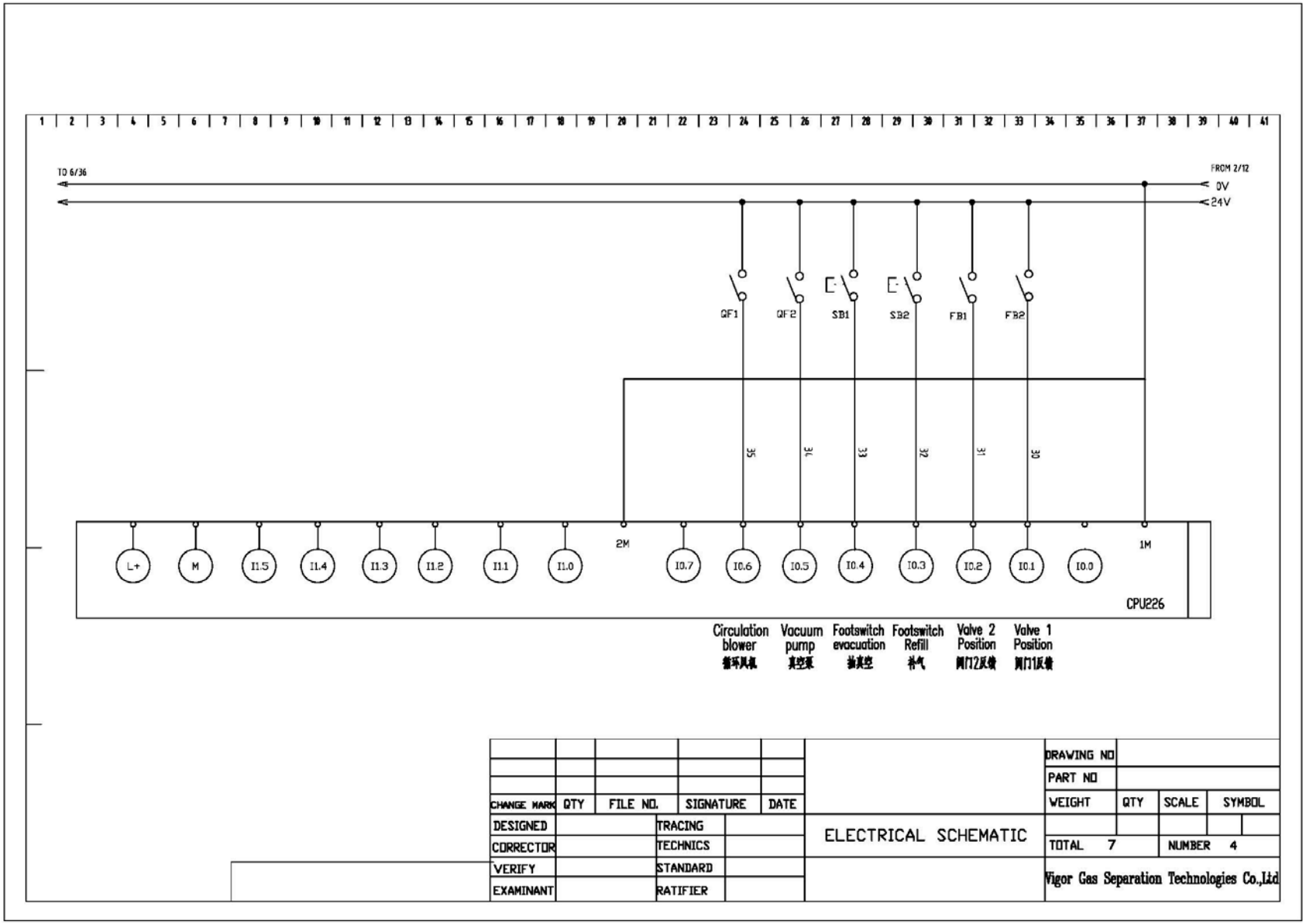
Glovebox PLC indicators, Relay, and Valves Controls

