Particulate Systems

HPVA Series

High Pressure Volumetric Analyzer

Operator's Manual

V4.0

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March 2011

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1. Introduction

This manual describes how to operate and maintain the HPVA High Pressure Volumetric Analyzer, Models HPVA-100 and HPVA-200.

Conventions

This manual uses the following conventions:

Indicates important information pertinent to the subject matter.

Provides information that helps you prevent actions that may damage the instrument.

Provides information that helps you prevent actions that may cause personal injury.

Blue words — **Indicate a link to additional information about the subject matter.**

Equipment Description

The High Pressure Volumetric Analyzer (HPVA) from Particulate Systems is designed to obtain high-pressure adsorption isotherms using gases such as hydrogen, methane, and carbon dioxide employing the static volumetric method. The volumetric technique consists of introducing (dosing) a known amount of gas (adsorptive) into the chamber containing the sample to be analyzed.When the sample reaches equilibrium with the adsorbate gas, the final equilibrium pressure is recorded. These data are then used to calculate the quantity of gas adsorbed by the sample. This process is repeated at given pressure intervals until the maximum pre-selected pressure is reached. Each of the resulting equilibrium points (volume adsorbed and equilibrium pressure) is then plotted to provide an isotherm. Excellent reproducibility and accuracy are obtained by using separate transducers for dosing the sample and for monitoring the pressure in the sample chamber.

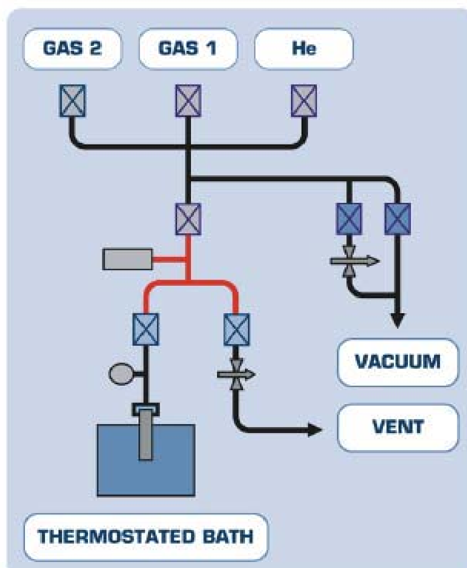
The HPVA Series of analyzers includes two models: the HPVA-100, capable of achieving pressures up to 100 bar and the HPVA-200, capable of achieving pressures up to 200 bar.

The HPVA Analyzer includes a separate degas port for drying the sample before testing. The furnace is capable of temperatures up to 500 °C, controlled with a routine which includes ramp and soak capabilities.

To protect the user, the HPVA includes a hydrogen gas sensor to detect excessive levels of H2 gas should a leak in the system occur. If tripped, the safety circuit places the HPVA unit in a safe condition by removing power to the unit and closing all valves.

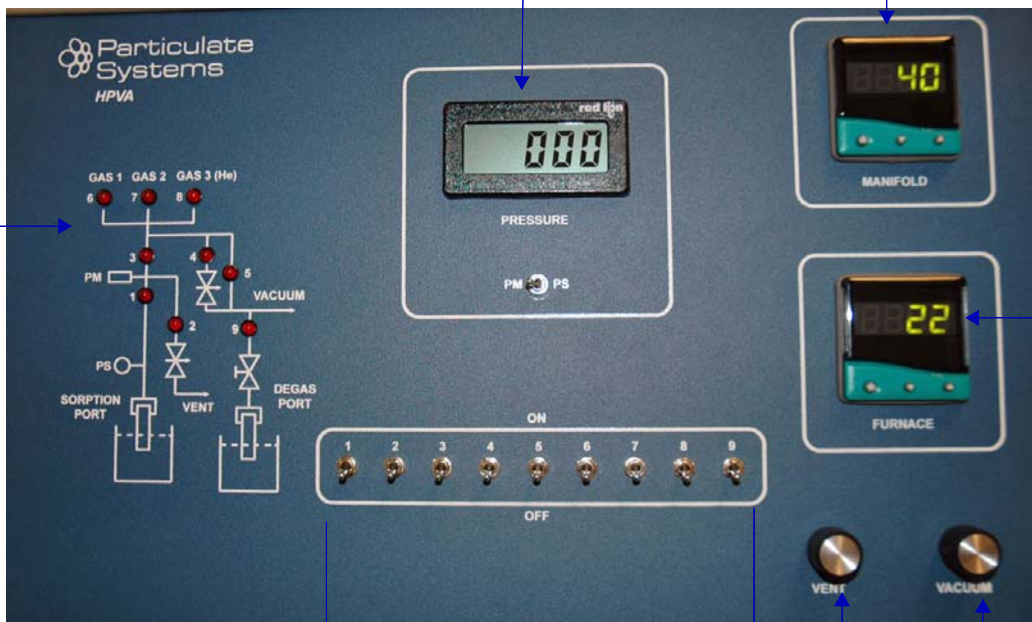
Analysis results are displayed real-time on the computer screen during an experiment and details can be viewed and printed via a Microsoft® Excel macro provided with the application software.

The standard system consists of a vacuum pump and gauge, a gas manifold maintained at a constant temperature (40 °C typically) with two pressure transducers, a sample chamber, an outgassing furnace with PID controller, a temperature control vessel that can be connected to a constant temperature bath, and two stainless steel sample holders with diaphragm shut-off valves. The following is a schematic of the HPVA system. A system schematic can be viewed from within the application software and can be used to manually control the valves if required.



Controls and Connectors

Upper Front Panel



Pressure	Displays the manifold pressure when set to **PM** and the sample pressure when set to **PS**. The display is useful as a quick reference; a more accurate reading of the pressure can be displayed on the software screen.
Manifold Temperature Control and Display	Displays the current manifold panel heater temperature in degrees Celsius. Also enables you to increase or decrease the set point temperature.
Furnace Temperature Control and Display	Displays the current furnace temperature in degrees Celsius. Also enables you to increase or decrease the set point temperature.

Vacuum Valve Control

Enables you to control the flow rate through vacuum valve 4.

Vent Valve Control

Enables you to control the flow rate through the vent valve.

Valve Switches

Enable you to open and close valves. You can also open and close valves 1 through 8 through the software, which is the recommended method of manual control.

Valve 9 can be controlled by the switch only.

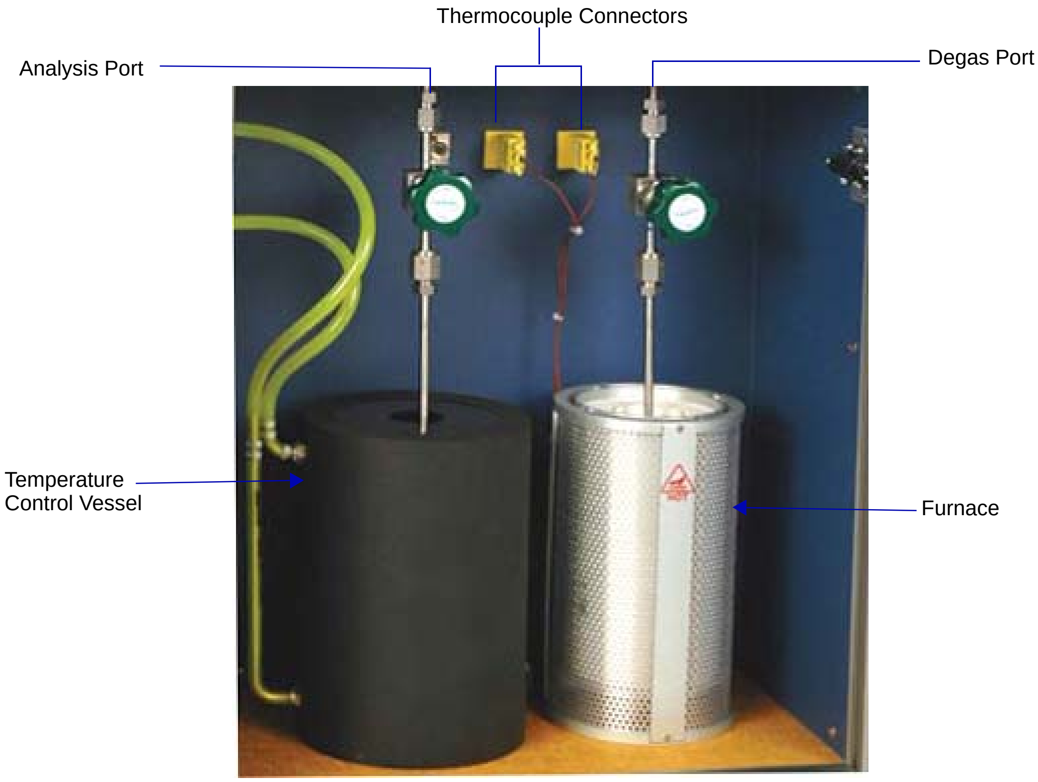
System Schematic

A schematic of the manifold, showing valve status. A valve indicator turns on when the valve is open and turns off when a valve is closed.

When the HPVA is performing an experiment or being operated in manual control mode, the valve switches on the front panel should be placed and remain in the closed (down) position.

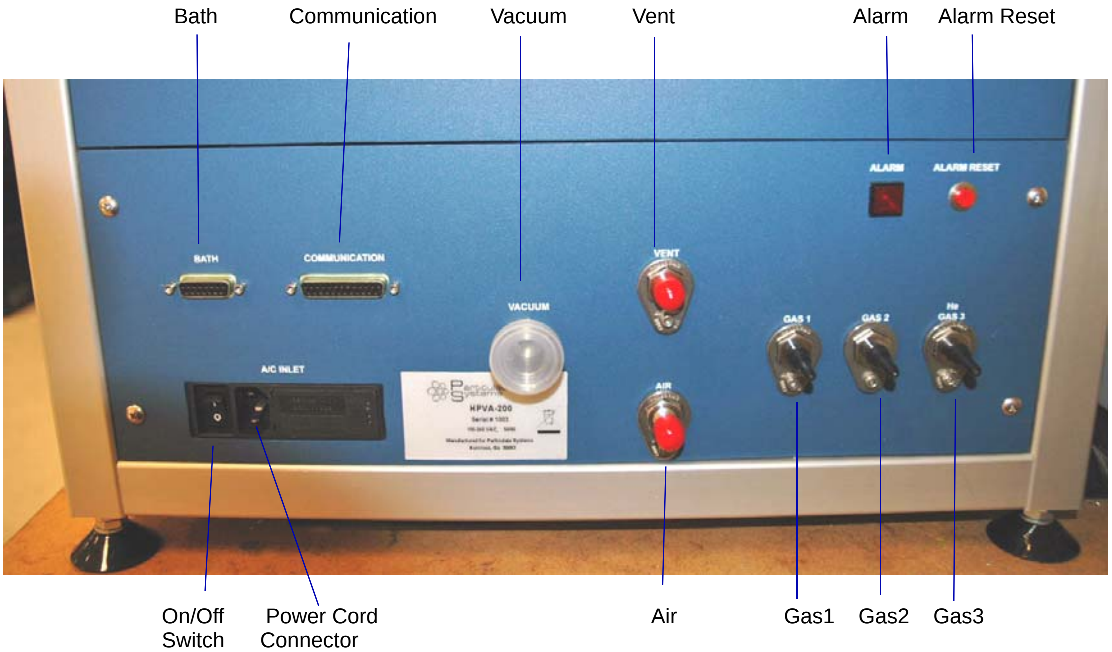
If a switch is placed in the opened position during a software operation, it will override the software and may damage the instrument.

Sample Compartment



Temperature Control Vessel	Maintains the temperature of the sample during an experiment.
Analysis Port	Port used to perform an experiment.
Thermocouple Connectors	Connect the furnace thermocouples to the instrument.
Degas Port	Port used to evacuate a sample (remove moisture and contaminants) prior to performing an experiment.
Furnace	Maintains the desired temperature during the preparation process.

Rear Panel



Bath	Connects the recirculating bath to the instrument.
Communication	Connects the computer to the instrument.
Vacuum	Connects the vacuum pump to the instrument.
Vent	Connects vent tubing to the instrument.
Alarm	Turns on when power is applied to the instrument or when the H2 sensor is triggered.
Alarm Reset	Must be pressed after the instrument On/Off switch is placed in the ON position and when the H2 sensor is activated, which shuts off power.
On/Off Switch	Turns power to the instrument on or off.
Power Cord Connector	Connects the power cord from the power source to the instrument.

Air	Connects the nitrogen or compressed air supply for the pneumatic valves to the instrument.
Gas1	Connects an analysis gas to the instrument.
Gas2	Connects another analysis gas to the instrument.
Gas3	Connects helium (backfill gas) to the instrument.

Turning the Analyzer On and Off

To turn on the analyzer:

1. Place the power switch on the rear panel of the analyzer in the ON (|) position. The alarm indicator will light indicating that power is applied to the instrument.
2. Wait about 20 seconds, then press the **ALARM RESET** button on the back panel of the analyzer.
3. The alarm indicator will turn off.

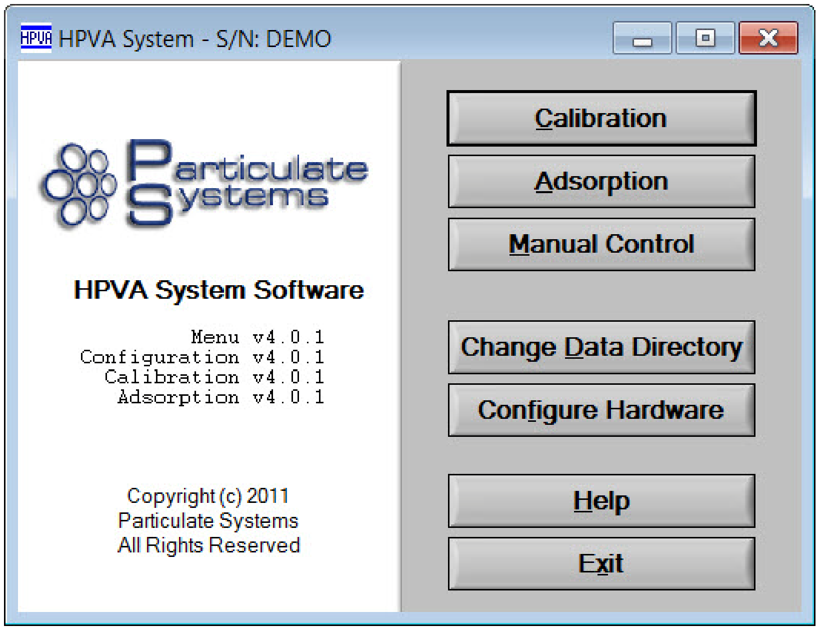
4. Wait 30 to 60 minutes to allow the manifold temperature to stabilize.

To turn off the analyzer:

1. Ensure that the analyzer is vented to atmosphere.
2. Place the power switch on the rear panel of the analyzer in the OFF (O) position.

Main Menu

The Main Menu enables you to access all software functions of the HPVA system.



Calibration	Used to calibrate the manifold volume. This function is used by authorized service personnel only.
Adsorption	Enables you to enter experiment conditions and to perform an experiment.
Manual Control	Enables you to manually control the system through a schematic displayed on the screen.
Change Data Directory	Enables you to change the directory in which experiment data are stored.
Configure Hardware	Enables you to change parameters that affect the operation of the analyzer.
Help	Displays the HPVA Series Operator's Manual on the screen. Use the Bookmarks and Table of Contents to find the information you need.
Exit	Closes the HPVA software.

Starting and Exiting the HPVA Software

To start the HPVA software, click the **HPVA** icon on your desktop or select the software using the appropriate Windows function.

To close the HPVA software, click the **Exit** button on the HPVA Main Menu.

File Names

The following table shows the file name extensions used by the HPVA software.

File Type	Extension
Data file - generated only if Advanced Mode is activated; primarily used to troubleshoot the instrument; contains data for everything that occurred during the analysis	dat
Long data file - contains the manifold and sample pressures and temperatures recorded over time	HIlo
Short data file - used for the isotherm calculations	HIsh
Helium free space data file - used to calculate free space in the sample tube	HIHe
Analysis parameters file -used to load previous analysis conditions prior to the experiment	HItm

Specifications

Characteristic	Specification
Environment	
Temperature:	10 to ºC, operating; -10 to 55 ºC, storing or shipping
Humidity:	20 to 80% relative, non-condensing
Physical	
Height	35 in. (88.9 cm)
Width:	20 in. (50.8 cm)
Depth:	20 in. (50.8 cm)
Weight:	60 lbs
Electrical	
Voltage:	115/230
Power:	15 Amps
Frequency:	50 to 60 Hz
Gases	
Handles typical adsorbates such as Nitrogen, Hydrogen, Methane, Argon, Oxygen, Carbon Monoxide, and Carbon Dioxide.	
Pressure	
Pressure range	HPVA-100: vacuum to 100 bar, HPVA-200: vacuum to 200 bar
Pressure transducer	Pressure reading accuracy is ±0.04% full scale with a stability of ±0.1%
Computer	
Minimum requirements:	Intel Core 2 Duo processor - 2.0 GHz or faster One DVD ROM drive 1 gigabyte of RAM 20-gigabyte hard disk space SVGA Monitor Windows® XP Professional or Windows 7; Microsoft® Excel 2002 or higher PCI slot for National Instruments control board One RS232 serial port for each attached instrument and accessory requiring an RS232 connection Mouse Printer that is IBM Graphics or Epson LQ compatible (optional) UPS for computer (optional)

2. Performing an Experiment

This chapter provides step-by step procedures for performing an experiment including:

- Preparing a sample
- Degassing a sample
- Entering experiment parameters
- Running an experiment

Preparing the Sample

Requirements

The following items are required to prepare a sample and perform an experiment with the HPVA analyzer.

Supplied by User	Supplied by Particulate Systems
• Ultrasonic bath	• Sample holder assembly
• Long-stemmed pipette	• 5/8-in wrench
• Drying oven	• 3/4-in. wrench
• Dry, compressed nitrogen or air to dry sample cylinder	• Funnel
• Isopropyl alcohol (IPA); acetone or deionized water may be used if IPA is unavailable	
• Balance	
• Detergent	

Cleaning the Sample Cylinder

1. Preheat the drying oven to 110 °C.

2. Clean the sample cylinder using a ratio of 5 grams of detergent (Alconox or a similar product) per 500ml of warm water. Fill the bowl of the ultrasonic unit with enough water to cover the sample cylinder. Ensure that the detergent is dissolved before placing the sample cylinder into the water.

3. Submerge the sample cylinder in the bath and fill a long-stemmed pipette with water. Pipe the water into the cylinder to remove all air.

4. Turn on the ultrasonic bath and allow it to run for approximately 30 minutes.

5. Using latex gloves, remove the sample cylinder from the bowl. Fill a pipette with hot water and then pipe the water into the cylinder to rinse it. Repeat this process several times.

6. Rinse the cylinder with Isopropyl Alcohol.

7. Using dry, compressed nitrogen or air dry the interior of the sample tube.

8. Place the sample cylinder into a drying oven that has been preheated to 110 °C.

9. Bake for two hours. After two hours remove the cylinder and allow it to cool.

Weighing the Sample

1. Tare a balance and allow it to stabilize at zero.
2. Place the sample cylinder on the balance and record the weight as ***Weight of empty sample cylinder***.

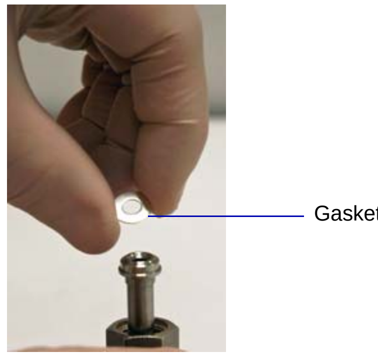
3. Remove the sample cylinder from the balance and place a funnel in the sample cylinder.
4. Slowly add the sample material to the cylinder.

5. Tare the balance and allow it to stabilize at zero.
6. Place the sample cylinder, with sample, on the balance. Record the weight as ***Weight of sample cylinder plus*** sample.

7. Subtract the ***Weight of sample cylinder*** (recorded earlier) from the ***Weight of sample cylinder plus sample.*** The value obtained is Weight of sample, which you will enter when starting the experiment.

Assembling the Sample Holder

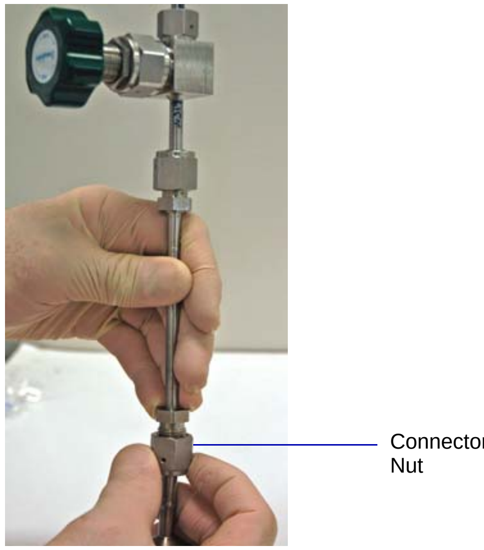
1. Assemble the sample holder by placing the gasket on the sample cylinder as shown below.



Two types of gaskets are available. One is filtered, the other one is not. It is best to use a filtered gasket for fine powders. The non-filtered gasket is sufficient for other types of materials.

Note that the gasket can be used only once. Reusing the gasket may cause leaks at the sample holder connection.

