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ASAP 2050

Extended Pressure Adsorption Analyzer

Operator's Manual

V1.01

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2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.

3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

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1. GENERAL INFORMATION

This manual describes how to install and operate the ASAP 2050 Xtended Pressure Adsorption Analyzer.

Organization of the Manual

The manual is divided into the following chapters:

Chapter 1   **GENERAL INFORMATION**
Provides a general description of the ASAP 2050 system as well as its specifications.

Chapter 2   **USER INTERFACE**
Provides basic instrument and software interface.

Chapter 3   **OPERATIONAL PROCEDURES**
Provides step-by-step instructions for operating the ASAP 2050.

Chapter 4   **SOFTWARE AND SETUP MODIFICATIONS**
Provides instructions for using the 2050 Setup program.

Chapter 5   **FILE MENU**
Provides a description of the commands on the File menu.

Chapter 6   **UNIT MENU**
Provides a description of the commands on the Unit menu.

Chapter 7   **REPORTS MENU**
Provides a description of the commands on the Reports menu and samples of reports.

Chapter 8   **OPTIONS MENU**
Provides a description of the commands on the Options menu.

Chapter 9   **TROUBLESHOOTING AND MAINTENANCE**
Provides instructions for troubleshooting hardware problems and for performing routine maintenance procedures.

Conventions

This manual uses the symbols shown below to identify notes of importance, cautions, and warnings.

Notes contain a tip or important information pertinent to the subject matter.

Cautions contain information to help you prevent actions which could damage the instrument.

Warnings contain information to help you prevent actions which could cause personal injury.

Online Manual

For your convenience, the Operator's Manual is available online. You can access the manual by selecting **Help**, then **Operator's Manual** from the analysis program main menu. The manual appears in an Adobe® Acrobat® Reader®.

Following are some tips to help you quickly locate the information you need in the manual. Refer to the Adobe Acrobat Help system (click the **Help** button on the Acrobat menu) for more information on the Acrobat features you can use while viewing the manual.

Using Bookmarks

Click the **Bookmarks** tab to list and access the topics included in the manual.
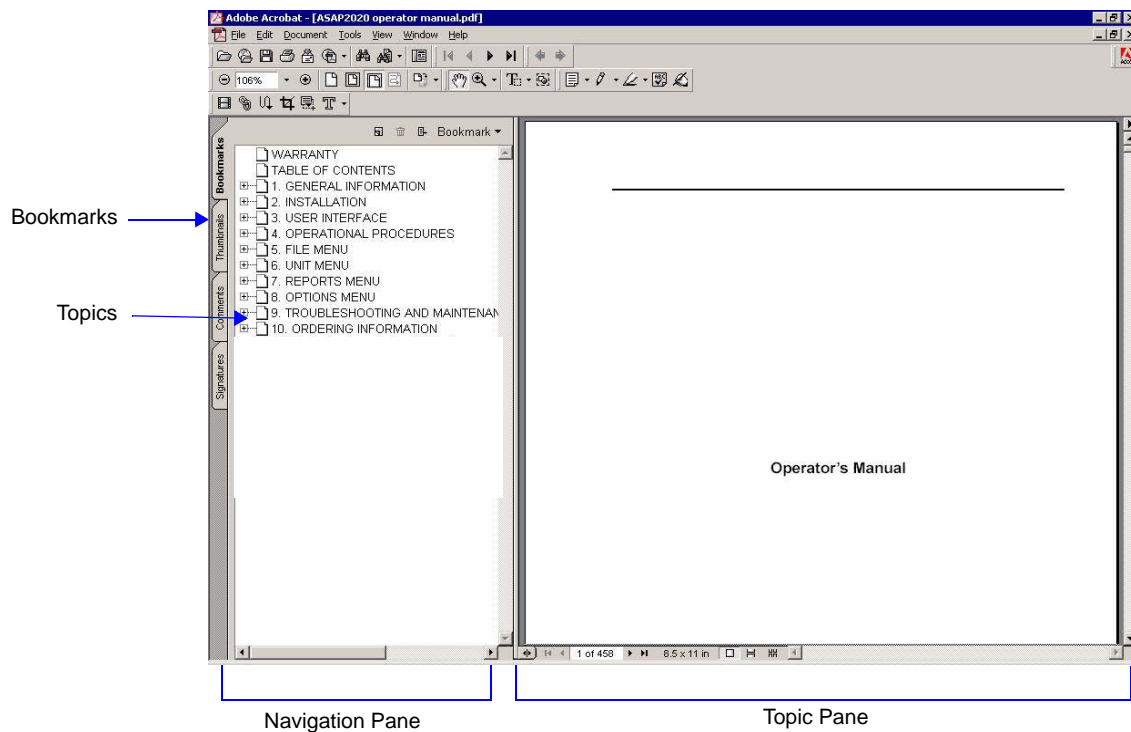
You can use the + and − buttons next to topics as they are used in Windows Explorer to expand or collapse the topic list.

To display a topic, click the topic name in the Bookmarks section. The related information appears in the topic pane of the window as shown in the following example.
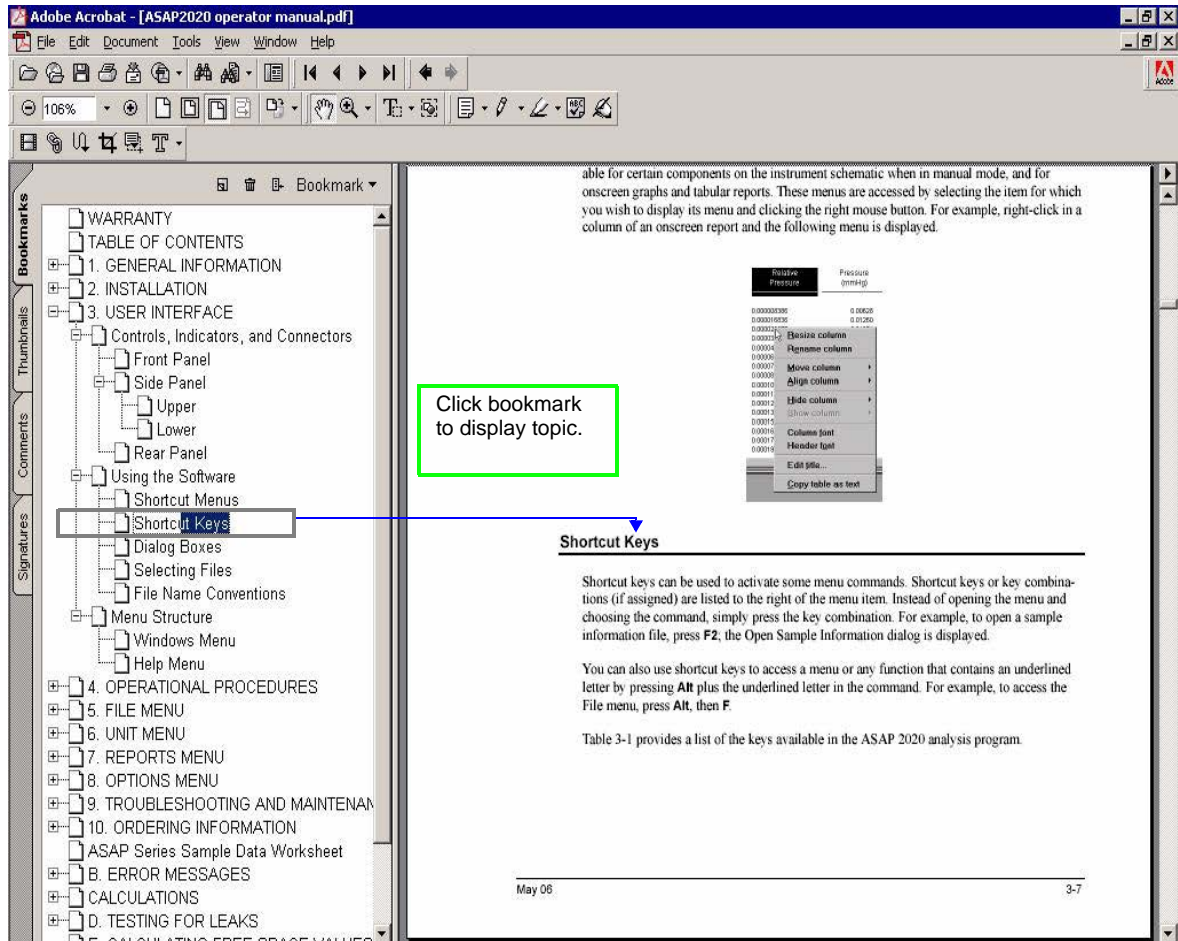
Using the Table of Contents, Index, and other Links

Links provide direct access to selected information. All links appear in blue type. Links are contained in:

• the table of contents
• index entries
• cross-references within the manual

Table of Contents

To display the table of contents, click **Table of Contents** in the **Bookmarks** section. When the table of contents is displayed, you can click an entry to display its associated page. For example, clicking **Using the Software** in the table of contents, displays the page containing information about the software.
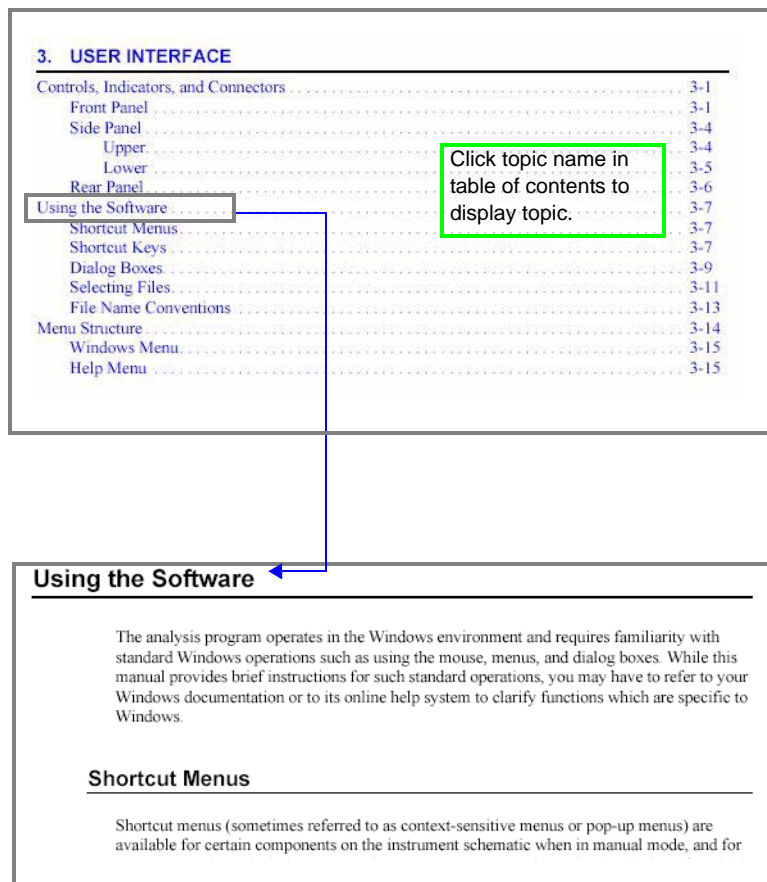
Index

To use the index in the online manual, click the **Bookmarks** tab, scroll down to **INDEX** (the last topic in Bookmarks), then click the + button to expand the index. The letters A through Z are displayed. Click a letter to display its corresponding index entries as shown in the following example.
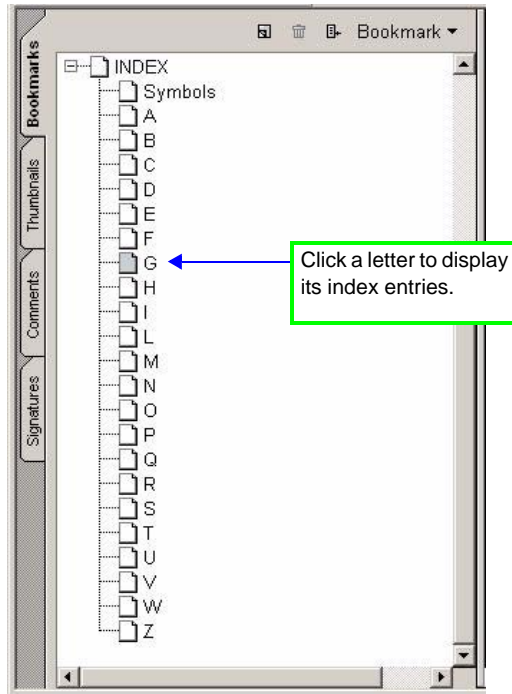
After you display the entries, locate the item of interest and click on the page reference to access the information.

Cross References

Cross-references work in the same manner. In the example below, clicking on the cross-reference, **FILE MENU** (shown on the screen in blue type) will display the first page of the chapter describing the commands found on the File menu.

FILE MENU

Provides a description of the commands available on the File menu.

Using the Find Command

The Adobe Acrobat **Find** command provides another method of easily accessing specific information. For example, suppose you want to know how the **Save as** command works. You could select **Edit > Find** from the Adobe Acrobat menu, then enter **Save as** in the Find dialog. The following example shows the results.
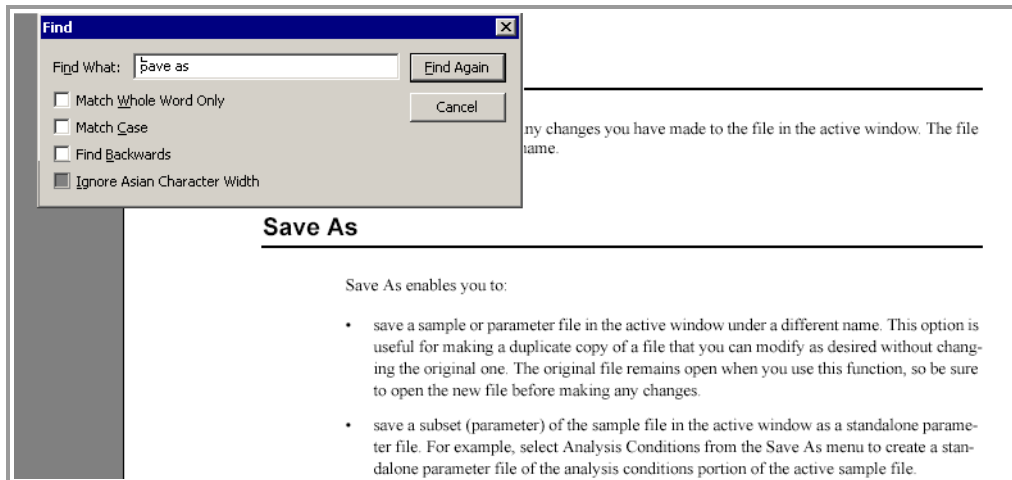
Printing

You can print the entire manual, a selected page, or range of pages. There are several options for printing. You can:

- Select the printer icon (🖨) on the Adobe Acrobat toolbar.

  A standard Print dialog is displayed. Select the page(s) to print, then click **OK**. When using this option (or the next one), be sure to enter the page number(s) displayed in Adobe Acrobat; do not use the page number(s) listed in the footer(s) of the manual.

Enter this number.

Do not use number in footer of the manual.
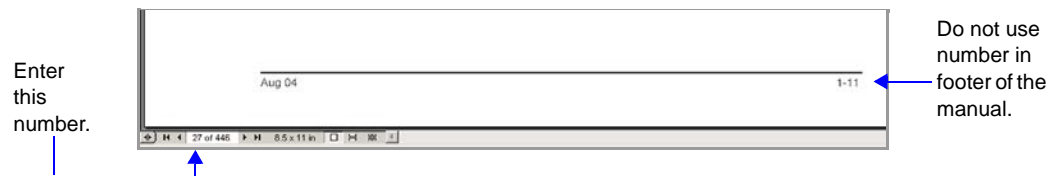
- Select **File > Print**.

  A standard Print dialog is displayed. Select the page(s) to print, then click **OK**.

- Click the **Thumbnails** tab.

  Thumbnails of manual pages are displayed.

  a. Click the pages you want to print.

  b. Right-click to display a shortcut menu, then select **Print Pages**.

  c. A standard Print dialog is displayed; click **OK**.