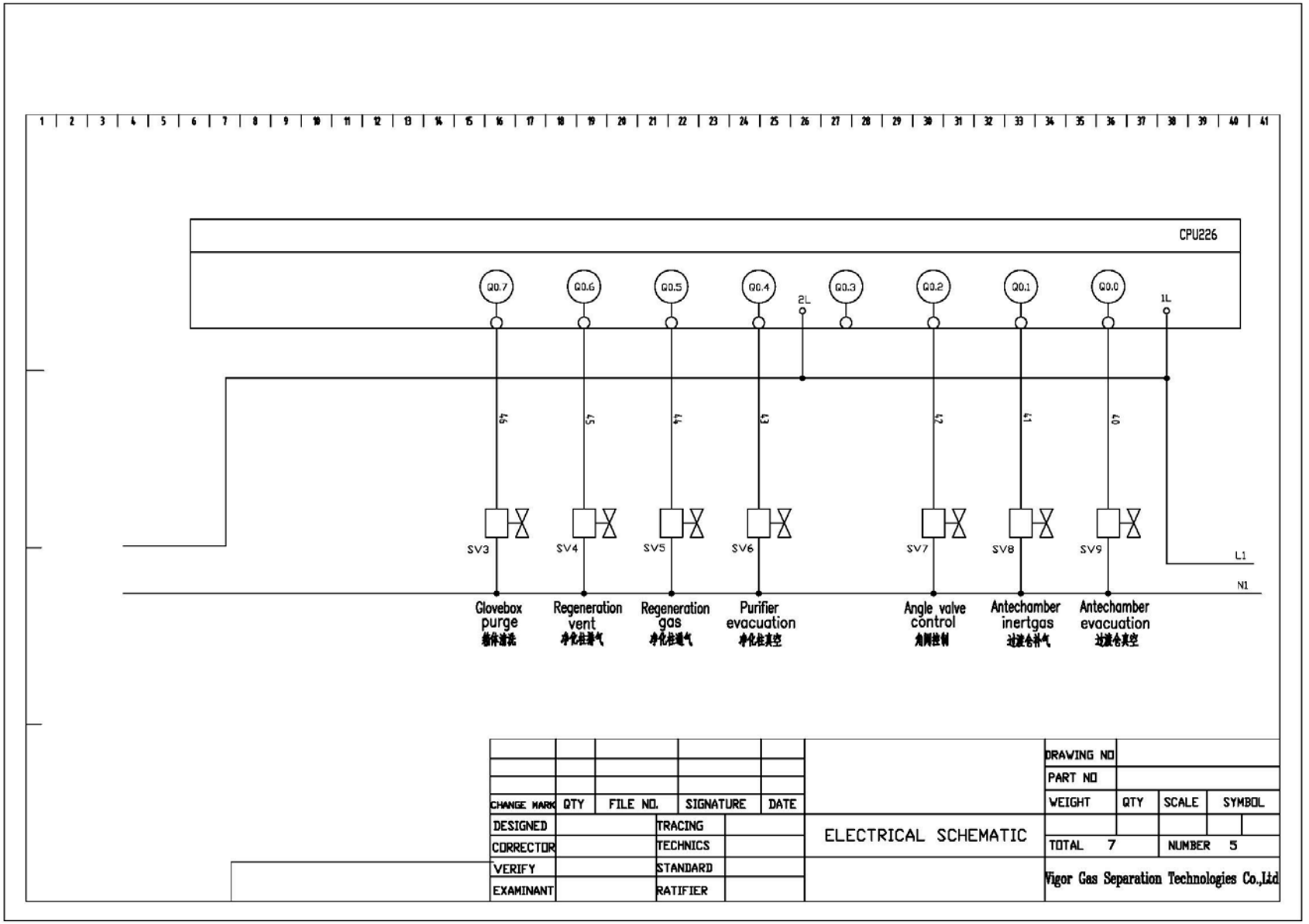
PLC Lights	*Wire #*	*Destination*	*Function Activated*
Q0.0	40	SV9	Antechamber evacuation
Q0.1	41	SV8	Antechamber refill
Q0.2	42	SV7	Open pneumatic valves P1 and P2
Q0.3	--	--	--
Q0.4	43	SV6	Purifier evacuation
Q0.5	44	SV5	Add regeneration gas
Q0.6	45	SV4	Vent regeneration gas
Q0.7	46	SV7.1	Open purge valve P3
Q0.8	--	--	--
Q0.9	--	--	--
Q1.0	50	SV2	Evacuate glovebox
Q1.1	51	SV1	Refill glovebox
Q1.2	52	KA4	Blower
Q1.3	53	KA3	Vacuum pump
Q1.4	54	KA2, QF3	Purifier heating
Q1.5	55	KA1, QF3	Lights
I0.1	30	FB2	P1 feedback signal
I0.2	31	FB1	P2 feedback signal
I0.3	32	SB1	Footswitch refill
I0.4	33	SB2	Footswitch evacuation
I0.5	34	QF2, 24V	Vacuum pump
I0.6	35	QF1, 24V	Blower