2. Place the upper portion of the sample holder on the cylinder and hand-tighten the connector nut.



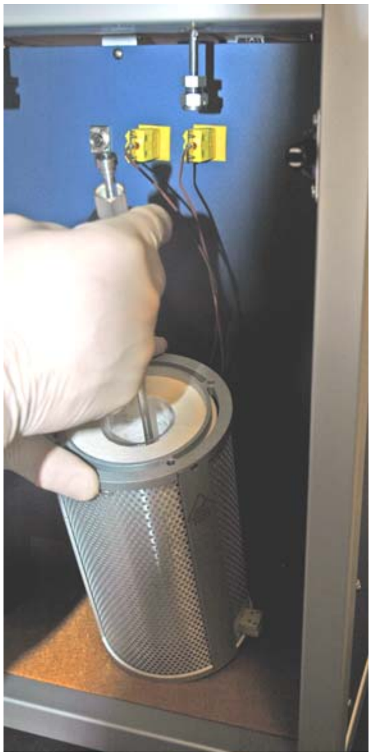
3. Use the two wrenches (5/8-in and 3/4-in) to tighten the connector an additional 1/8 turn.

Degassing the Sample

1. Insert the furnace thermocouple cables into the thermocouple connectors inside the sample compartment.

2. Plug the furnace power cord into the power connector inside the sample compartment.

3. Tilt the furnace, then insert the sample holder assembly into the furnace.



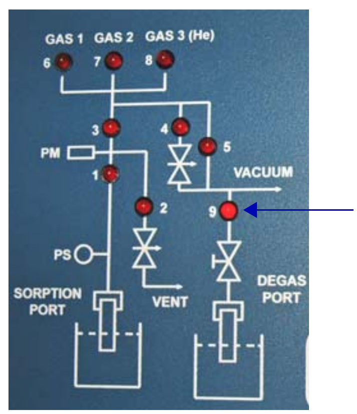
4. Connect the sample holder to the degas port by first ensuring that the O-ring is in place in the degas port.

5. Attach the sample holder to the degas port and hand-tighten the connector nut.

6. Using two wrenches (3/4-in and 5/8-in) tighten the nut just until snug.

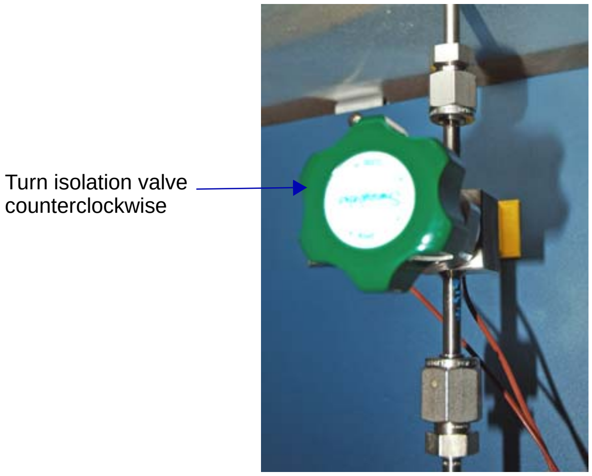
Do not over tighten the connector nut, doing so could damage the port fitting.

7. Begin the sample evacuation by opening valve 9 as shown. Note that the valve 9 indicator on the front panel must turn on.



Valve 9
Indicator

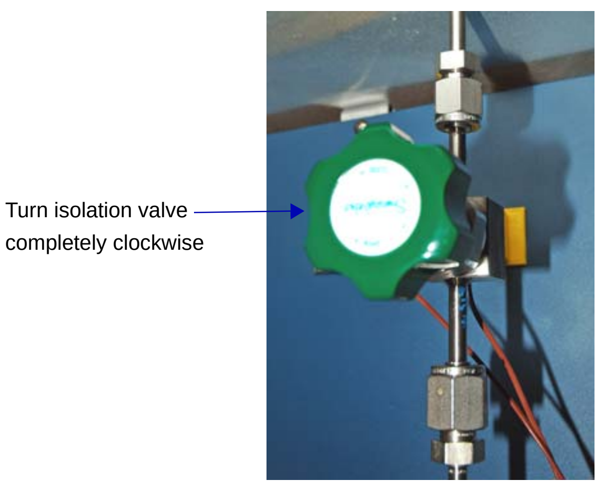
8. Slowly open the isolation valve on the sample holder by turning it completely counterclockwise.



9. Monitor the external vacuum gauge to ensure that vacuum is reached.

10. Set the temperature to the desired level by holding the left **FURNACE** button and the **up** or **down** button as needed. Allow the sample to degas as needed for the material to be analyzed.

11. To end the degas procedure, close the isolation valve by turning it completely clockwise.



12. Close valve 9. The valve 9 indicator on the front panel will turn off.

13. Set the furnace to 0 °C and allow the sample to cool to room temperature.

14. Remove the sample holder from the degas port after the sample has cooled to room temperature by using two wrenches (3/4-in and 5/8-in) to loosen the connector nut.

15. Remove the sample holder from the degas port.

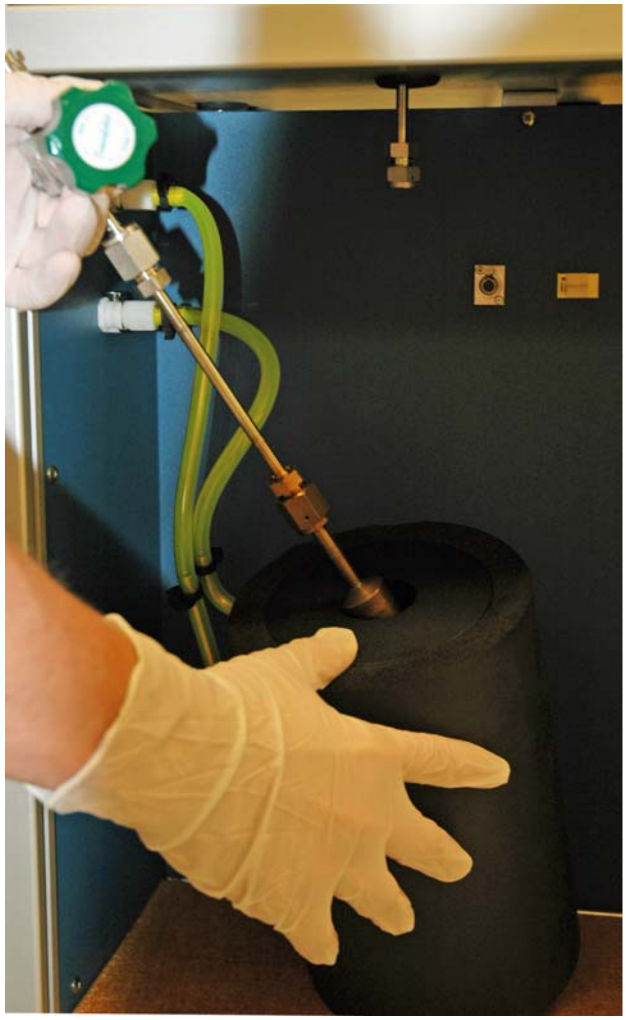
16. Ensure that the O-ring remains in place in the degas port, and then place a cap on the port.

17. Tilt the furnace, and then remove the sample holder assembly from the furnace.

Attaching the Sample Holder to the Analysis Port

1. Connect the temperature control vessel by attaching the **supply** and **return** hoses from the temperature control vessel to the ports inside the sample compartment. Make sure the hoses are connected to the appropriate input and output ports from the circulating bath.

2. Place the sample holder in the temperature control vessel by tilting the temperature control vessel and placing the sample holder in the vessel.



3. Connect the sample holder to the analysis port by first ensuring that the O-ring is in place in the port.

4. Attach the sample holder to the analysis port and hand-tighten the connector nut.

5. Using two wrenches (3/4-in and 5/8-in) tighten the nut just until snug.

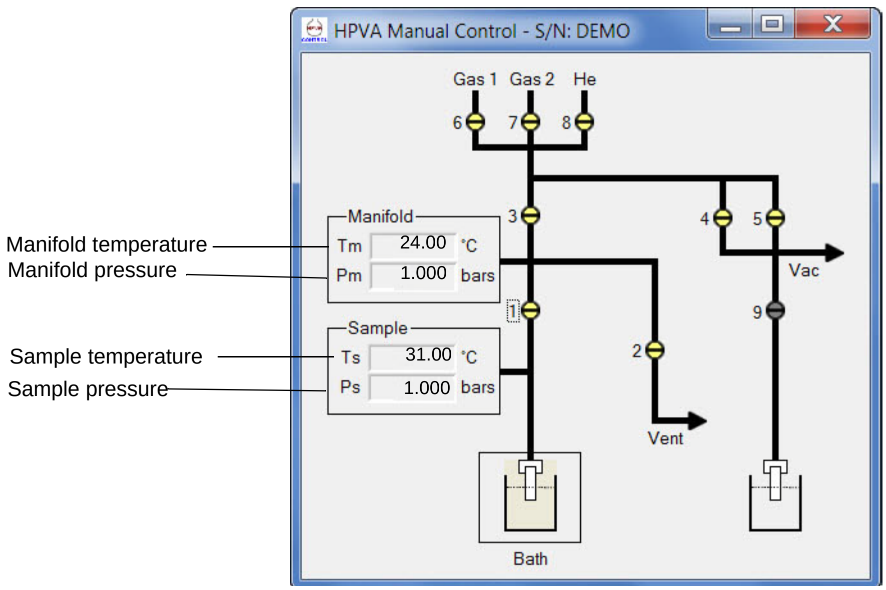
Do not over tighten the connector nut, doing so could damage the port fitting.

Purging the System

When the HPVA is performing an experiment or being operated in manual control mode, the valve switches on the front panel should be placed and remain in the closed (down) position.

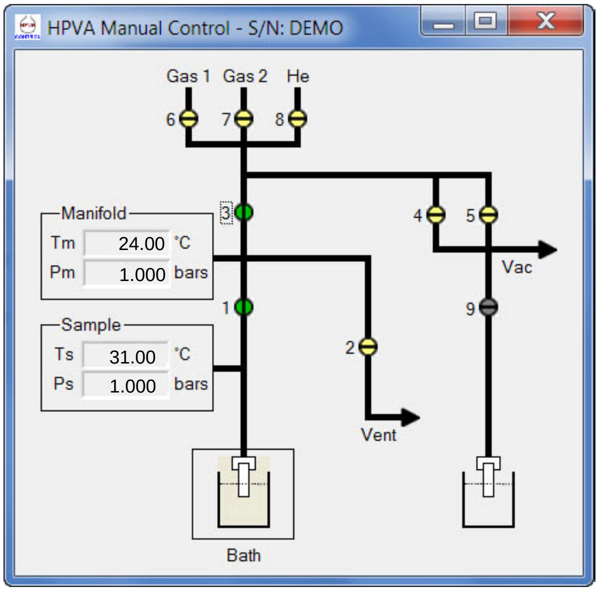
If a switch is placed in the opened position during a software operation, it will override the software and may damage the instrument.

1. Click **Manual Control** from the Main Menu to display the Manual Control screen.



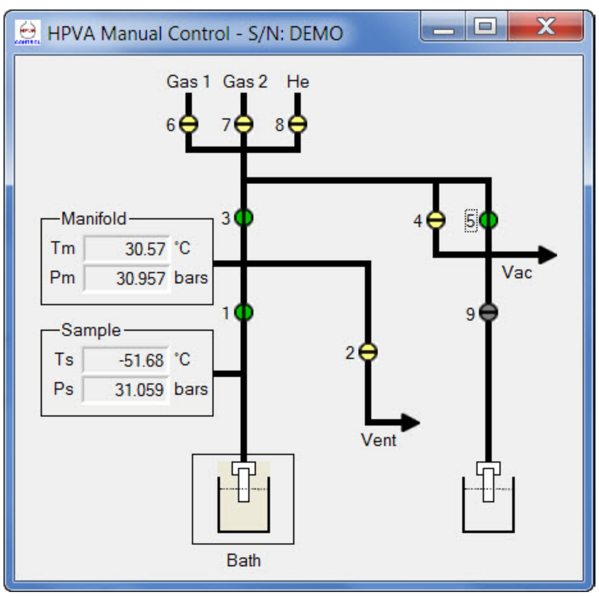
You can open and close a valve by double-clicking the left mouse button on the valve icon or clicking the valve icon then pressing the space bar. Yellow indicates a closed valve and green indicates an open valve.

2. Open valves 1 and 3 as shown below. Check the **Manifold** pressure displayed on the screen. There should be no pressure on the manifold (reading should be 1 bar or less).



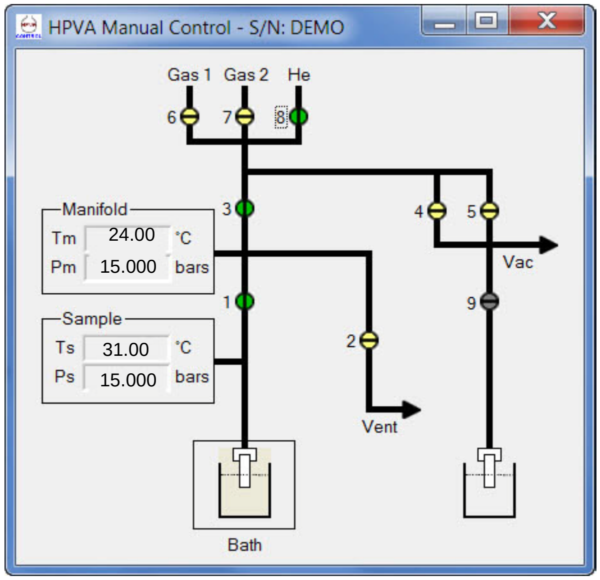
3. Open valve 4 until the pressure reaches about 0.5 bar, then close valve 4.

4. Open valve 5. Leave the valves open until the external Vacuum Gauge reads less than 5 microns.



5. Close valve 5.

6. Open valve 8 and pressurize the instrument until the **Sample** pressure displayed on the screen reads 15 bar.



7. Close valve 8.

8. Open valve 2.

9. Adjust the **Vent** control knob on the front panel of the instrument to allow pressure to vent.

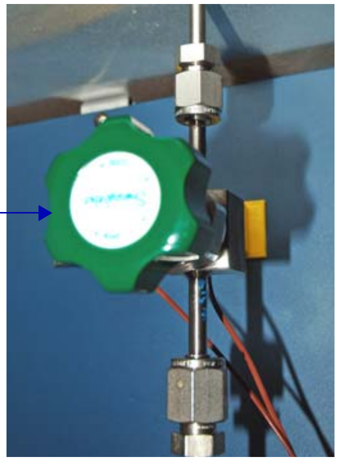
10. When the pressure reaches 1 bar or less, close valve 2

11. Repeat steps 2 through 9 three times, then proceed to step 11.

12. Open valve 4 until the pressure reaches about 0.5 bar, then close valve 4.

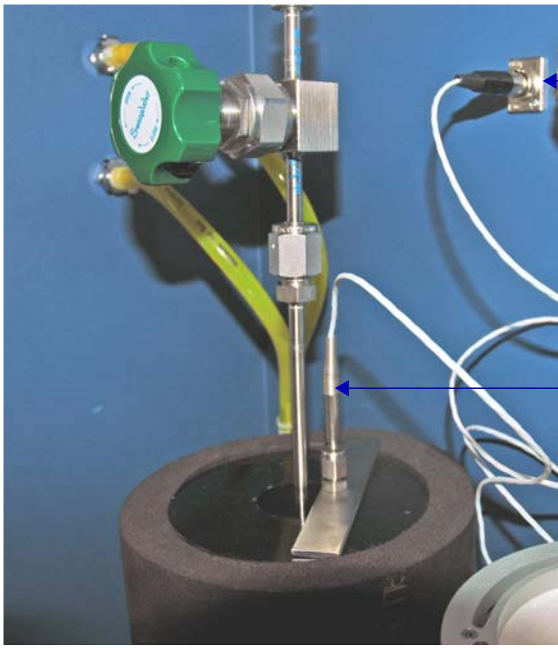
13. Perform the unrestricted evacuation by opening valve 5.

14. When the system reaches full vacuum, open the isolation valve on the sample holder by turning it completely counterclockwise.



15. Plug the temperature probe cable into the connector on the instrument.

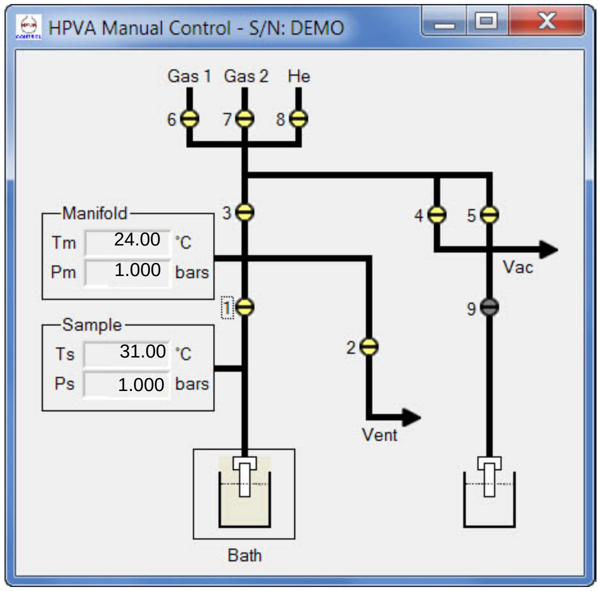
16. Insert the temperature probe into the temperature control vessel.



17. Add the appropriate liquid to the temperature control vessel until the level reaches about ½ inches from the top.

18. Double-click the Bath icon and set the circulating bath controller to the desired temperature.

19. Close all valves.



20. You are now ready to run the experiment.

Running the Experiment

The Adsorption option on the Main Menu is used to define and perform experiments. This function involves two windows:

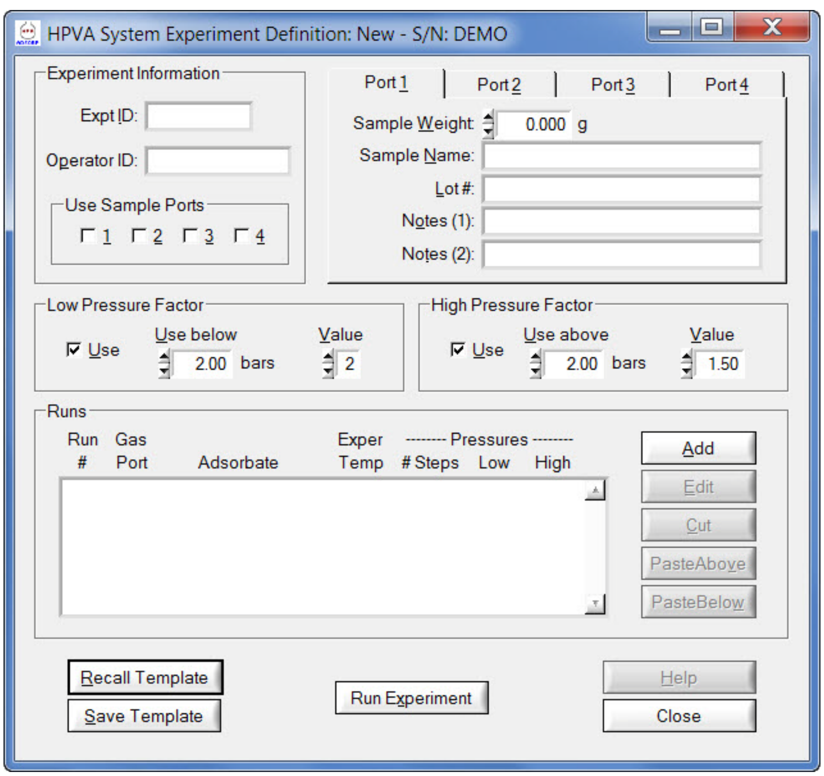
- The System Experiment Definition window allows you to enter a sample ID and sample information.
- The System Step Isotherm Define Run window allows you to specify the experiment parameters.

The HPVA software allows you to perform multiple experiments, or runs, in sequential order for one sample. For example, if you have a sample you wish to analyze at different temperatures, you can add multiple runs with the desired temperatures.You can edit the order of the runs using the buttons to the right of the run list.

The software also enables you to save experiment parameters in a *Template*. Once a template is created, you can use it for other experiments by just loading it into the System Definition window and entering a unique Experiment ID. You can modify the template as needed and save the changes as another template if desired.

The following is a general procedure for entering experiment parameters, starting the experiment, and viewing the isotherm. Refer to "Definitions of Fields in the System Windows" on page 2-27 for details about the fields on this window.

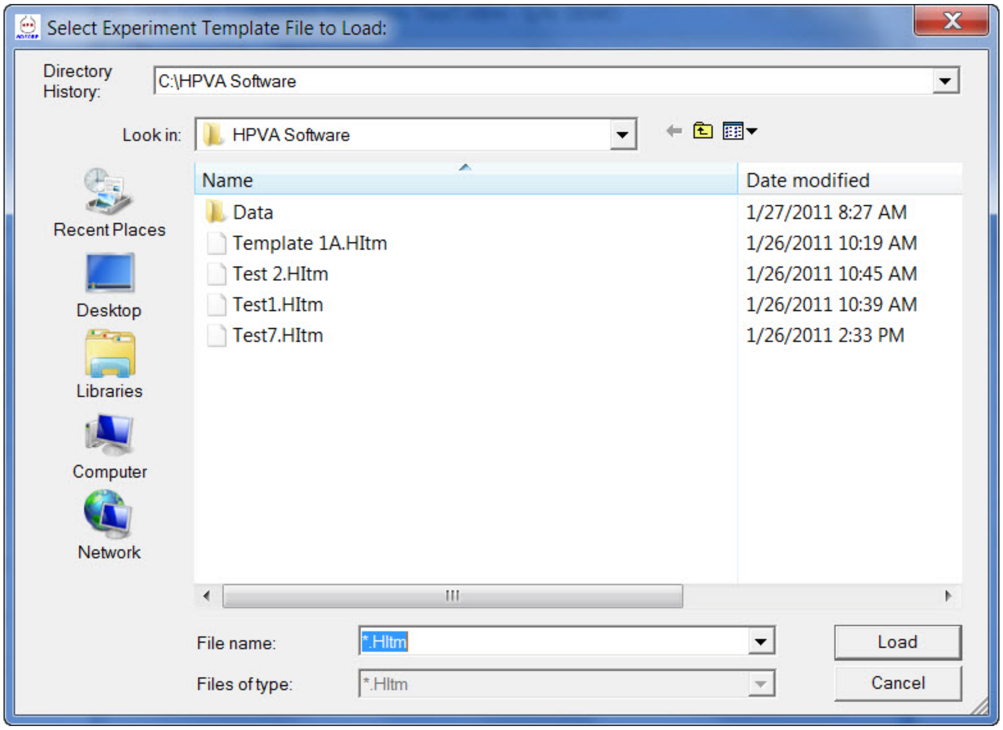
1. Click **Adsorption** on the Main Menu to display the System Experiment Definition window.



