Summary of VPAL Set Up Procedures and Exercises

Selected VPAL-A Specifications

Base pressures (typical):

- 150 mTorr (plastic chamber)
- <50 mTorr (glass chamber)

Pump:

- 2-stage rotary vane pump with isolation valve and gas ballast

Gauges:

- 0-30 in Hg atm referenced Bourdon gauge
- <1 mTorr - 1000 Torr absolute MKS micro-Pirani™ transducer
- 1-1600 Torr Vernier gas pressure sensor

Valves:

- Foreline bellows valve for pump isolation and exhaust conductance control
- Needle valve for venting and control of gas inlet

Chamber connection fittings:

- NW40 KF main chamber flange with o-ring and screen
- 1-3/8" compression fitting for glass chambers

Nominal Component Volumes:

Manifold:	50cc
Plastic Bell Jar:	4955cc
Glass Chamber:	190cc

Understanding the Pressure Gauges

Overview

The VPAL-A system is designed to accommodate 3 vacuum gauges:

Bourdon dial gauge: The Bourdon pressure gauge is an atmosphere-referenced direct measuring gauge. The "0" mark is the local atmospheric pressure. Increasing values indicate a lower pressure. To be proper, the readings on the gauge are negative pressures i.e. negative with respect to the ambient. Conversely, positive pressures would be pressures in excess of the local ambient. This gauge is gas-type insensitive. The useful resolution of the gauge is 0.5 in Hg, which corresponds to about 13 Torr.

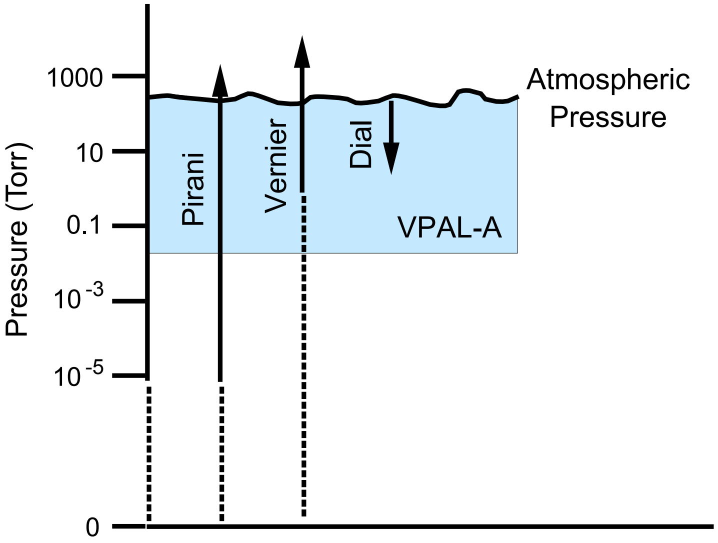
MKS 925C micro-Pirani Transducer: This gauge is an absolute pressure instrument and has a useful operating range of 10^{-5} Torr to atmospheric pressure. It is an indirect gauge and is gas-type sensitive. The operating principle is thermal conduction by the gas from a heated filament.

Vernier Gas Pressure Sensor: This gauge is an absolute pressure instrument that has a useful operating range of approximately 1 Torr to 1600 Torr with a resolution of 1.6 Torr. It is a direct gauge and is gas-type insensitive. The sensing element is a strain gauge.

The figure below shows the operating range and reference point for each gauge and the operating pressure for the VPAL-A.

In terms of the absolute pressure scale, standard sea-level pressure is defined as 760 mmHg (Torr) or 29.92 inches of mercury (in Hg). These values correspond to 101.3 kilopascals (kPa), 1013 millibar (mbar) or 14.7 pounds per sq. inch (psi).

Briefly explain the origins of these measurement units. Which are proper expressions of pressure as Force/Unit Area?



For each of the following applications, state whether atmosphere-referenced or absolute pressure measurement is more appropriate and briefly state why.

- *Medical respirator*

- *Aircraft Altimeter*
-
- *Tire pressure*
- *To calculate the number of molecules of gas contained in a particular volume*
- *To determine the efficiency of a vacuum cleaner*
- *To determine the proper pressure to fill a neon sign tube with gas*
- *To determine whether a vacuum flask (Dewar) has been properly evacuated*
- *Optimum pressure to open a vacuum chamber*

Converting Gage (Atm Reference) to Absolute Pressure

In order to relate gage pressure to absolute pressure we need to know the local atmospheric pressure. The pressure is dependent on local meteorological conditions (reflected in the current barometric pressure) and elevation above sea level. Therefore, the zero reference of our Bourdon gauge is dependent upon location and the weather and is therefore constantly changing.