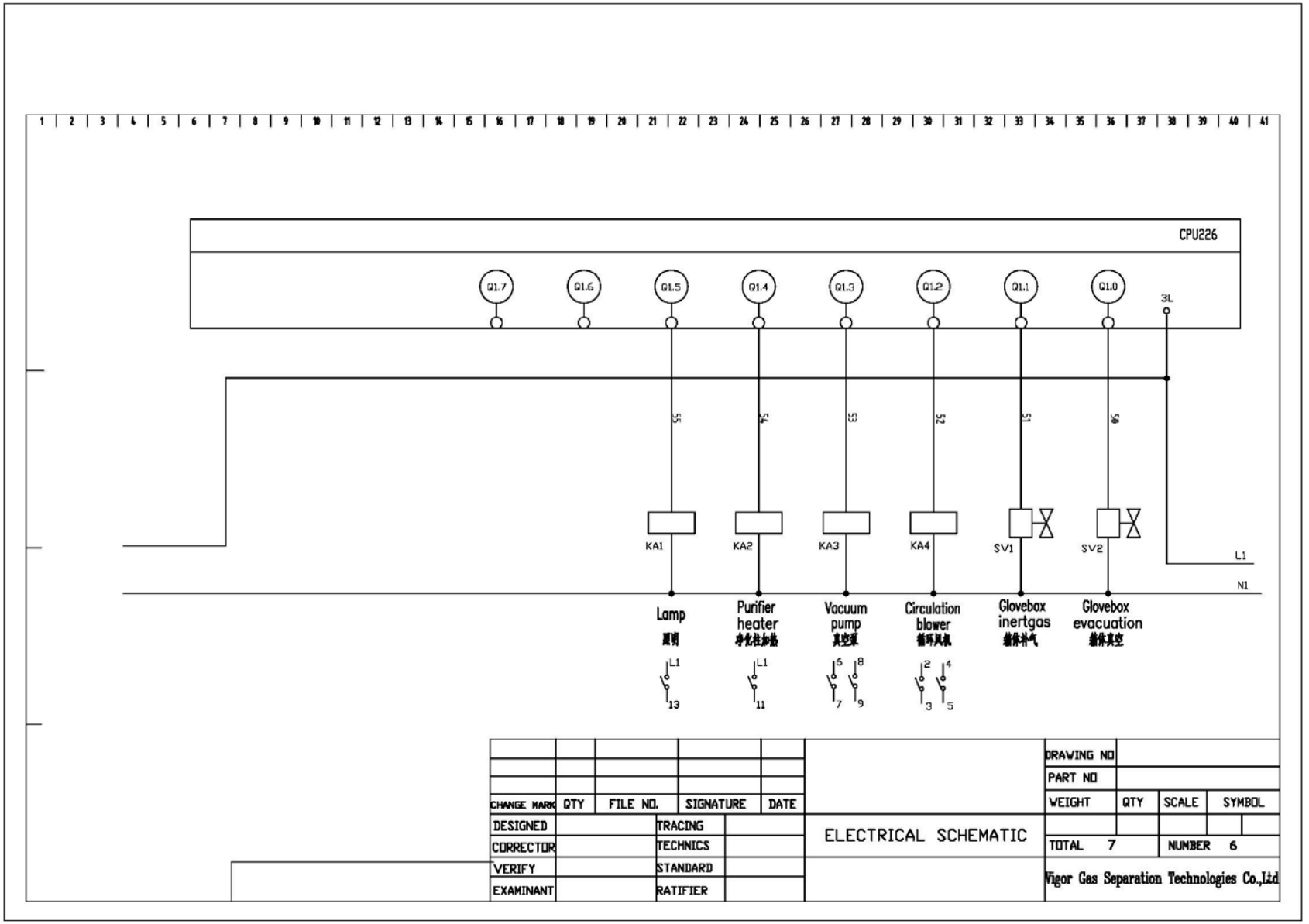


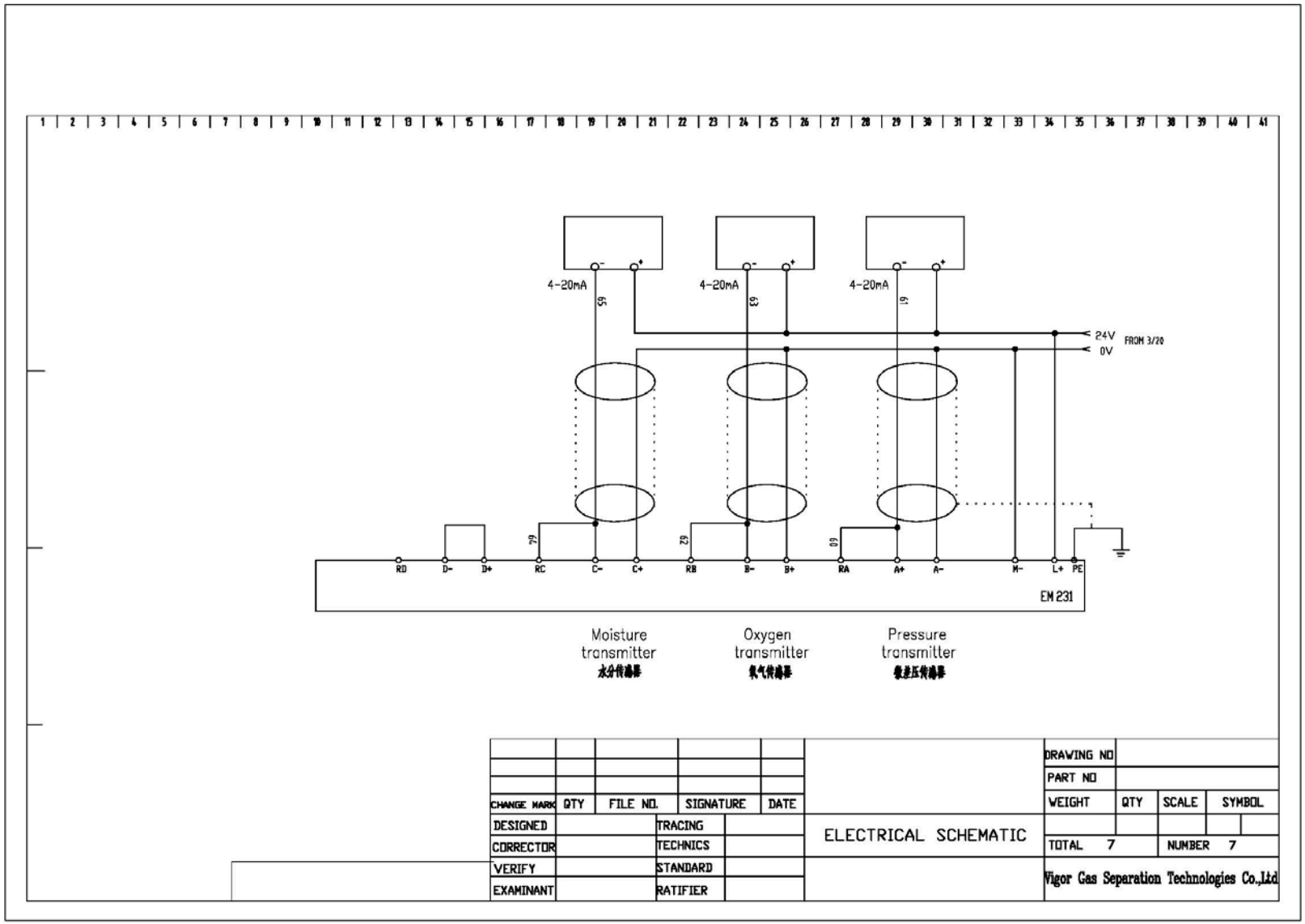




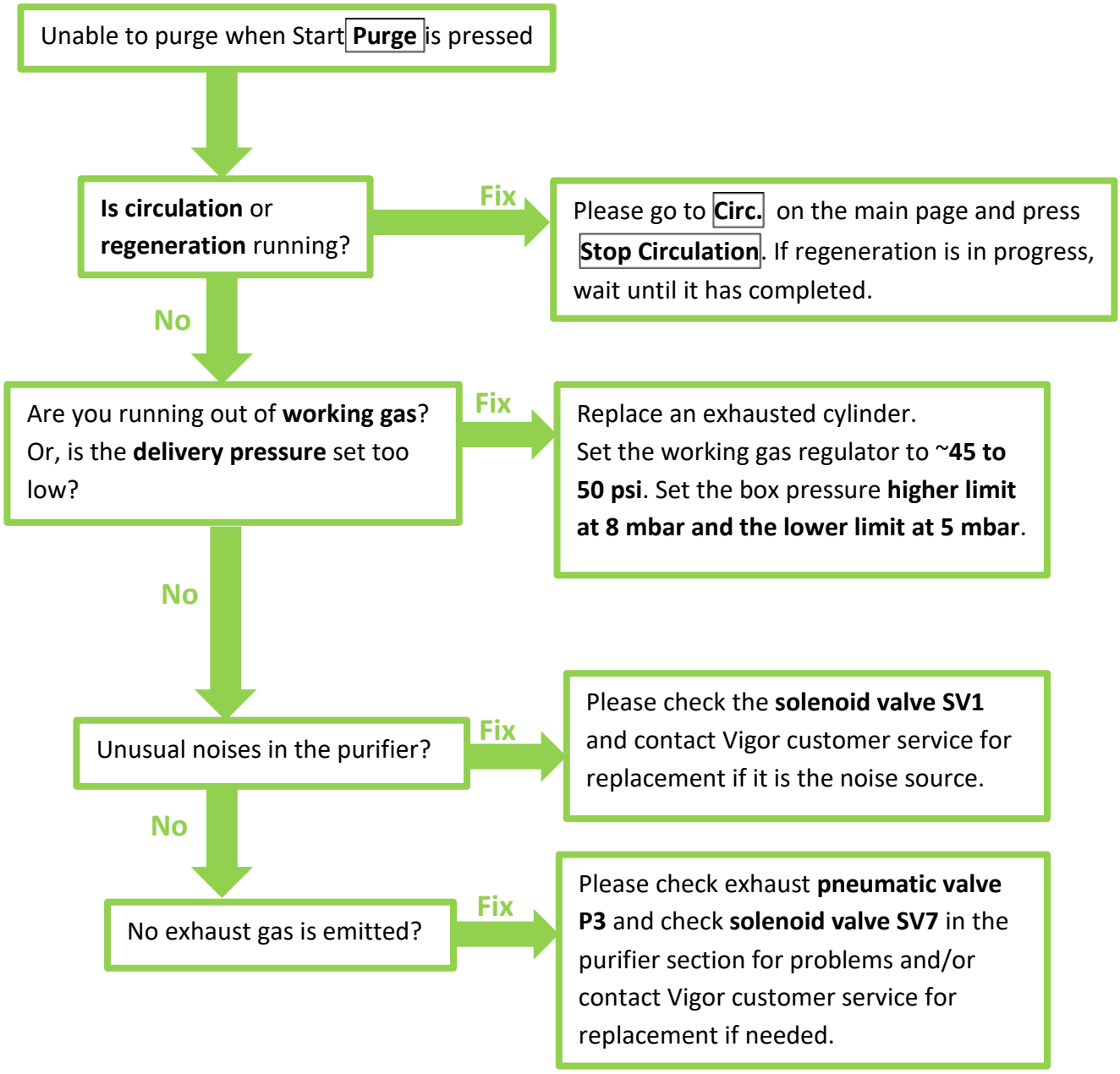




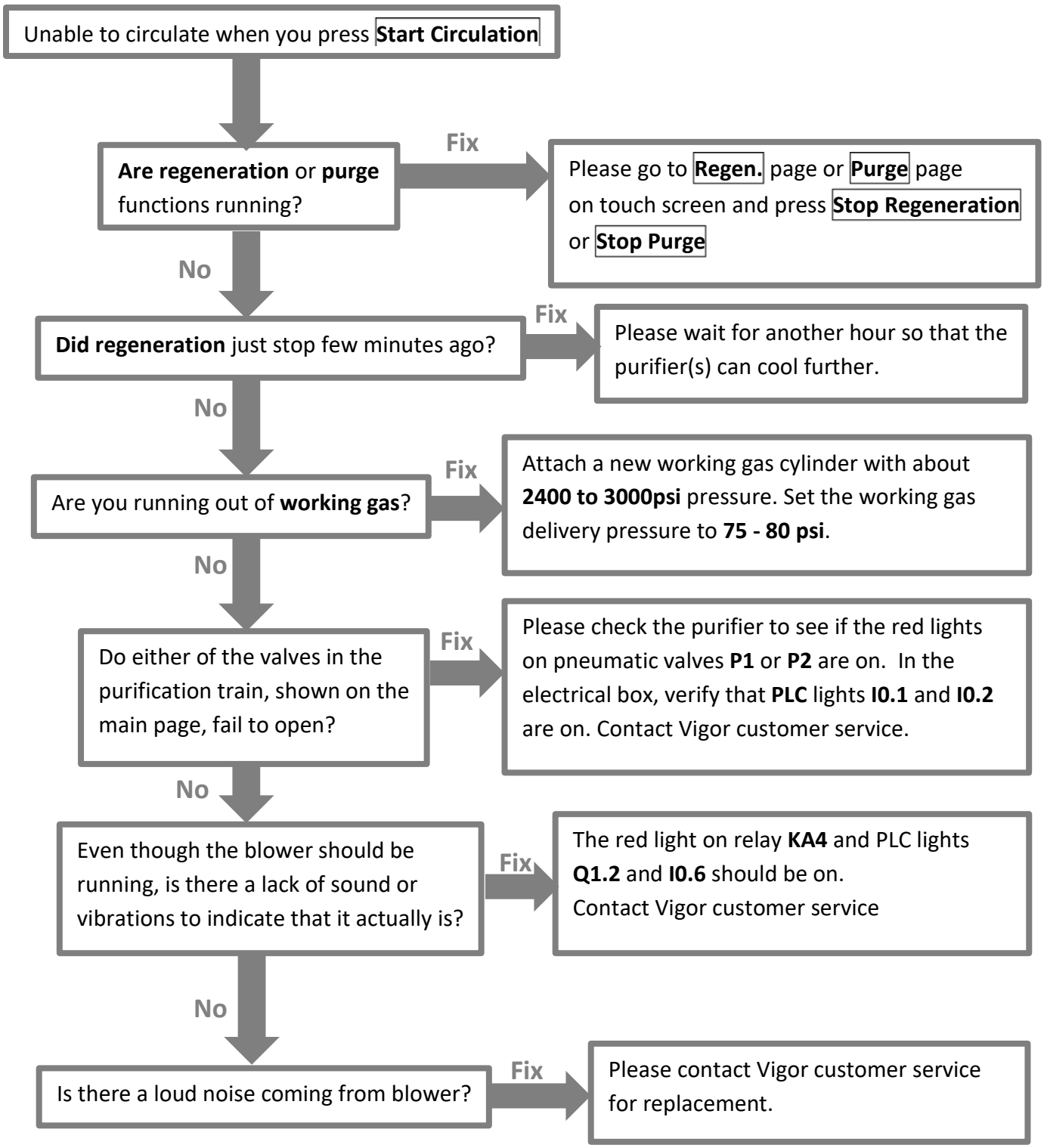




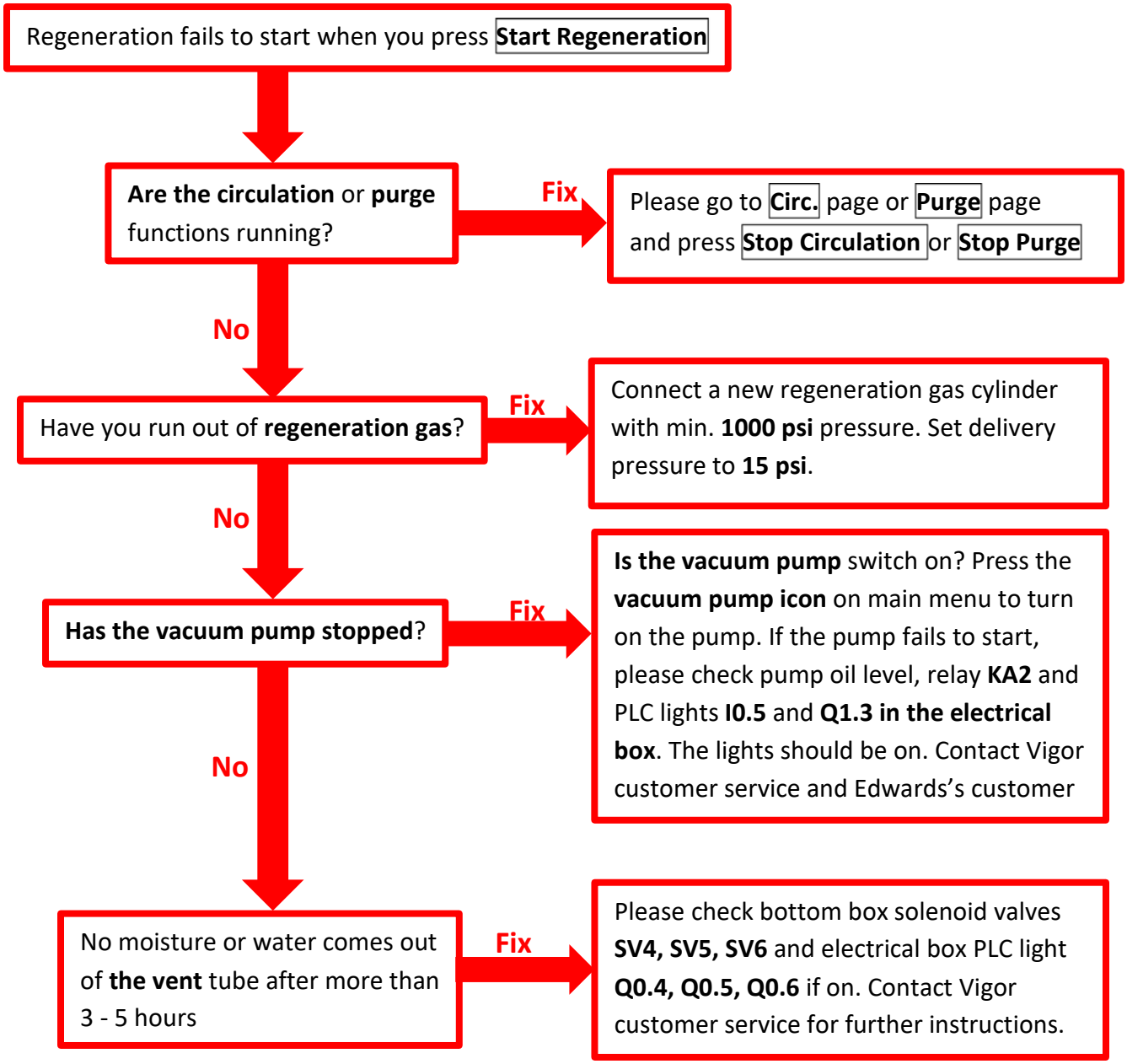
5.2 Purging Problems (Troubleshoot by following the flowchart sequence below)



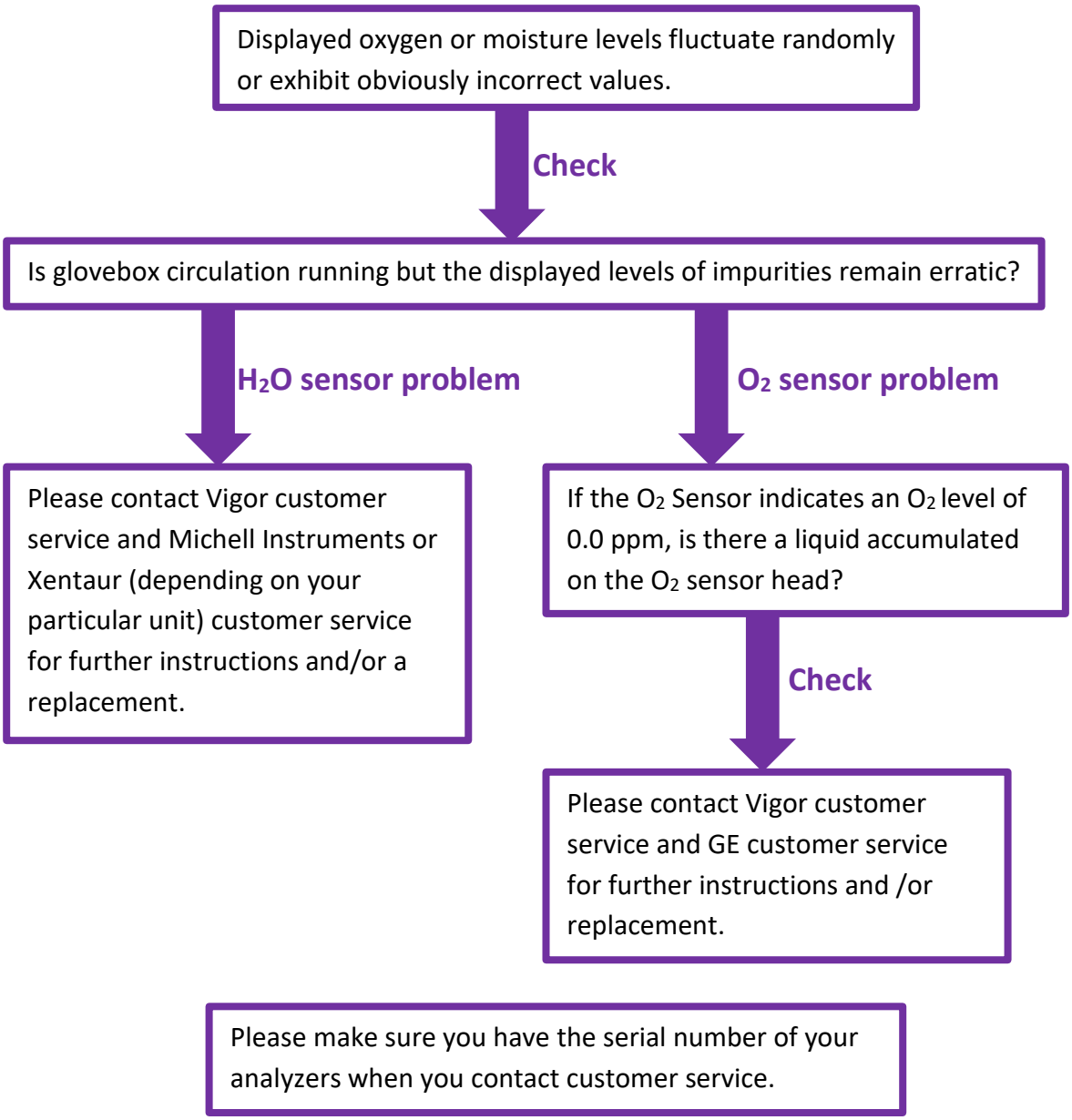
5.3 Circulation Problems (Troubleshoot by following the flowchart sequence below)



5.4 Regeneration Problems (Troubleshoot by following the flowchart sequence below)



5.5 Analyzers Problems



5.6 Other Common Problems

5.6.1 Glovebox Leakage

A simple way to test glovebox leaks (with Oxygen analyzer installed)

Stop **circulation** and **set glovebox to maintain a pressure of 0 mbar**

Wait 30 minutes

Record the displayed oxygen level in ppm

Start circulation

Oxygen level increases with time

Leak in purification train

Possible leakage locations

1. Solenoid valves
2. Inlet gas line (Including regulators)
3. Valve fittings
4. Pneumatic valves (Including clamps)
5. All purifier connections

Soapy water is the simplest method to diagnose leaks.