2. Choose one of the following:

   - To enter a new set of experiment parameters, proceed to step 3.
   - To use an existing set of experiment parameters:

   a. Click **Recall Template** to display the Select Experiment Template File to Load window.



   b. Select the template name, then click **Load.**

   c. Enter an experiment ID in the **Expt ID** field.

   c. Make any desired changes.

   d. Proceed to Step 10.

3. Enter an ID for the experiment.

4. Enter an Operator ID for the experiment.

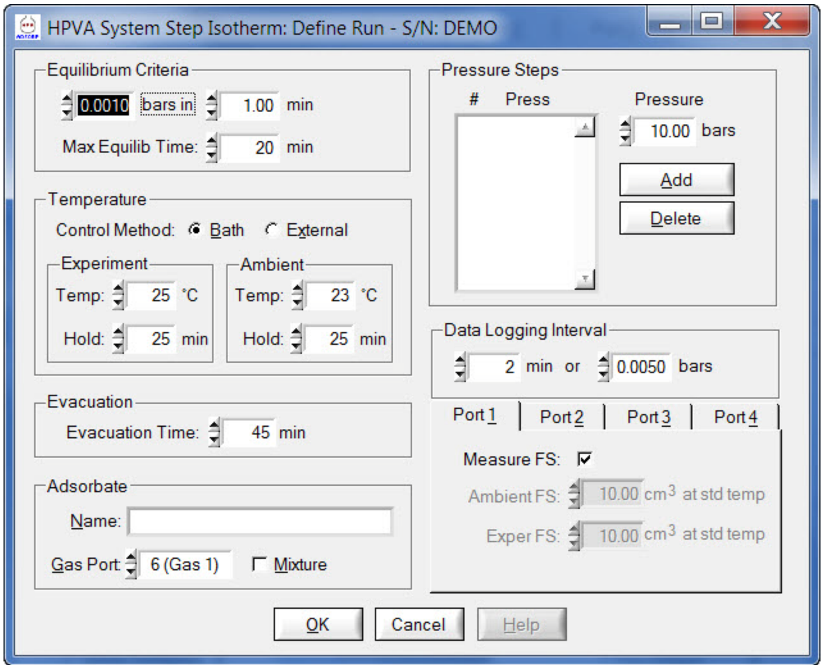
5. Under **Use Sample Ports**, click 1. The window shows ports 1 through 4; however, the models covered in this manual, the HPVA-100 and HPVA-200, have only one sample port.

6. Enter the weight of the sample that was calculated when preparing the sample.

7. Enter the sample name and, optionally, the lot number and notes.

8. Enter Low and High Pressure Factors if desired.

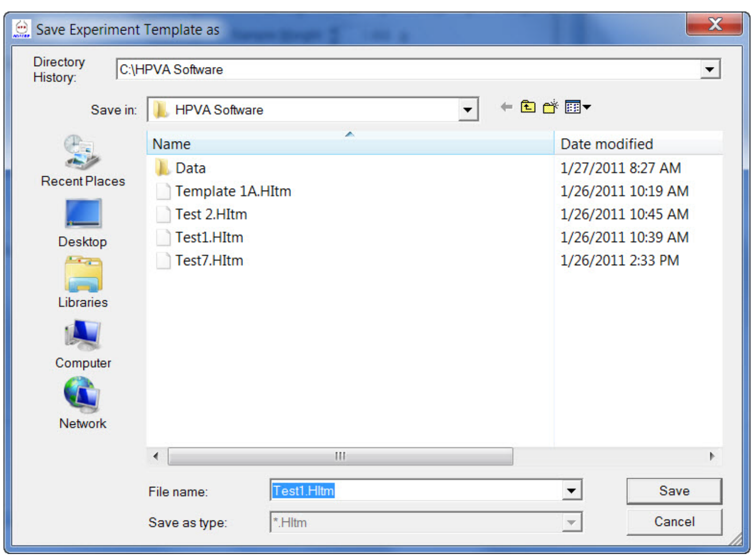
9. Click **Add** to add run parameters to the experiment. The HPVA System Step Isotherm: Define Run window is displayed.



10. Add the parameters for the run, then click **OK**. Refer to Define Run Window, page 2-29 for a description of the fields on this screen.

11. Repeat steps 9 and 10 for each run you wish to add to the experiment.

12. When you have finished entering information, click **Run Experiment**. The HPVA Step Isotherm window is displayed and you are prompted to save the template.



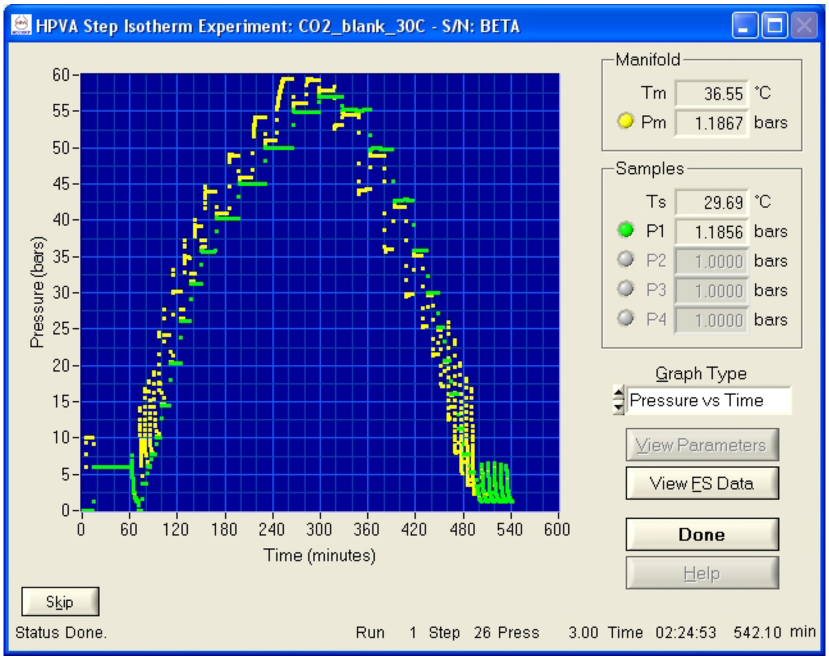
13. Enter a name for the template if you would like to save it, then click **Save**.

14. You can view the isotherm as the experiment progresses as described in the next topic.

Viewing the Experiment

Displaying the Isotherm

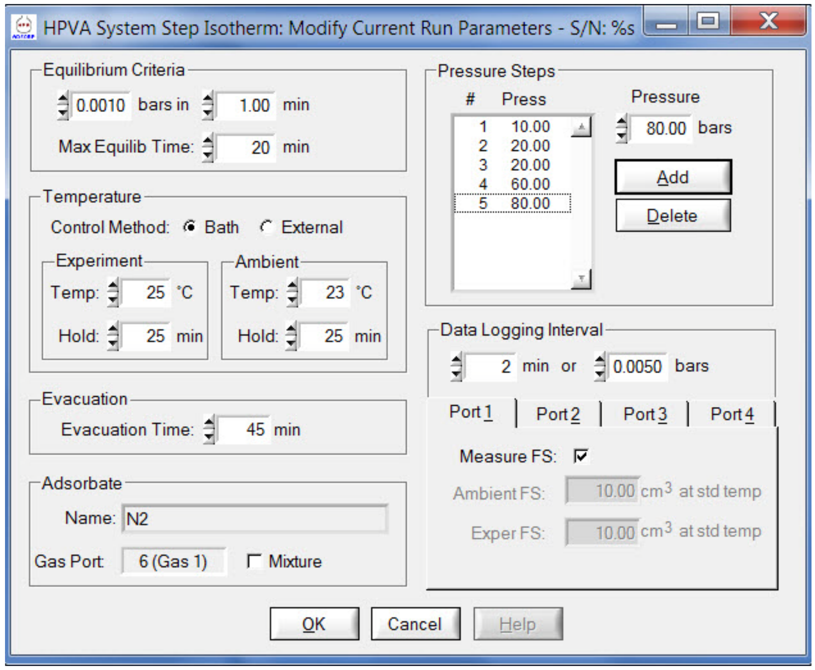
During an experiment, a screen similar to the example below is displayed. If you would like to zoom into an area of the graph, hold the **Ctrl** key and drag, selecting an area with the left mouse button. To return to the full graph, double-click in the graph.



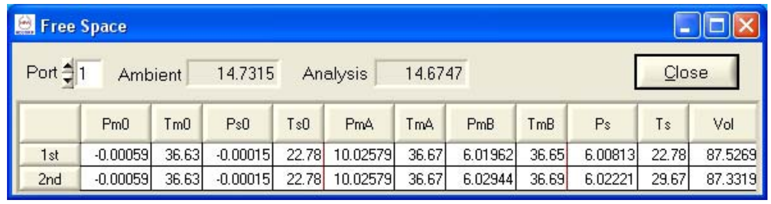
Manifold	The current Manifold temperature and pressure are displayed.
Samples	The current Sample temperature and pressure are displayed.
Graph Type	Select the type of graph you wish to display. The choices are: • Temp vs Time • Volume vs Pressure • Pressure vs Time

View Parameters

This command displays the Modify Current Run Parameters window, which enables you to change parameters for any step that has not completed.



View FS Data This command enables you to view free space data.



Ambient: the free space at ambient temperature is shown.

Analysis: the free space at the analysis temperature is shown.

The 1st row shows values at ambient temperature.

The 2nd row shows values at analysis temperature.

The columns are described below:

Pm0: Initial manifold pressure

Tm0: Initial manifold temperature

Ps0: Initial sample pressure

Ts0: Initial sample temperature

PmA: Manifold pressure before dosing

TmA: Manifold temperature before dosing

PmB: Manifold pressure after dosing

TmA: Manifold temperature after dosing

Ps: Sample pressure after dosing

Ts: Sample temperature after dosing

Vol: quantity dosed into sample

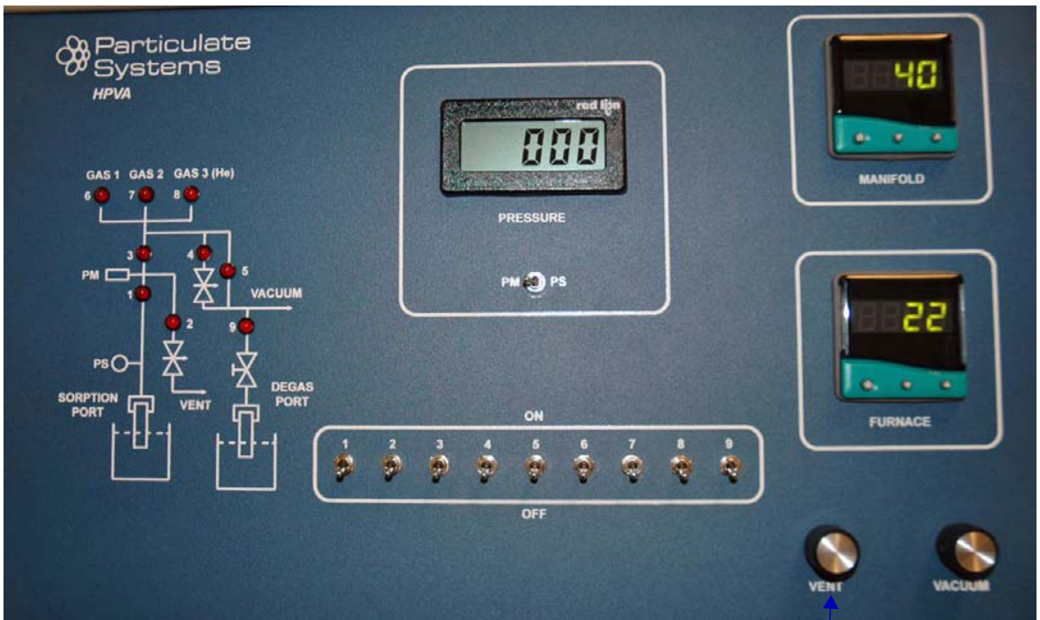
Done This command closes the Experiment window.

Help This command displays the HPVA Operator’s Manual.

Adjusting Flow Valves

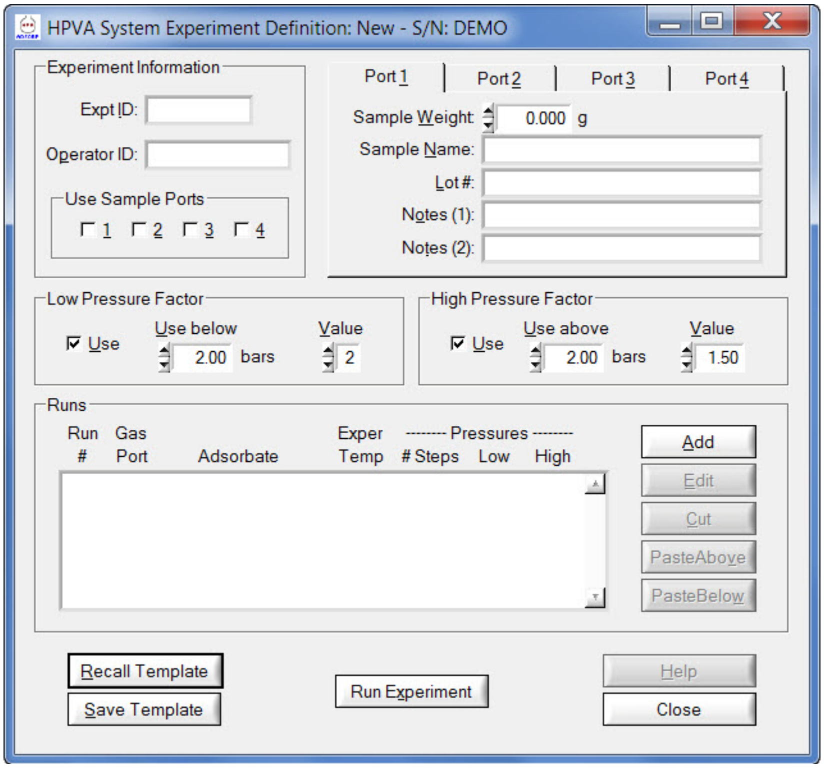
The Vent valve control on the instrument's front panel adjusts the flow rate through the vent valve. If the flow rate is high, you may not be able to achieve accurate dosing pressures. If the flow rate is low, the length of the experiment will be increased. Close the valve slightly if the instrument is having difficulty achieving dosing pressures within the specified tolerance. Open the valve slightly to speed up the experiment.

The vent valve rate should be adjusted prior to the experiment to achieve a range between 5 to 15 seconds per 1 bar decrease by monitoring the pressure on the manual control screen while adjusting the Vent valve control.



Definitions of Fields in the System Windows

Define New Experiment Window



Exp ID

Enter the experiment identifier. The data files created will use this name.

Operator ID

Enter the name or initials of the operator.

Use Sample Ports

Check port 1. The window shows ports 1 through 4; however, the models covered in this manual, the HPVA-100 and HPVA-200, have only one sample port.

Sample Weight

Enter the sample weight after degas.

Sample Name

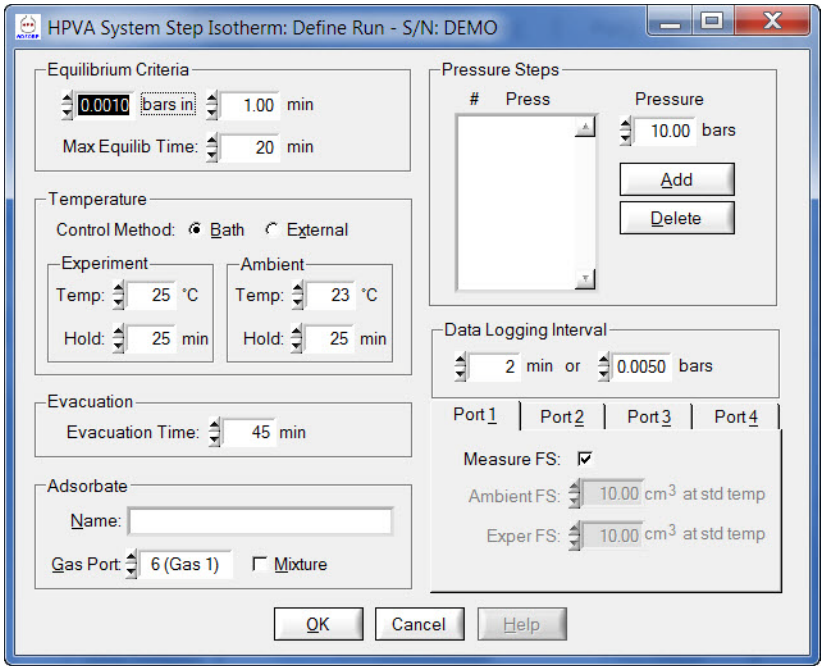
Enter the name you wish to assign to the sample.

Lot #

If the sample has a designated identification number, enter it here.

Notes (1) and (2)	Enter any additional notes about the sample here, for example, the degas time.
Low Pressure Factor	If you check this option, the system will not dose the manifold to a pressure exceeding the Value times the desired sample pressure in the pressure table if it is below the entered pressure. This is typically used below 2 bar with a factor of 2. If you select this option, use the arrow keys to select the pressure in the **Use below** field and the value in the **Value** field.
High Pressure Factor	If you check this option, the system will not dose the manifold to a pressure exceeding the Value times the desired sample pressure in the pressure table if it is above the entered pressure. This is typically used above 2 bar with a factor of 1.5 If you select this option, use the arrow keys to select the pressure in the **Use below** field and the value in the **Value** field
Recall Template	Click this button to load a saved template.
Save Template	Click this button to save the current experiment definition for future experiments.
Add	Click this button to add a run to the experiment. Refer to "Define Run Window" on page 2-29 for field descriptions.
Edit	Click this button to edit the selected run. Refer to "Define Run Window" on page 2-29 for field descriptions.
Cut **Paste Above** **Paste Below**	Use these button to rearrange the order of runs during the experiment.
Run Experiment	Click this button to start the experiment.
Close	Click this button to close the System Experiment Definition window and return to the Main Menu.

Define Run Window



Equilibration Criteria

Specifies when the system has reached equilibrium after each step in the adsorption/desorption procedure.

Enter the pressure to be reached and the number of minutes in which it should be reached.

Enter the maximum equilibration time in minutes in the **Max Equilib Time** field.

Control Method

Select the temperature control method to be used during the experiment.

Click **Bath** if a recirculation bath or cryostat is to be used.

Click **External** if the furnace or a dewar is to be used.

Experiment

Temp: Enter the temperature for the experiment in °C.

Hold: Enter the number of minutes the system will wait once the sample RTD reaches the experiment temperature. A hold time of at least 60 minutes is recommended if a liquid nitrogen bath is used.

Ambient	**Temp:** Enter the temperature of the air in the laboratory in °C. **Hold:** Enter the number of minutes the system will wait once the sample RTD reaches the ambient temperature.
Evacuation Time	Enter the number of minutes you wish to evacuate the sample prior to running the free space routine.
Adsorbate Name	Enter the name of the gas to be used for the experiment. It should match the name under the **Adsorbate** column on the **Gases** tab of the Excel macro spreadsheet.
Gas Port	Select the port (either 6 or 7) to which the adsorbate gas tank is attached to the instrument. If the adsorbate is a gas mixture, check **Mixture**.
Pressure Steps	This table lists the pressures the sample holder will reach during the experiment when the standard dosing mode is used. To enter a pressure step, enter the pressure in bars in the **Pressure** field, then click **Add**. If you wish to delete a pressure step, click the step in the table, then click **Delete**.
Data Logging Interval	This field enables you to specify how often data points are collected either by time or by pressure. Enter a number of minutes in the **min** field or a pressure in the **bars** field. This is typically set to every 2 minutes or whenever the system pressure changes by 0.005 bars.
Port 1 through Port 4	Click **Port 1**.
Measure FS	Check this box if you wish to have a free space analysis conducted prior to the isotherm experiment.
Ambient FS	If the Measure FS box is not checked, enter the ambient free space volume of the sample tube in this field.

Exper FS	If the Measure FS box is not checked, enter the experiment free space volume of the sample tube in this field.
OK	Click **OK** to save the run parameters and return to the System Experiment Definition window.
Cancel	Click **Cancel** to discard your entries and return to the System Experiment Definition window.