Thumbnails tab
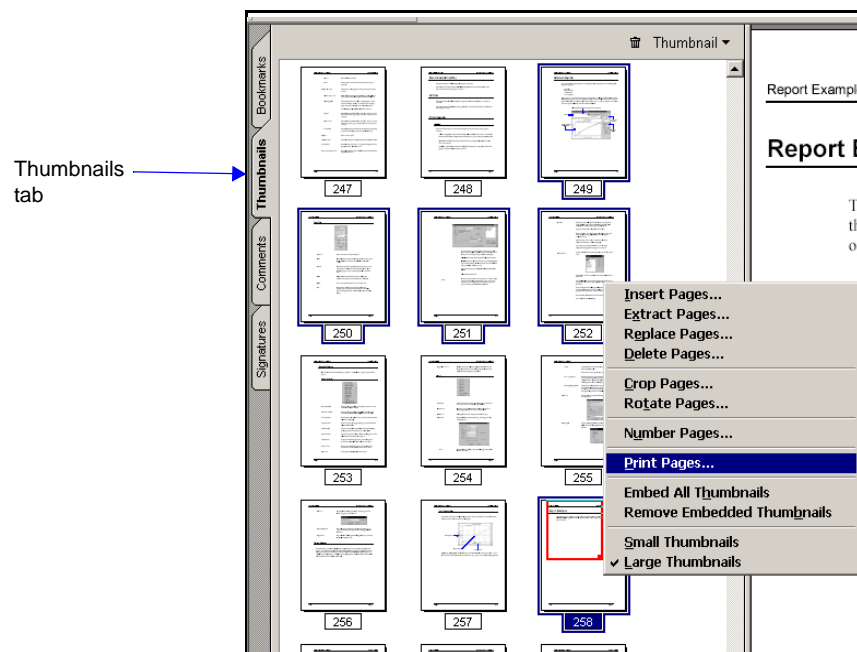
Internet Access

Visit **www.micromeritics.com** to learn more about Micromeritics, our products, and applications. Our site is user-friendly, easy to navigate, and informative. Its content is summarized below.

About Micromeritics A brief history of Micromeritics, office locations, awards/certifications, career opportunities, and a virtual tour of its headquarters

Products Product information and printable brochures

Applications Application Notes, Product Bulletins, Tech Tips, Technical Articles/papers, and important application links

Online Catalog Catalog of instruments and accessories, allowing you to place your order online

News and Press Press releases, Events calendar, microReports, and latest Micromeritics news updates

Lab Service Provides laboratory tips and access to the Micromeritics Analytical Services web site

Customer Support Customer support contacts, product registration, instrument training information, Material Safety Data Sheets, and account registration

Grant Program Details of the Grant Program established for non-profit organizations and universities

Contact Us Contact information, office locations, maps and driving directions to the Micromeritics facility, and registration for the microReport newsletter

Be sure to browse our site to see the many ways in which we can assist you.

Equipment Description

> ⚠ **The ASAP 2050 can operate in a pressure range from 0 to 10 atm; therefore, ensure that the sample tube is not under pressure when removing it from the instrument. Use caution appropriate for these types of devices.**

The ASAP 2050 analyzer is equipped with two independent vacuum systems — one for sample preparation and one for sample analysis. Having two separate systems, as well as separate preparation ports, allows sample preparation and sample analysis to occur concurrently without interruption. Inline cold traps are located between the vacuum pump and the manifold in both the analysis and the degas systems. The sample saturation pressure (Psat) tube is located next to the sample analysis port. Gas inlet ports and cable connections are located conveniently on the side panel of the analyzer for easy access.

The ASAP 2050 is equipped with an elevator that raises and lowers the analysis bath fluid Dewar automatically. A removable shield to enclose the Dewar is also included for safety purposes.

The ASAP 2050 system includes Micromeritics' patented Isothermal Jackets for the sample tube. The Isothermal Jacket maintains a stable thermal profile along the full length of the sample and Psat tubes.

Gas Requirements

Compressed gases are required for analyses performed by the ASAP 2050 analyzer. Gas bottles or an outlet from a central source should be located near the analyzer.

Appropriate two-stage regulators which have been leak-checked and specially cleaned are required. Gas regulators are available from Micromeritics; refer to **Ordering Information**, page **10-1**.

Gases that be used with the ASAP 2050 include argon, carbon dioxide, nitrogen, hydrogen, oxygen (with oxygen-compatible vacuum system), and other suitable gases

In order to use oxygen with the ASAP 2050, your 2050 analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin® (or a suitable equivalent) pump oil. Failure to use the proper vacuum system could result in hazardous conditions including fire and personal injury.

Analysis Program

The ASAP 2050 analysis program is designed to operate in a Windows 2000 or Windows XP Professional environment. The Windows environment provides a user-friendly interface for performing analyses and generating reports.

The ASAP 2050 System software monitors and controls the analyzer. It enables you to perform automatic analyses with just a few key strokes, and collects and reports analysis data. You can choose from a variety of reports, which can be printed automatically after an analysis and/or stored and printed later.

Report System

The ASAP 2050 software includes a report system which allows you to manipulate and customize reports. You can zoom in on portions of the graphs or shift the axes to examine fine details. Scalable graphs can be copied to the clipboard and pasted into other applications. Reports can be customized with your choice of fonts and a company logo added to the report header for an impressive presentation. Refer to **"REPORTS MENU" on page 7-1** for the options available for reports.

Specifications

The ASAP 2050 system has been designed and tested to meet the specifications provided below.

Characteristic	Specification
──────── **UTILITY REQUIREMENTS** ────────	
Electrical:	100/115/230 VAC; 50 or 60 Hz, 800 VA, exclusive of vacuum pumps, which are powered separately
──────── **Operating Environment** ────────	
Temperature:	10 to 35 °C operating - 10 to 55 °C storage or shipping
Humidity:	Up to 90% (non-condensing) for instrument 20% to 80% for computer system
──────── **PRESSURE MEASUREMENT** ────────	
Range:	0 to 10 atm
Resolution:	
Analysis transducer	0.00015 psi
Accuracy (Analysis system only):	0.1% BFSL (Best Fit Straight Line)
Includes nonlinearity, hysteresis, and nonrepeatability. Transducer manufacturer's specifications.	
──────── **VACUUM SYSTEM** ────────	
Vacuum Pump:	Mechanical, two-stage, ultimate vacuum 5 x 10^{-3} mmHg; 2 pumps (one analysis and one degas)

Characteristic	Specification

————— MANIFOLD TEMPERATURE TRANSDUCER —————

Type: Platinum resistance device (RTD)

Accuracy: $\pm$ 0.02 ^{o}C (by keyboard entry)

Stability: +0.1 ^{o}C per month

————— DEGAS SYSTEM —————

Temperature Range: Ambient to 450 ^{o}C

Selection: 1 ^{o}C increments

Accuracy: Deviation less than $\pm$10 ^{o}C of set point at thermocouple

Backfill Gas: User-selectable, typically helium or nitrogen

————— SYSTEM CAPACITY —————

Sample Preparation: 2 degas ports, each with independently-controlled heating mantle, using servo-controlled restricted evacuation

Analysis: 1 sample port; 1 saturation pressure tube port

Total Operating Capacity: Up to two complete analysis units can be controlled independently by one computer

————— CRYOGEN SYSTEM —————

Special Features: Patented Isothermal Jackets effectively maintain cryogen level constant on sample tube and Po tube during analysis while evaporation of cryogen occurs

Capacity: 3-Liter Dewar, which provides at least 50 hours of unattended analysis.

Analysis Time: Unlimited. Dewars may be refilled without affecting the accuracy of results.

Characteristic	Specification

—————— **SAMPLE SIZE** ——————

1/2-inch straight-wall 316 stainless steel sample tubes.

—————— **GASES** ——————

Normal: Argon, carbon dioxide, nitrogen, hydrogen
                               oxygen, and other suitable gases

—————— **PHYSICAL** ——————

Height: 99 cm (39 in.)

Width: 85 cm (33.5 in.)

Depth: 61 cm (24 in.)

Weight: 115 kg (250 lbs)

—————— **COMPUTER** ——————

Minimum requirements: Pentium 333 MHz computer (or equivalent)
                               CD-ROM drive
                               128 megabytes of main memory
                               1-gigabyte hard drive
                               SVGA monitor (800 x 600 minimum
                               resolution)
                               RS232 port
                               Windows 2000 or Windows XP Professional

2. USER INTERFACE

This chapter contains information to familiarize you with the hardware and software of the ASAP 2050 system. It is recommended that you read this chapter before attempting to operate the ASAP 2050 system.

Controls, Indicators, and Connectors

This section contains a description of the controls, indicators, and connectors located on the front, side, and rear panels of the ASAP 2050 system.

Front Panel

Power indicator light

Vacuum pump panel

Elevator
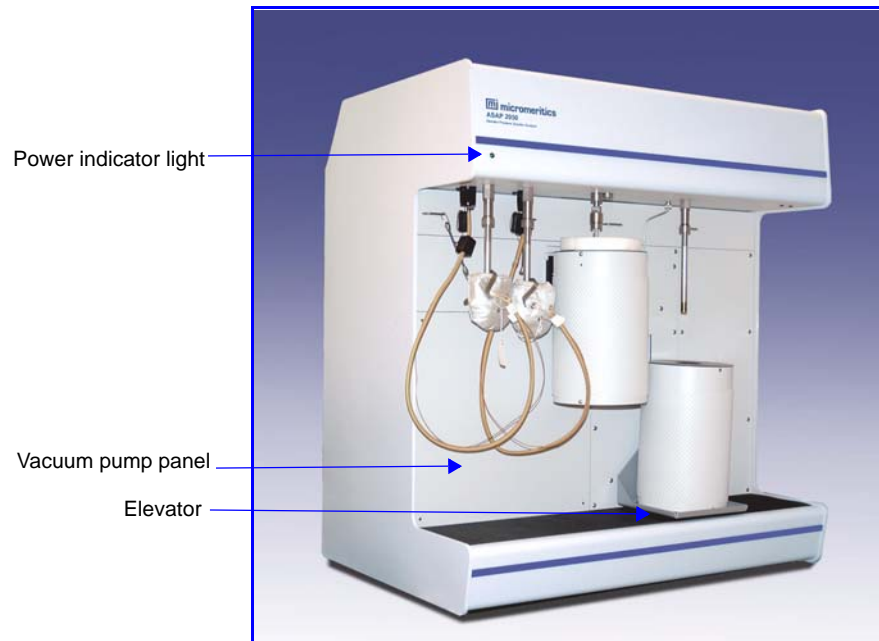
Green power indicator light   Illuminated when power is applied to the analyzer.

Vacuum pump panel   Allows access to the vacuum pumps. Remove this panel when you need to service the pumps.

It is not always necessary to remove the panel to inspect the condition of the fluid or the fluid level. To accomplish either of these tasks, remove (or fold over to the right) the protective rubber mat and lift off the metal cover; this exposes the vacuum pump sight window.

Drain   Vacuum pump
sight window
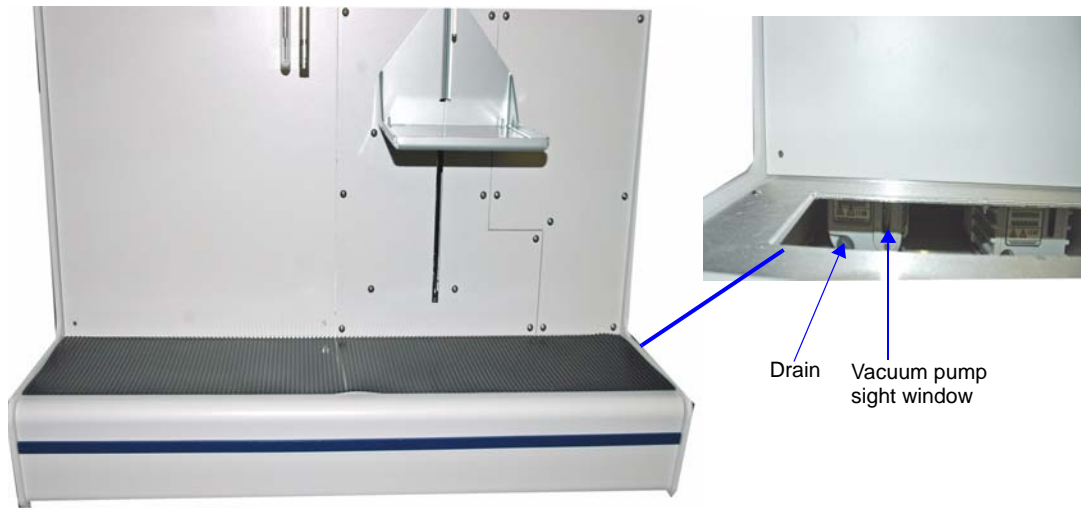
Vacuum pump sight window	The vacuum pump sight windows enable you to inspect the oil levels in the degas and analysis pumps.
Vacuum pump drain	Provides a convenient method of draining fluid from the pump when service is required. (Pump must be removed from analyzer in order to drain.)
Elevator	Allows placement of the Dewar around the sample tube.

The components listed here are located on the underside of the upper extension of the front panel.
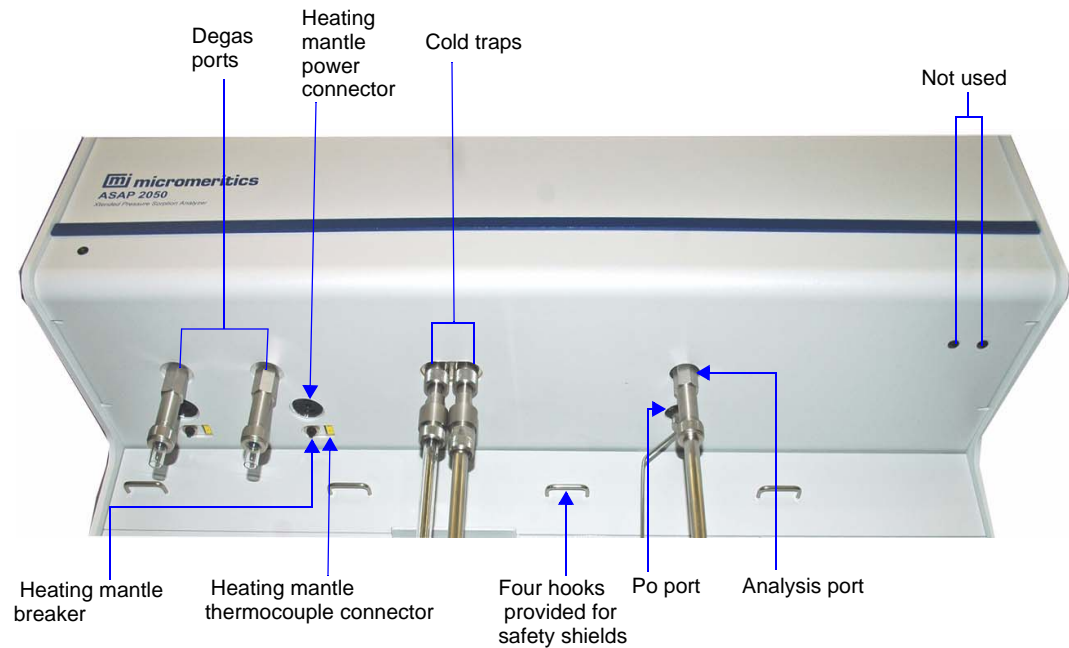
Degas ports	Allow you to degas up to two samples. Each degas port has connections for a heating mantle.
Heating mantle thermocouple connector	Allows connection of a heating mantle thermocouple (one for each degas port).
Heating mantle power connector	Allows connection of the heating mantle power cord (one for each heating mantle).
Heating mantle breaker	Protects the circuitry for the heating mantle in the event of a failure (one for each heating mantle). If the circuit breaker trips (pops out), call your Micromeritics service representative.
Cold Traps	Two cold traps are provided; one for degassing and one for analysis.
Analysis port	For installing the sample tube containing the material you wish to analyze.
Po port	For installing a Po (saturation pressure) tube.

Side Panel

Upper
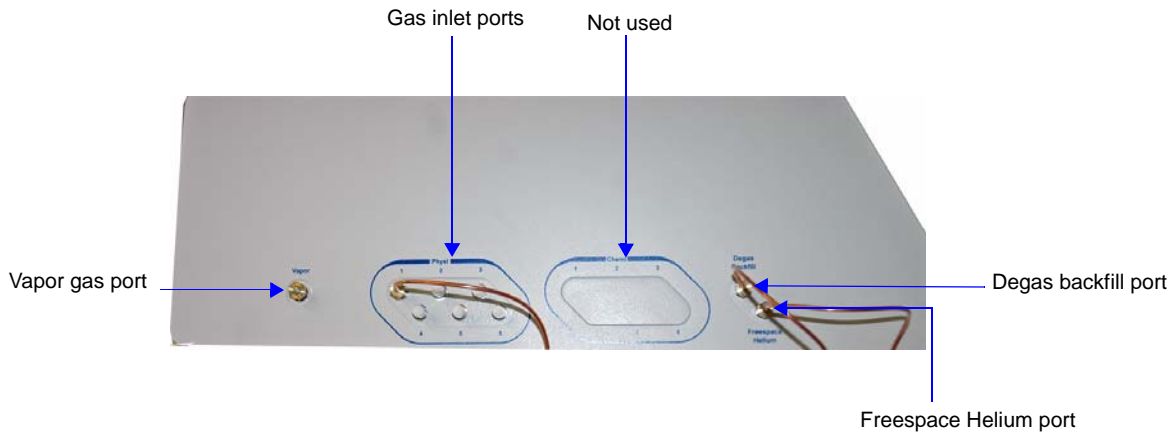
Gas inlet ports	Used to connect gas supplies to the analyzer.
Vapor gas port	For attaching the water vapor option, or connecting a vapor gas. Refer to **Ordering Information** on page **10-1** for the part number of the water vapor option.
Degas Backfill port	Allows connection of a gas to use for degassing the sample.
Freespace Helium port	Allows connection of helium to use for measuring the free space.