Leave circulation off

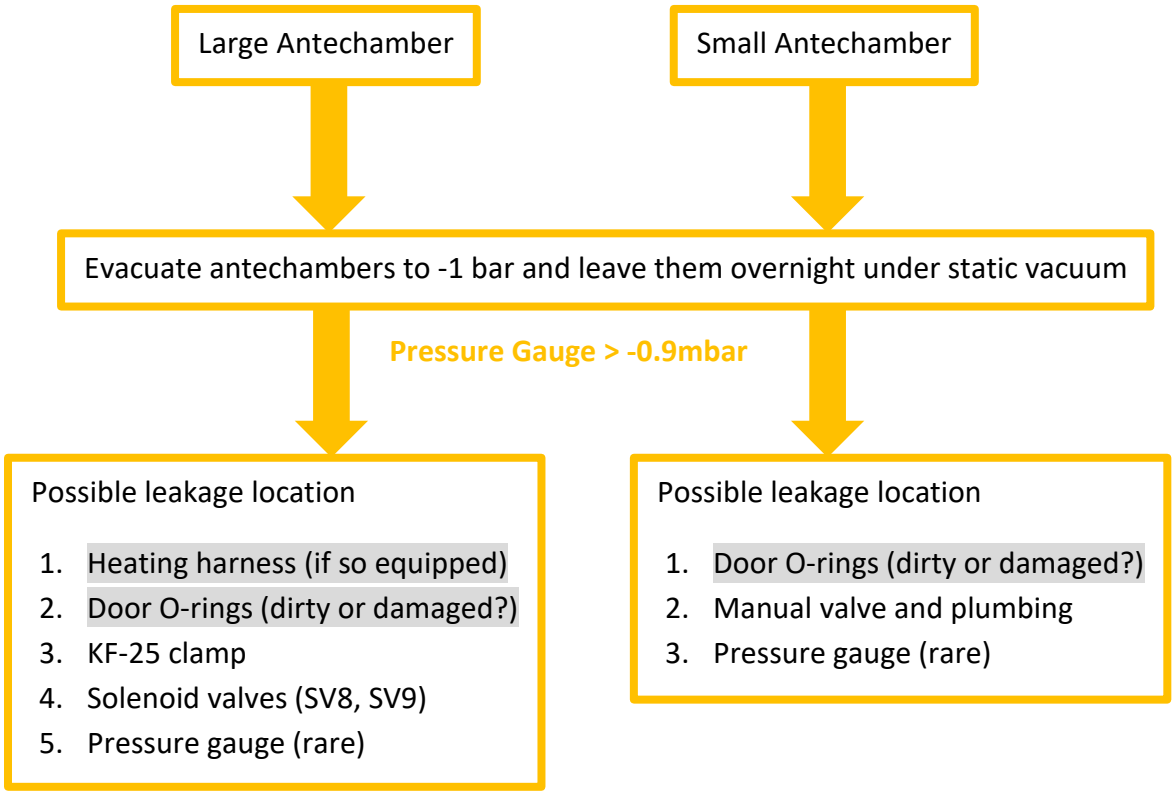
Oxygen level increases with time faster than 1 ppm/hr., which is the expected rate with the circulation off due to diffusion through gloves.

Glovebox leak

Possible leakage locations

1. Purge valve (P3)
2. Gloves (the most common leak)
3. Feedthroughs including clamps
4. Interfaces with analyzers
5. Fittings of antechamber refill tubing
6. Antechamber O-rings
7. Regulators (common with older units)

5.6.2 Antechamber Leakage

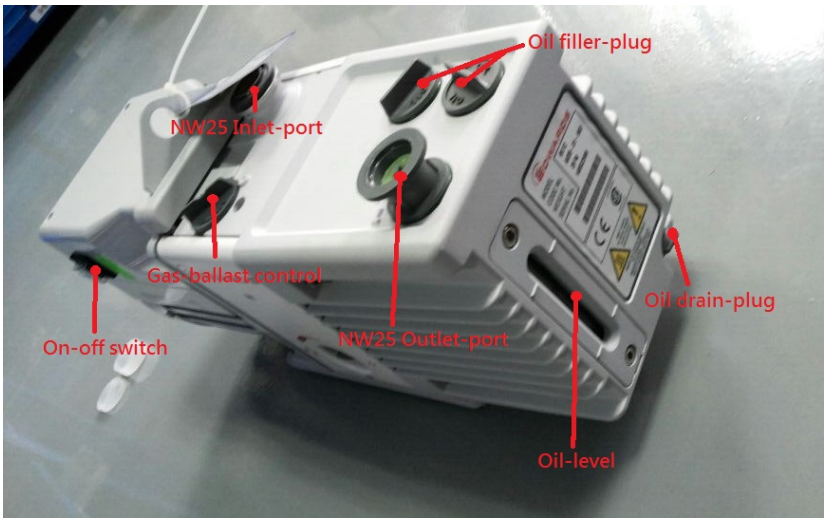


5.7 Error Messages

When **Alarm** is flashing on the main menu page, it means the system encountered some difficulty and the corresponding error message(s) is (are) displayed on the **Alarm** page.