As an example, let's say the current barometric pressure in Oshkosh, WI is 29.80 inHg. Oshkosh has a published elevation of 770 feet above mean sea level. We can compute the absolute pressure that our gauge's zero corresponds to as follows:

1. Determine the pressure reduction related to elevation above sea level:
    - For elevations between 0 and 5000 ft, apply a correction of -1.006 inHg per 1000 feet

- For elevations between 5000 and 10,000 ft. there is a further correction of -0.862 inHg per 1000 feet.

2. Calculate using: Absolute pressure = Gage pressure + Atmospheric pressure

From (1) the actual pressure at Oshkosh at the time of the barometer reading is:

29.80 inHg – (770 ft x 1.006 inHg/1000 ft) = 29.80 inHg – 0.77 inHg = 29.03 inHg

From (2) our gauge's zero corresponds to 29.0 inHg absolute and 0 inHg absolute is –29 inHg on the gauge's dial.

In this example, what does the range on the gauge's dial between –29 inHg and –30 inHg represent?

Calibrating the Dial Gauge for Today's Local Conditions

Using available resources, acquire the following information:

Your Elevation above Sea Level:
Current Barometric Pressure:

From the above, calculate the ambient atmospheric pressure in inches of mercury (inHg):

Fill out the table on the next page. These relationships will be helpful:

1 inHg = 25.4 mmHg = 25.4 Torr
1 inHg = 3386.4 Pa (N/m^2)

Dial Gauge Reading (inHg)	Corresponding Absolute Pressure (inHg)	Corresponding Absolute Pressure (Torr)	Corresponding Absolute Pressure (kPa)
0			
-1			
-2			
-3			
-4			
-5			
-6			
-7			
-8			
-9			
-10			
-11			
-12			
-13			
-14			
-15			
-16			
-17			
-18			
-19			
-20			
-21			
-22			
-23			
-24			
-25			
-26			
-27			
-28			
-29			
-30			

Pumpdown and Vent Procedures
Initial Vacuum Test

This covers the general procedure for pumpdown and venting and also provides a quick test of vacuum integrity. For this familiarization the system will be equipped with the plastic bell jar and plate. The dial gauge will be used to monitor pressure.

- Check that all fittings are tight, that the bell jar is properly seated on the base plate and gasket, vent valve is closed and main exhaust isolation valve is closed.
- Put on protective eyewear.
- Check the following on the pump: oil level, gas ballast closed (tight), cap on auxiliary inlet port closed (tight), vent unblocked, pump isolation valve open (handle up and down).
- Turn on the pump. It should immediately begin gurgling and then, as the air is removed from the vacuum line, the gurgling should subside.
- Open the VPAL-A manifold's isolation valve several turns. Since air is now being introduced to the pump, the gurgling will resume. The needle on the dial gauge should quickly move off of zero.
- After a minute or two of pumping the gauge should indicate a value close to full vacuum (30 inHg if the system is near sea level).
- When the pressure stabilizes, open the needle (vent) valve just a little bit. The pressure should raise somewhat. Open the needle valve a little more to observe a further rise. This illustrates how partially opening the needle valve can be used to control pressure. Return the needle valve to the closed position.
- Now close the main exhaust valve to isolate the pump. If the system is leak free the pressure should not rise. Open the valve again.

In the following steps the system will be brought back to atmospheric pressure.

For repeated evacuations where the mechanical pump is kept running:

- Close the main exhaust valve to isolate the pump.
- Open the needle (vent) valve fully.
- Observe that the pressure, as indicated by the Bourdon gauge, returns to "0."
- When it is desired to repump the system, simply close the vent valve and open the main exhaust valve.

When it is desired to vent the system fully and shut the pump down:

- Close the isolation valve that is located on the pump.
- Open the needle (vent) valve fully.
- Observe that the pressure, as indicated by the Bourdon gauge, returns to "0."
- Turn off the pump.
- Open the pump's isolation valve.

Improper operation can damage your system! Observe the following:

- Do not start the pump under vacuum. The motor may not have enough startup torque to come up to speed.

Never turn off the pump with the system under vacuum. The oil in the pump will be sucked into the manifold. At the minimum this will require a time consuming clean up. If the MKS micro-Pirani is attached to the system, oil can get into the gauge and destroy the sensor. If for any reason the pump stops while the system is under vacuum, quickly close the pump's isolation valve, the VPAL-A isolation valve and open the needle (vent) valve fully.