Lower
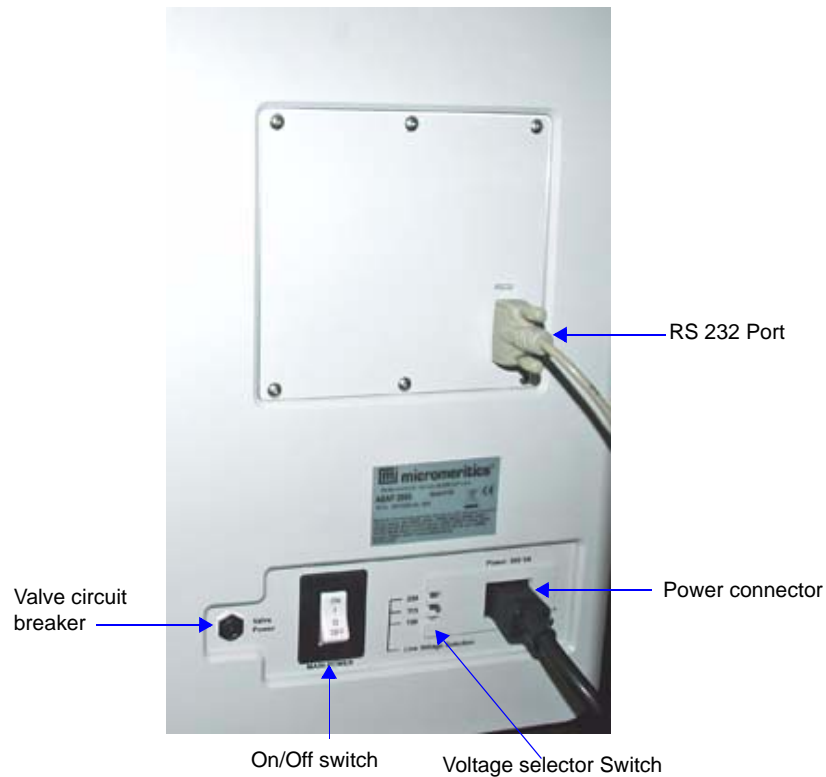
| On/Off switch | For turning the analyzer on and off. This switch also serves as the main breaker for the analyzer; it switches off automatically in the event of an electrical fault. |

On/Off switch

For turning the analyzer on and off. This switch also serves as the main breaker for the analyzer; it switches off automatically in the event of an electrical fault.

Power connector

For connecting the analyzer to the electrical supply.

RS232 port

For connecting the analyzer to a computer.

Valve circuit breaker

Protects the circuitry for the valves in the event of a failure. If the circuit breaker trips (pops out), call your Micromeritics service representative.

Voltage selector switch

For setting the analyzer to the correct incoming AC line voltage.

Rear Panel

Vacuum pump
location
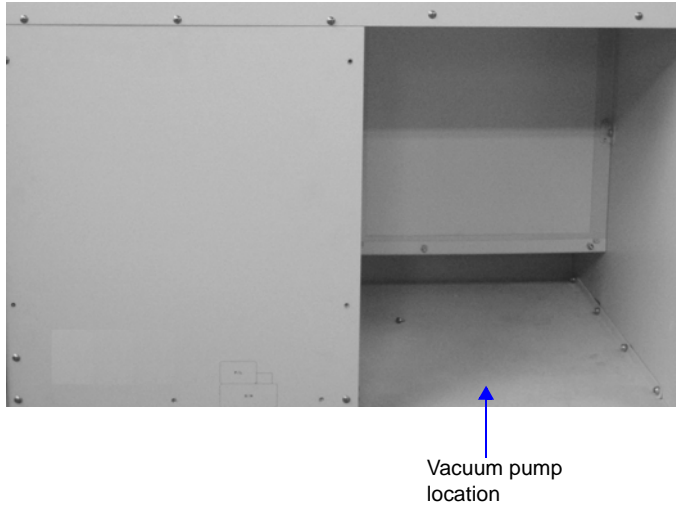
Vacuum pump recess   Provides for placement of the vacuum pumps. Vacuum
pumps can be serviced from the front of the instrument by
removing the vacuum pump panel located on the lower left
side of the front panel (see **Front Panel** described
previously).

Turning On the System

1. Place the ON/OFF switches for the computer and all peripheral devices in the ON
   position.

2. Place the analyzer ON/OFF switch in the ON position; verify that the green power
   indicator on the front panel is illuminated.

Turning Off the System

⚠ **Always exit the ASAP 2050 program and/or Windows before turning off the computer.
Failure to do so could result in loss of data.**

1. Select **Close** from the System Menu or **Exit** from the File menu.

2. If you exit the ASAP 2050 program with analyses in progress, you will be warned of the
   operation. If desired, you can continue with exiting the application and the analyses will
   proceed and continue to collect data. Reports that are queued under the Print Manager will

print. If, however, a power failure occurs and an uninterruptible power supply (UPS) is not attached to the computer, the data collected after exiting the ASAP 2050 System program are lost.

3. Place the computer, monitor, and printer ON/OFF switches in the OFF position.

4. Place the analyzer Main Power switch in the OFF position.

Using the Software

The analysis program operates in the Window^{TM} environment and requires familiarity with standard Windows operations such as using the mouse, menus, and dialogs. While this manual provides brief instructions for such standard operations, you may have to refer to your Windows documentation or to its online help system to clarify functions which are specific to Windows.

Shortcut Menus

Shortcut menus (sometimes referred to as *context-sensitive* menus or *pop-up* menus) are available for certain components on the instrument schematic when in manual mode, and for onscreen graphs and tabular reports. These menus are accessed by selecting the item for which you wish to display its menu and clicking the right mouse button. For example, right-click in a column of an onscreen report and the following menu is displayed.
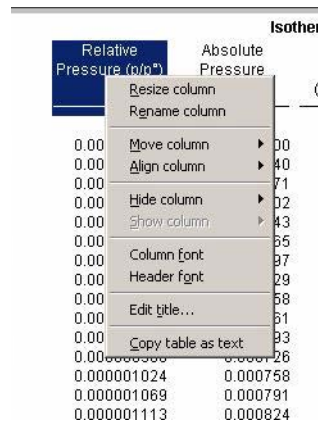
Shortcut Keys

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (if assigned) are listed to the right of the menu item. Instead of opening the menu and choosing the command, simply press the key combination. For example, to open a sample information file, press **F2**; the Open Sample Information dialog is displayed.

You can also use shortcut keys to access a menu or any function that contains an underlined

letter by pressing **Alt** plus the underlined letter in the command. For example, to access the File menu, press **Alt**, then **F**.

Table 2-1 provides a list of the keys available in the ASAP 2050 analysis program.

Table 2-1. Shortcut Keys

Key(s)	Function
F1	Access online operator's manual
F2	Open a sample information file
	Clear the field of existing date (Select Dates dialog)
F3	Open an analysis conditions file
	Insert the current date (Select Dates dialog)
F4	Open an adsorptive properties file
	Display a calendar from which to choose a date (Select Dates dialog)
F5	Open a report options file
F6	Tile open windows
F7	Cascade open windows
F8	Start report
F9	Close all open reports
F10	Generate a Heat of Adsorption report
Alt + F4	Exit the ASAP 2050 program
Shift + F2	List sample information files
Shift + F3	List analysis conditions files
Shift + F4	List adsorptive properties files
Shift + F5	List report options files
Shift + F9	Access shortcut menu of (1) selected component on instrument schematic, or (2) onscreen reports

Dialog Boxes

Dialog boxes (referred to as dialogs) are displayed when a menu item followed by an ellipsis (...) is selected. Subdialog boxes are displayed when certain push buttons are selected. Both types of boxes may contain one or more of the items listed below.
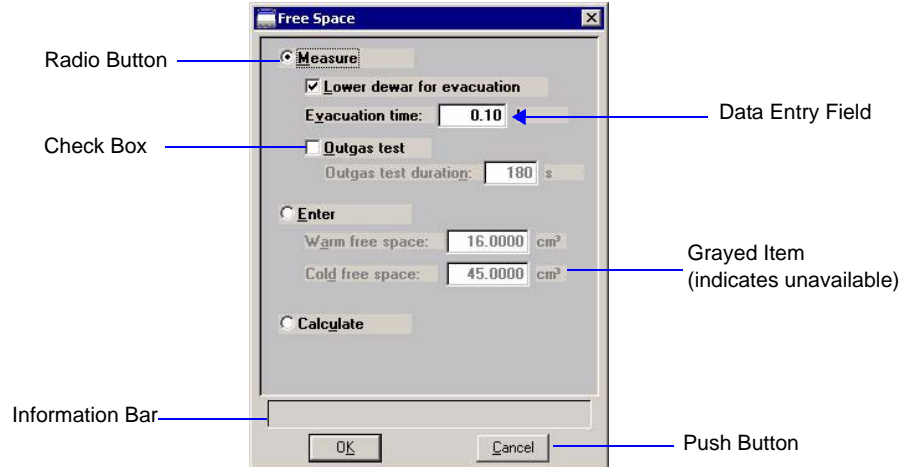
The following describes elements that are often included in dialogs. If an element is shown in gray instead of black, the element is currently unavailable.

Data Entry Fields	A Data Entry field is used to enter text; either numeric (numbers only) or alphanumeric (numbers, letters, or printable characters).
	If an invalid entry is made, an error message is displayed, for example, if you attempt to enter text in a numeric field or a number outside of the range shown in the information bar.
Information Bar	Some dialogs contain information pertinent to the selected field in an information bar across the bottom of the dialog. For example, a range is shown for fields in which numeric entries are required.
Radio buttons	Radio buttons are provided in groups of two or more and are used to make a choice; only one radio button can be selected. Simply click the desired option with your mouse pointer. A black dot indicates the item is selected.
Check boxes	Check boxes allow you to choose multiple options from a group of options. Click the desired option(s) with your mouse pointer. An **X** in the box indicates the item is selected; remove the **X** in the same manner.

Push Buttons   A push button is used to display a subdialog box in which to enter additional information about the subject matter, or to invoke an action.

For example, if you click **Date Range** on the Open Sample Information dialog, the Select Dates subdialog is displayed. If you click **Cancel** on the Open Sample Information dialog, you invoke the action of canceling and closing the dialog.

Close   Closes the active dialog. If the dialog contains unsaved changes, you will be prompted to save them before the dialog closes.

Any information entered in *subdialog*s (refer to the next section of this chapter), is discarded also.

Save   Saves the information entered in the current session; the dialog remains open.

Replace   Allows you to replace the contents of the current file with those from an existing file. For example, if you are creating an analysis conditions file, you can save time by clicking and choosing the file containing the values you wish to use. These values are copied into the current file automatically. And since the values are actually just copied into the file, you can edit them in any way you wish. The file from which they were copied remains intact and ready for the next use.

Cancel   Discards everything you entered in the dialog and any subdialogs, and closes the dialog. A warning message is displayed before closing.

Drop-down list   A drop-down list contains a list of options and is indicated by a down arrow to the right of the field. If there are more items than can fit in the box, a scroll bar is provided for navigating through the list.

Selecting Files

Sample information is stored in files and saved under file names. Certain dialogs contain a **Files** list box which displays a list of files available for that particular operation. For example, the Open Sample Information File dialog.

The Status drop-down list does not appear on the Open dialog for parameter files.

Displays all files of the type in the **Status** field.

Displays the current directory.
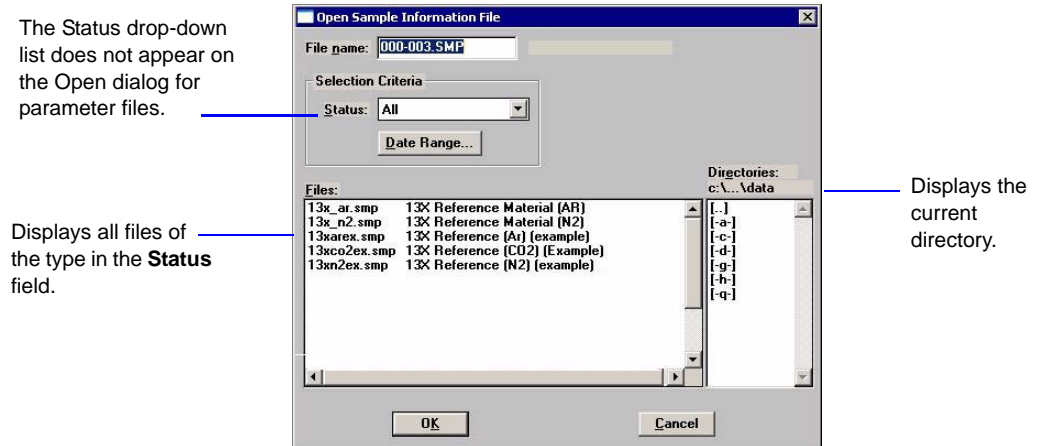
A default string appears in the **File name** field. To select a file, simply move the mouse pointer to the desired file in the list and double-click.

You can limit the list of files displayed by choosing one or more of the following options:

- Use wildcard characters in the path name entered in the **File name** field. Standard and wildcard characters (such as ***** and **?**) can be used to filter file names. For example, you could limit the list of files shown in the dialog above to those beginning with **13x** by entering **13x*.SMP** in the file name field.

- Enter a range of dates. Click **Date Range**; the Select Dates dialog is displayed.

Select **Show Date Range** to enable the **From** and **To** fields, then enter a beginning and ending date. Or you can double-click in each field to display a calendar to select a date. The range of dates remains the default until you change the dates or select **Show All Dates**.

For convenience, the following shortcut keys are available when the Select Dates dialog is displayed:

F2   Clears the date
F3   Inserts the current date
F4   Displays a calendar from which you may select a date

• Select a file status from the **Status** drop-down list. For example, choose **Complete** from the drop-down list and only files that have been used in an analysis are displayed. The table shown below describes each file status.

> **The Status drop-down list does not appear on the File > Open dialogs for parameter files.**

Table 2-2. File Status and Description

Status	Description
All	All sample information files in the specified directory and within the specified range of dates
Analyzing	Sample information files that are currently being used with an analysis
Complete	Sample information files that have been used with an analysis
Entered	Sample information files that contain manually entered data
No analysis	Sample information files that have not been used in a degassing operation or an analysis
Prepared	Sample information files that have been used in an automatic degassing operation and are ready for analysis
Preparing	Sample information files that are currently being used in an automatic degassing operation

• Navigate to a different directory. The current directory is displayed just above the **Directories** list box. You can change directories by double-clicking a directory in the **Directories** list box, double-clicking **[..]** to move up one level, or by entering the desired directory in the **File Name** field. For example, enter C:\2050files\sample*.smp to display sample files in the **2050files\sample** directory on your local drive.

File Name Conventions

For sample information and parameter files, a wildcard character (*) and a default extension display.

The following table shows the file name extensions for the ASAP 2050 program.

Table 2-3. Default File Name Extensions

File Type	Extension
Sample Information	SMP
Sample Tube	STB
Degas Conditions	DEG
Analysis Conditions	ANC
Report Options	RPO
Adsorptive Properties	ADP
Export to disk (ASCII)	ISO
Report to disk	RPT
List to disk	LST
Thickness curve	THK
Alpha-s curve	ALS
The following types are available for reports saved from the Report window	
Report	REP
Spreadsheet	XLS
ASCII	TXT

Menu Structure

All functions for the ASAP 2050 are located on menus which are accessed from the Menu bar. Each menu contains commands, and in some cases a submenu. A submenu is indicated when the command is followed by an arrow.
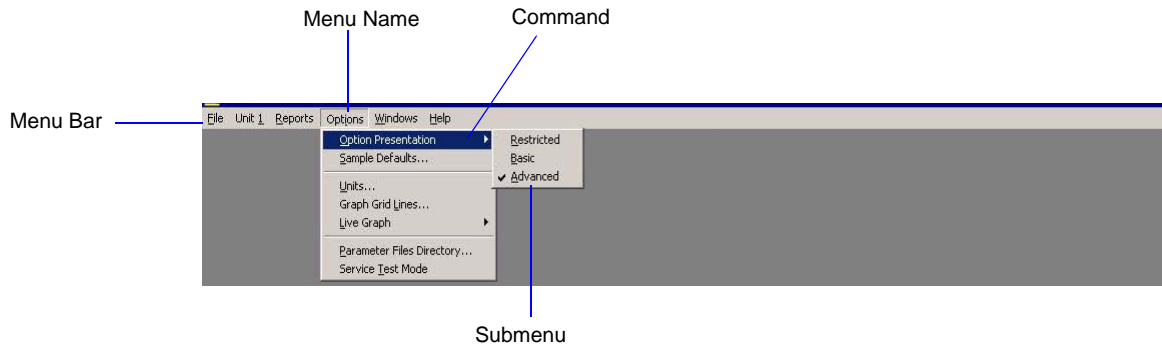
Brief descriptions of each menu are provided below; refer to the chapter given in parentheses for a detailed description of the commands contained on that menu.