3. Viewing Experiment Results

Experiment data are viewed in the Microsoft® Excel Macro that was installed during installation of the HPVA. The macro preforms the following:

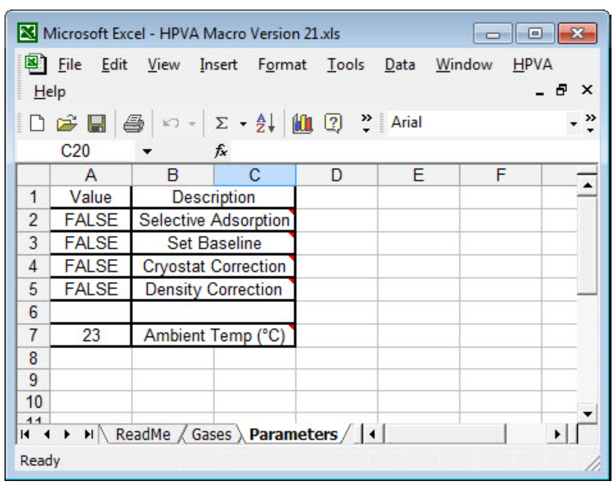
- Calculates free space data for both the ambient volume and the analysis temperature volume.
- Calculates the volume of gas adsorbed during an experiment.
- Displays data isotherms.
- Displays the raw data collected by the HPVA program during an experiment.
- Enables you to copy and paste data into other programs such as Microsoft Word or to save data in a variety of formats using Excel features.

Running the Macro

In order to use the Macro, you must select these features in Excel:

- When prompted by Excel, select **Enable Macros.**
- Make sure the **Macro Security (**Excel Options window) is set to **Medium** or lower.

When the Macro is opened, the following window is displayed.



The window contains three tabs:

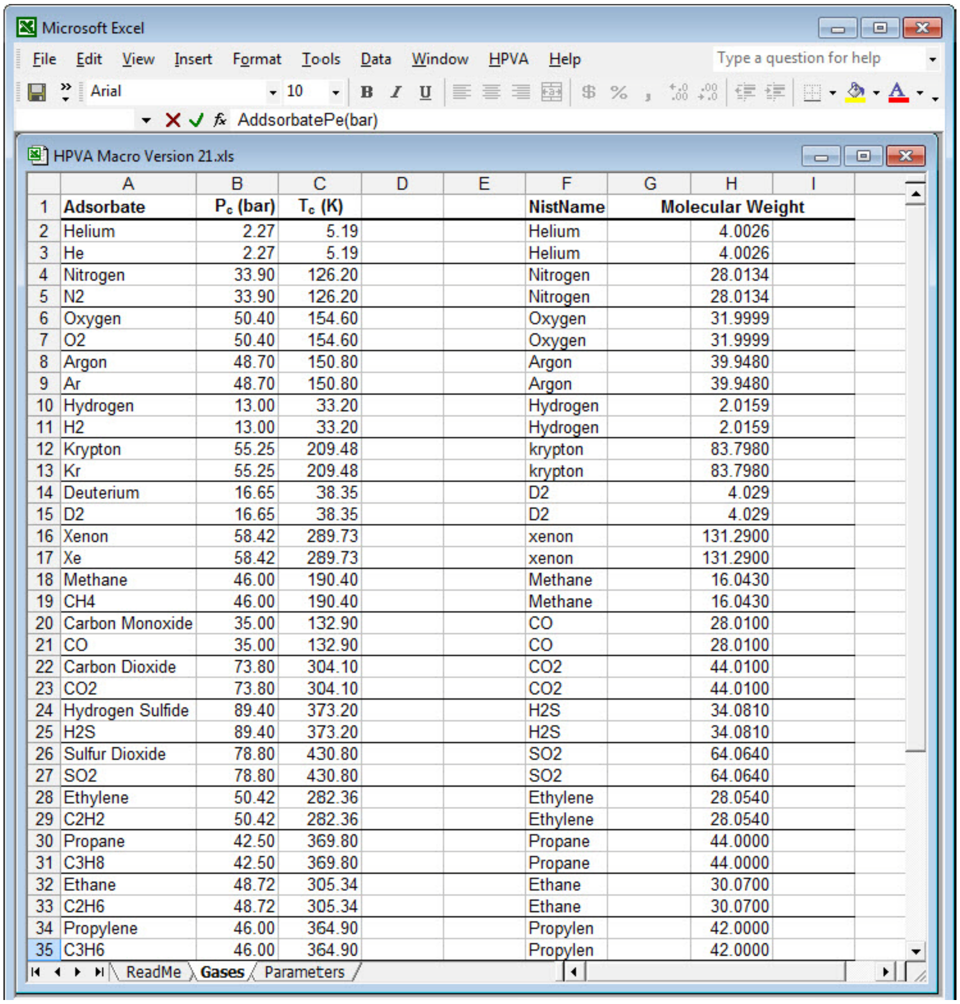
- ReadMe
- Gases
- Parameters

Read Me Tab

The ReadMe tab displays information about the HPVA Macro.

Gases Tab

The Gases Tab displays the gas properties used in calculations.



	A	B	C	F	H
1	Adsorbate	P_c (bar)	T_c (K)	NistName	Molecular Weight
2	Helium	2.27	5.19	Helium	4.0026
3	He	2.27	5.19	Helium	4.0026
4	Nitrogen	33.90	126.20	Nitrogen	28.0134
5	N2	33.90	126.20	Nitrogen	28.0134
6	Oxygen	50.40	154.60	Oxygen	31.9999
7	O2	50.40	154.60	Oxygen	31.9999
8	Argon	48.70	150.80	Argon	39.9480
9	Ar	48.70	150.80	Argon	39.9480
10	Hydrogen	13.00	33.20	Hydrogen	2.0159
11	H2	13.00	33.20	Hydrogen	2.0159
12	Krypton	55.25	209.48	krypton	83.7980
13	Kr	55.25	209.48	krypton	83.7980
14	Deuterium	16.65	38.35	D2	4.029
15	D2	16.65	38.35	D2	4.029
16	Xenon	58.42	289.73	xenon	131.2900
17	Xe	58.42	289.73	xenon	131.2900
18	Methane	46.00	190.40	Methane	16.0430
19	CH4	46.00	190.40	Methane	16.0430
20	Carbon Monoxide	35.00	132.90	CO	28.0100
21	CO	35.00	132.90	CO	28.0100
22	Carbon Dioxide	73.80	304.10	CO2	44.0100
23	CO2	73.80	304.10	CO2	44.0100
24	Hydrogen Sulfide	89.40	373.20	H2S	34.0810
25	H2S	89.40	373.20	H2S	34.0810
26	Sulfur Dioxide	78.80	430.80	SO2	64.0640
27	SO2	78.80	430.80	SO2	64.0640
28	Ethylene	50.42	282.36	Ethylene	28.0540
29	C2H2	50.42	282.36	Ethylene	28.0540
30	Propane	42.50	369.80	Propane	44.0000
31	C3H8	42.50	369.80	Propane	44.0000
32	Ethane	48.72	305.34	Ethane	30.0700
33	C2H6	48.72	305.34	Ethane	30.0700
34	Propylene	46.00	364.90	Propylen	42.0000
35	C3H6	46.00	364.90	Propylen	42.0000

The columns contained on this sheet are:

Adsorbate The name of the adsorbate is listed on one line and its symbol on the next line. Both the name and symbol are listed so the macro will recognize the adsorbate, whether its name or its symbol was entered in the **Define Run** window prior to the experiment.

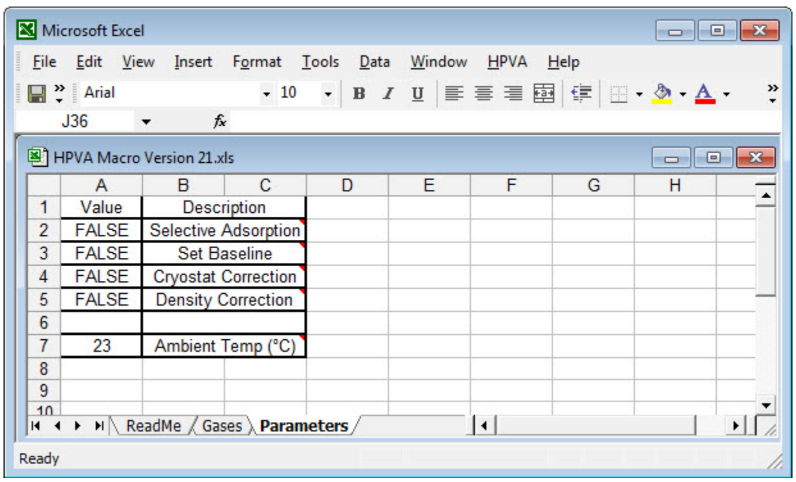
P_c(bar) The critical pressure expressed in bar.

T_c(K)	The critical temperature expressed in Kelvin.
NistName	The National Institute of Standards name of the adsorbate. This is the name the REFPRO software recognizes when called for compressibility data.
Molecular Weight	The molecular weight of the adsorbate.

You may add gases to this list if required.

Parameters Tab

The Parameters tab displays the parameters used in calculations.



The columns contained on this sheet are:

Value	The current value set for the parameter.
Description	A description of the parameter.

You can adjust the parameters if required. The settings are described in the following table.

Parameter	Settings
Selective Adsorption	Set this option to TRUE if the analysis was conducted using the Selective Adsorption routine. Otherwise, set it to FALSE. When set to TRUE, this option accounts for the evacuation of the sample cell after each adsorption/desorption point during the selective adsorption routine when performing calculations.

<table>
<tr><th>Parameter</th><th>Settings</th></tr>
<tr><td>Set Baseline</td><td>Set this option to TRUE if a blank tube analysis was performed. Otherwise, set it to FALSE.

When set to TRUE, this option corrects errors in an analysis by using data collected from a blank tube analysis.

When set to TRUE, when you select Read Data File from the HPVA menu, you will be prompted to:

• Select the analysis sample file
• Enter the dry mass of the sample
• Select the blank tube analysis sample file

To perform the correction, the slopes and intercepts of the lines of best fit for the adsorption and desorption curves are calculated. The system corrects the sample analysis file by subtracting out the error found with the slope and intercept at each pressure point in the isotherm using a volumetric basis.

This correction should only be used if the blank sample tube analysis was performed at the same temperature, in the same pressure range, and with the same sample tube and analysis gas as the analysis performed on the sample.</td></tr>
<tr><td>Cryostat Correction</td><td>Note: This setting applies to a ColdEdge cryostat. It has not been verified with other brands.

Set this option to TRUE only if a cryostat was used for the analysis. Otherwise, set the option to FALSE.

When a cryostat is in use with the instrument, the instrument reads the sample temperature from the control box of the cryostat. The control box reads the temperature from a temperature probe within the sample well of the cryostat. The reading of this temperature probe is approximately 1°C less than the true temperature within the sample cell. This correction adds 1°C to all sample temperature readings prior to all calculations.</td></tr>
</table>

Parameter	Settings
Density Correction	If you entered free space data in the HPVA Define Run window from an empty sample tube prior to the analysis, set this option to TRUE. The program will prompt you for the density of the sample. The program will use the density and mass of the sample to correct the free space volume for the physical volume of the sample. Set this option to FALSE if the free space data was acquired with the sample in the tube.
Ambient Temp.	Enter the approximate temperature of the environment surrounding the instrument. Since the instrument is unable to read the ambient temperature when the Sample RTD is in the bath or dewar, this value must be approximated. Typical air conditioned labs should have this parameter set to 22 or 23 °C.

Compile Error Message

Occasionally the following Excel error message may appear when you are trying to open a file: "Compile Error: Can't find project or library".

If this occurs, perform the following steps to clear the error.

1. Open the Excel macro workbook
2. Press Alt + F11 to open the Visual Basic Editor.
3. On the Tools menu, click **References**. The References dialog is displayed.
4. Clear the check box for the type library or object library marked as "Missing" - this should be SOLVER.XLS

Displaying Experiment Data

Experiment data can be displayed in one of two formats:

- Long Data File
- Short Data File

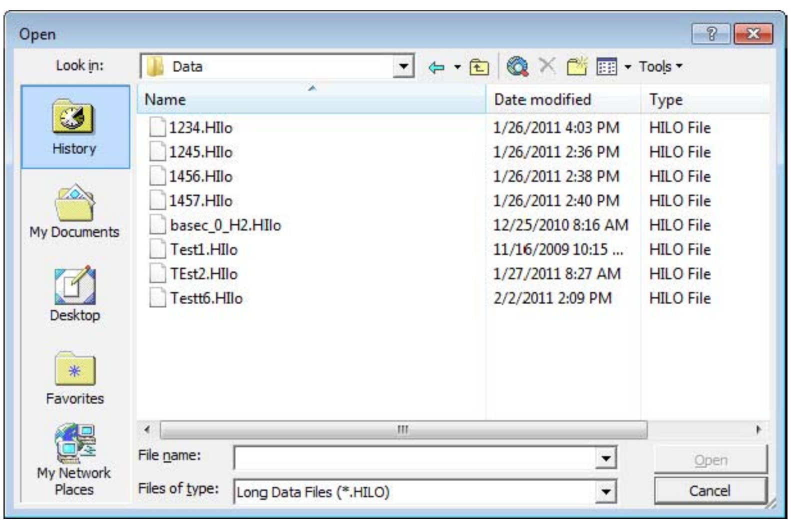
When you select **Read Long Data File** from the HPVA menu, a spreadsheet containing all the raw data from the experiment is displayed.

When you select **Read Short Data File**, the following are displayed:

- Volume Adsorbed isotherm
- PCT
- Information about the experiment
- Calculations for volume dosed
- Calculations for volume adsorbed

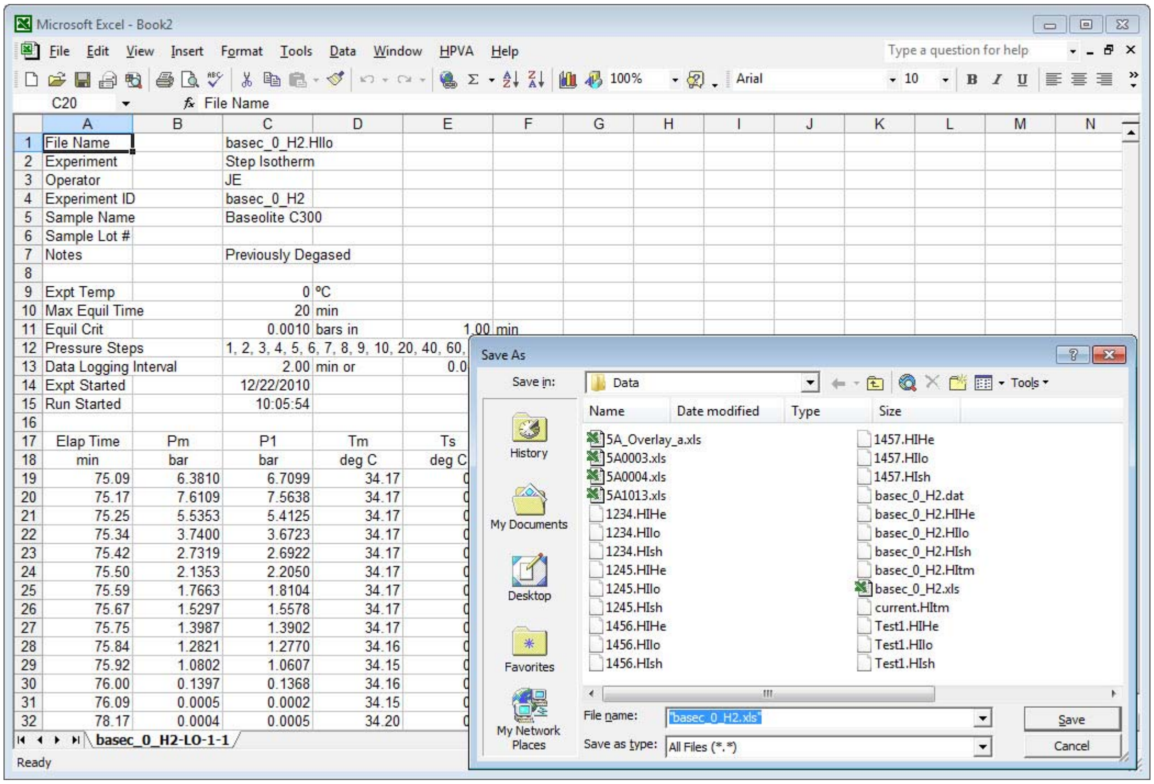
Read Long Data File

1. Select **Read Long Data File** from the HPVA menu in the Excel Main Menu bar. The Open dialog is displayed.



2. Select the desired file (long data files have an extension of HIlo), then click **Open**.

3. A sheet similar to the one shown below is displayed.



4. Information about the sample is displayed in the first several rows. Following the experiment information is a table of the data collected.

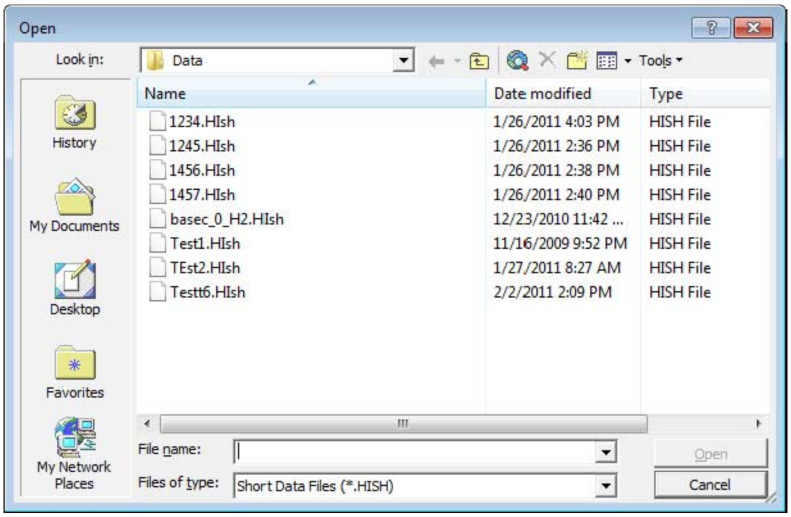
Column Heading	Description
Elap Time min	Elapsed time in the experiment
Pm bar	Manifold pressure
P1 bar	Sample pressure
Tm deg C	Manifold temperature
Ts deg C	Sample temperature

5. The Save As dialog is also displayed. If you would like to save the data in an Excel spreadsheet, enter a file name or accept the default name, then click **Save**.

Read Short Data File

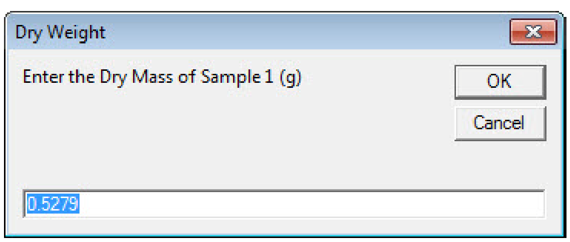
The Short Data file displays two graphs, as well as information about the experiment and calculations. When the graphs are displayed, you can use Excel charting functions to change curve colors, add symbols, modify the legend, etc. You can also use Excel functions such as Cut, Copy, and Save As to transfer data to other programs. Refer to your Excel documentation for more information.

1. Select **Read Short Data File** from the HPVA menu in the Excel Main Menu bar. The Open dialog is displayed.



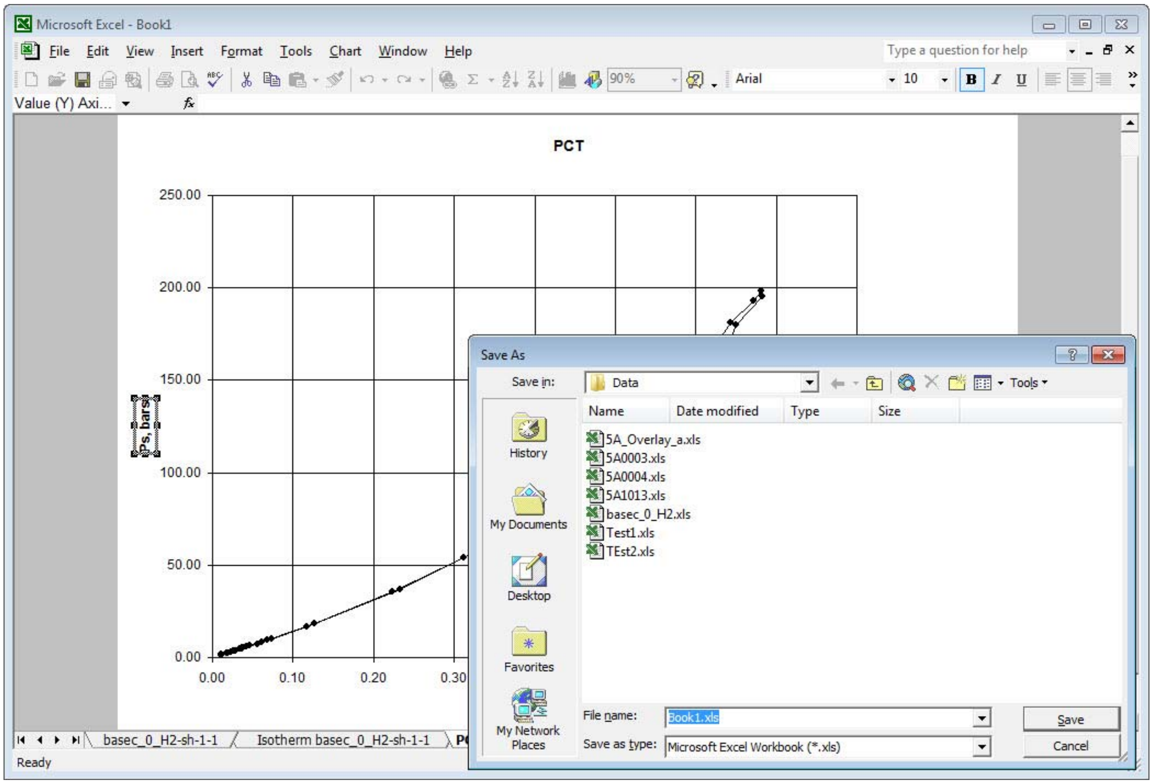
2. Select the desired file (short data files have an extension of HIsh), then click **Open**.