Pumpdown Time Constant

Relationships

Pumpdown curve:

$$P = P_0 e^{-(St/V)}$$

where P is the current pressure (Torr), P_0 is the initial pressure, S is the pumping speed (liters/sec), t is time in seconds and V is the system volume in liters.

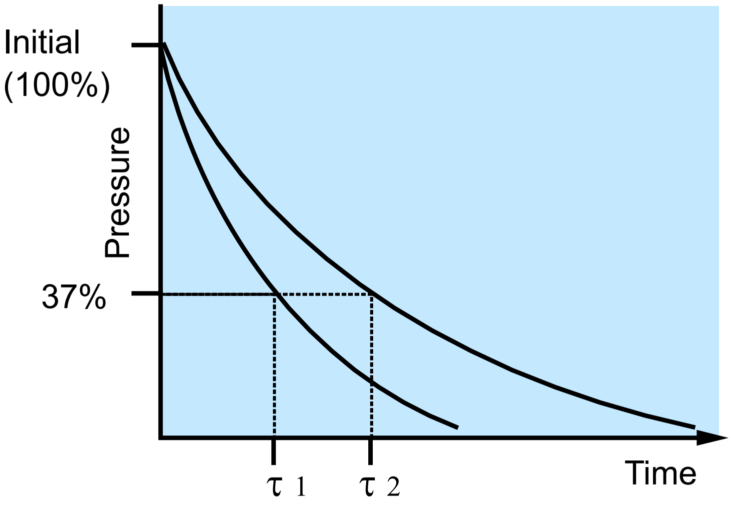
System time constant:

$\tau = V/S$

Time to reach a particular pressure:

$$t = \frac{V}{S}\ln\left[\frac{P_1}{P_2}\right]$$

where t is the time (seconds) to pump from P_1 to P_2, V is the system volume (liters), S is the pumping speed (liters/sec).



How long does it take to pump a system down?

What "real world" factors influence the ideal pumpdown curve?

The first equation may be rewritten as

$$\ln P = -\left[\frac{S}{V}\right]t + \ln P_0$$

If ln$P$ is plotted vs time (this can be done in Vernier Logger *Pro* 3), a straight-line curve will result. The slope of the curve is $-S/V$.

Intuitively one would think that the faster the pumpdown the better. Are there some circumstances where this is not the case?

Simple Boyle's Law Demonstrations (PV = Constant)

Expansion of a partially filled balloon placed under vacuum

1. Partially inflate a rubber balloon (select a balloon that is as spherical as possible).
2. Determine the initial volume of the balloon.
3. Place the balloon within the vacuum chamber, close the pump isolation valve and turn on the pump.
4. Open the isolation valve and bring the chamber to a pressure slightly (10%) below atmospheric pressure. Measure the balloon's volume as accurately as possible. If the pressure undershoots, use the needle valve to raise the pressure.
5. Repeat for successively lower pressures.

6. Plot the actual measured volumes as a function of pressure. Compare the measured data with the expansion as predicted by theory.

Discuss:

Sources of error in the results:

Reasons for the differences between the curve resulting from measurements and the curve as predicted by theory: Exclude errors that aren't related to the measurements.

De-airing of a liquid

This demonstrates a practical application of Boyle's Law.

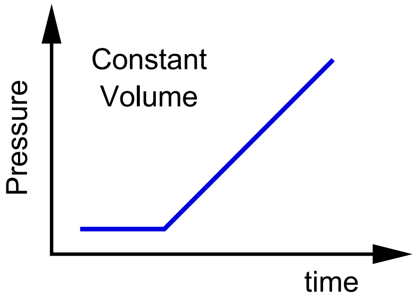
1. Obtain a small quantity of clear oil (mineral oil, fresh vacuum pump oil). Shake the oil to develop some bubbles within the oil.
2. Place a small amount (about ½ inch deep) in a small clear plastic cup (the cup should be about 3-4 inches tall).
3. Place the cup within the vacuum chamber, close the pump isolation valve and turn on the pump.
4. Open the isolation valve slightly and slowly reduce the pressure while monitoring the dial gauge. Observe the oil carefully for an increase in size and density of bubbles. Once bubble formation is noted, be careful in any further pressure decreases to avoid "burping" of the oil.
5. Let the process continue until the bubbling substantially ceases.

Describe what is happening and the relationship to Boyle's Law.