File   Enables you to maintain system files. (Chapter 5, **FILE MENU**)

Unit [n]   Enables you to perform analyses and other instrument operations. (Chapter 6, **UNIT MENU**)

Reports   Enables you to generate, customize, and close reports. (Chapter 7, **REPORTS MENU**)

Options   Enables you to select data presentation formats, and enter system default values. (Chapter 8, **OPTIONS MENU**)

Windows   Enables you to arrange the windows and icons on your screen. It also displays the names of all open files. (this chapter, page **2-15**)

Help   Displays Help information. (this chapter, page **2-15**)

Windows Menu
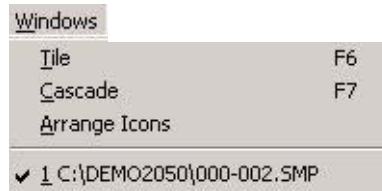
Tile   Resizes all open windows and arranges them side by side so
                                  that the contents of all open windows are visible.

Cascade   Resizes all open windows and arranges them in a stacked
                                  fashion. The active window is positioned on top of the stack.
                                  Each window's title remains visible, making it easy to select
                                  other windows.

Arrange Icons   Arranges the symbols for all minimized windows in an
                                  orderly manner.

Open file names   Displays all open files; the active window is preceded with a
                                  check mark.

Help Menu
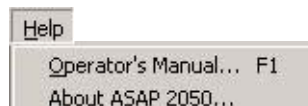
Operator's Manual   Displays the operator's manual in PDF format.

About ASAP 2050   Displays information about the ASAP 2050 analysis
                                  program.

3. OPERATIONAL PROCEDURES

This chapter contains brief step-by-step instructions on how to:

- specify sample file defaults, this page
- create sample information files, page **3-6**
- define parameter files, page **3-9**
- prepare the sample page, **3-16**
- perform an analysis page, **3-37**
- generate a list of information on a sample or parameter file, page **3-40**
- print contents of a sample or parameter file, page **3-39**
- export data in a sample information file to an ASCII file, page **3-41**
- overlay graphs, page **3-42**

This chapter does not contain detailed descriptions of the dialogs used to perform these procedures. Refer to Chapters 5 through 8 for dialog descriptions. Use the index to assist you in locating the appropriate dialog.

Sample Information Files

Sample information files contain information used to control the analysis. Therefore, an analysis cannot proceed until it has been assigned a sample information file. A sample information file consists of the following:

- sample identification
- sample tube criteria
- analysis conditions
- adsorptive properties
- report options
- collected/entered data (after an analysis has been completed)

Portions of the sample information file can also exist as standalone parameter files. Having these files exist independently allows you to use them as many times as you wish. For example, if you typically use the same analysis conditions for many of your analyses, you can create an analysis conditions file containing the desired conditions. Then when you create your sample file, select that file for your analysis conditions; the values will be copied into the current sample file. After it becomes part of the new sample file, you can edit it as desired. The file from which the values were copied remains intact and ready for the next use.

Sample information files reside in a folder. The more files in a folder, the longer it may take to access a file. Therefore, it is a good practice to limit the number of files in a folder to approximately 200. You may create additional folders as needed.

Sample information files can be created and presented in the Advanced, Basic, or Restricted format.

- **Advanced**

  Presents all parts of the sample information file in a tabbed dialog. Each tab opens its associated dialog, allowing you to edit conditions. You can also switch to the Basic format if desired.

- **Basic**

  Presents all parts of the sample information file as a single dialog. This format allows you to quickly create a sample information file using predefined parameter files. You can also switch to the Advanced format if desired.

- **Restricted**

  Identical to the Basic format except that you cannot switch to the Advanced format for editing; certain functions are also disabled.

Specifying Sample File Defaults

You can specify sample defaults in the Basic or Advanced format. The defaults you specify are the ones you see when you create a new sample file in the respective format. Therefore, it is best to specify (or enter) parameters that you plan to use most frequently. For example; specify defaults for your most commonly analyzed sample material. You can always edit parameters in the sample file when it is created. The ASAP 2050 system automatically generates sample information file names and uses the values you specify as the defaults.

Basic Format

Perform the following steps to define defaults for a sample information file in the Basic format.

> **Select *Option Presentation* on the Options menu and ensure that Basic is selected as the format.**

1. Select **Options** > **Sample Defaults**; the default Basic Sample Information dialog is displayed.

2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File > Open > Sample information**. You can use up to eight characters.

3. In the field on the right of the **Sample** line, enter a format for the sample identification. Be sure to include the $ symbol if you want the sample file number included as part of the identification. You can use up to 42 alphanumeric characters.

> **You also can edit the word Sample. For example, you may prefer to use *Material* or *Test*. You can enter up to 15 characters in this field.**

4. Enter a default value in the **Mass** field. An approximate weight is sufficient; a more accurate weight can be entered later.

5. The sample density is used when free space is calculated; enter a default for your sample's density if you typically use a calculated free space. Otherwise, this value is ignored.

6. Select the down arrow to the right of the parameter fields to choose default parameter files:

    • Sample tube
    • Degas conditions
    • Analysis conditions
    • Adsorptive properties
    • Report options

7. Click **Save**, then **Close.**

Advanced Format

The Advanced Sample Defaults dialog resembles a set of index cards. You can move from one set of parameters to another by clicking the parameter tab or by using **Next** and **Prev**. The values you specify in the parameter portions of the sample file (Analysis Conditions, Degas Conditions, Adsorptive Properties, and Report Options) are saved as the defaults for newly created parameter files.

For example, after specifying defaults for a sample file in the Advanced format:

- Select **File > Open > Sample Information**, **Yes** to create the file, and the defaults you specify display for all parameters.

- Select **File > Open > Analysis Conditions**, enter a name, then **Yes** to create the file, and the defaults you specify in the Analysis Conditions portion of the Advanced Sample Defaults dialog display in the fields.

> **Select *Option Presentation* on the Options menu and ensure that *Advanced* is selected as the format.**

1. Select **Options** > **Sample Defaults**, the Advanced Sample Defaults dialog is displayed.

2. In the **Sequence** field, specify a default string for the sample file number; you can use up to eight characters. This is the number that appears in the File name field when you select **File > Open > Sample information**.

3.   In the right-side field of the **Sample** line, enter a format for the sample's identification. You can enter up to 42 characters. Be sure to include the **$** symbol if you want the sample file number (Sequence) included as part of the identification.

4.   Edit the **Operator** and **Submitter** lines as desired. Or have them omitted entirely by selecting **Omit**.

5.   Enter a default value in the **Mass** field. An approximation in this field will suffice; a more accurate value for the sample mass can be entered at the time of analysis.

6.   If you plan to use a calculated free space, enter the sample's density in the **Density** field.

7.   Choose whether you wish to have data collected automatically or if you plan to enter the data. This option can be changed (if desired) at the time of analysis.

8.   Click the **Sample Tube** tab. Enter criteria for the sample tube you most commonly use, then click **Save**.

You can click Save on each dialog as you specify parameters, or you can click Save on any dialog after all parameters have been specified. All dialog defaults will be saved.

---

9.   Click the **Degas Conditions** tab. Specify degassing criteria, then click **Save**.

10. Click the **Analysis Conditions** tab. Choose the analysis conditions appropriate for your most commonly analyzed material, then click **Save**.

11. Click the **Adsorptive Properties** tab. Specify gas characteristics, then click **Save**.

12. Click the **Report Options** tab. Choose desired reports, using **Edit** to specify details; then click **Save**.

13. Click **Close** to close the dialog.

Creating Sample Information Files

A sample information file must be assigned to every sample that is analyzed. When you create a sample file, you can accept the default values specified using Sample Defaults, or you can edit them as desired. You can create a sample information file using the Advanced, Basic, or Restricted format.

Advanced Format

The Advanced format allows you to customize the parameters of a sample information file. Chapter 5 provides a description of the fields associated with creating sample information files.

1. Select **File > Open > Sample information**; the Open Sample Information File dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Sample Information dialog is displayed.

The defaults that appear in the fields are the ones specified in **Sample Defaults**.

4. Accept the default identification in the **Sample** field or change it to an appropriate one. The dialog above shows the sample file number because the dollar ($) symbol was used when specifying sample defaults.

> **If a sample information file already exists containing the values you want to use in this file, you can click _Replace all_ to copy those values into this one. You can still edit the values after they are loaded.**

5. Edit the **Operator** and/or **Submitter** fields as needed. If these fields are not displayed, they were selected to be omitted in **Sample Defaults**.

6. Enter the sample's mass (if different from the default value) in the **Mass** field.

7. Choose whether you want data automatically collected by the system or whether you wish to enter the data.

> **Use the Comments window to record specifics of the analysis or its conditions. Anything you enter in this window is displayed in the report header.**

8. Click **Save** to save the information you entered.

9. The steps for completing the remaining parameters of the sample information file are explained in subsequent sections:

    • Specifying Sample Tube Criteria
    • Defining Degas Conditions
    • Defining Analysis Conditions
    • Defining Adsorptive Properties
    • Defining Report Options

    Simply click on the tabs to open the associated dialog.

Basic and Restricted Formats

Sample information files are created in the Basic and Restricted formats using predefined parameter files. Chapter 5 provides a description of the fields associated with creating sample information files.

1. Select **File > Open > Sample information**; the Open Sample Information File dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Sample Information dialog is displayed. The defaults that appear in the fields are the ones specified in **Sample Defaults**.

Use this push button to copy parameters

Not displayed on the Restricted dialog.
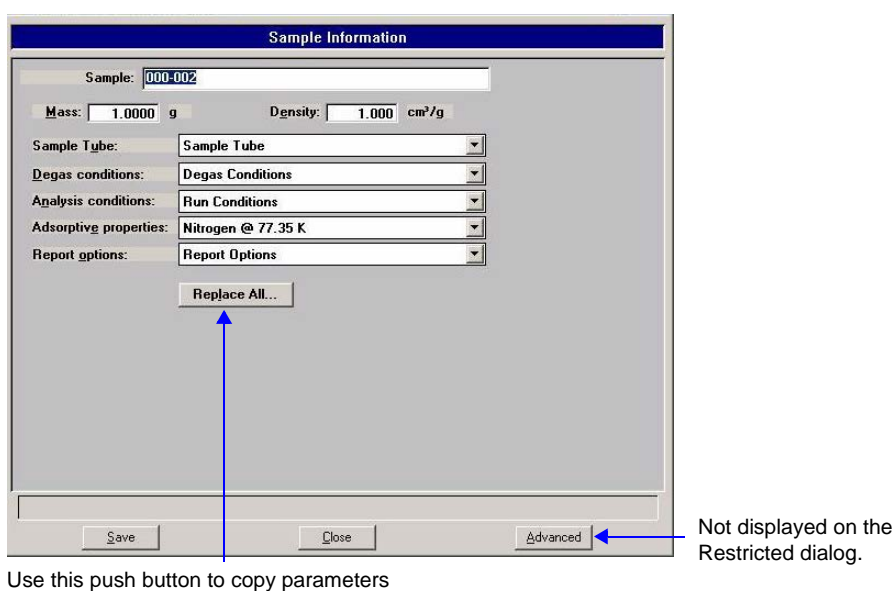
4. Accept the default identification or change it to an appropriate one.

5. Enter the sample's mass (if different from the default value) in the **Mass** field.

6. Using the down arrows to the right of each parameter field, select an appropriate file (or accept the defaults). You can review or edit the contents of these files by switching to the Advanced format if desired.

> **If you are using the Restricted format, you cannot switch to the Advanced format.**

7. Click **Save**, then **Close**.

Defining Parameter Files

The following file types can exist as part of the sample information file or as an individual parameter file:

- Sample tube
- Degas conditions
- Analysis conditions
- Adsorptive properties
- Report options

Having these files exist independently allows you to use them over and over again. Several predefined parameter files (located in the params directory) are included with the ASAP 2050 analysis program. Although these files may come close to the needs of your laboratory, you may wish to define additional ones. Or you can use a predefined file as a starting point. This is easily accomplished by creating a new file and then selecting **Replace**. A dialog is displayed so that you can select the existing file containing the values you wish to use. After the values are copied into the current file, you can edit the values as desired; the original file remains intact and ready for the next use.

If you wish to have parameter files display in the drop-down list on the Basic Sample Information dialog, be sure to save them to the directory specified as the Parameter Files Directory. (Refer **Parameter Files Directory** on page **8-12** for additional information.)

Sample Tube

1.   Select **File** >**Open > Sample Tube**; the Sample tube dialog is displayed.

2.   Enter a name in the **File name** field, then click **OK.**

3.   Click **Yes** to create the file; the Sample Tube dialog is displayed
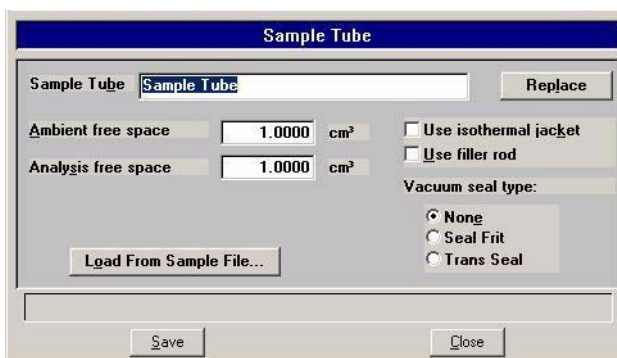
4.   Enter a description in the **Sample Tube** field. Be sure to use an intuitive description so that you can recognize it easily.

5.   Click **Load from Sample File**; the Open Sample Information File dialog is displayed.

6. Select the file you used in the blank run with this sample tube, then click **Open** to copy the ambient and analysis free space into the Sample Tube dialog.

7. If a vacuum seal of some type was used, select the appropriate option or leave the default of **None** selected.

8. Click **Save**, then **Close**.

Degas Conditions

Degas Conditions files contain degassing information for preparing samples.

Perform the following steps to define a degas conditions file:

1. Select **File** > **Open > Degas Conditions**; the Degas Conditions dialog is displayed.

2. Enter a name in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Degas Conditions dialog is displayed.

4. Enter a description in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily.

5. Specify values for the evacuation and heating phases for degassing the sample

6. Click **Save**, then **Close**.

Analysis Conditions

Analysis conditions specify the data used to guide an analysis. An analysis conditions file may be assigned a unique name, and you can direct any sample to be analyzed according to the conditions in any existing analysis conditions file. Chapter 5 provides a detailed description of the fields on this dialog.

1. Select **File** > **Open > Analysis Conditions**; the Open Analysis Conditions File dialog is displayed.

2. Enter a name (up to eight characters) in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Analysis Conditions dialog is displayed.
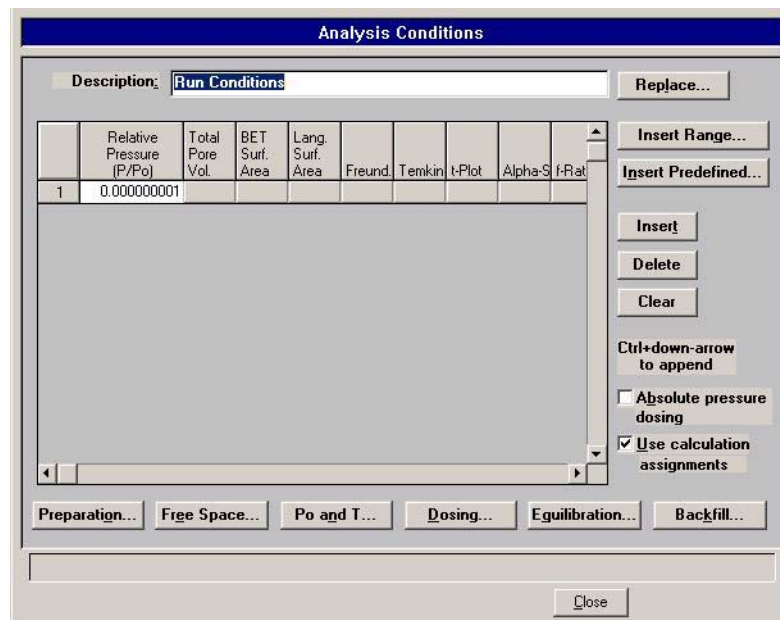
4. Enter a description (up to 42 characters) in the **Description** field. Use an intuitive description, one that will help you identify the type of sample you plan to analyze using these analysis conditions.

5. Specify points for your pressure table. Use the push buttons adjacent to the table to assist you in creating your table.

6. Click **Preparation** to specify evacuation and leak test values.

7. Click **Free Space** to specify how the free space is to be measured.

8. Click **Po and T** to specify how the saturation pressure (Po) is to be measured, and the analysis bath temperature. If you select **Absolute pressure dosing**, options 1 through 5 are disabled. The Po will be calculated using the analysis bath temperature; enter the appropriate temperature in the field provided.

9. Click **Dosing** to specify dosing options.

10. Click **Equilibration** to specify the equilibration interval and its delay time.

11. Click **Backfill** to specify backfill options.

12. If you wish to use absolute dosing, click the **Absolute pressure dosing** option.

13. Click **Save**, then **Close**.

Adsorptive Properties

Adsorptive properties specify the characteristics of the gases used in the ASAP 2050 System. Chapter 5 provides a detailed description of the fields on this dialog.