3. If a blank tube analysis was performed, the Dry Weight dialog is displayed.



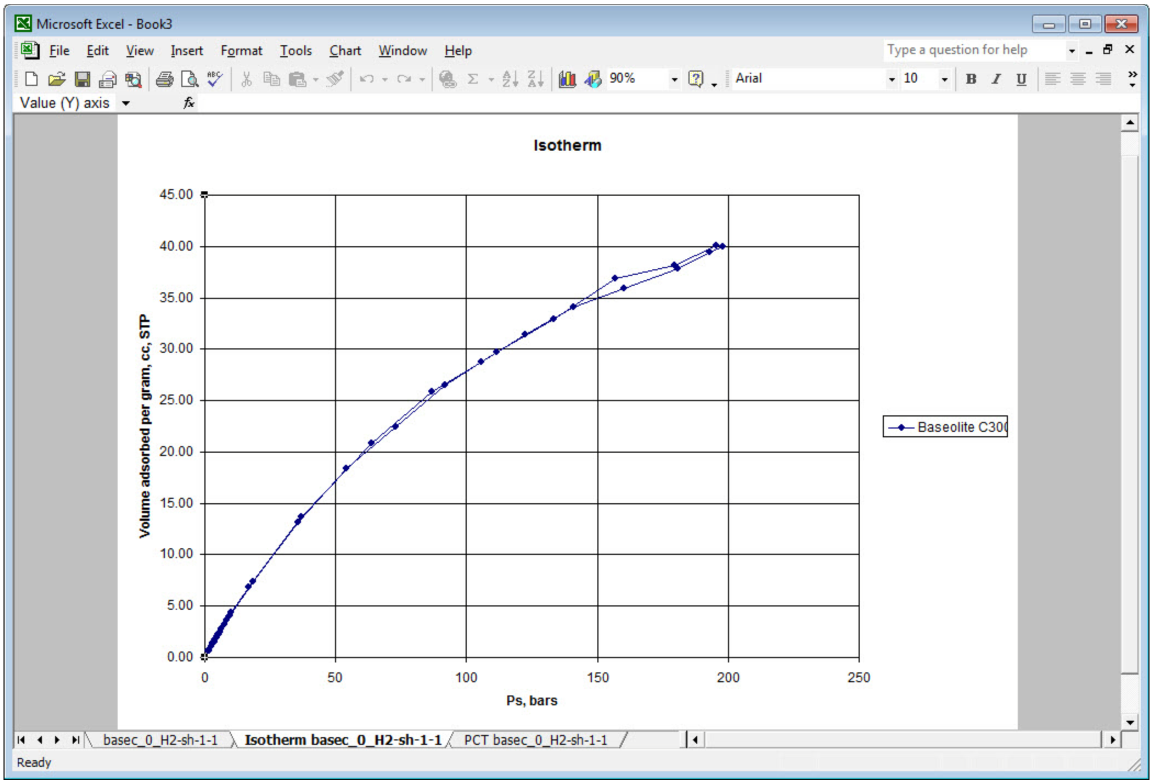
Enter the mass of the sample after analysis in grams, then click **OK.**

4. A spreadsheet similar to the following is displayed.

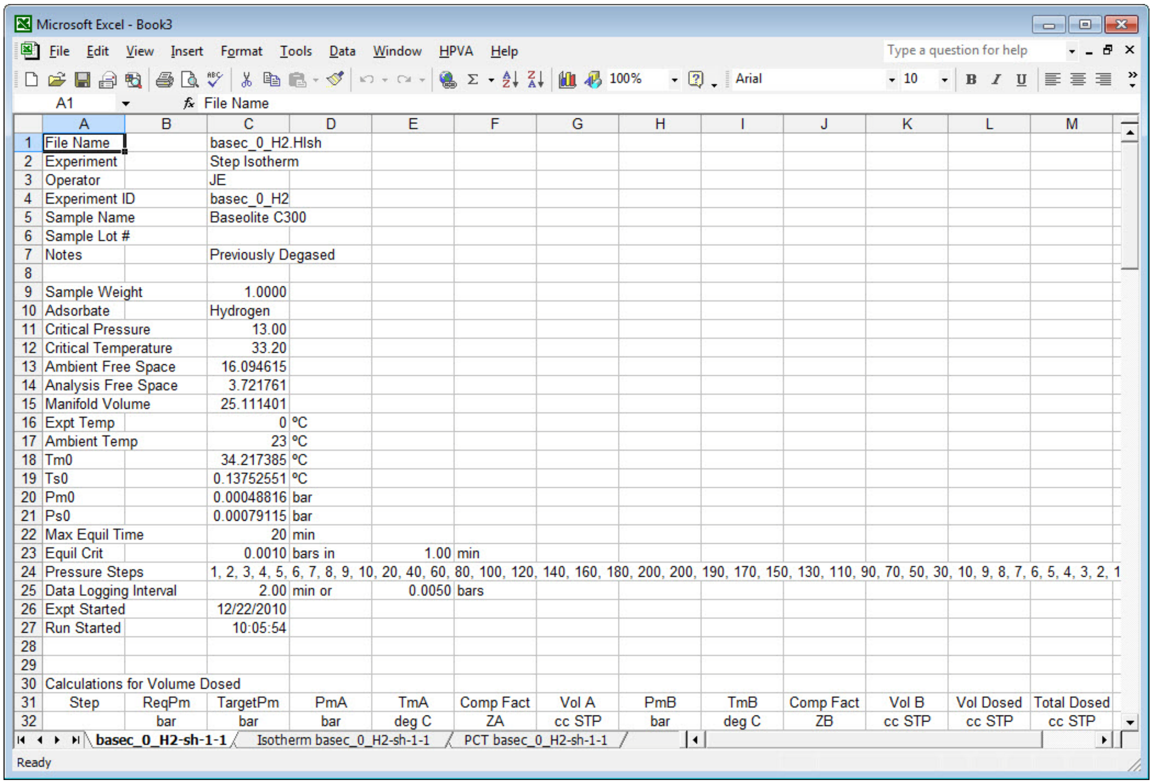


A PCT graph of the experiment data is displayed and the Save As dialog is displayed. If you would like to save the data in an Excel spreadsheet, enter a file name or accept the default name, then click **Save**.

5. You can click the isotherm tab to display an isotherm of the experiment data. An example is shown below.



6. You can click the file name tab to display experiment data and the volume dosed and volume adsorbed calculations.



The columns in the calculation tables are described below.

Column Heading	Description
ReqPm	Requested pressure by the user
Target Pm	Manifold target pressure for dosing
PmA	Pressure of manifold before dosing
TmA	Temperature of manifold before dosing
ZA	Compressibility of adsorbate at PmA and TmA
Vol A	Volume of adsorbate in manifold before dosing
PmB	Pressure of manifold after dosing
TmB	Temperature of manifold after dosing
ZB	Compressibility of adsorbate at PmB and TmB
Vol B	Volume of adsorbate in manifold after dosing

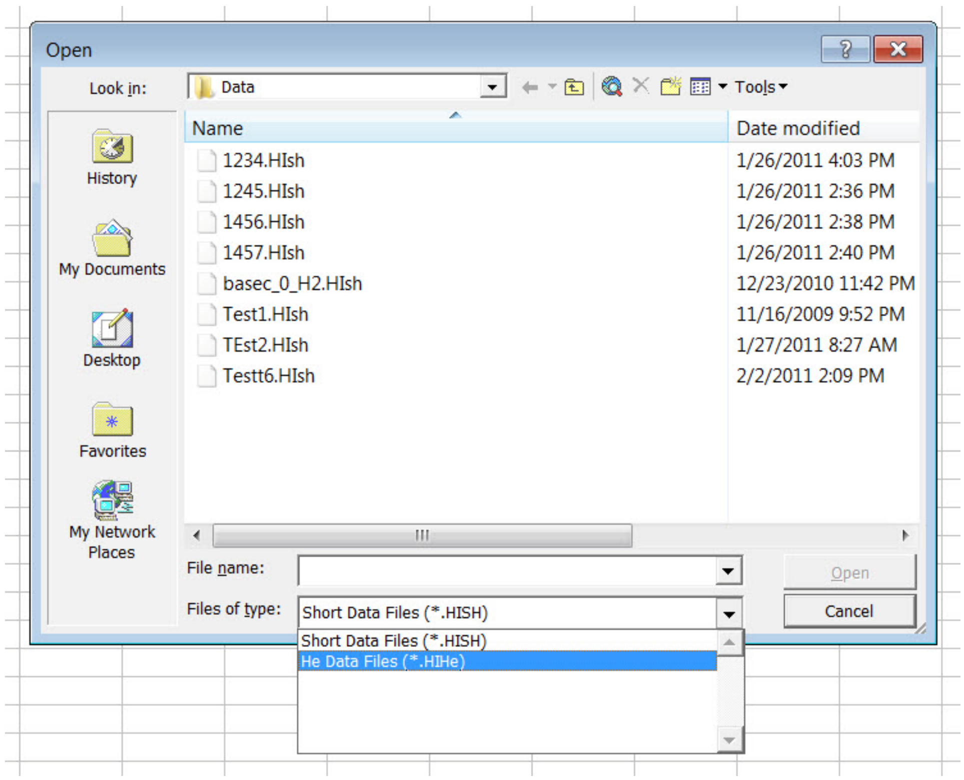
Column Heading	Description
Vol Dosed	Volume of adsorbate dosed to sample this step
Total Dosed	Total volume of adsorbate dosed to sample
Ps	Pressure of sample after dosing
Ts	Temperature of sample after dosing
ZS	Compressibility of adsorbate at Ps and Ts
Vs NAds	Volume of adsorbate in sample cylinder after dosing
ZXL	Compressibility of adsorbate at Ps and TmA
Vxl	Volume of adsorbate in lower stem after dosing
ZXU	Compressibility of adsorbate at Ps and TmB
Vxu	Volume of adsorbate in upper stem after dosing
Vol NAds	Total volume of adsorbate below value 1 after dosing
Vol Ads	Volume adsorbed by sample
Vol Ads/g	Volume adsorbed by sample/sample mass
wt%	Percentage of sample weight that is adsorbate

Read Helium Data File

The Helium Data file displays two graphs, as well as information about the free space analysis and calculations. When the graphs are displayed, you can use Excel charting functions to change curve colors, add symbols, modify the legend, etc. You can also use Excel functions such as Cut, Copy, and Save As to transfer data to other programs. Refer to your Excel documentation for more information.

Follow the instructions for Read Short Data File, page 3-8 with the following exception.

After you select **Read Short Data File** from the HPVA menu in the Excel Main Menu bar. The Open dialog is displayed. Click the down arrow next to the **Files of type** field and select **He Data Files (*.HIHe)**.

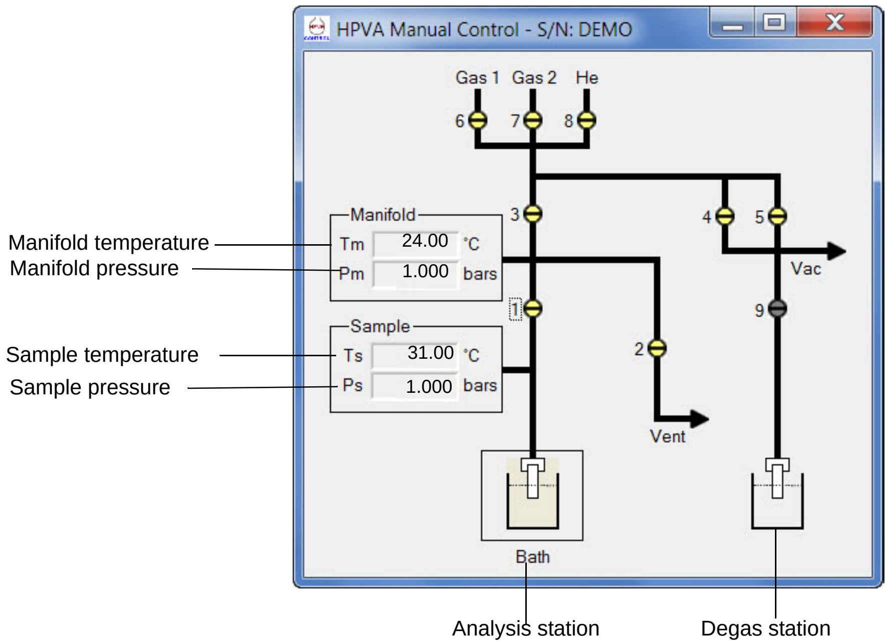


Select the desired HIHe file. The file will have the same name as the Short File for the experiment with an extension of HIHe.

4. Using the Manual Control Window

Description

The Manual Control Window shows a schematic of the HPVA System and can be used to manually open and close valves and, if a temperature control bath or cryostat is used, to set the sample temperature.



The manifold and sample pressures and temperatures are displayed in real time and are updated approximately every second.

Controlling Valves

The nine valves are represent by numbered icons. You can open and close a valve by double-clicking the left mouse button on the valve icon or by clicking the valve icon then pressing the space bar. Yellow indicates a closed valve and green indicates an open valve.

Do not open the vacuum valve when there is high pressure (pressure greater than 1.5 bar) in the manifold. The software cannot control the pressure in the manifold when the valves are manually operated.

The valves in the schematic are described below:

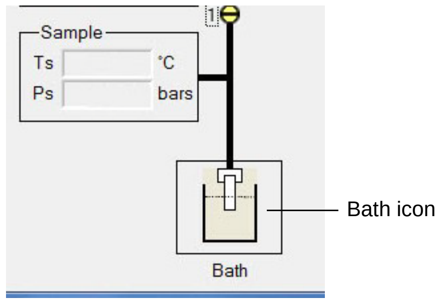
Valve	Description
1	Analysis port valve
2	Vent valve
3	Manifold valve
4	Fine Vacuum valve
5	Course Vacuum valve
6	Adsorptive gas 1 valve
7	Adsorptive gas 2 valve
8	Helium gas valve
9	Degas port valve*

*Valve 9 cannot be opened and closed on the schematic window. It can be controlled only by the switch on the instrument's front panel. Press the switch up to open the valve or down to close the valve.

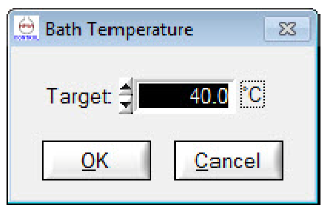
Setting the Sample Bath Temperature

If a temperature control bath or cryostat is configured with the instrument, you can set the bath temperature using the Manual Control screen as follows:

1. Double-click the left mouse button on the Bath icon.



2. The Bath Temperature dialog is displayed



Enter the temperature, then click **OK**.

5. Configuring the System

This chapter describes how to use the following options on the Main Menu:

- Calibration
- Change Data Directory
- Configure Hardware

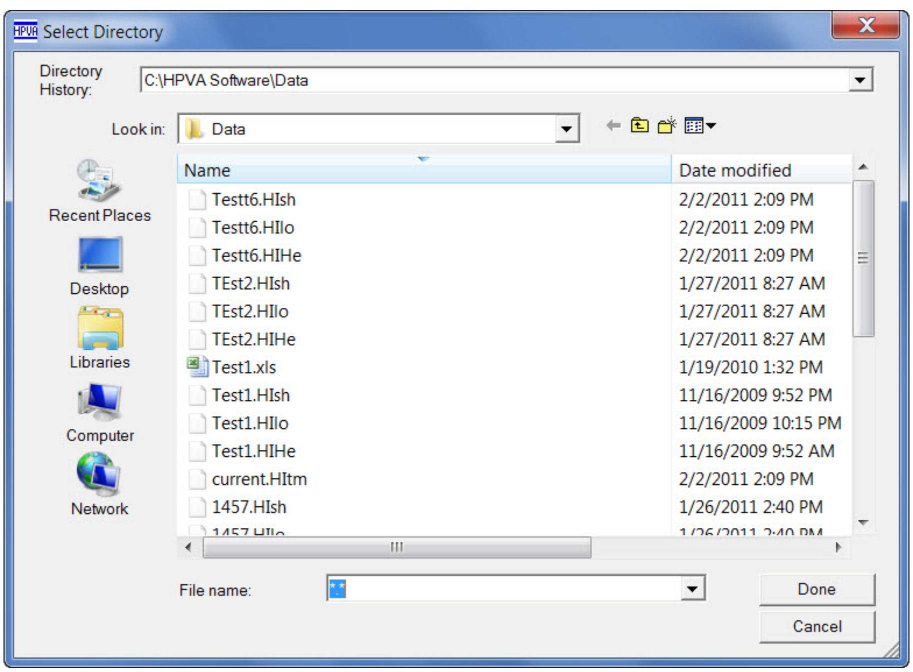
Calibrating the System

The Calibration option requires the use of a known volume cylinder. Calibration should be performed only by authorized service personnel.

Changing the Data Directory

The Data Directory is the folder in which the files produced by the system are placed. If you wish to change the location for the files:

1. Select **Change Data Directory** from the Main Menu. The following window is displayed.



2. Select the desired folder, then click **Done**.

3. The window closes and the Main Menu is displayed.

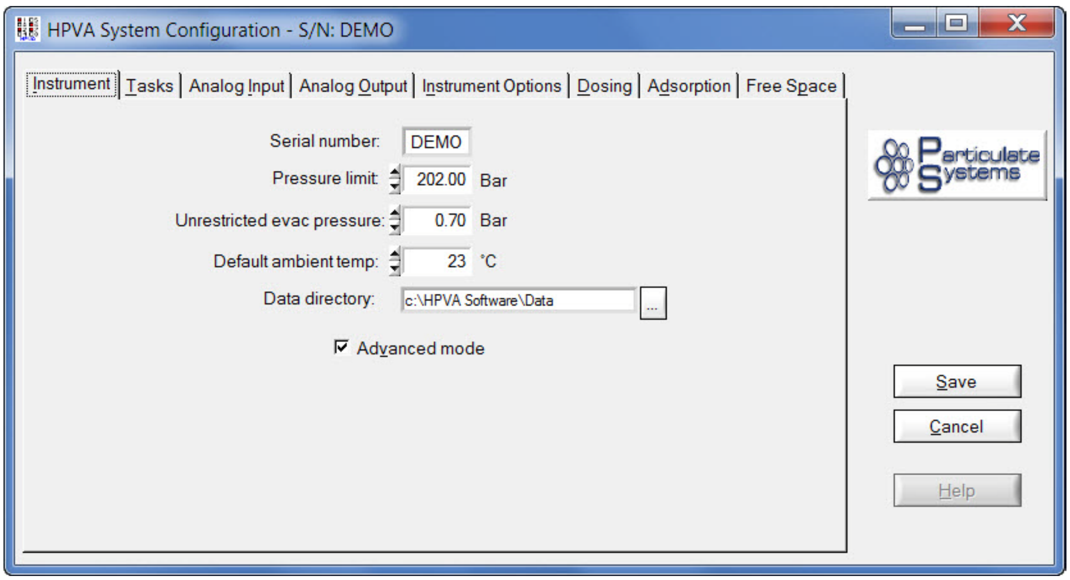
Configuring the Hardware

When you select **Configure Hardware** on the Main Menu, the System Configuration window is displayed.

System Configuration Window

The System Configuration window contains 8 tabs of parameters that you can edit prior to performing an experiment. The tabs and their associated dialogs are described below.

Instrument Tab



Serial Number Enter the instrument’s serial number. If a number is not entered or a wrong number is entered, the software will be unable to find the instrument’s calibration data.

Pressure Limit Enter the highest pressure the manifold will be allowed to reach – for a HPVA-100, the maximum should be set no higher than 103 bar and for a HPVA-200, the maximum should be set no higher than 202 bar.

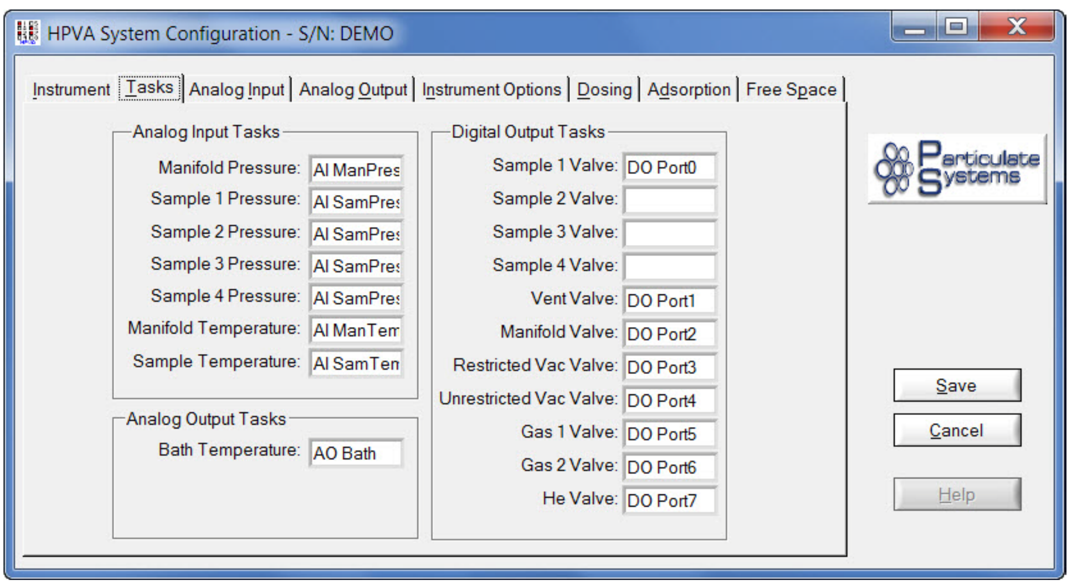
Unrestricted Evac Pressure When the system vacuums down the manifold, the restricted vacuum will be used until this pressure is reached; when this pressure is reached, the system will open Valve 5, allowing a strong vacuum to be applied to the system.