Identify practical applications for this process.

Characterizing and Quantifying Leaks using the Pressure Rate-of-Rise Method

The general form of a leak in terms of pressure vs time is represented here:

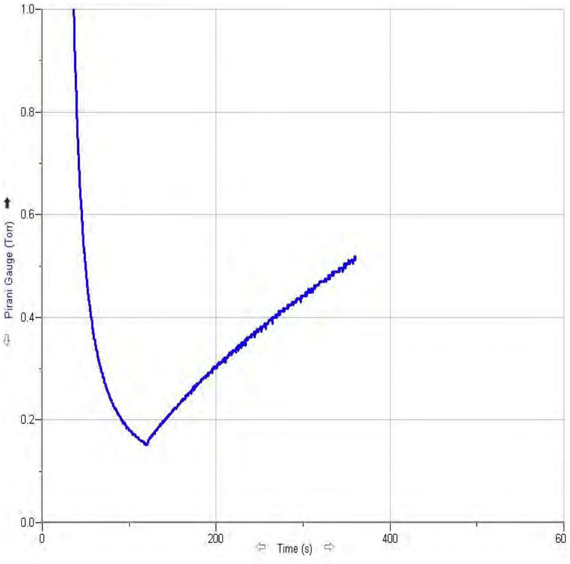


The leak rate may be quantified using this expression:

$$Q = 79\left[\frac{273}{273+T}\right]\left[V\frac{\Delta P}{t}\right]$$

where Q is in standard cc/min (sccm), 79 is the conversion factor from torr-liter/sec units to sccm, T is the ambient temperature in Celsius, V is the chamber volume in liters, ΔP is the change in pressure in torr over the time t in seconds.

An example “real” curve is shown below:



Does this represent a real or virtual leak?

Run three successive rate-of-rise tests. In each case the 925C transducer will be used to monitor the pressure. Set the x-axis of Logger Pro 3 for a sufficient duration to run several tests (about 1800 seconds.)

1. Plastic chamber:

- Attach the plastic chamber to the VPAL manifold. Pump the system to a pressure of about 0.5 Torr, close the vacuum isolation valve and observe the pressure rise.
- After several minutes open the isolation valve and repump the system until the base pressure is noticeably lower than in the first run and repeat the test.

- Repeat a third time

What differences are seen between the pressure rise curves for each of the above runs?

2. Glass chamber:

- Repeat #1 using the glass chamber. In the second and third run pump the system to base pressures <0.1 Torr before starting the test. In the third run try to get well below 0.1 Torr i.e. as close to the system's base pressure as possible.

Describe the differences between #1 and #2. To what can the differences be attributed?

3. Insert several inches of silicone rubber tubing into the glass chamber and rerun #2.

4. Remove the silicone tubing from the glass chamber and attach the tubing to the hose connector on the needle/vent valve. Place a pinch clamp at the extreme end of the tubing.

- Pump the system until the pressure stabilizes. *How does the new base pressure compare to the base pressure in #2?*
- Close the isolation valve and monitor the pressure rate-of-rise. Let this continue for several minutes. *How does the form of the curve compare with the curve when the tubing was contained within the chamber?*

What effect does the silicone tubing have on the results? Could silicone rubber be a good vacuum material? Under what circumstances?

Quantify the leak rates using the rate-of-rise equation and the known volumes of the system components.

Determining the Size of an Orifice Leak

Included with the VPAL are two orifice leaks.

Attach the 200 micron leak to the hose connector on the needle/vent valve. Install the air filter on the open end of the orifice to prevent the orifice from clogging. Install the plastic bell jar and base plate to the manifold. For this exercise monitor the pressure with both the dial gauge and the Vernier gas pressure sensor.

Close the valve and pump the system to base pressure.

- Recheck the pressure rate-of-rise to ensure that the system is relatively leak free.
- Open the vent valve so that air flows through the orifice. Is the orifice in choked (sonic) flow?
- Let the pressure rise to atmospheric pressure. *What happens to the shape of the rate-of-rise curve*?
- Calculate the choked flow rate using the rate-of-rise equation.

Determining Mass Flow Rate with a Bubble Meter

The bubble meter is a simple mass flow meter that uses the change in volume at constant pressure to determine mass flow rate. This type of device is commonly used to calibrate electrical mass flow meters.

A bubble meter is supplied with the VPAL-A. It consists of an open ended graduated cylinder with a stopper and hose connector at the upper end as shown in the figure on the next page.