Error Message	*Possible Reasons*	*Solutions*
Vacuum Overloaded	1. The trip current is set too low on adjustable breaker QF2. 2. The pump oil level is too low. 3. The pump oil is dark and/or thickened.	1. Adjust the current dial on the breaker to higher amperage level. 2. Add more pump oil. 3. Change the pump oil.
Blower Overloaded	1. Circulation filters blocked 2. The trip current is set too low on adjustable breaker QF1. 3. PLC generates this message when you try to start circulation but the pneumatic valves have failed to send feedback confirming that they're open. (see FCHZ1, FCHZ2 no feedback)	1. Change filters if they are significantly restricted. 2. Adjust the current dial on the breaker to higher amperage level. 3. Working gas is depleted or the delivery pressure is too low (<70 psi). Replace and/or adjust.
FCHZ1, FCHZ2 no feedback	1. The working gas is depleted or the delivery pressure is too low (< 70 psi) to operate the pneumatic valves. 2. P1 and P2 feedback signal sensors are not in the correct position.	1. Replace cylinder or change delivery gas pressure to at least 70 psi. 2. Adjust the signal sensors positions so that the red indicator lights are on (on = feedback says valve is open).
O_2 exceeds the set value	O_2 concentration exceeds the user-specified threshold value.	Inspect the box and supply gas cylinder connections and pressures. Take corrective actions to bring the oxygen level down.
H_2O exceeds the set value	H_2O concentration exceeds the user-specified threshold value.	Inspect the box and supply gas cylinder connections and pressures. Take corrective actions to bring the moisture level down.

5.8 Vacuum Pump Maintenance (*information is from BOC Edwards RV12 instruction manual)



Tools for Oil Change:
1. Two liter of Ultragrade 19 oil
2. Pliers
3. An empty two-liter container
4. A funnel

Completely Turn off Purge / Regeneration/ Large/ Small Antechamber Evac. and wait for 1 hour until the pump has cooled.

Switch off and unplug pump power and uninstall connections to NW25 inlet and outlet barb.

Open two oil filler-plugs and then open the oil drain-plug to drain the pump oil out. Tilt the pump slightly to completely drain the oil.

Close the oil drain-plug and fill the pump with new oil until the oil level reach the maximum suggested level (marked with lines near the oil-level window).

Close both oil filler-plugs and re-install NW25 inlet bellows tubing and outlet vinyl tubing.

Plug the power back in and switch the pump back on.

Section 6 Technical Information

I. What is the leakage rate? How is it calculated?

The leakage rate is one of most vital parameters for a glovebox. The definition of the leakage rate, when maintaining the glovebox at a constant negative pressure over a period of time, is the rate of the diffusion of impurities into glovebox. Usually, we express the leakage rate as a percentage of the total volume of the glovebox leaked per hour, also known as %Vol/hr. For example, suppose a standard single length glovebox (~750L) is maintained at a pressure of -10mbar over 4 hours and 1 L of impurities are found to have diffused in. The leakage rate is 1 L/ 750 L/ 4 hr. = 1/3000 = 0.03%Vol/hr.

II. A common misperception: When the glovebox is at a positive pressure, air or other atmospheric impurities will not flow into the glovebox.

There are two type of gas flow models. First, pressure differences drive gas flows from high pressure regions to low pressure regions. Second, concentration differences drive gas flows from regions of high concentration to regions of low concentration. Generally, pressure is a larger driving force for flowing gasses.

If there is leakage from a hole which is larger than 0.1mm, pressure will dominate the physics and gas flowing from the inside to the outside of the glovebox will succeed in stopping the ingress of impurities. However, when a hole is on the order of 0.001mm, the pressure-driven process will encounter a large resistance to gas flow through the hole. The diffusion defined process dominates in this situation. The difference between the oxygen concentrations outside and inside of the glovebox is more than about 200K, so the oxygen and moisture will gradually diffuse into the glovebox under the influence of this large gradient.

III. When the purifier is turned off, the oxygen and moisture levels are seen to slowly rise. But, why do impurity levels rise before falling again when circulation is restarted?

The oxygen analyzer detects the oxygen concentration existing around the sensor cell. The measured oxygen level in the glovebox only depends on the concentration of the oxygen, rather than the rate of working gas flow. When a user turns off the purifier system, the oxygen level rises because of air leakage into the glovebox. Therefore, the sealing technology is especially important when circulation is turned off. With a Vigor glovebox, whose purifier is not

currently turned on, oxygen levels can be expected to rise at ~1 ppm/hr. Nearly all of this is diffusion leakage through the gloves and is unavoidable with standard butyl rubber gloves. In this constant pressure environment, with no circulation gas flowing, diffusion will dominate in controlling the oxygen movement inside the glovebox, and this is a comparatively slow process. When circulation is restarted, these oxygen leaks which have "pooled" in tubing and in the gloves, will be mixed into the main glovebox chamber. The oxygen level will increase very briefly and then decrease.

IV. Why do glovebox purifier systems need regeneration? What is the suggested frequency?

The purifier materials function by chemically removing oxygen and physically removing moisture (via adsorption) from the glovebox atmosphere. Fresh adsorbents have the largest impurity removal abilities, but this slowly decreases with time as more impurities are scavenged. When the purifier materials become saturated, the glovebox oxygen and moisture levels will no longer be brought lower by running the circulation. At this point, the Cu-based oxygen absorbent needs to be reduced back to its original metallic form and the zeolites (and carbon, if so equipped) need to be stripped of their adsorbed impurities by regeneration.

There are several factors that influence the time needed to regenerate the purifier:

1. The purifier materials usually scavenge from 20 to 60 liters of oxygen and moisture before they need to be regenerated. Regeneration frequency depends on the quantities of purifier materials present and the quantities of impurities they must remove.
2. The leakage rate and amount of impurities also play an important role. Vigor gloveboxes may attain oxygen leakage rates as low as 0.001%Vol/hr. – 0.005 %Vol/hr. Lack of significant leakage means the absorption abilities of the purifiers will not be bled away at a significant rate. Therefore, the Vigor customer service team recommends regeneration be performed about once a year, under normal circumstances, to maintain efficient impurity removal from the glovebox.

V. How are leakage rates measured?

We use two methods to measure the glovebox leakage rate. One is an oxygen-based method (ISO10648-2) and the other is based on pressure. Either variable is recorded as a function of time during testing. The two methods are complimentary.

Oxygen-based method (used to determine accurate leakage values of oxygen into the glovebox, not as good to detect leakage out of the glovebox. Only usable in low oxygen conditions.)

Although temperature and pressure fluctuations may occur during any leakage test, the oxygen-based method is still very accurate as these variables exert negligible effects on the oxygen analyzer. If you wish, you can perform the following steps to manually test the leakage rate of your Vigor glovebox by the oxygen-based method, however it is much easier to simply allow the glovebox to perform the tests automatically and report the result on the Settings page.