Default Ambient Temp Enter the air temperature of the lab.

Data Directory By clicking the **…** button next to the **Data directory** field you can change the directory in which data files are written (this can be done more conveniently by selecting **Change Data Directory** from the Main Menu).

Advanced Mode Check this box to run the instrument in **Advanced Mode**. To prevent damage to the system, this option should only be enabled at the direction of a Micromeritics/Particulate Systems service representative.

Tasks Tab

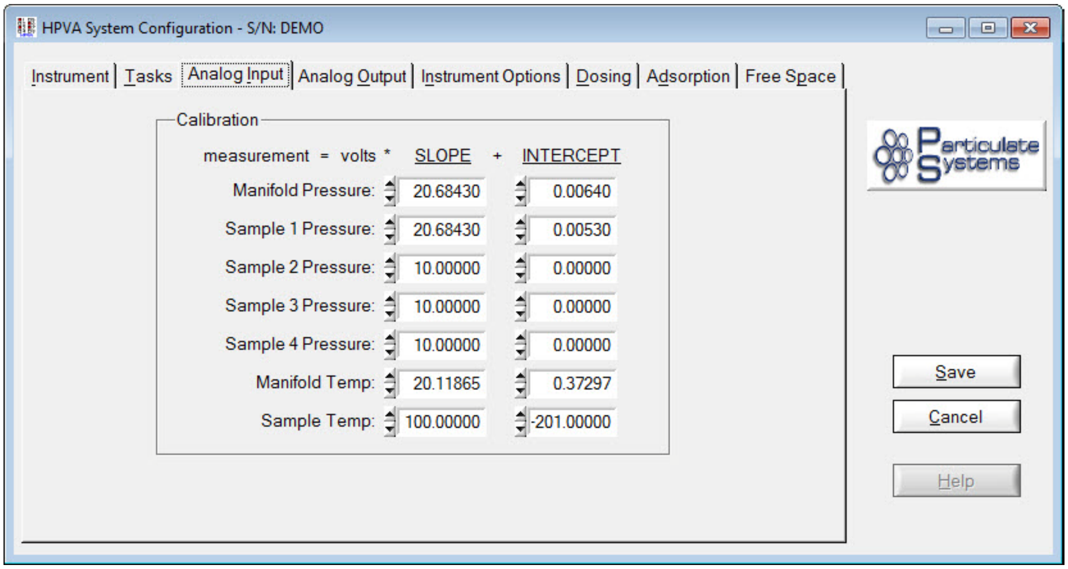


Analog Input Tasks Lists the tasks configured with the NI MAX software used to read data from the instrument. These tasks are configured during software installation.

Analog Output Tasks Shows the task associated with the control of a recirculation bath. This task is configured during software installation.

Digital Output Tasks Lists the tasks configured with the NI MAX software used to control the instrument valves. These tasks are configured during software installation.

Analog Input Tab



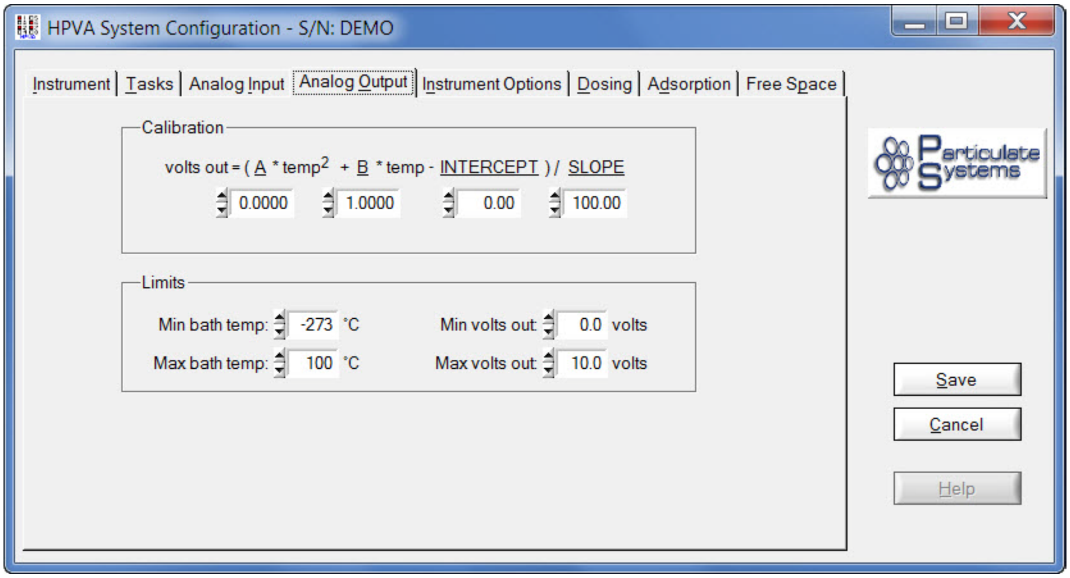
Manifold Pressure Shows the slope and intercept for the linear calibration of the manifold transducer.

Sample 1-4 Pressure Shows the slope and intercept for the linear calibration of the sample port transducers. Models HPVA-100 and HPVA-200 have only one sample port, Sample ports 2 through 4 are not applicable for these models.

Manifold Temp Shows the slope and intercept for the linear calibration of the manifold RTD.

Sample Temp Shows the slope and intercept for the linear calibration of the sample RTD.

Analog Output Tab



Calibration Enter the values to be used for the temperature calibration of the temperature control bath.

Limits

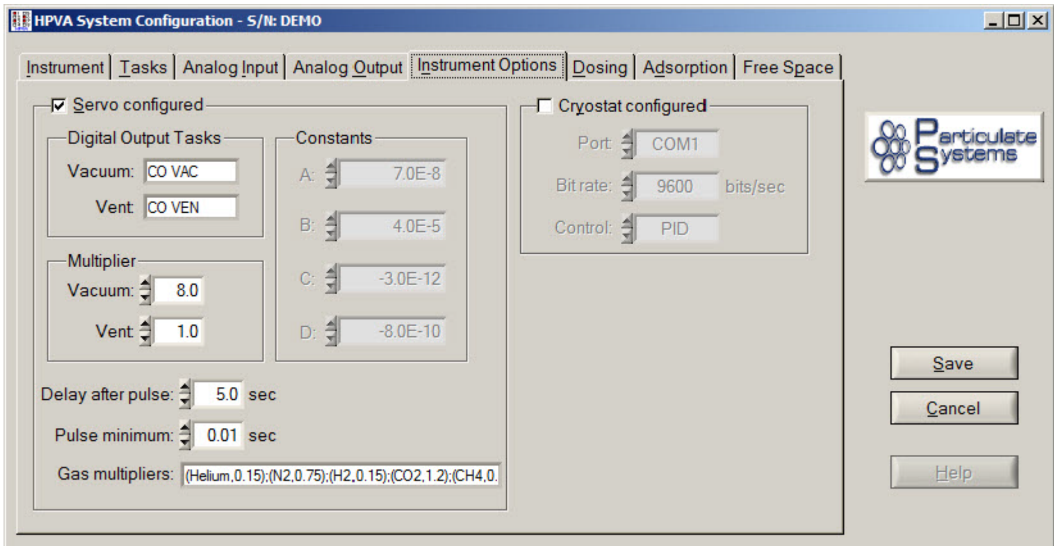
Min Bath Temp Enter the minimum analysis temperature allowed if **Bath** is selected on the System Step Isotherm window when preparing for an experiment.

Max Bath Temp Enter the maximum analysis temperature allowed if **Bath** is selected on the System Step Isotherm window when preparing for an experiment.

Min Volts Out Enter the minimum voltage the system is allowed to output to the bath.

Max Volts Out Enter the maximum voltage the system is allowed to output to the bath.

Instrument Options Tab



Servo Configured If the instrument is configured with servo valves, this option must be selected. If the instrument is configured with needle valves, this option must be cleared.

Digital Output Tasks

Vacuum The NI MAX task associated with the vacuum servo valve.

Vent The NI MAX task associated with the vent servo valve.

Multiplier

Vacuum Increase this value to increase the speed of the vacuum servo valve; decrease it to increase the servo precision.

Vent Increase this value to increase the speed of the vent servo valve; decrease it to increase the servo precision.

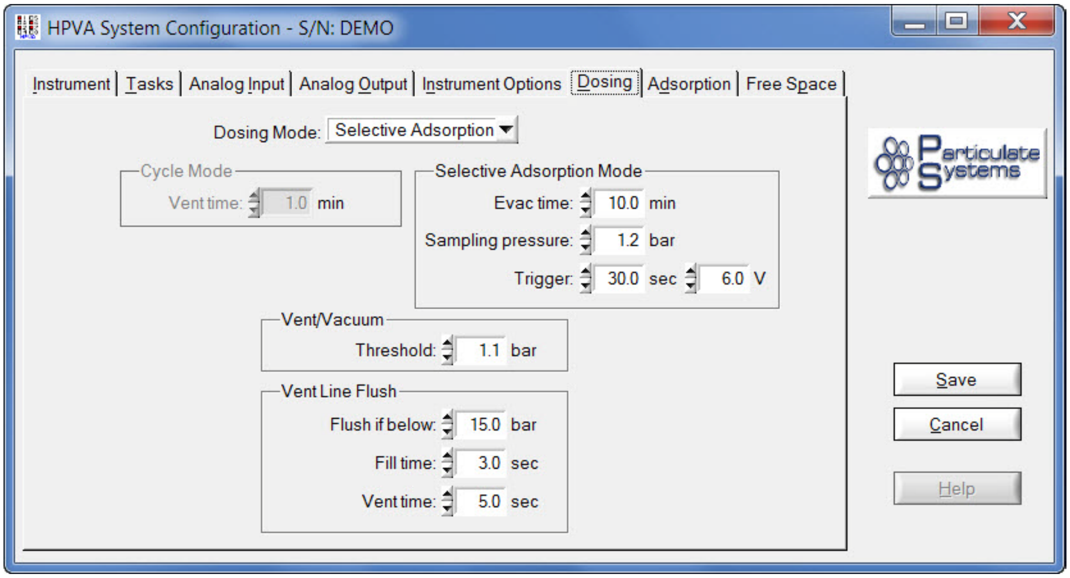
Constants These are fixed values used for the servo algorithm, they cannot be changed by a user.

Delay After Pulse Enter the time between each pulse made when using the servo valves.

Pulse Minimum Enter the minimum amount of time the servo valve will remain open during a pulse

Gas Multipliers	Enter the values used to control the servo valve based on the gas in the system.
Cryostat Configured	If a cryostat is to be used during analyses, check this box.
Port	Enter the serial port used to connect the cryostat controller to the computer.
Bit Rate	Enter the speed of the connection between the cryostat controller and the computer (default 9600 bits/sec).
Control	Select the type of control used by the cryostat to maintain temperature (PID or PID Table); refer to the cryostat manual for more information.

Dosing Tab



Dosing Mode

Select on of three dosing mode options from the drop-down list:

Standard
The system will dose the sample to the desired pressure points listed in the System Step Isotherm window without evacuating the sample tube between points.

Cycle
The system will dose the manifold to the desired pressure points listed in the System Step Isotherm window and vent the sample after each pressure point – the Low and High Pressure Factors will be ignored.

Selective Adsorption
The system will dose the manifold to the desired pressure points listed in the System Step Isotherm window and will evacuate the sample tube between points and trigger an external device to collect said evacuated gas for composition analysis.

Cycle Mode Vent Time

Enter the amount of time the vent will be open after each pressure step when using the Cycle Dosing Mode.

Selective Adsorption Mode

Evac Time The amount of time the system evacuates the sample tube between pressure points when Selective Adsorption Mode is enabled.

Sampling Pressure After equilibration, the system will vent the manifold to this pressure before triggering the external device to collect data.

Trigger The voltage and the amount of time the trigger signal is sent to the external device.

Vent/Vacuum Threshold Above this pressure the system will use the vent servo or needle valve to reach desired pressures, below this pressure the system will use the vacuum servo or needle valve to reach desired pressures.

Vent Line Flush

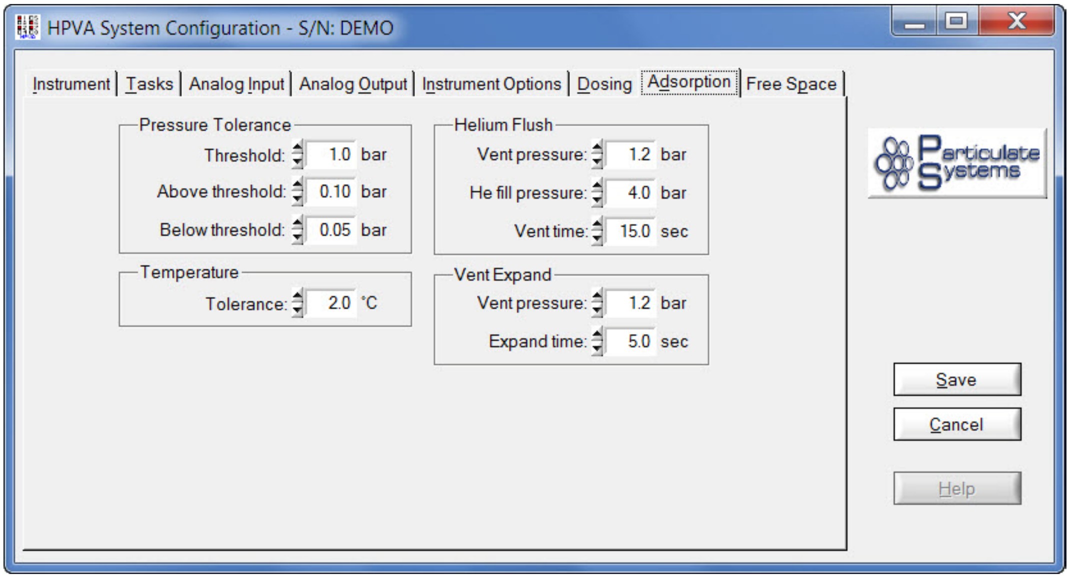
Flush if Below If any dose pressure in the isotherm analysis is below this value, the system will flush the vent line with the analysis gas before pressurizing the manifold to the dose pressure.

This prevents contamination of the manifold during venting at near-ambient pressures.

Fill time The amount of time the system will fill the manifold with the analysis gas prior to the vent line flush.

Vent time The amount of time the system will open the vent valve to flush the analysis gas through the vent line.

Adsorption Tab



Pressure Tolerance

Threshold	The pressure at which the dose pressure tolerance below (this value) is different from the pressure tolerance above (this value).
Above Threshold	The dose pressure must be within ± the value entered here for the system to continue during dosing.
Below Threshold	The dose pressure must be within ± the value entered here for the system to continue during dosing.
Temperature Tolerance	The sample temperature must be within ± this value to be considered at the target temperature.
Helium Flush	The system will cycle helium through the manifold and sample tube three times after the last experiment to allow safe removal of the sample.
Vent Pressure	After pressurization with helium, the sample tube and manifold will be vented to this pressure.
He Fill Pressure	The helium fill pressure.

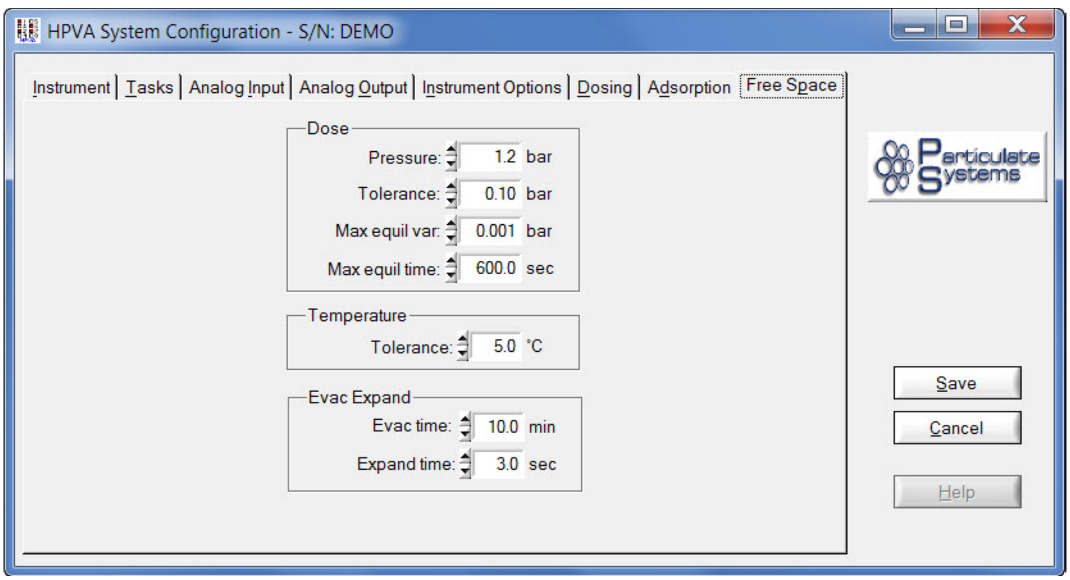
Vent Time The amount of time the vent remains open after the third helium fill/vent cycle.

Vent Expand

Vent Pressure During the adsorption experiment, the system will be considered vented if at or below this pressure.

Expand Time During the analysis, the system will open valve 1 to dose the sample for this amount of time before closing the valve and waiting for equilibration.

Free Space Tab



Dose

Pressure The pressure the manifold will dose to prior to the free space analysis. The recommended value is 10 bar.

Tolerance The tolerance of the dosing pressure for the free space analysis ($\pm$ this value).

Max Equil Var The system is considered at equilibrium if the pressure does not vary by more than this pressure during a one minute period.

Max Equil Time The maximum time allowed for equilibration during the free space analysis.

Temperature Tolerance The temperature tolerance allowed for the ambient and analysis temperatures during the free space analysis (± this value).

Evac Expand

Evac Time The amount of time the system will evacuate the sample tube prior to the free space analysis.

Expand Time The amount of time valve 1 is opened to allow the gas in the manifold to expand into the sample tube during free space analysis.

6. Troubleshooting and Maintenance

The HPVA system has been designed to provide efficient and continuous service. However, certain maintenance procedures should be followed to obtain the best results over the longest period of time. This chapter includes troubleshooting and maintenance procedures.

Troubleshooting

Most operational problems are caused by:

- Leaks (commonly around the sample tube O-ring at the analysis port)
- Sample weighing errors
- Impure gas supply

Always check these first when expected experiment results are not obtained. Some common operational problems, which are not indicated on the video monitor screen, and their respective causes and solutions are provided in the following table.

What Happened	Why	What To Do
Vacuum error, such as vacuum gauge cannot reach lower limit	Vacuum pump hose leaking around an O-ring.	The vacuum pump hose uses four O-rings: • Two on the tee-connector/ hose connection • One on the hose/instrument connection on the rear panel of the instrument • One on the hose/vacuum pump connection These O-rings must be properly greased and must not be worn or cracked. Check the O-rings and grease or replace as necessary.
	Vent valve 2 is open allowing ambient air to be ingested.	Close Vent valve 2, flush with helium, then vacuum.

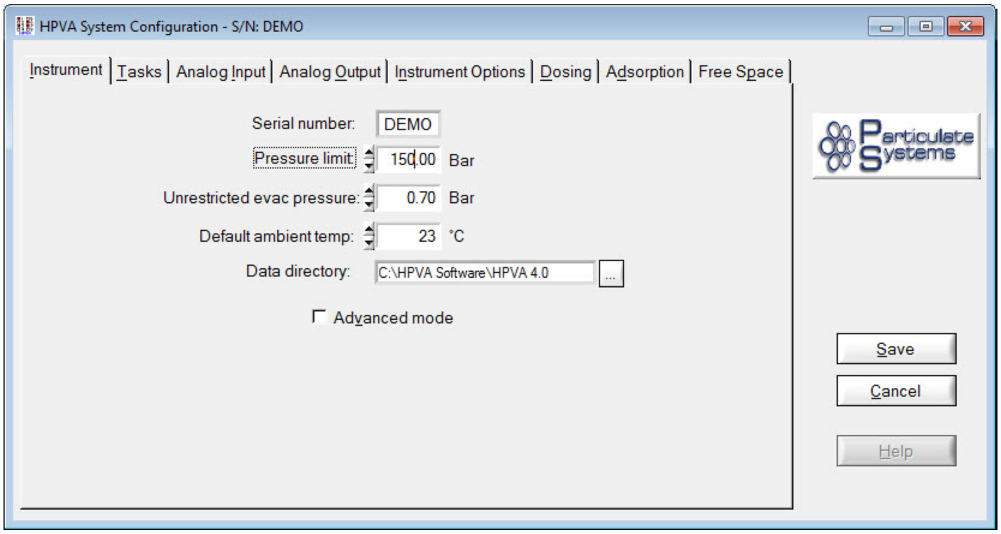
What Happened	Why	What to Do
Low degas adsorption	Contaminated gas lines.	Place gas bottles close to the analyzer. Using gas line extenders on gas bottles located in remote areas may degrade gas quality and reduce pressure.
Instrument shuts off unexpectedly	Hydrogen sensor has been triggered, indicating that there is a hydrogen leak in the instrument. May also be triggered by other hydrocarbon sources in the lab.	Clear the hydrocarbon source, then turn the instrument back on.
Valves will not operate	Gas bottle depleted For nitrogen or compressed air, the pressure on the gas line is less than 75 psi.	Replace the gas bottle. Refer to Connecting a Replacement Gas Bottle, page 6-8.
When the system is under vacuum, a value of 0 is not shown in the pressure display.	The transducers may need to be zeroed.	Call you service representative.
Inaccurate temperature reading shown in the one of the temperature displays	RTD's may need to be recalibrated.	Call you service representative.
Pressure cycling	Gas is flowing into the manifold too fast.	Adjust the metering valves. Refer to Adjusting the Metering Valves, page 6-11.