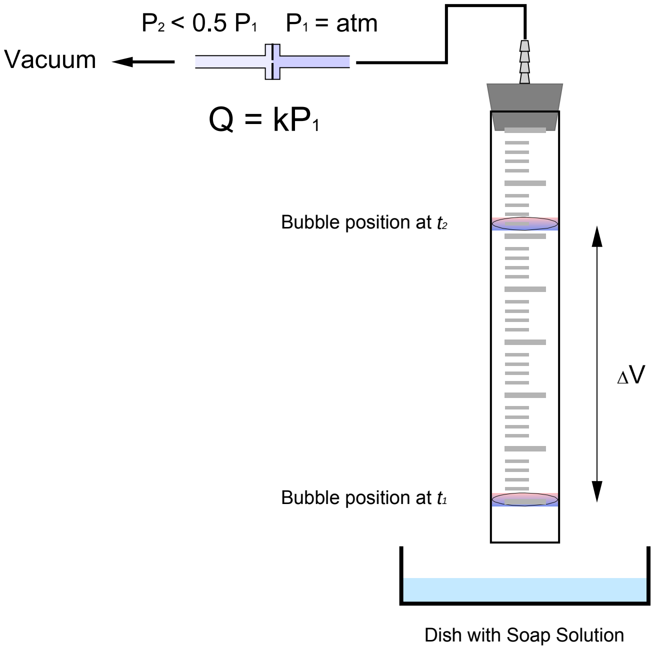
Mass flow is calculated as follows:

$$Q = \left[\frac{P}{760}\right]\left[\frac{\Delta V}{\Delta t}\right]\left[\frac{273}{273+T}\right]$$

Prepare the system as shown in the figure using bubble solution or a pure dishwashing soap mixed with water. Take care to not let water or soap solutions get into the manifold. It is advisable to remove the MKS 925C transducer during this exercise. Wetting the inside surface of the cylinder helps to prolong the life of the soap film.

- Pump the system with the needle/vent valve fully closed.
- Dip the end of the cylinder into the soap solution. When a film has formed, open the needle/vent valve fully.
- Using a stopwatch, time the movement of the film between two widely separated graduations.
- Repeat the test one or more times to check consistency.
- Calculate the flow rate in sccm.

How does the leak rate by this method compare with the leak rate as determined by the pressure rate-of-rise method?



Calculating Effective Pumping Speed and Simple Pressure Control

Now that the orifice has been sized in terms of sccm, we can use the orifice to calculate the effective pumping speed of our vacuum pump.

Attach the orifice to the hose connector. Install the air filter on the open end of the orifice to prevent the orifice from clogging. Replace the plastic bell jar and base plate with the glass chamber. Monitor pressure with the MKS 925C transducer.

- With the needle/vent valve closed, pump the system to a pressure below 0.1 Torr.
- Open the needle/vent valve.

From the pressure vs time plot, describe what happens when the valve is opened.

What is the new base pressure?

Compute the effective pumping speed using the relationship Q = P x S where Q is the flow through the orifice, P is the new base pressure (assumed to be much greater than the original base pressure with no flow) and S is the effective pumping speed.

How does the effective pumping speed compare to the manufacturer's stated pump speed?

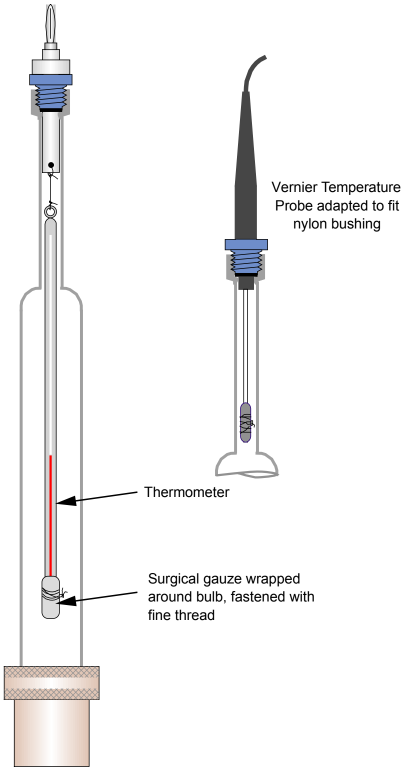
Evaporative Cooling and the Phases of Water

In this exercise the phase and vapor pressure characteristics of water will be examined.

The water sample is in the form of a water soaked piece of gauze that is attached to either the bulb of a liquid thermometer or, preferably, to a Vernier stainless steel temperature probe if available. Both arrangements are shown in the figure below.