1. Install the oxygen analyzer.
2. Go to “Tasks” on the menu bar.
3. Press “Leakage Test” on the drop-down menu
4. Press the “Test Method” button and leave it on “Oxygen Method”
5. Set the “Test Mode” button to “Manual”.
6. Set the “Test Duration”. A minimum of 5 minutes is satisfactory, however 30 minutes is the default value.
7. You can leave the other values at their default values.
8. Press “Start Manual test”. After 6 + (Test Duration) minutes, the test ends and a new leakage value appears at the bottom of the settings page.

If you want the test to run automatically every night;

1.) Set the “Test Mode” button to “Auto”.
2.) Set the desired time in the “Auto Test Time”.

The maximum passing value is 0.05%Vol/hr.

Pressure-based method (used in any atmosphere, even air, to detect gross leaks of gas out of the glovebox)

1. The glovebox should maintain at a relatively constant pressure over time. However, the internal glovebox pressure is dependent on temperature (1 degree C change= 3 mbar pressure change) and on atmospheric conditions. Since the pressure-based test measures pressure as a function of time, these variations need to be minimized. The most important consideration is holding T steady. The blower is the largest single factor in determining glovebox temperature. The blower should be off for 30 minutes prior to a pressure-based leakage test, ideally. Results are nearly as reliable if the blower has been on for at least 30 minutes, also.
2. Control is via the same screen as the oxygen-based test, but the “Test Method” button is left on “Pressure Method”.

The maximum passing value is 0.5%Vol/hr.

VI. What kinds of oxygen analyzers are commonly used for gloveboxes?

There are two kinds of oxygen analyzers commonly used in gloveboxes. One type is the electrochemical fuel cell sensor. The other is a solid-state Zirconium dioxide sensor. The working characteristics of these analyzers are quite different.

1. Electrochemical fuel cell sensor (EFC)
   When oxygen diffuses past an oxygen permeable Teflon membrane and into the cell, it encounters a potassium hydroxide electrolyte where it is reduced to hydroxide ions by electrons furnished by the simultaneous oxidation of the lead anode. This creates an electric current between the lead anode and the gold-plated cathode through an external load resistance. The current produced is proportional to the concentration (partial pressure) of oxygen present.
   The electrochemical reactions are as following.
   Positive electrode: $2Pb + 4OH^- \rightarrow 2PbO + 2H_2O + 4e^-$
   Negative electrode: $O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$
   Total cell reaction: $2Pb + O_2 \rightarrow 2PbO$ (which implies that the cell is exhausted when the lead anode is depleted)

   Lifetime: In theory, sensor life is limited by the amount of anode material present. In actual practice, because the cell must interface with the glovebox atmosphere to measure it, the electrolyte may eventually dry out over a period of years.

   Advantages
   1. Easy to calibrate. If there is no oxygen gas, there will be no current flow.
   2. The electrochemical reaction, which is just a simple room temperature redox reaction, doesn't interact with organic solvent vapors.
   3. They need no high power supporting circuitry.
   4. Cheaper to replace. Users only need to replace the EFC sensor unit, not the entire unit.

Disadvantages

1. Exposure to high oxygen concentrations for long periods will consume the sensor's capacity to measure oxygen. The whole fuel cell sensor should be protected by an inert gas environment when not installed.
2. Can't be used in high temperature environments.

2. Zirconium dioxide sensor (ZO)

The ZO sensor has porous platinum electrodes at the anode and cathode with a zirconium dioxide solid state membrane/electrolyte between them. When the electrolyte is heated to a high temperature (400 -700^{o}C), oxygen ions becomes mobile within the membrane, generating a voltage which is dependent upon the differences between the partial pressures of the oxygen in the sample and the oxygen in a reference gas (generally air) and is determined by using the Nernst equation.

Advantages

1. A ZO sensor is more sensitive than an EFC sensor. It has a short response time at high temperature and at various pressures.
2. The sensor can be stored in the air and air can be the standard reference gas.

Disadvantages

1. ZO sensor are sensitive to organic solvents because they operate at high temperatures and contain platinum. Hydrocarbon solvents, catalyzed by hot platinum, will react with oxygen thus giving an oxygen reading which is artificially lower than the actual oxygen level.
2. Because air is the standard reference for the ZO sensor, when used for detection of oxygen levels in the 1 ppm O2 range, the great difference between in concentrations between air and 1 ppm O2 (more than 200,000) means that the ZO sensor has to be recalibrated frequently to maintain accuracy.
3. The sensor is expensive and, if broken, needs to be replaced.
4. Needs high power supporting circuitry for heating the zirconia. Since warm-up is slow, this circuitry usually needs to be left on.

VII. What does the vacuum pump gas ballast control do?

The Edwards pumps, provided with Vigor gloveboxes, have gas ballasts.

Glovebox vacuum pumps may pump both permanent gas and vapors. Permanent gases are expelled, but vapors (usually water) can condense to the liquid state during the compression cycle and contaminate the oil. The effects of liquids mixed with the oil may include the reduction of vacuum performance and, in extreme cases, lubrication loss and corrosion leading to possible pump damage.

Opening the Gas ballast valve will have the immediate effect of allowing a small air flow into the compression part of the pumping cycle to dilute the condensable vapors which suppresses their condensation to a liquid phase. Over several hours this can exhaust condensed vapors from the oil.

When opened, the gas ballast will also have these effects; the pump noise and operating temperature will increase as the ultimate vacuum falls somewhat, some oil is lost to misting and the vapor pumping capacity of the pump actually increases due to the temperature rise of the oil (harder to liquefy vapors).

VIII. What are the differences between regular regeneration and solvent regeneration?

	Regular Regeneration	*Solvent Regeneration*
Gas Types[1]	Working gas plus 5% hydrogen gas	Working gas or working gas plus 5% hydrogen gas
Regeneration Time	20 to 24 hours	18 to 20 hours
Heating Temperature	300 to 360°C[2]	180 to 190°C
Catalyst Types	Copper/Zeolite	Active carbon
Purifier Capacities After Regen. [3]	60 Liter of Oxygen 1.5 Kg of Water	Depends on the types of solvents.
Life Time [4]	5 to 10 years	5 to 10 years

[1] 99.995 to ultra-high purity gas, 99.999%
[2] Regular regeneration heating curve, temperature versus time
[3] Standard single length glovebox SG1200/750TS, about 750 L.
[4] The **lifetime of the catalyst** depends on the application

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