1. Select **File > Open** > **Adsorptive properties**; the Open Adsorptive Properties File dialog is displayed.

2. Enter a name (up to eight characters) in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Adsorptive Properties dialog is displayed.

.

4. Enter the name of the adsorptive gas in the **Adsorptive** field, then enter its mnemonic.

5. Click **Psat vs T** to edit the Psat vs Temperature table. Edit any other field(s) on this dialog as required, then click **OK**.

6. Select **Non-condensing Adsorptive** if the gas is non-condensing.

7. Enter the appropriate information (or accept the defaults) in the following fields.

   Maximum manifold pressure
   Density conversion factor

Therm. tran. hard-sphere diameter
Molecular cross-sectional area

8. Click **Open** and select the compressibility factor table for gas to be used for analysis.

9. Choose your preferred dosing method; choose **Normal** for gases under pressure.

Report Options

Report options specify the types of reports to be generated from an analysis or manually entered data. They also help you customize details of reports such as axis scale, axis range, column headings, and components of thickness curve equations.

You can tailor report options files to accommodate the requirements of your analyses. For example, you can generate a simple report that lets you determine the basic characteristics of the sample. Then use that report to make choices about the variables you want to include in lengthier, more sophisticated reports. You can specify for reports to be generated automatically after each analysis, or you can generate reports at any time during or after an analysis. However, a report generated during an analysis only includes data collected up to the time of the report. Chapter 5 provides a detailed description of the fields on this dialog.

1. Select **File > Open** > **Report Options**; the Open Report Options File dialog is displayed.

2. Enter a name (up to eight characters) in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Report Options dialog is displayed.
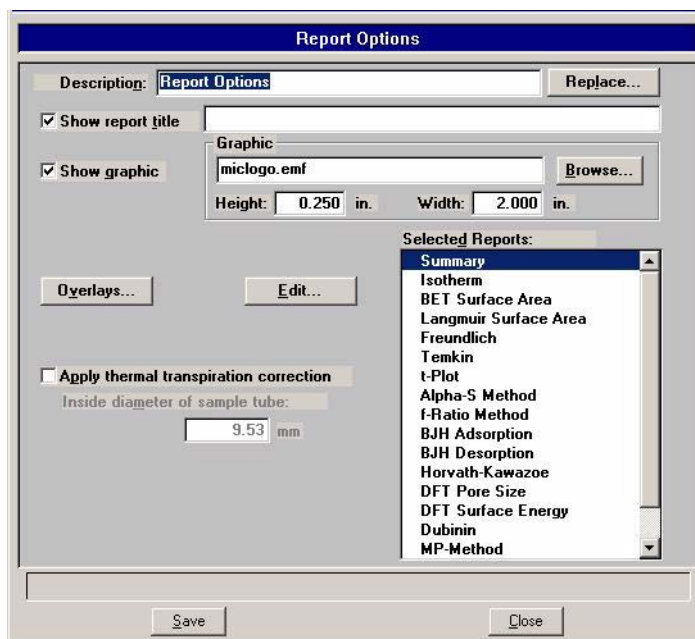
4. Enter a description (up to 42 characters) in the **Description** field. Enter an identifier that gives a more intuitive description of the file's contents. For example, **BJH Adsorption Report Options**.

5. Select **Show report title** and enter the title you wish to appear at the top of the report. Or deselect this option if you prefer not to have a report title.

> **If your company logo exists as a bitmap (bmp) or enhanced metafile (emf), you can have it display in the report header by selecting *Show graphic*. Click *Browse* to select the file; use the *Height* and *Width* fields to specify the size.**

6. If you wish to compare the same type of graph from multiple files, click **Overlays** and choose the files. Then be sure you edit the graph from the Selected Reports window and choose **Samples** from the Overlay drop-down list.

7. Select **Thermal transpiration correction** if you want correction made for thermal transpiration in the sample tube, then enter the inside diameter of the sample tube.

    Thermal transpiration correction should be used for analyses where the sample temperature is more than 100 $^{\circ}$C below the manifold temperature.

8. The reports that may be generated are listed in the Selected Reports list. Select reports by double-clicking on the desired report. Reports are deselected in the same manner. A report is selected when it is preceded by a check mark.

9. You can edit some reports by highlighting the desired report and clicking **Edit**.

10. Click **Save,** then **Close** to save the information and close the dialog.

Using the Cold Trap

The cold trap helps remove oil vapors from the system. It can be used when preparing samples on the degas ports or when analyzing samples on the analysis port. If should not, however, be used with gases that could freeze in the cold trap, for example CO_2 at liquid nitrogen temperatures.

1. Make sure the Dewar contains enough analysis bath fluid. (Refer to **"Filling the Dewar" on page 3-25**.)

2. Hang the cold trap Dewar around the cold trap port as shown below.

Cold trap
port
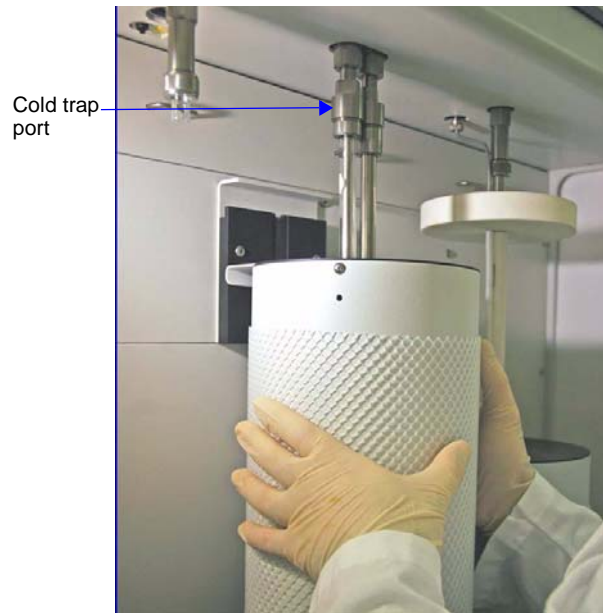
3.   Place the cover over the Dewar opening.

Dewar
cover

Preparing Samples

Care should be taken in conditioning and filling sample tubes. The following guidelines help ensure accurate, reproducible results.

The weight of the isothermal jacket may vary slightly and cannot be considered as constant; therefore, do not weigh it with the sample tube set.

The sample tube should be cleaned (using an ultrasonic cleaning unit) and adequately dried before each analysis to prevent the cross-contamination of samples. The following materials are recommended for cleaning sample tubes:

- Ultrasonic cleaning unit
- Suitable detergent
- Latex gloves or lint-free cloth
- Isopropyl alcohol (IPA) is preferred for cleaning; acetone or deionized water may be used if IPA is unavailable
- Nitrogen or helium
- Sample tube brush

1. Ensure that the ultrasonic cleaning unit is clean.

2. Using a ratio of 5 grams of detergent per 500 mL of warm water, fill the bowl of the ultrasonic unit with enough water to cover the entire sample tube.

3. Ensure that the detergent is dissolved before placing the sample tube into the water. If too much detergent is used, it may be difficult to rinse from the sample tube.

4. Fill the sample tube with warm water and place it in the bowl of the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.

5. Using latex gloves or a lint-free cloth, remove the sample tube from the bowl.

6. Clean the interior of the sample tube with the brush supplied in the accessories kit.

7. Rinse the sample tube thoroughly with hot water. Then rinse it with isopropyl alcohol.

If isopropyl alcohol is unavailable, acetone or deionized water may be used to rinse the sample tube. Do not use any corrosive cleaning agents. They may reduce the integrity of the stainless sample tube.

8. Using dry, compressed nitrogen or air, dry the interior of the sample tube. Use a tubing extension long enough and small enough in diameter to fit inside the sample tube.

9. Wipe a stopper or seal frit with a lint-free cloth. Place the stopper or seal frit on the sample tube.

Determining the Mass of the Sample

> **A form to help you determine the sample's mass is provided in Appendix A. Make copies of this form and use it for your convenience.**

1. Place the sample tube (with stopper or seal frit) onto the balance. Record the value as the mass of the empty sample tube.

2. Remove the sample tube from the balance.

3. Place a sample container onto the balance. Zero the balance and allow it to stabilize.

4. Slowly add the sample to the container. This step enables you to control the size of the sample before it is poured into the prepared sample tube. Do not record the sample mass at this point.

5. Remove the stopper from the tube. Carefully pour the sample from the container into the
    sample tube. Use a pipe cleaner to clean the walls of the sample tube before weighing the
    sample.

6. Replace the stopper or seal frit on the sample tube.

7. Place the sample tube onto the balance. Determine the mass of the sample tube (with
    stopper) and sample. Record the value as the **Sample plus sample tube mass** if you are
    using the Sample Mass form from Appendix A.

Degassing Samples on Degas Ports

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The sample is heated and placed under vacuum to remove moisture and other contaminants. This process is referred to as degassing the sample. You may degas up to two samples at one time using the degas ports.

1. Insert the heating mantle thermocouple plug into the thermocouple connector above the degas port to be used. Insert the heating mantle power plug into power connector and twist to lock securely.

Heating mantle thermocouple

Heating mantle power plug
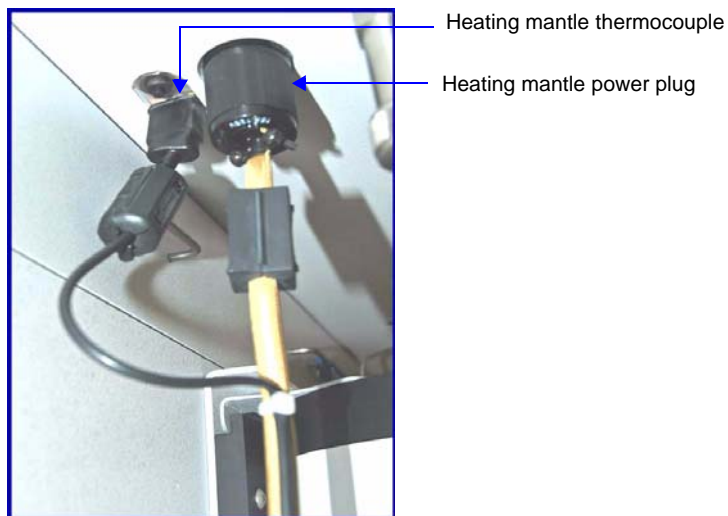
2. While holding the degas port plug, remove the connector nut and plug from the degas port by turning the connector nut counterclockwise.

Connector nut

Port plug
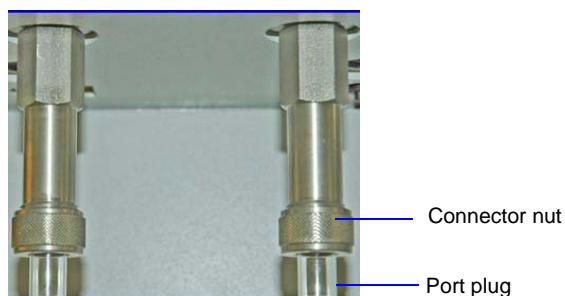
3.   Place the connector nut, and, if used, seal frit, onto the sample tube. Make sure the O-ring is in place on the sample tube as shown below.
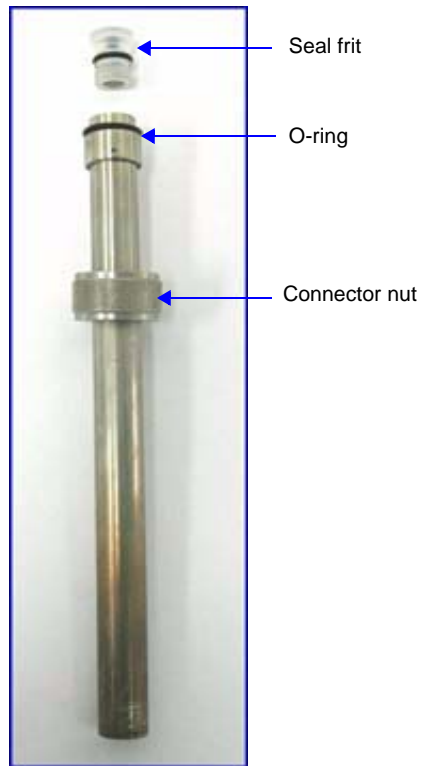
4.   Attach the sample tube to the degas port. Be sure to push the sample tube in to a full stop. Secure the sample tube in place by sliding the connector nut up to the degas port and turning the connector nut clockwise. Tighten the nut securely by hand.

5. Slide the heating mantle around the sample tube with the heating mantle hook toward the back.

Heating mantle hook
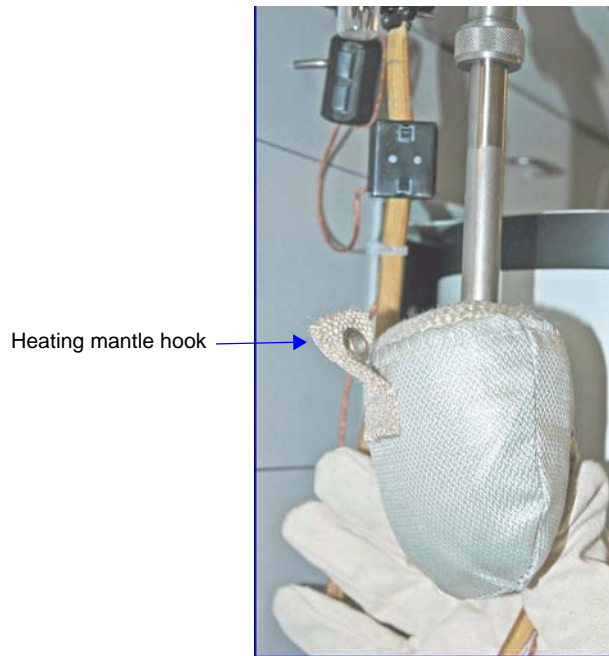
6. Attach the elastic cord to the hook on the heating mantle and to the hook on the front panel of the instrument; then secure the mantle with a heating mantle clip as shown below.

Front panel hook

Elastic cord

Heating mantle hook

Heating mantle clip
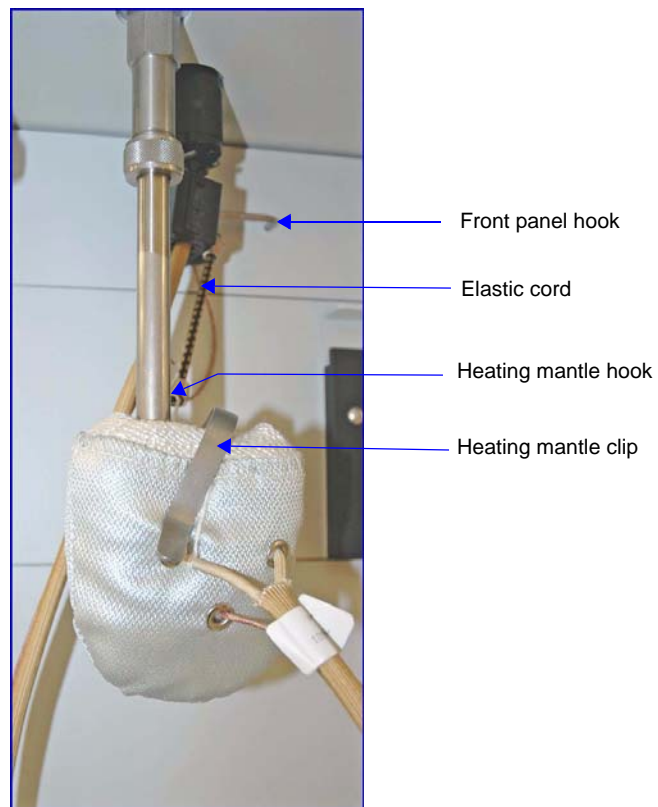
7. Select **Start Degas** from the Unit menu; the Start Degas dialog is displayed.
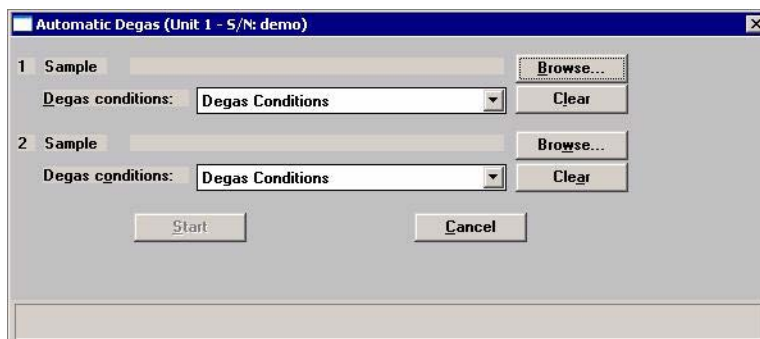
8. Click **Browse** to the right of the **Sample field** to choose your degas file.

    Repeat this step if you are degassing two samples.

9. Click **Start** to begin the degassing operation.

> **Observe the status bar of the degassing operation to determine when degassing is complete.**

10. After degassing has completed, allow the heating mantle and sample tube to cool.

> **Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube or heating mantle could result in burns.**

11. After the heating mantle has cooled, carefully remove the heating mantle and allow the sample tube to cool to room temperature (approximately fifteen minutes).