Pressure will be monitored with the MKS 925C gauge. If the Vernier temperature probe is used, pressure and temperature should be monitored simultaneously.

Only a small amount of water is required, just enough to saturate the gauze. The pump is best operated without the gas ballast in order to achieve a sufficiently low pressure (below 2 Torr).



Set up the apparatus, let the temperature stabilize and then pump the system to about 30-40 Torr. Resume pumping and note the interrelationship between the pressure and temperature curves.

As the water cools and turns to ice, what happens to the system pressure and the temperature? Explain in terms of phase change and latent heat of vaporization.

Describe how water vapor could be a source of defects in high purity process systems.

The Freeze Drying Process

Purpose

The VPAL-A can be used to freeze dry small samples of organic matter. The process demonstrates evaporative cooling at low pressures, sublimation and the effect of gas load on vacuum system operating pressure.

Method

The VPAL-A is configured with a Vernier Surface Temperature Probe that is inserted into the Nalge bell jar through the base plate side port. Pressure is monitored with the MKS 925C transducer.

A convenient sample is a high moisture content vegetable, for example a quarter slice of cucumber between 1/8 and 3/16 inch thick. The sample is frozen using a freezer or "quick cool" cooling spray.

The frozen sample is placed in the vacuum chamber with the tip of the temperature probe placed in direct contact with the sample. The chamber is then evacuated. The operating pressure has to be below the triple point for sublimation to occur and will typically be in the vicinity of 1 Torr. The process is run until evaporative cooling ceases and the sample returns to room temperature.

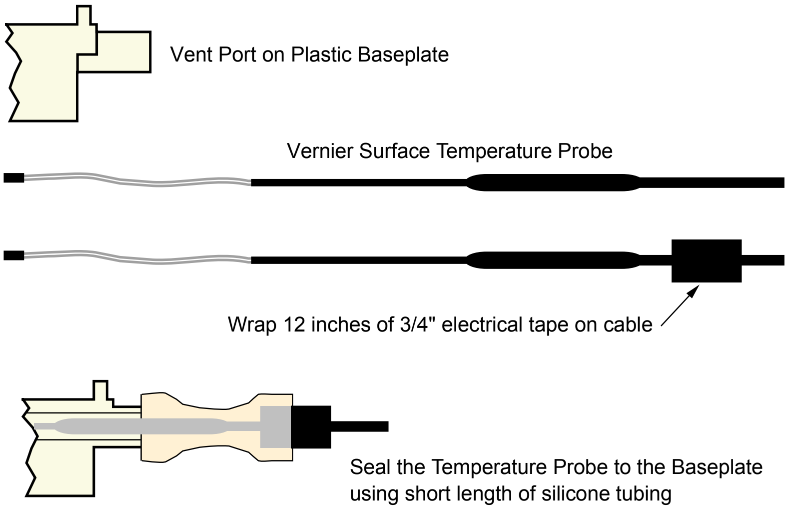
The vacuum pump will probably not be able to achieve a low enough pressure with the gas ballast on. With the gas ballast off, the pump oil will accumulate some water. This will not cause any damage (in moderation), but the oil will be contaminated. When the experiment is over the pump should be run with the ballast open until the oil clears and no longer has a milky appearance.

The full drying process requires an overnight run.

Preparing the Surface Temperature Probe

Prepare the Vernier Surface Temperature Probe as shown in the illustration on the next page.

Wrap the cable just behind the thick section with approximately 12 inches of vinyl electrical tape. The diameter of the tape wound cable should make a tight fit in a short length of ¼ inch inside diameter silicone tubing. Slide the temperature probe through the base plate's side port and slip the other end of the tubing over the base plate's side port tubulation.



Wrap 12 inches of 3/4" electrical tape on cable

Seal the Temperature Probe to the Baseplate using short length of silicone tubing