What Happened	Why	What to Do
Pressure cycling *(continued)*	Gas is venting out of the manifold too fast.	Adjust the **Vent** valve on the instrument’s front panel. 
Target pressure not reached	Gas level in tank is too low.	Replace the gas bottle. Refer to Connecting a Replacement Gas Bottle, page 6-8.
	The pressure limit entered in the Instrument Configuration window does not match the pressure set on the regulator. For example, if you are using CO_2 or methane, the pressure limit must be set below the maximum output of the regulator.	Enter the correct pressure limit. Refer to Resetting the Pressure Limit, page 6-4.
Ambient pressure not reached	Slope set incorrectly in the Hardware Configuration window.	Enter the correct slope. Refer to Entering the Slope, page 6-5.
Degas or analysis not progressing as it should	O-rings in ports are missing, worn, or damaged.	Replace the port O-ring. Refer to Replacing Degas and Analysis Port O-Rings, page 6-6.

Resetting the Pressure Limit

1. Select **Configure Hardware** from the HPVA main menu.

2. Make sure the Instrument tab is selected.



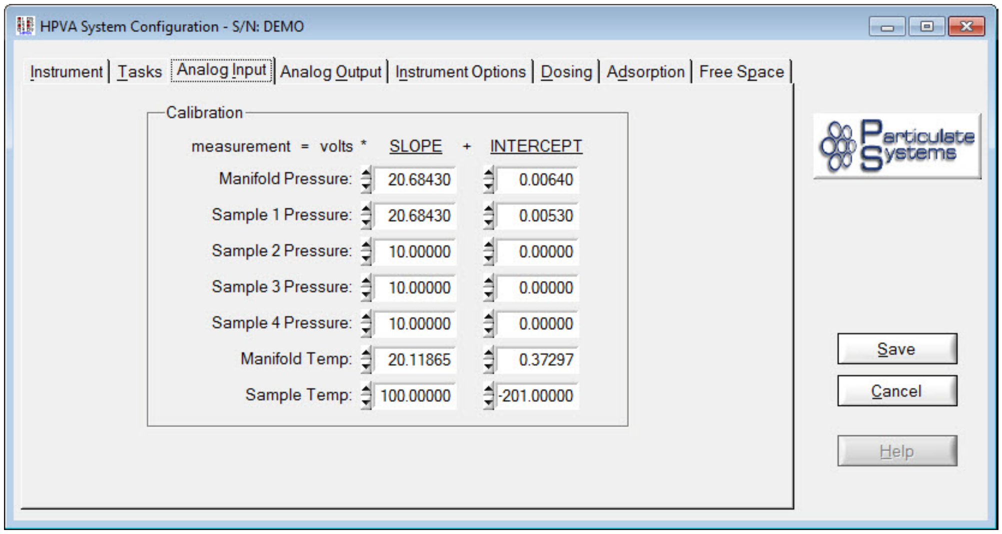
3. Enter the maximum pressure for the gas you are using in the **Pressure limit** field.

4. Click **Save**.

Entering the Slope

1. Select **Configure Hardware** from the HPVA main menu.

2. Make sure the Analog Input tab is selected.



Enter the correct slope in **Manifold Pressure** and **Sample 1 Pressure** fields as follows:

HPVA-100 = 13.7895
HPVA-200 = 20.6843

The HPVA-100 and HPVA-200 contain only one analysis port, so the fields Sample 2 Pressure - Sample 4 Pressure do not apply.

3. Click **Save**.

Replacing Degas and Analysis Port O-Rings

Each of the ports, degas and analysis, contains an O-ring that must be present when degassing or analyzing a sample. Ensure that the O-ring is present before performing either procedure.

If an O-ring becomes worn or damaged, it should be replaced. A damaged O-ring may cause leaks at the port, and inaccurate results.

Connecting Gases

Guidelines for Connecting Gases to the Analyzer

Use these guidelines when installing regulators and gas lines:

- Place gas bottles close to the analyzer. Using gas line extenders on gas bottles located in remote areas may degrade gas quality and reduce pressure.
- Use a retaining strap (or other appropriate tether) to secure the gas bottle.
- Carefully route the gas lines from the bottle to the analyzer,; avoid overlapping or entangling lines.
- Label the gas line at the instrument inlet for proper identification and maintenance.
- Ensure that the gas bottle is closed before connecting to the analyzer.

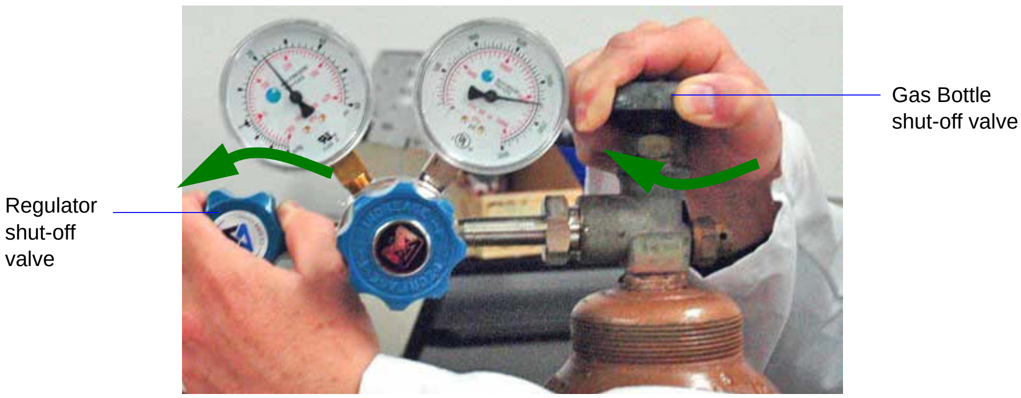
The following instructions describe a typical installation. Some configurations require additional components, such as regulator expansion kits, when one gas source will be used for several operations or when the gas bottle cannot be located close to the analyzer.

Required Items.

Particulate Systems Supplied	User Supplied
Stainless steel gas tubing Two 7/16-in. wrenches	Analysis gas (recommended purity 99.999%) Helium (recommended purity 99.999%) Clean compressed air and air line Regulators for gas bottles

Disconnecting the Depleted Bottle

1. Close the gas bottle shut-off valve, then open the regulator shut-off valve.



2. Both gauges should read at or near zero; if not, open the gas inlet valve (3) and the vent valve (2) and allow the pressure to purge from the lines.

3. Use an appropriate wrench to loosen the nut at the regulator/gas bottle connection, then remove the regulator from the bottle.

You do not have to disconnect the gas line from the regulator or the instrument.

4. Replace the protective cap on the depleted bottle, disconnect the retaining strap, and remove the bottle from its current location.

Connecting a Replacement Gas Bottle

Move the replacement bottle close to the instrument and tether it into place.

When connecting hazardous gases, be sure to vent properly and follow the safety procedures established for your lab.

A power failure or loss of cryogen can result in dangerous pressures in the sample tube. The HPVA uses pressure relief valves to vent this pressure into the instrument cabinet and return the instrument to a safe condition. When using toxic or flammable gases, additional venting of the cabinet may be required.

1. Use an appropriate cylinder wrench to remove the protective cap from the replacement bottle.

2. Attach the gas regulator to the connector on the gas bottle. Hand-tighten the nut, then use an appropriate wrench to tighten an additional 3/4 turn.

Do not overtighten the fitting; doing so may cause a leak.

3. Check for leaks at the high-pressure side of the regulator and in the connector.

a. Turn the regulator control knob fully counterclockwise.

b. Slowly open the gas bottle shut-off valve, then close it.

c. Observe the pressure on the high-pressure gauge.

d. If the pressure is stable, proceed with the next step.

If the pressure decreases, tighten the regulator connector nut until it becomes stable.

5. Purge the air from the lines.

a. Turn the regulator shut-off valve counterclockwise to open.

b. Open the gas bottle shut-off valve to flow gas.

c. Close the regulator shut-off valve to stop flow.

d. Close the gas bottle valve.

5. Set the instrument pressure.

a. Turn the regulator control knob clockwise until the low-pressure gauge reads 15 psi (1.03 bar).

b. Open the regulator shut-off valve.

c. Open the gas bottle shut-off valve and flow gas for 10 to 30 seconds.

d. Close the gas bottle shut-off valve.

5. If you disconnected the gas line to the instrument inlet, reconnect it now.

6. Ensure that the gas pressure is set to the following:

Analysis gas	1500 psi (103 bar) for an HPVA-100 3000 psi (206 bar) for an HPVA-200
Helium gas	500 psi (34.4 bar)
Compressed air	75 to 80 psi (5.2 to 5.5 bar)

7. Adjust the gas metering valve for proper gas flow. Refer to the next topic.

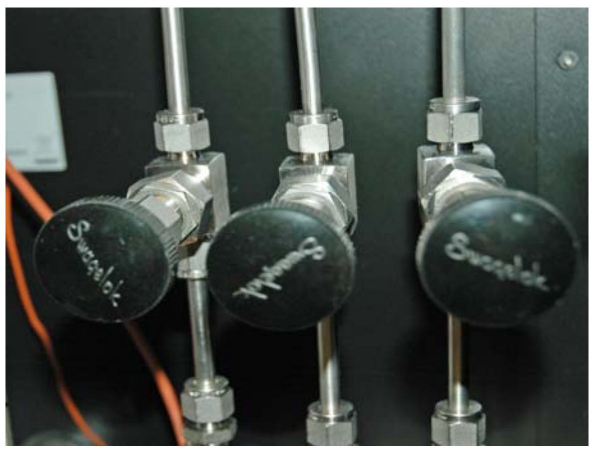
Adjusting the Metering Valves

The metering valves control the pressure of the gas flow into the unit. It is very important that they are set correctly to ensure proper dosing without overshooting pressure points.

There are three metering valves inside the instrument; one for each gas line. When you change gases or need to adjust the flow of a gas follow these procedures.

1. Remove the rear panel from the instrument.

2. The metering valves are located on the lower right side of the instrument.

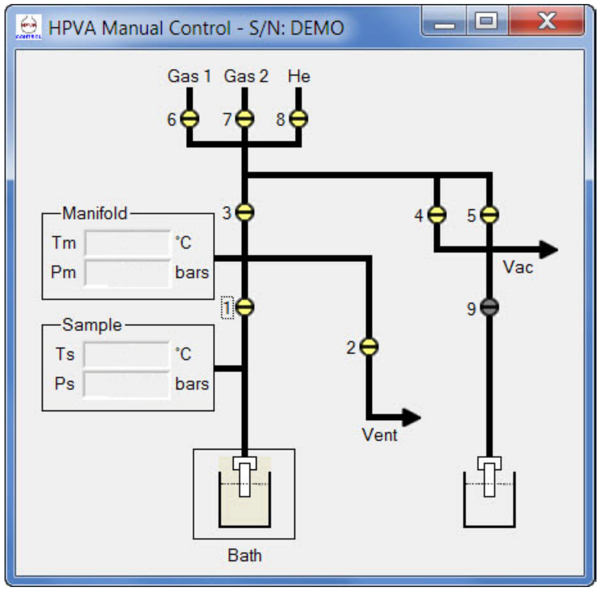


   The valves are, from let to right, Gas 1, Gas 2, and Gas 3 (Helium).

   Fully close the metering valve for the appropriate gas line.

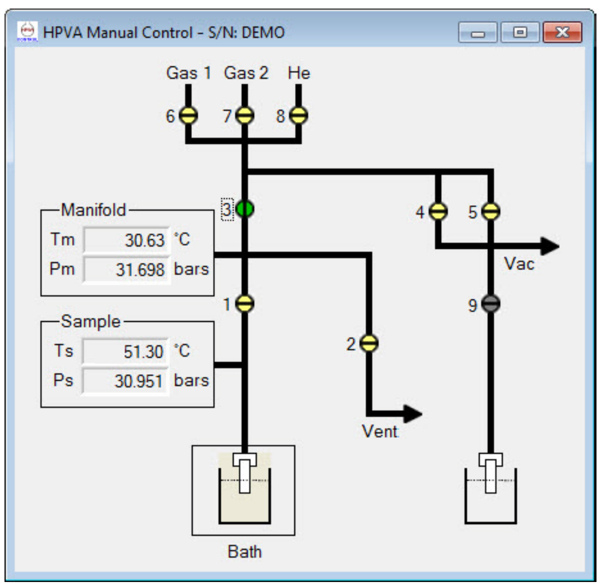
3. Select **Manual Control** from the HPVA main menu.

4. Make sure all the shut-off valves shown in the schematic are closed.

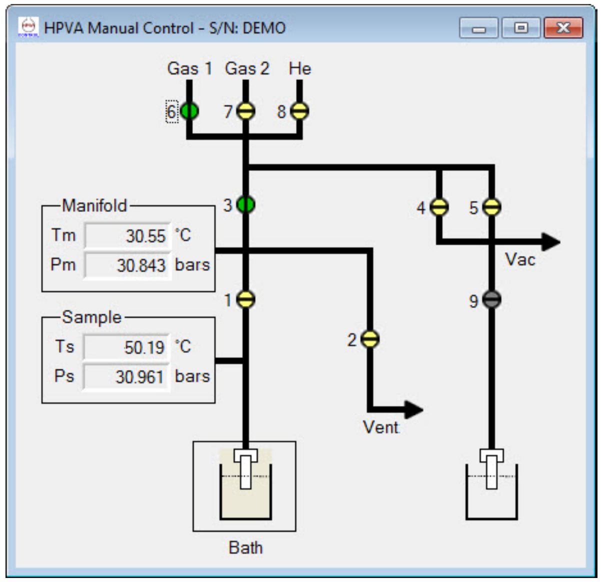


5. Turn on the gas at the gas bottle.

6. On the Manual Control screen, open valve 3 so that pressure can be read.



7. On the Manual Control screen, open the gas line valve, for example, valve 6 for Gas Line 1.

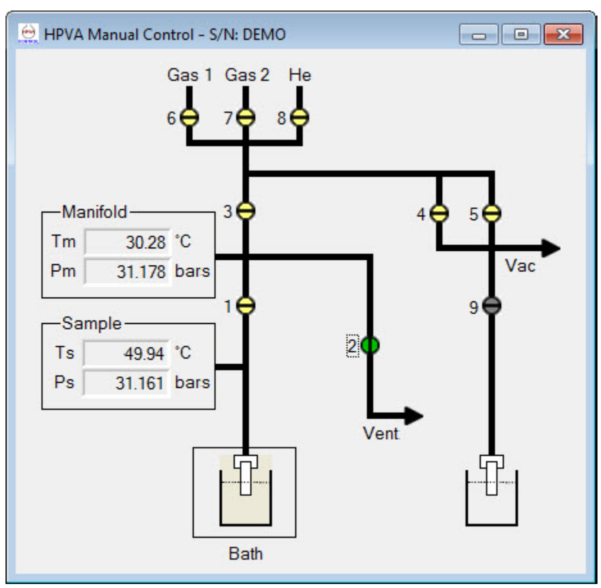


8. Slightly open the metering valve.

9. Observe the Manifold pressure (**Pm**) shown on the Manual Control screen. The pressure should rise approximately 1 bar every 10 to 20 seconds.

10. Adjust the metering valve as necessary to obtain the appropriate gas flow.

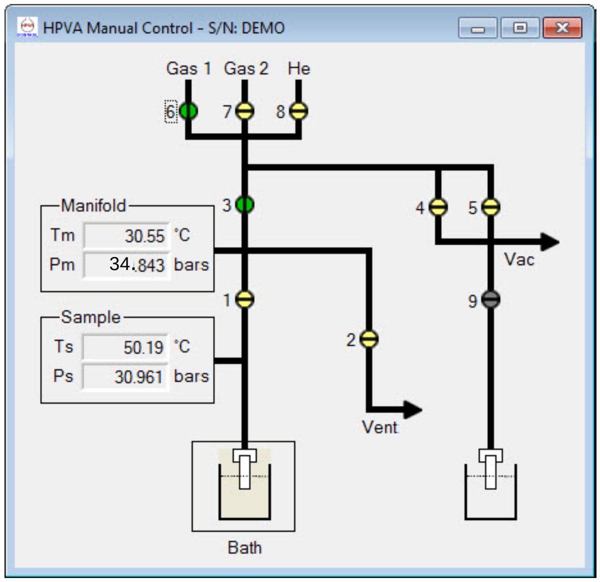
11. Close the valves on the Manual Control screen, then open valve 2 to vent.



12. To recheck the pressure:

    a. Close valve 2.

    a. Open valve 3 and observe the pressure as you open the gas valve (valve 6 in our example). There should be an initial rapid increase followed by a steady pressure increase.



2. After observing a steady pressure increase, close valves 3 and 6.

3. Open valve 2 to vent.

4. After venting, close the Manual Control screen.

7. Ordering Information

Components for the HPVA Series analyzers can be ordered using one of the following methods:

- Call Customer Service at (770) 662-3636
- Contact your local sales representative

When ordering, please use the information provided below to place your order.

Part Number	Item and Description
P02-25805-00	Gas inlet line, stainless steel, 8 ft.
P02-31701-00	Furnace insulator top, half
003-51123-00	Fuse, 6.25 Amp, 3AG, Slow Blow
003-51134-01	Fuse, 5.0 Amp, 5x20 mm, Slow Blow
004-25040-01	Gasket, 1/4 in., stainless steel, silver plated; for sample cylinder
004-27056-01	Filtered gasket, 60 micron; for sample cylinder when fine powders used
P02-25821-00	Sample cylinder, 2 cc
P02-25827-00	Sample cylinder, 10 cc
P02-25846-00	Sample tube funnel
004-25466-02	O-ring -010 60 Duro Viton F, for degas or analysis port
P02-25820-00	Sample isolation valve
P02-25823-00	Sample tube stem, used with sample isolation valve
P02-33012-00	Cryogenic Kit, includes Dewar and accessories

A. Error Messages

This appendix contains the error messages that may be encountered while operating the HPVA Series instruments; they are listed in alphabetical order.

Changes made to the pressure steps table affect steps that have already run.

Cause: You attempted to update the pressure table that modified pressure points already collected.

Action: You can modify only the entries in the pressure table which have not been collected.

Error (number) while preparing to write (path-to-ini-file)

Cause: The application initialization file is missing from the HPVA System applications directory.

Action: Verify that all HPVA System applications and the HPVASys.ini file are in the same directory.

Instrument .ini file could not be found: (path-to-ini-file)

Cause: The application initialization file is missing from the HPVA System applications directory.

Action: Verify that all HPVA System applications and the HPVASys.ini file are in the same directory.

One or more hardware errors were found. Unable to continue.

Cause: Initialization of the National Instruments data acquisition board failed.

Action A: Verify proper installation of the HPVA software and that no additional updates from National Instruments have been installed.

Action B: Verify that the board and external cables are properly connected.

Trigger output voltage (V) is out of range. Trigger will not be set.

Cause: While in the selective adsorption dosing mode, the requested trigger signal voltage was over 10 volts or under -10 volts.

Action: Verify that the selective adsorption trigger voltage is within the range of +10 to -10 volts.

Trigger signal is not ready.

Cause: While in the selective adsorption dosing mode, the trigger signal to the external analytical instrument (GC or mass spec) could not be enabled.

Action A: Verify that the selective adsorption dosing mode was properly selected.

Action B: Verify proper installation of the HPVA software and that no additional updates from National Instruments have been installed.

Action C: Verify that the board and external cables are properly connected.

Unable to load UI.

Cause: The application initialization failed to load due to unavailable resources.

Action: Verify that there is available free memory on the computer to run the HPVA software.

Unable to open instrument log <(path-to-log)>

Cause: The instrument log file failed to open.

Action: Verify that you have write permissions to the directory specified for your data files.

Unable to open template file.

Cause: The selected template file has been corrupted or is not a proper template file.

Action: Select another template file for analysis.

Unable to read info from .ini file

Cause: The application initialization file is missing from the HPVA System applications directory.

Action: Verify that all HPVA System applications and the HPVASys.ini file are in the same directory.

Unable to write calibration file (file name)

Cause: Writing of the calibration file failed.

Action A: Verify that there is available disk space to write the calibration file.

Action B: Verify that all HPVA System applications and calibration file are in the same directory.

Action C: Verify that you have write permissions to the directory in which the HPVA System applications are installed.

Unable to write template file to the data directory: (file path)

Cause: The template file failed to write to the specified data directory.

Action A: Verify that there is available disk space to write the template file.

Action B: Verify that you have write permissions to the specified data directory.

Unable to write the .ini file (path-to-ini-file)

Cause: Writing of the application initialization file failed.

Action A: Verify that there is available disk space to write the application initialization file.

Action B: Verify that all HPVA system applications and the HPVASys.ini file are in the same directory.

Action C: Verify that you have write permissions to the directory in which the HPVA System applications are installed.

You must enter a valid Expt ID before you can run. (list of unavailable characters)

Cause: The entered Experiment ID contains invalid characters.

Action: Use an alternate experiment ID.