12. When the sample tube has cooled, transfer it to the analysis port (refer to **"Transferring a Degassed Sample to the Analysis Port" on page 3-26**).

Filling the Analysis Dewar

Precautions

> **Always handle Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution.**

We recommend the following be observed when handling Dewars containing liquefied gases:

- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.

- When pouring liquefied gases from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the liquefied gas slowly to prevent splashing, and 3) vent the receiving container to the atmosphere.

- Use a plastic stirring rod when stirring substances in a Dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some form of protective coating.

- Do not handle heavy objects above the Dewar. If unavoidable, place a protective cover over the Dewar's opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

- Always install the Dewar cover before performing an analysis. The cover reduces the accumulation of ice. Accumulated ice could cause the Dewar to bond to the sample tube.

Filling the Dewar

1. Fill the analysis Dewar with the cryogen to about 5 cm (2 inches) from the top.

> **Incorrect fluid levels can lead to measurement errors. Do not overfill the Dewar.**

2. Check the analysis bath fluid level with the dipstick as shown below.

Analysis Dewar

Maximum Level
Indicator Mark

Wetness or frozen
condensation indicates
bath liquid level.
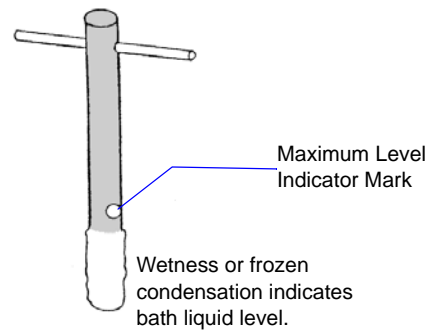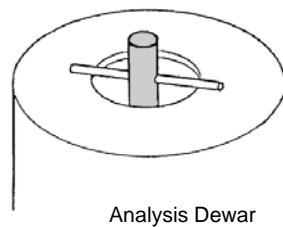
Transferring a Degassed Sample to the Analysis Port

The sample tube must be removed from the degas port, weighed and then installed onto the analysis port for analysis.

> **If the sample tube is not mounted to the analysis port immediately, either leave it on the degas port or remove it and insert the rubber stopper (unless you are using a seal frit) into the sample tube opening.**

1. Make sure the sample tube has cooled to room temperature.

2. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If you are not using a seal frit, insert a stopper immediately. Remove the connector nut from sample tube stem.

3. Weigh the sample tube set. Enter the weight on the Sample Data Worksheet as *Mass of sample tube plus sample (After Degas)*.

4. Subtract the *Mass of empty sample tube (Before Degas)* from the *Mass of sample tube plus sample (After Degas)* to determine the mass of the sample. Record this value as the *Mass of sample (After Degas)*.

5. Remove the connector nut, and, if installed, the port plug, from the analysis port by turning the connector nut counterclockwise.

Connector nut

Port plug
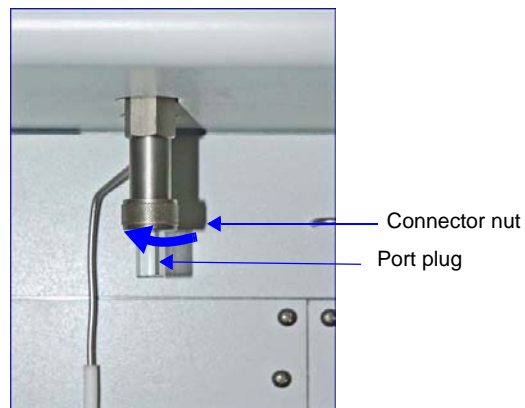
6. Remove the stopper from the sample tube and immediately attach the sample tube to the analysis port, pushing it fully up into the port. Secure it in place by screwing the connector nut onto the analysis port connector; hand-tighten the connector nut.

You do not have to remove the seal frit (if one is used).

7. If you wish to degas the sample on the analysis port, proceed to **"Degassing a Sample on the Analysis Port" on page 3-31**.

    If you wish to perform an analysis, slide the isothermal jacket up over the sample tube. Make sure the saturation pressure tube is close to the sample tube.

8. Place the analysis Dewar on the elevator as shown in the following illustration.

9. Place the Dewar cover over the sample tube stem just above the isothermal jacket as shown below.

10. Attach the safety shield to the two hooks above the port.

11. Begin the analysis. Refer to **"Performing an Analysis" on page 3-37**.

Degassing a Sample on the Analysis Port

In addition to preparing a sample on a degas port, you may further evacuate the sample on the analysis port prior to starting an analysis.

1. With the sample tube attached to the analysis port (refer to **"Transferring a Degassed Sample to the Analysis Port" on page 3-26**), select **Unit [n] > Show Analysis Schematic**, then select **Unit [n] > Enable Manual Control**. The Manual Control screen is displayed.

2. Open valves 7, 9 and 2.

3. Wait until the value displayed for the transducer is below 10 mmHg.

4. Open valve 1.
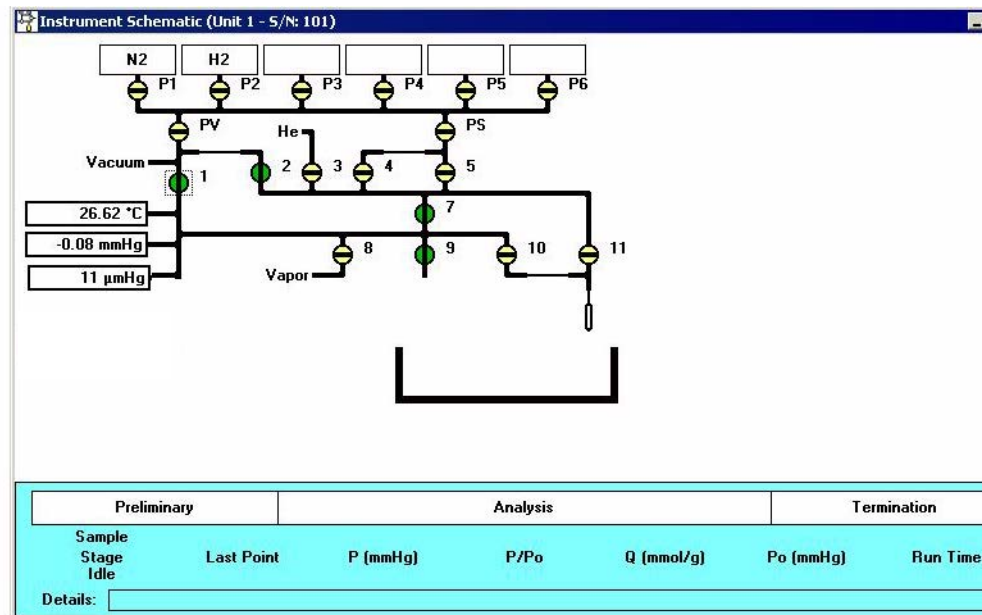
5. Evacuate until the vacuum gauge reaches vacuum and no leaks are indicated on the gauge.

6. Insert the heating mantle thermocouple plug into the thermocouple connector behind one of the degas ports. Insert the heating mantle power plug into the associated power connector and twist to lock securely.

Heating mantle thermocouple

Heating mantle power plug
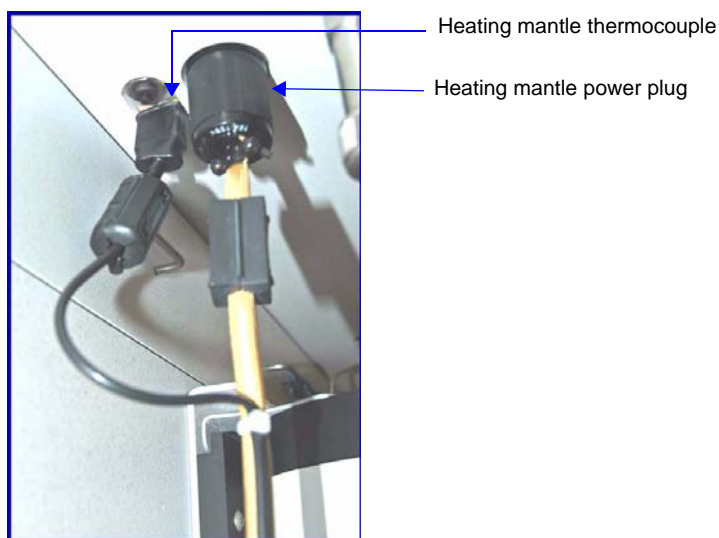
7. Slide the heating mantle around the sample tube with the heating mantle hook toward the back.

8. Attach the elastic cord to the hook on the heating mantle and to the hook on the front panel of the instrument, then attach a heating mantle clip as shown below.

Instrument hook

Elastic cord

Heating mantle clip
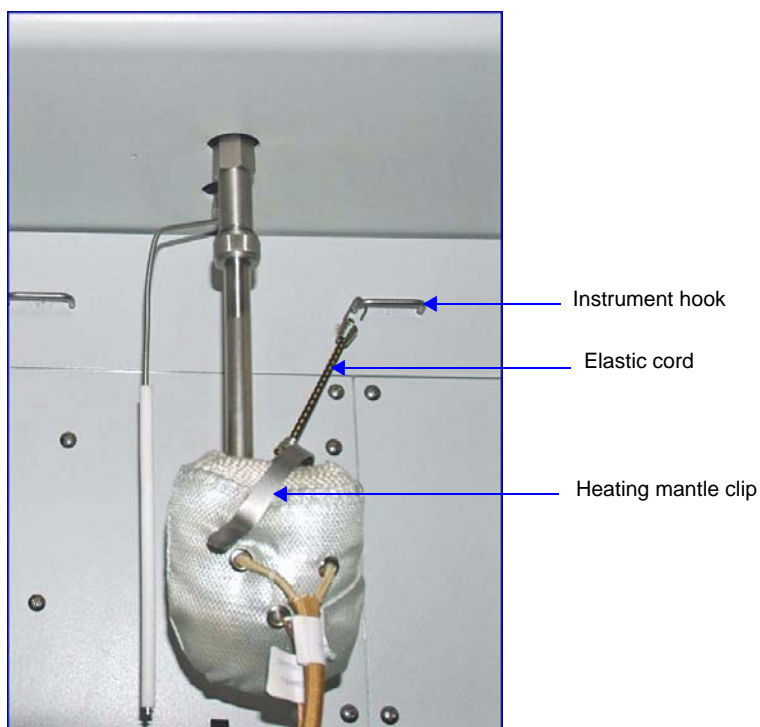
9. Gently heat the sample:

a. Select **Unit [n] > Degas > Show Degas Schematic** to display the Degas Schematic.

b. If the analyzer is not in manual control mode, select **Unit [n] > Degas > Enable Manual Control.**

c. Right-click on the degas port to which the heating mantle power plug and thermocouple are connected, then select **Set**.
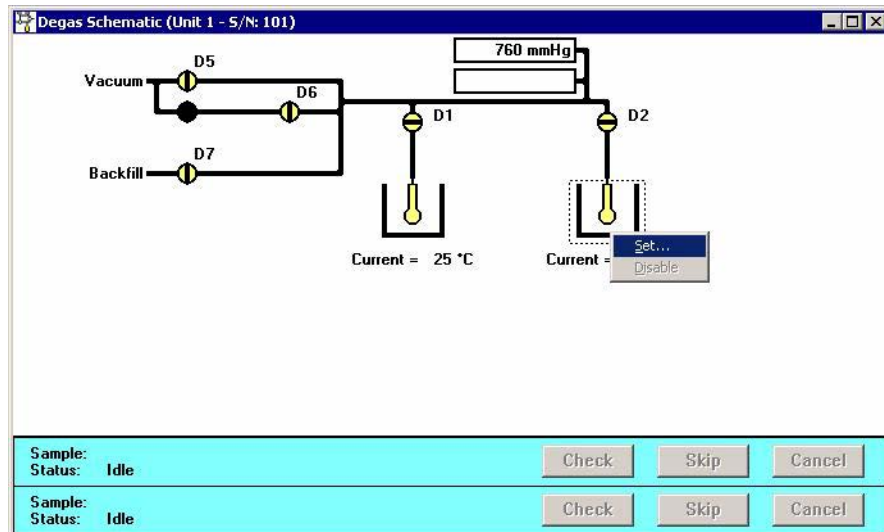
d. The Heater Settings dialog is displayed. Enter 10 ^{o}C/min. for **Ramp rate**.

e. Enter 150 ^{o}C for **Target temperature**.
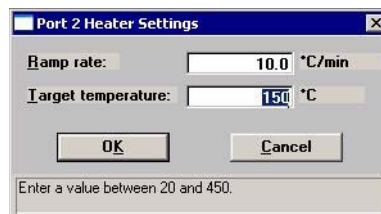
f. Click **OK**.

10. Allow the sample to degas for a minimum of one hour. (Additional time may be required to fully degas the sample.)

11. Right-click on the degas port sample tube, then select **Disable**.
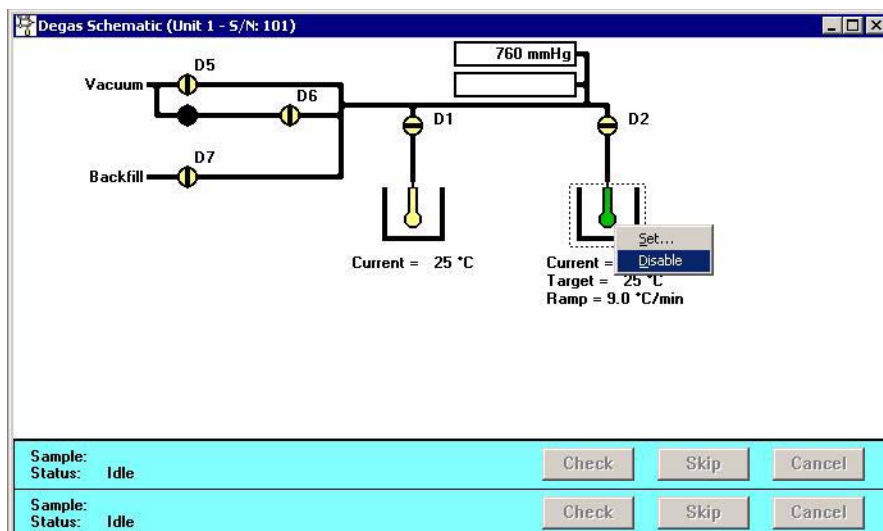
Do not touch the sample tube or the heating mantle until they have cooled. Touching the sample tube, heating mantle, or heating mantle clip before they have cooled could result in burns.

12. When the heating mantle is cool enough to handle, remove it from the sample tube.

13. Allow the sample to cool to room temperature.

14. Slide the isothermal jacket up over the sample tube. Make sure the saturation pressure tube is close to the sample tube.

15. Place the analysis Dewar onto the elevator as shown in the following illustration.

16. Place the Dewar cover over the sample tube stem just above the isothermal jacket as shown below.

17. Attach the safety shield to the two hooks above the port.

18. Begin the analysis. Refer to **"Performing an Analysis" on page 3-37**.

Performing an Analysis

After the sample has been degassed and transferred to an analysis port, you may begin an analysis.

> **Before beginning an analysis, make sure the tank pressure for the gas regulator is at least 200 psig (1379 kPa). Pressures less than 200 psig (1379 kPa) may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.**

Ensure that the analysis gas and the Psat gas specified in the sample file match the system configuration gases; if they do not, correct the sample information file or correct the unit configuration gas.

Perform the following steps to begin an analysis:

1. Select **Unit** > **Start Analysis**; the Analysis dialog is displayed with the Start Analysis dialog positioned on top.

2. Choose a file for your analysis and click **OK**; the Analysis dialog containing the parameters of the selected file is displayed.

3. Verify parameters and make any changes you feel necessary.

4. To generate a report automatically after the analysis, click **Report after Analysis** and choose report output options. You can generate reports to the screen (Preview) or directly to a printer.

> **Make sure the elevator is all the way down. If it is not all the way down, hazardous conditions can result.**

5. Click **Start** to begin the analysis.

6. Until data collection begins, the following message is displayed on the screen.

No Isotherm Data Available.