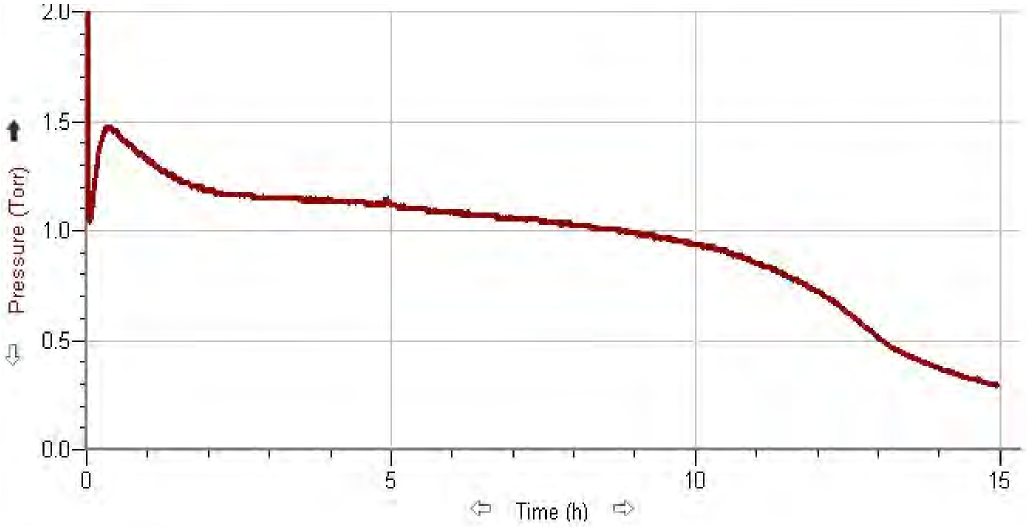
Results

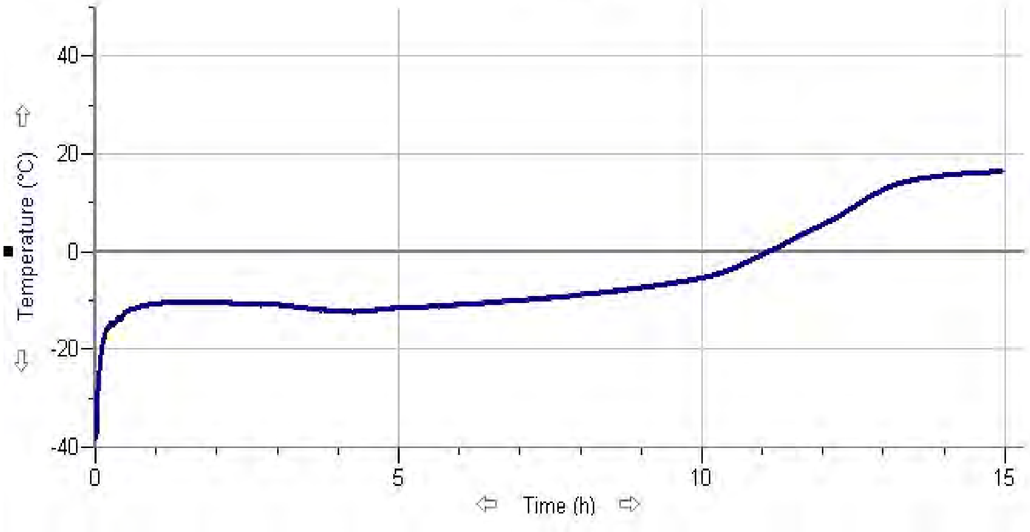
A representative result is shown on the next page.

Explain what is happening during the first ½ hour

Explain the main features of the process during the balance of the cycle.

What methods could be employed to reduce the time to dry the sample?


2.0
1.5
1.0
0.5
0.0
Pressure (Torr)
0
5
10
15
Time (h)


40
20
0
-20
-40
Temperature (°C)
0
5
10
15
Time (h)

The Glow Discharge and Sputter Deposition

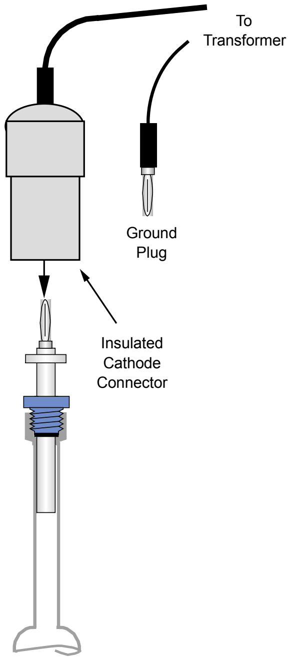
Glow discharges are the agents behind plasma processes.

In these exercises we will examine some characteristics of the DC glow and then use the glow discharge phenomenon to demonstrate the sputter deposition of copper.

A current regulated high voltage power supply is required for these exercises. The supply that is supplied for the VPAL-A is a transformer of the neon sign type and has a peak output of -4 kV at 20 mA.

The high voltage connector is contained within a plastic insulated housing that mates with the banana plug that projects from the VPAL's electrode.