You must enter an Expt ID before you can run.

Cause: The Experiment ID is blank.

Action: Enter a valid Experiment ID before continuing.

You need to define at least one run.

Cause: You attempted to proceed with an isotherm experiment with no defined experimental runs.

Action: Define the conditions for at least one experimental run before continuing.

B. Performing a Cryogenic Analysis

Description

This appendix contains instructions for performing an analysis with the optional Cryogenic Kit, available from Particulate Systems.

Due to helium interaction with samples at cryogenic temperatures, it is difficult to find the true analysis free space of the sample tube with the sample present. Therefore, we recommend that you first find the ambient and analysis free space volumes of the empty sample tube to be used in the analysis. After you perform the free space routine, you can then put the sample into the sample tube and prepare it for analysis. The HPVA software allows you to enter the ambient and analysis free space values previously found prior to the adsorption/desorption routine.

When the volumetric calculations are performed by the HPVA Excel macro, the mass and density of the sample are used to find the sample's physical volume. This volume is subtracted from the ambient and analysis free space values originally entered into the HPVA software. This corrects the blank sample tube free space values for the volume of the sample and provides accurate volumes to be used for the isotherm calculations.

When the HPVA is performing an experiment or being operated in manual control mode, the valve switches on the front panel should be placed and remain in the closed (down) position.

If a switch is placed in the opened position during a software operation, it will override the software and may damage the instrument.

Measuring Free Space

Performing a Blank Tube Experiment

1. Clean and dry the sample cylinder. (Refer to **Cleaning the Sample Cylinder,** page **2-2**.)
2. Assemble the sample holder. (Refer to **Assembling the Sample Holder,** page **2-5**.)
3. Place an isothermal jacket on the stem of the sample holder.
4. Place the dewar stand and empty dewar under the analysis port, then plug in the temperature probe.

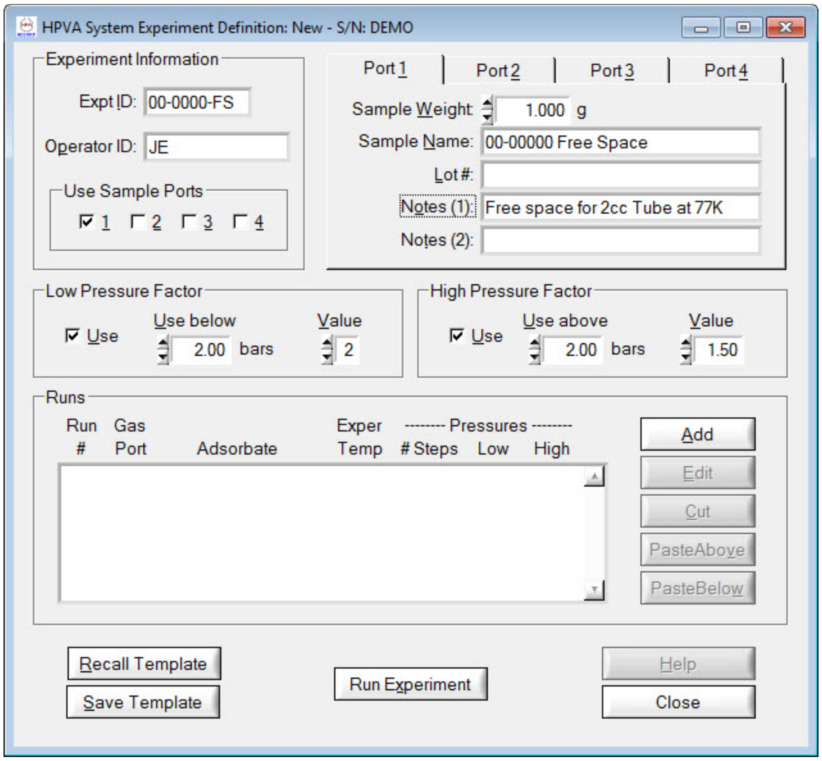
5. Ensure that the O-ring is in place in the analysis port.

6. Tilt the dewar and insert the sample holder.

7. Attach the sample holder to the sample port and hand-tighten the connector nut.

8. Using two wrenches (3/4-in and 5/8-in) tighten the nut just until snug. Note, do not over tighten the connector nut, doing so could damage the port fitting.

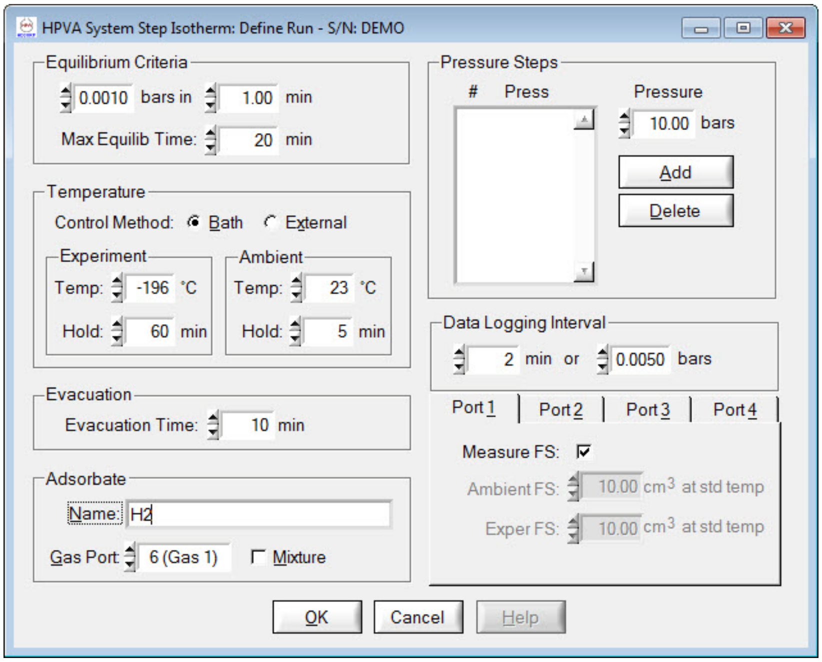
9. Select the **Adsorption** option from the HPVA main menu. The Experiment Definition: New window is displayed.



10. Enter information in the fields in this window. (Refer to **Define New Experiment Window,** page **2-27**).

It is important to set the Sample Weight to 1 (or a value greater than 0) or the analysis will not be able to start

11. Click **Add** to enter the analysis conditions for the free space measurement. The Define Run window is displayed.



12. Select the **External Temperature Control Method** radio button.

13. Enter the **Experiment** and **Ambient** temperatures.

14. Set the **Experiment Hold** time to about 60 minutes, but no less than 45 minutes. This is necessary because when the warm sample tube is submerged into a liquid cryogen bath, it takes at least 45 minutes for the sample tube to equilibrate and create a stable temperature within the tube.

15. Click **OK**. The Experiment Definition: New window is displayed.

16. Open the sample isolation valve on the sample tube.

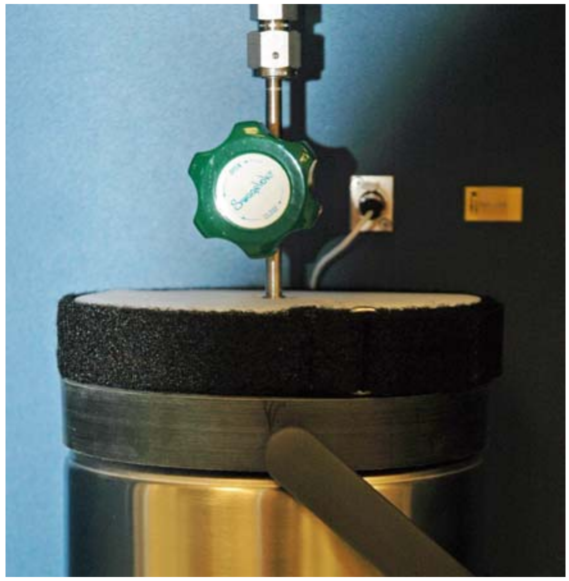
17. Click **Run Experiment**.

18. The software will find the ambient temperature free space volume. When this step is completed, you will be prompted to make the temperature change.

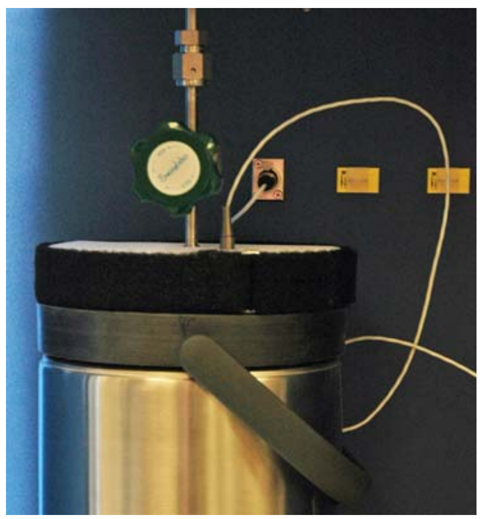
19. Fill the dewar with liquid nitrogen to the top of the isothermal jacket.

20. Place the dewar cover on top of the dewar.

21. Place the strap around the dewar cover and tighten.



22. Insert the temperature probe into the dewar.



23. Click **OK**.

Do not remove the sample tube from the analysis port between the ambient temperature volume analysis and the analysis temperature volume analysis. Doing so will cause the free space analysis to be compromised.

24. When the analysis is complete, the results will be available in the Excel HPVA macro as described in the next topic.

Displaying the Free Space

1. Open the Excel Macro.
2. Select **Read Short Data File** from the HPVA menu.
3. Select He Data Files (*HIHe) from the File Type drop-down list.
4. Select the helium data file created during the free space analysis. A report, which includes Ambient Free Space and Analysis Free Space (measured in cc STP) is displayed. Below is an example, with the free space volumes highlighted.

File Name	00-0000-FS.HIHe	
Experiment	Step Isotherm	
Operator	JE	
Experiment ID	00-0000-FS	
Sample Name	00-0000 Free Space	
Sample Lot #		
Notes	Free Space for 2cc Tube	
Sample Weight	1.0000	
Adsorbate	Nitrogen	
Critical Pressure	33.90	
Critical Temperature	126.20	
Ambient Free Space	14.203300	
Analysis Free Space	3.224400	
Manifold Volume	25.111401	
Expt Temp	-196	ºC
Ambient Temp	23	ºC
Tm0	-196.01236	ºC
Ts0	22.9819912	ºC
Pm0	0.00127263	bar
Ps0	-0.0167417	bar
Max Equil Time	20	min

5. The free space volumes of the sample tube can be used for future adsorption/desorption analyses as long as the analysis temperature does not change. We recommend that you record these volumes for future use.

Preparing the Sample

1. Remove the sample holder from the analysis port.
2. Remove the isothermal jacket.
3. Allow the sample holder to return to room temperature.
4. Remove any condensation that formed on the exterior of the sample holder.
5. Disassemble the sample holder.
6. Weigh the sample. (Refer to **Weighing the Sample,** page **2-4**.)
7. Assemble the sample holder. (Refer to **Assembling the Sample Holder,** page **2-5**).
8. Degas the sample to remove any moisture or CO_2 from the sample. (Refer to **Degassing the Sample,** page **2-7**).

Make sure the isothermal jacket is removed from the sample holder before degassing the sample. The isothermal jacket cannot withstand the heat during the degas process.

9. When the degas is complete, close the Sample isolation valve on the sample holder.

10. Cool the sample holder to room temperature.

Conducting the Adsorption/Desorption Analysis

1. Place the isothermal jacket on the sample tube.
2. Place the dewar stand and empty dewar under the analysis port, then plug in the temperature probe.

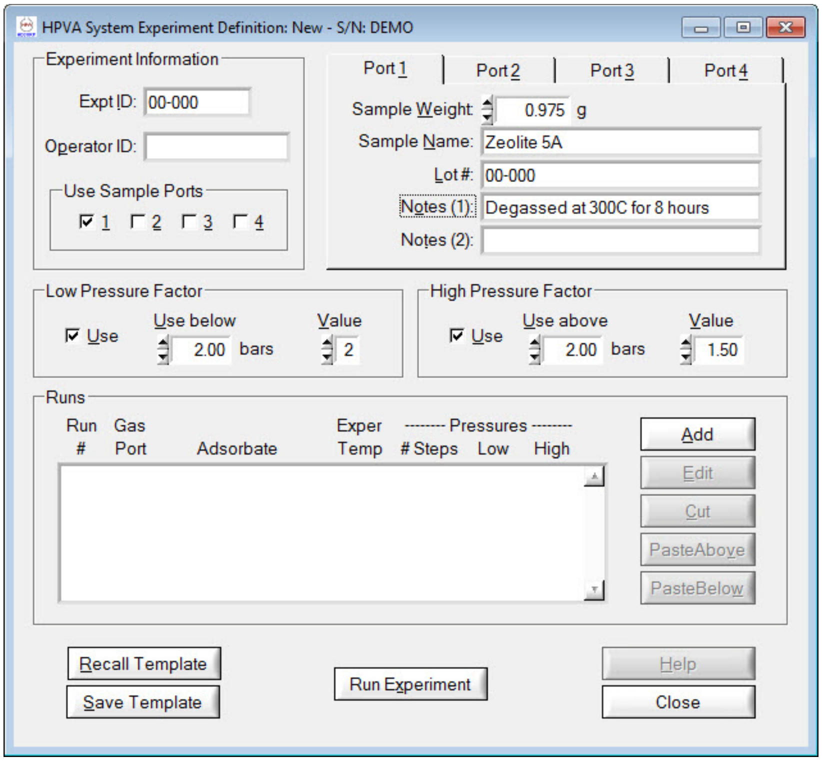
3. Ensure that the O-ring is in place in the analysis port.

4. Tilt the dewar and insert the sample holder.

5. Attach the sample holder to the sample port and hand-tighten the connector nut.

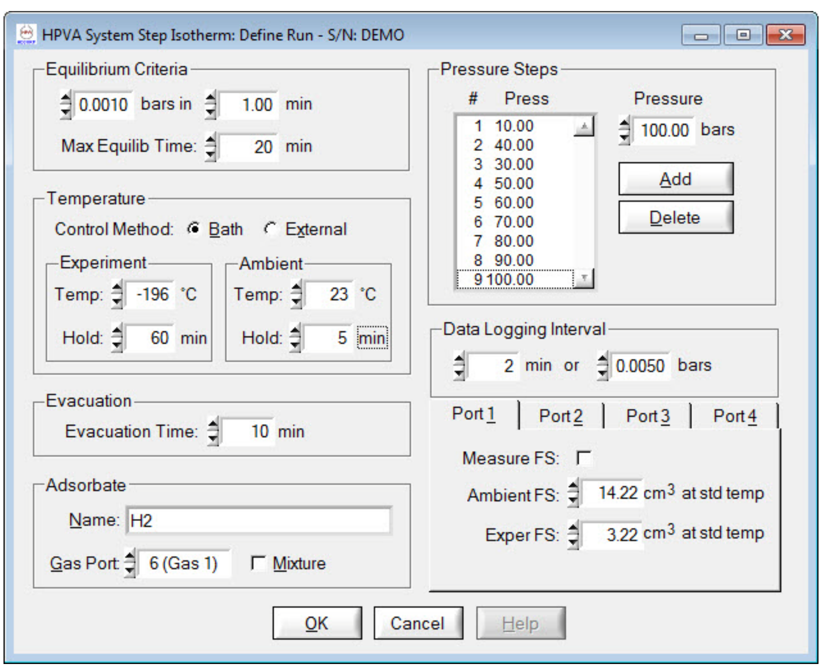
6. Using two wrenches (3/4-in and 5/8-in) tighten the nut just until snug. Note, do not over tighten the connector nut, doing so could damage the port fitting.

7. Select the **Adsorption** option from the HPVA main menu. The Experiment Definition: New window is displayed.



8. Enter information in the fields in this window. (Refer to **Define New Experiment Window,** page **2-27**).

9. Click **Add** to display the Define Run window.

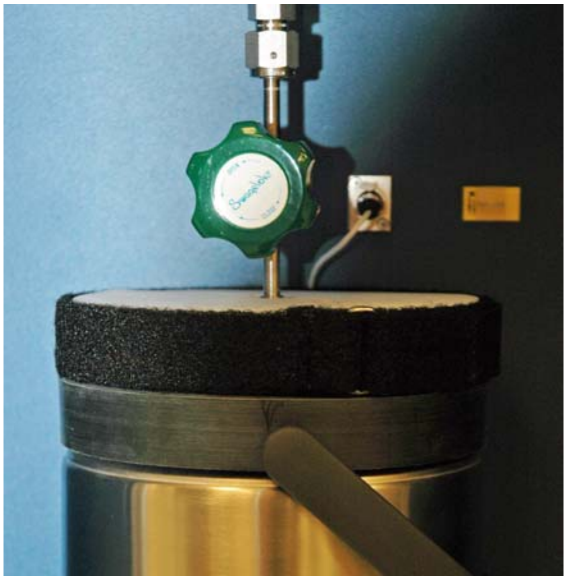


10. Select the **External Temperature Control Method** radio button.
11. Enter the pressure steps that are desired for the sample to reach during analysis:
    a. Enter or select a pressure in the **Pressure** field.
    b. Click **Add** to add the pressure to the table.
12. Enter temperature settings similar to those used for the free space analysis.
13. Uncheck the **Measure FS** box.
14. Enter the Ambient free space from the free space analysis in the **Ambient FS** field.
15. Enter the Experiment free space from the free space analysis in the **Exper FS** field.
16. Set the **Hold** time to about 60 minutes, but no less than 45 minutes. This is necessary because when the warm sample tube is submerged into a liquid cryogen bath, it takes at least 45 minutes for the sample tube to equilibrate and create a stable temperature within the tube.
17. Click **OK**. The Experiment Definition: New window is displayed.
18. Open the sample isolation valve on the sample tube.

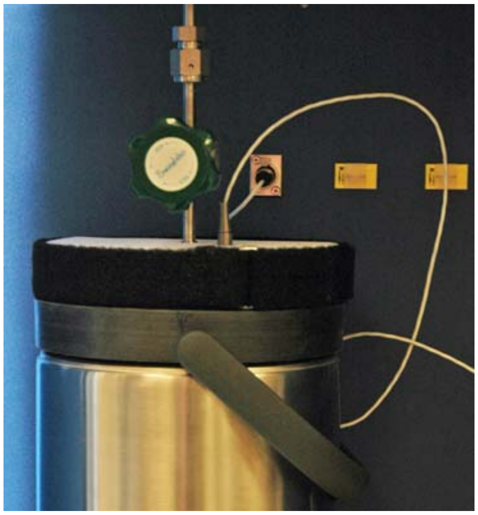
19. Click **Run Experiment**.
20. The software will prompt you to bring the sample tube to the desired analysis temperature.
21. Fill the dewar with liquid nitrogen to the top of the isothermal jacket,

22. Place the dewar cover on top of the dewar.

23. Place the strap around the dewar cover and tighten.



24. Insert the temperature probe into the dewar.



25. Click **OK**.

26. Once the system has reached the desired temperature and the hold time has elapsed, the HPVA will begin the analysis.

27. When the analysis is complete, the results will be available in the Excel HPVA macro as described in the next topic.

28. After the analysis has completed, close the sample isolation valve.

29. Remove the sample from the analysis port and allow it to cool to room temperature.

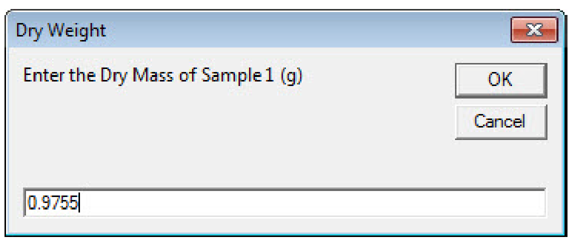
30. The sample container can then be removed from the sample holder and reweighed to find the dry mass of the sample.

Analyzing the Results

1. Open the Excel Macro.
2. Click the Parameter tab if the Parameter page is not already displayed.
3. Make sure the Density Correction is set to TRUE.

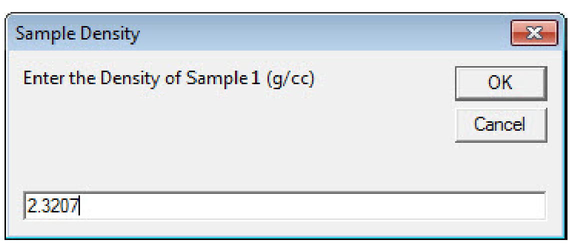
	A	B	C
1	Value	Description	
2	FALSE	Selective Adsorption	
3	FALSE	Set Baseline	
4	FALSE	Cryostat Correction	
5	TRUE	Density Correction	
6			
7	23	Ambient Temp (°C)	
8			

4. Select **Read Short Data File** from the HPVA menu.
5. Select the file short data file (extension HISH) from the analysis you performed, then click **OK.**
6. You will then be prompted to enter the dry mass of the sample.



Enter the mass of the sample after the analysis, then click **OK**.

7. You will then be prompted to enter the sample density.



Enter the sample density, then click **OK**.

Since the macro knows the mass and density of the sample, it simply divides the sample mass by the density to find the physical volume of the sample. This volume is subtracted from the entered ambient and analysis temperature volumes that were entered into the HPVA software prior to analysis. These corrected values are used for the calculations for constructing the volume of gas absorbed at each pressure step of the experiment.

8. A PCT graph of the experiment data is displayed and the Save As dialog is displayed. If you would like to save the data in an Excel spreadsheet, enter a file name or accept the default name, then click **Save**.

9. You can click the isotherm tab to display an isotherm of the experiment data or the file name tab to display experiment data and the volume dosed and volume adsorbed calculations.

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