7. As data are collected, a graph will be drawn in the window.
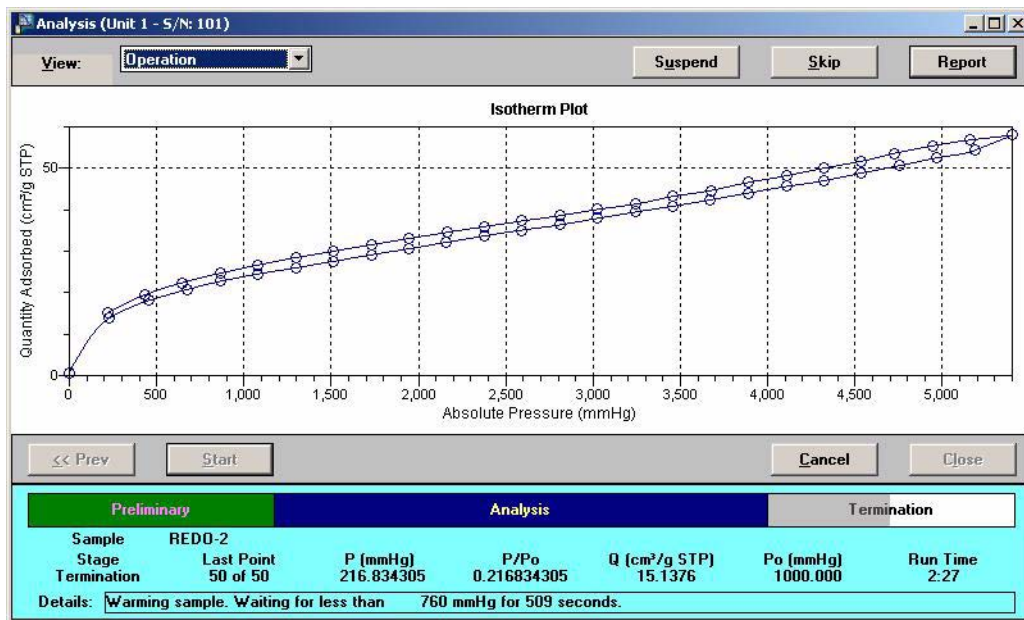
8. If you wish to perform another analysis after the current analysis is completed, click **Next**; the first view of the Analysis dialog is displayed.

> **After the elevator rises, do not place any items under the elevator platform. Objects under the platform will prevent the elevator from fully lowering, and could cause damage to the elevator mechanism.**

> ⚠ **The ASAP 2050 can operate in a pressure range from 0 to 10 atm; therefore, ensure that the sample tube is not under pressure when removing it from the instrument. Use caution appropriate for these types of devices.**

Printing File Contents

You can print the contents of one or more sample or parameter files.

1. Select **File** > **Print**; a drop-down list containing file types is displayed.

2. Choose the file type; a dialog similar to this one is displayed.
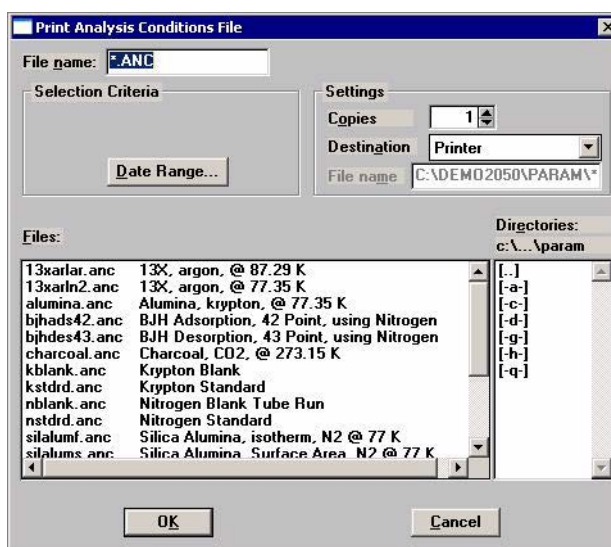
3. From the **Files** list box, select the desired file. If you plan to print multiple files, hold down **Ctrl** while clicking on the desired files.

> ✏ **You can use the Status drop-down list and/or the Date Range push button to limit the files displayed in the Files list.**

4. Choose whether you wish to print the contents to the screen (Preview) or to a printer (Print). If you choose Print, the **Copies** field is enabled allowing you to print up to four copies.

5. Click **OK**; the file is printed to the specified destination.

Listing File Statistics

You can generate a list of the following information on a sample file or parameter file.

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status

1. Select **File** > **List**.

2. From the List drop-down menu, select the type of file on which you wish information. A dialog similar to the one shown below is displayed:

Displays the type of file on which you requested information; in this example, a sample information file
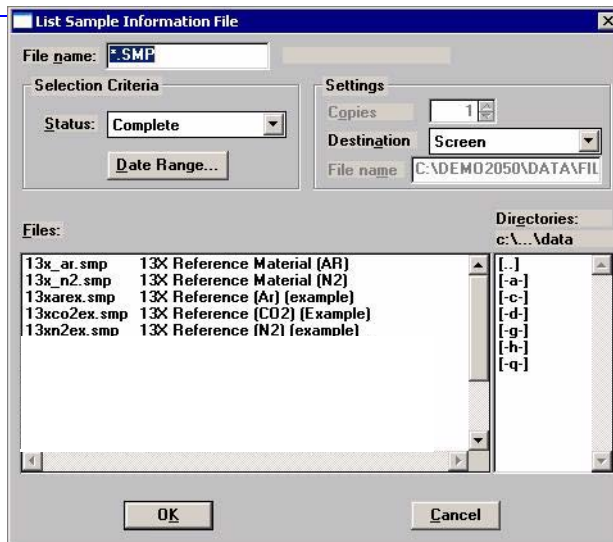
3. From the **Files** list box, choose the desired file(s). If you wish to include all files, leave all files *deselected*.

4. Choose whether you wish to print statistics to the screen (Preview) or to a printer (Print). If you choose Print, the **Copies** field is enabled allowing you to print up to four copies.

5. Click **OK**, a list for the requested file(s) is sent to the specified destination.

Exporting Isotherm Data

The Export option on the File menu allows you to copy the isotherm data in the sample information file and reformat it in ASCII text. If saved to a **File**, the data can be imported into applications, such as spreadsheets. The output file consists of four columns containing the absolute pressure, relative pressure, specific volume adsorbed, and the elapsed time.

1. Select **File** > **Export**; the Export Sample Information dialog is displayed.
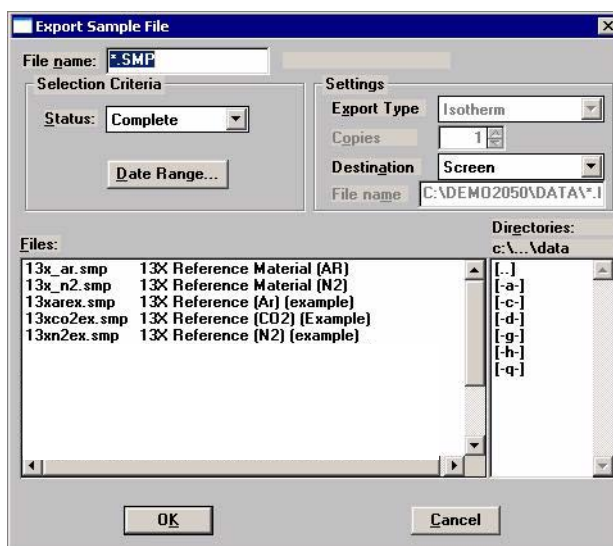
2. From the **Files** list box, choose the file(s) you wish to export. You can select multiple files by holding down **Ctrl** while clicking on the desired files.

> **You can use the Status drop-down list and/or the Date Range push button to limit the files displayed in the Files list.**

3. Choose an output destination. When you choose **File** as the destination, the **File name** field is enabled so you may enter a name (or you can accept the default). If you have selected multiple files, each file is exported as its file name with the extension **.ISO**.

4. Click **OK**; the file(s) is exported to the specified destination.

Generating Graph Overlays

Use graph overlays when you wish to compare graphically results for multiple samples or multiple graphs for one sample. Graphical lines are differentiated by the use of varying symbols and reported in a legend on the report. If color output capability is available, different colors are used instead of symbols.

Graph overlays can be implemented in two ways:

- **Multiple Sample Overlays**
  Overlay results for up to eight samples on top of a previously selected sample.

- **Multiple Graph Overlays**
  Overlay two different types of graphs from one sample. The multiple graph overlay capability exists for:

  BJH Adsorption/Desorption
  DFT Pore Size/Surface Energy

You cannot overlay graphs if your sample file presentation is in the Basic format. You must switch to the Advanced format before proceeding.

Multiple Samples Overlay

1. Select **File > Open > Sample Information** to display the Open Sample Information File dialog.

2. Select a sample on which to overlay graphs of other samples, then click **OK**; the Sample Information dialog is displayed.

3. Click the Report Options tab to display the Report Options dialog.
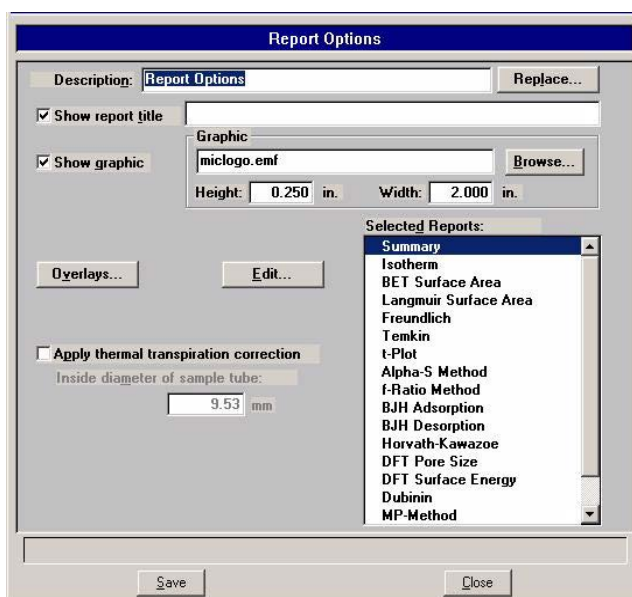
4. Make your report selections (or deselect any you do not wish to generate).

5. Choose the type of report for which you wish overlays; perform the steps indicated:

If overlaying this type of report...	Then...
Alpha-S Isotherm BET/Langmuir surface area F-ratio Freundlich Temkin t-Plot	Select the report for which you want an overlay, then click **Edit**. A report options dialog for that report is displayed.
	Select **Overlay Samples** and do any other editing you wish.
	Click **OK**; the main Report Options dialog reappears.
Dubinin	Select Dubinin, then click **Edit**; a Report Options dialog for Dubinin is displayed.
	Select the desired graph, then click **Edit**. An options dialog for that graph is displayed (for example, dV/dw Pore Volume).
	Select **Overlay Samples** and do any other editing you wish.
	Click **OK**; the options dialog for Dubinin reappears.
	Click **OK**; the main Report Options dialog reappears.
BJH Adsorption BJH Desorption DFT Pore Size DFT Surface Energy	Select the report for which you want an overlay, then click **Edit**. A Report Options dialog for that report is displayed.
	Select the desired report, then click **Edit**. An options dialog for that graph is displayed.
	Click on the down-arrow at the **Overlay** field and select **Samples**; do any other editing you wish.
	Click **OK**; do any other editing you wish.
	Click **OK**; the main Report Options dialog reappears.

6.   Click **Overlays**; the Graph Overlay Samples dialog is displayed.
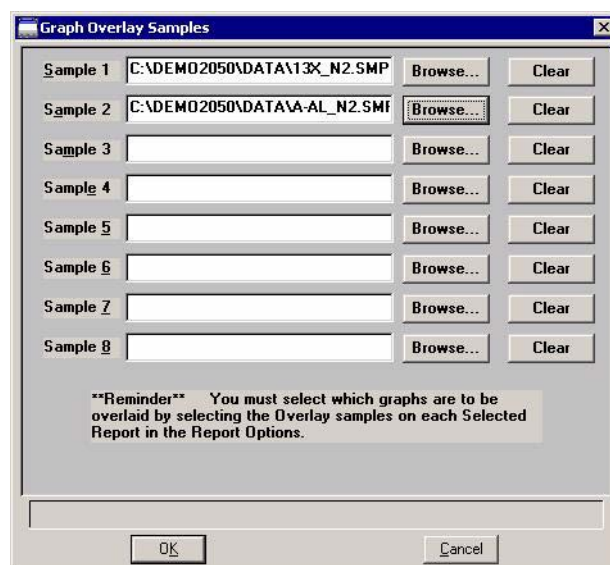
7.   Click **Browse** to the right of the **Sample 1** field; the Plot Overlay Sample Selection dialog
     is displayed.
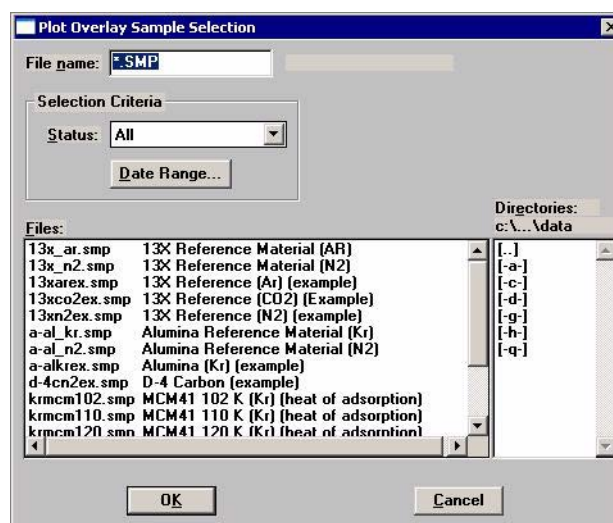
8.   Choose a sample file, then click **OK**. You may choose up to eight files in this manner.

9.   After selecting the desired number of sample files, click **OK**; the main Report Options
     dialog reappears.

10. Click **Save** if you wish to save your Selected Reports list. Even if you don't save, all of the
     options you selected regarding overlays and other items are included in the report you
     generate.

11. Select **Reports** > **Start Report**; the Start Report dialog is displayed with the name of your
     primary file highlighted.

12. Click **OK**; the Select Reports dialog is displayed.

13. Ensure that the desired graph is selected (preceded with a check mark), then click **OK**.

Multiple Graphs Overlay

Multiple graph overlays can be produced only for:

- BJH Adsorption
- BJH Desorption
- DFT Pore Size
- DFT Surface Energy

1. Select **File > Open > Sample Information** to display the Open Sample Information File dialog.

2. Select a sample for which you want to overlay two graphs; then select **Edit**; the Sample Information dialog is displayed.

3. Click the **Report Options** tab to display the Report Options dialog.
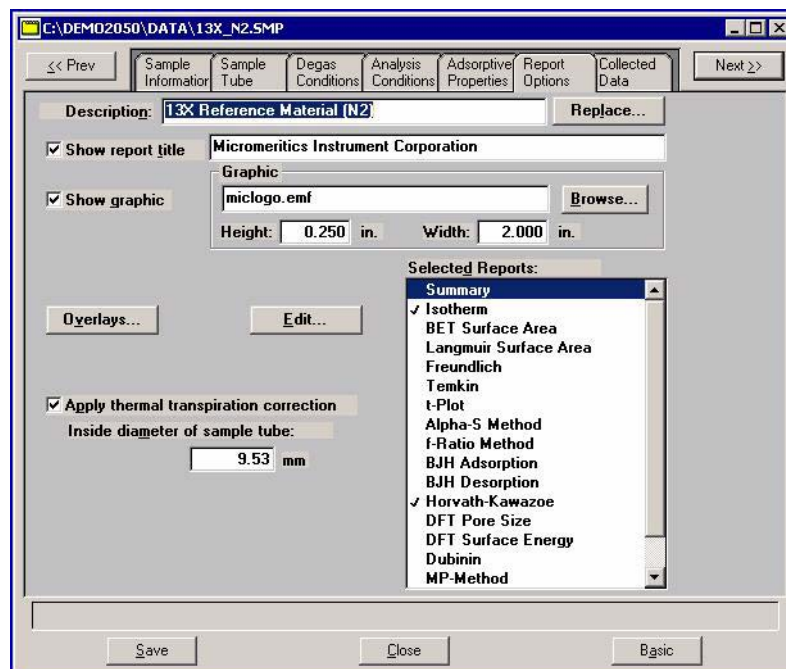
4. Make your report selections (or deselect any you do not wish to generate).

5. From the **Selected Reports** list, select the report for which you want multiple graph overlays, then select **Edit**; a Report Options dialog for that report (or graph) is displayed (this example shows BJH Adsorption).
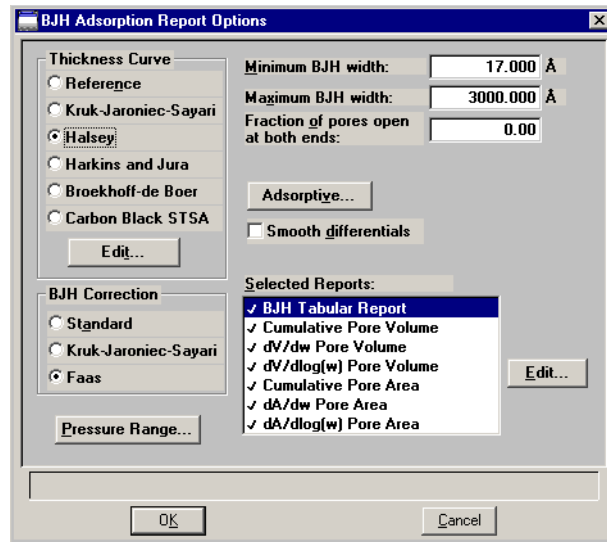
6. Select a report type, then click **Edit**; an options dialog for that specific item is displayed (this example shows Cumulative Pore Volume).
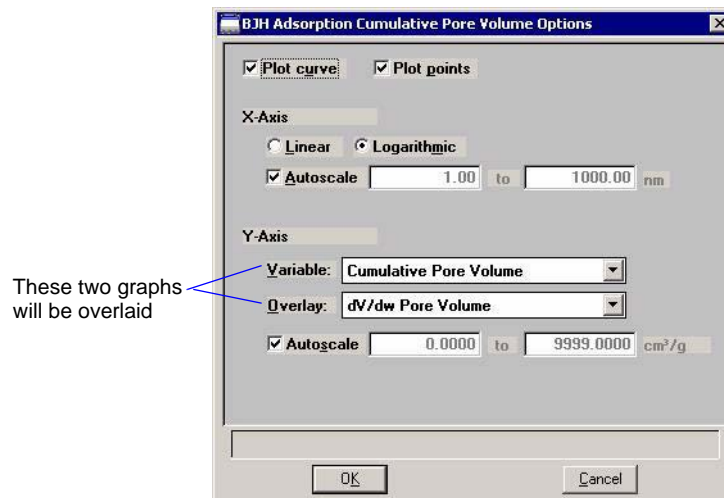
7. Click the down arrow at the **Overlay** field and select a graph type from the list. This selection will be overlaid on the graph selected in the **Variable** field.