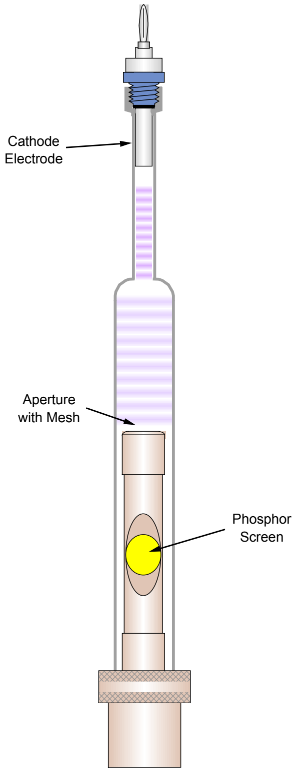
A separate ground electrode must be inserted into the ground connector on the VPAL unit's base. The transformer must also be plugged into a grounded outlet. Failure to properly ground the system may result in a high potential appearing on the exposed parts of the apparatus and may damage the electronic components.



Glow Discharge Electron Gun

This exercise will show the characteristics of the glow discharge at various pressures. The apparatus will also produce a glow discharge generated electron beam. The effect of increasing mean free path will be revealed as the electron beam becomes highly collimated at low pressure.

Assemble the components as shown in the figure on the next page.



Monitor pressure with the MKS 925C transducer.

With the system at atmospheric pressure, turn on the pump and power supply. Using the isolation valve, slowly decrease the pressure in small increments and note when the glow discharge first strikes. Continue to lower the pressure noting the appearance of the various regions of the discharge, especially the cathode dark space, the positive column, coloration and intensity.

As the pressure declines an electron beam will form, eventually illuminating the phosphor screen.

What can be implied by the change in coloration of the glow discharge?

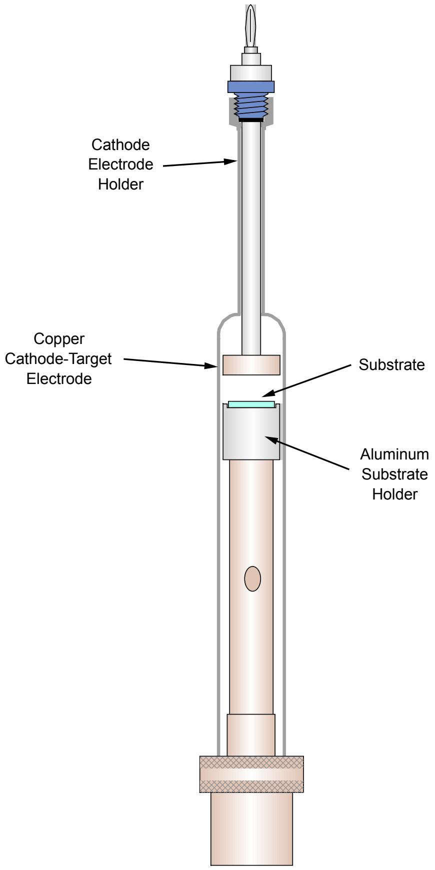
Describe and explain the appearance and characteristics of the electron beam.

Use a magnet placed near the tube to deflect the electron beam. Why does the beam move?

If the pressure were to continue to decline, what would happen to the glow discharge?

Sputter Deposition

By changing electrode assemblies the VPAL-A can be used to produce metal films by means of the DC sputter deposition process. The configuration is shown in the figure below.



With the glass chamber removed from the chamber fitting, place the substrate holder pedestal assembly within the fitting. It will be resting on the screen of the center ring. Place a glass substrate (microscope cover glass or similar) within the grooved slot of the substrate holder.

The target electrode assembly has to be inserted through the large bore of the chamber. Assemble everything so that there is a ~3/8 inch gap between the target electrode and the glass substrate.

Ideally argon would be used as the control and cover gas but, for demonstration purposes, air will work satisfactorily.

When assembled, pump the system down to a stable base pressure below 0.1 Torr. Turn on the power supply and very carefully open the needle/vent valve. The discharge will strike. Adjust the pressure in small increments to increase the width of the cathode dark space. If the pressure is too low, the cathode dark space will fill the gap and the discharge will become obstructed and will go out.

Once the pressure and conditions are stable, let the process proceed for several minutes. Sputtered copper will also appear on the glass chamber walls.

Turn off the power supply and return the system to atmospheric pressure. Examine the film for uniformity and density. Additional runs may be made with different times and conditions.