8. Select **OK** to return to the BJH Adsorption Report Options dialog; do any editing you wish.

9. Click **OK** to return to the main Report Options dialog.

10. Click **Save** if you wish to save your Selected Reports list. Even if you don't save, all of the options you selected regarding overlays and other items are included in the report you generate.

11. Select **Reports** > **Start Report**; the Start Report screen is displayed with the name of your primary file highlighted.

12. Click **OK**; the Select Reports dialog is displayed.

13. Ensure that the desired graph is selected (preceded with a check mark), then click **OK**.

4. SOFTWARE AND SETUP MODIFICATIONS

This chapter provides instructions for:

- Installing the software
- Using the Setup Program for other functions

Installing the Analysis Program

Power option features must be properly set to enable communications between the analyzer and the computer. Set these features as follows:

1. From the Windows Start menu, select **Settings > Control Panel > Power Options**.

2. Select **Never** from the drop-down lists for all Power Scheme options except the monitor. You may select any setting you wish for the monitor.

Initial Installation

The ASAP 2050 System program is supplied on a CD. Perform the following steps to install the program:

1. Turn on the analyzer.

2. Insert the CD into the CD-ROM drive.

> **Proceed to Step 4 if AutoPlay is enabled on your computer; Setup will start automatically and display the New Installation dialog.**

3. Select **Start** from the Status bar, then **Run** from the Start menu.

   Enter the drive designator for the CD-ROM drive, followed by setup. For example:

   e:setup

   Alternatively, you can click **Browse**, navigate to the CD-ROM drive, and select **setup.exe**.

4. Click **OK**; the New Installation dialog is displayed.

Displays the amount of current disk space and the amount required for installation of the application. Also displays the directory into which the application will be installed.

Select this option to add the ASAP 2050 icon to your desktop for quick access to the application.

You may cancel the installation at any time by selecting Exit. If you do so, you must start the installation program from the beginning to install the analysis program.

The **Destination Folder** group box displays the amount of current disk space, the amount of disk space required for the analysis program, and the directory into which the application will be installed. If you wish to install the application into a different directory, click **Browse** to choose the directory.

5. If you want to run the application from the desktop, select the check box just below the Destination Folder group box to add an icon.

6. The ASAP 2050 icon is added to the Micromeritics folder by default. If you prefer a different folder, enter or select one from the drop-down list.

7. Click **Next**; the Analyzer Configuration dialog is displayed.

In the Step 1 group box, click the radio button for the number of analyzers to be attached to this computer. If you are attaching two analyzers, make sure your computer has two serial ports.

Choose 0 (zero) if you are installing this program for data reduction on a computer other than the one controlling the analyzer.

8. In the Step 2 group box, enter the analyzer serial number and the communications port to which it will be attached for each analyzer.

9. Click **Next**; the Calibration File Installation dialog is displayed. Read the information in the dialog and proceed accordingly.

If you selected zero (0) as the number of instruments to install, the Calibration dialog is not displayed.

10. After the calibration files are installed, the Installation Complete dialog containing the Readme file is displayed.

11. Use the scroll bar if you want to read the contents of the file, then click **Finish** to close the dialog.

12. Remove the Setup CD and store in a safe place. The original Setup CD contains the calibration files specific to your instrument. Upgrade CDs do not contain calibration files. Therefore, it is important that you maintain your original Setup CD in a secure location in the event calibration files need to be reinstalled.

Using the Setup Program for Other Functions

After initial installation of the ASAP 2050 analysis program, the application setup program can be used to:

- Upgrade software
- Add an analyzer
- Move an analyzer from one computer to another computer
- Remove an analyzer from the computer
- Change the analyzer setup
- Reinstall calibration files
- Uninstall the analysis program

To start the application setup program:

1. Ensure that the analysis program is not operating.

2. Insert the CD into the CD-ROM drive.

Proceed to Step 4 if AutoPlay is enabled on your computer; Setup will start automatically and display the New Installation dialog.

3. Select **Start** from the Status bar, then **Run** from the Start menu.

   Enter the drive designator for the CD-ROM drive, followed by setup. For example:

   e:setup

   Alternatively, you can click **Browse**, navigate to the CD-ROM drive, and select **setup.exe**.

4. Click **OK**; the setup Welcome screen showing the options available is displayed.
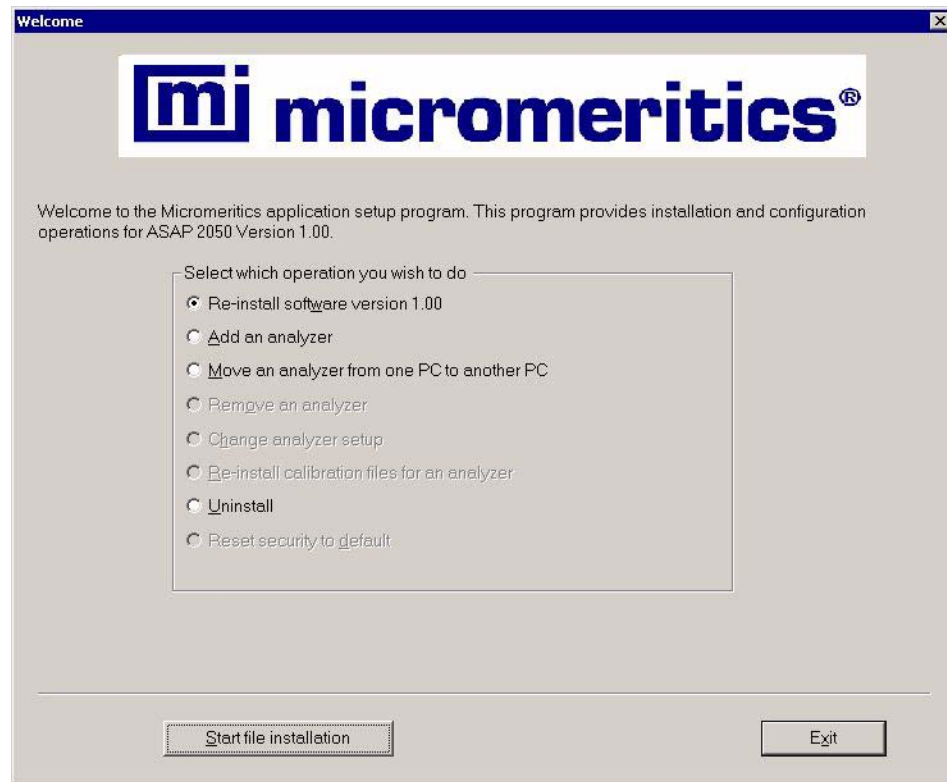
Installing Subsequent Software Versions

When you install a software upgrade, the system installs all of the application files and any status files that do not already exist on the computer. Existing analyzer status files are not affected and default and data files are not overwritten. There are three types of subsequent installation; the software version controlled by the setup program is:

- a later version than the version installed on the computer
- the same version as the version installed on the computer
- an earlier version than the version installed on the computer
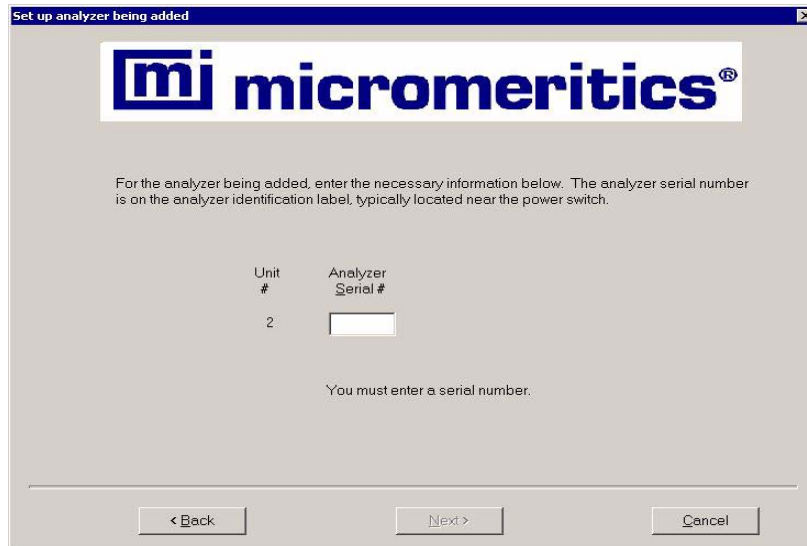
The setup program automatically detects which type of installation applies and customizes the selection in the Setup dialog accordingly.

1. Start the Setup program. Choose the software option; remember, only the applicable option will display; it will be one of the following:

   - Upgrade software to version (number) from version (number)
   - Reinstall software version (number)
   - Downgrade software to version (number) from version (number)

2. Click **Start File Installation**; the application installs the software and redisplays the setup Welcome dialog. If no other operations are desired using this dialog, click **Exit** to close the dialog.

Adding an Analyzer

Add an analyzer to the existing application as follows:

1. Start the Setup program. Select **Add an analyzer**, then click **Next**; the Setup analyzer dialog is displayed.

2. Enter the serial number of the analyzer being added, then the communications port to which it is to be connected.

3. Click **Next**; the Calibration Installation dialog is displayed.

4. Select the location of the calibration source files. If the calibration files are located in a directory other than the one displayed, click **Browse** to select the directory. Click **Finish**; a media change dialog is displayed.

5. Click **OK** to install the calibration files. A dialog containing the Readme file is displayed after the calibration files are installed.

6. Click **Exit** to close the dialog.

Moving an Analyzer from one PC to another PC

You can move an analyzer, along with its status and calibration files, from one computer (Source PC) to another computer (Destination PC).

This operation does not move sample or parameter files. To move these files, use a file management program such as Explorer or a backup/restore utility.

1. Install the application program on the destination computer. Refer to **Installing the Analysis Program** on page **4-1**.

    If the analysis program is already installed on the destination computer, proceed to Step 2.

2. Start the application setup program on the source computer. Refer to **Using the Setup Program for Other Functions** on page **4-4**.

3. In the Setup dialog, select **Move an analyzer from one PC to another PC**, then click **Next**; the Move analyzer operation dialog is displayed.
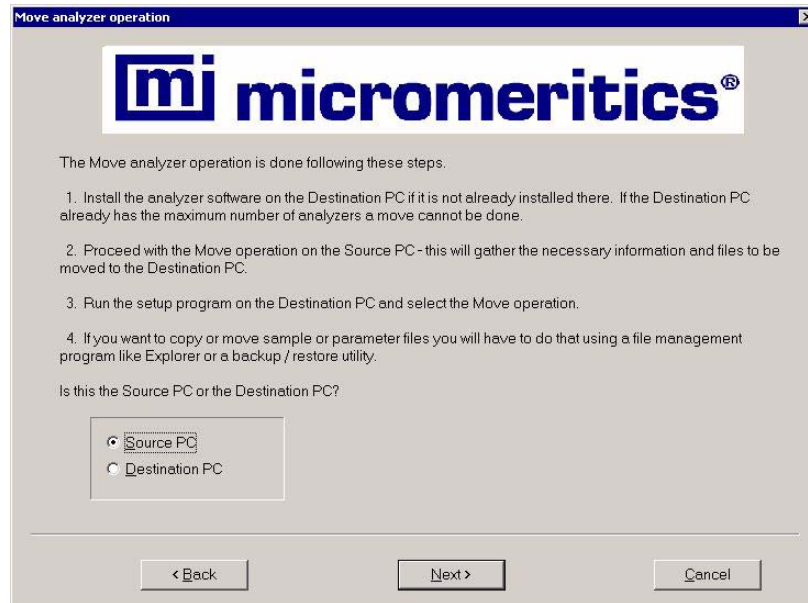
4. Select **Source PC,** then click **Next**; the following dialog is displayed.
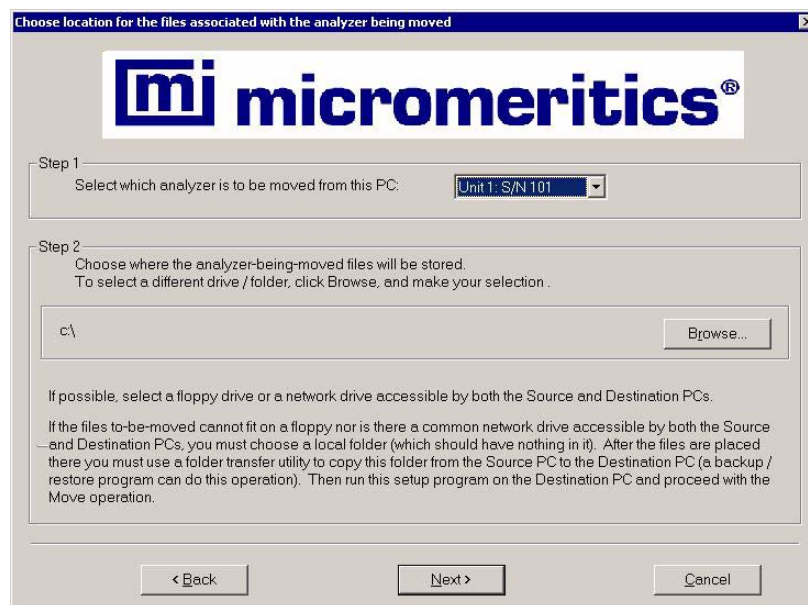
5. In the Step 1 group box, select the analyzer that is to be moved.

6. In the Step 2 group box, choose a location in which the moved files will be stored. If possible, choose a diskette drive or a shared network drive. If this is not possible, select a local folder. After the files are placed there, use a folder transfer utility (for example, Backup and Restore) to copy this folder from the Source PC to the Destination PC.

7. Click **Finish**; the files are moved and the setup Welcome screen is displayed.

8. Start the application setup program on the destination computer.

9. In the Setup dialog, select **Move an analyzer from one PC to another PC**; the Move analyzer operation dialog is displayed (shown on previous page).

10. Select **Destination PC**, then click **Next**; the following dialog is displayed.

11. In the Step 1 group box, enter the serial number of the analyzer being moved.

12. In the Step 2 group box, click **Browse** and choose the location of the moved files.

13. Click **Next**; the files are moved and the setup Welcome dialog is displayed.

Removing an Analyzer

You can remove an analyzer from the computer as follows. When you remove an analyzer, the status files are removed as well.

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **4-4**.

2. From the Setup dialog, select **Remove an analyzer**, then click **Next**; the Remove an analyzer dialog is displayed.
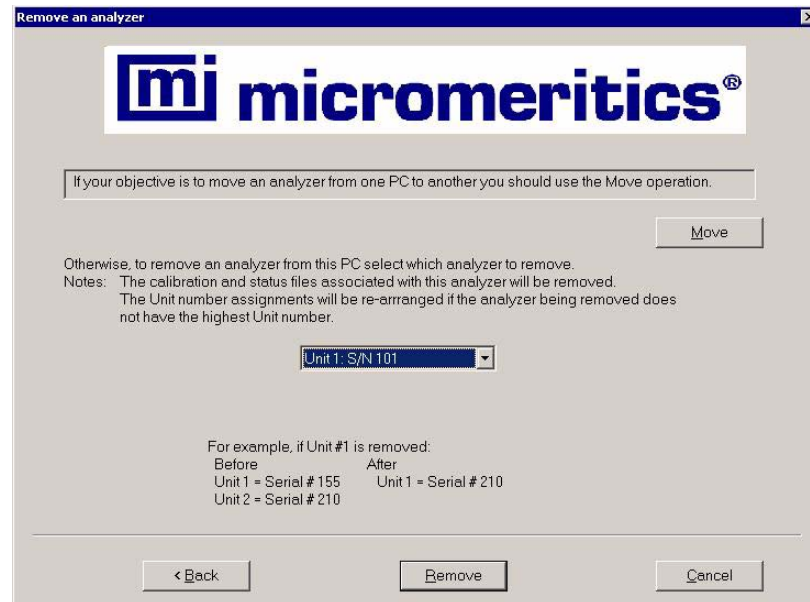
3. From the drop-down list, choose the serial number of the analyzer you want to remove.

4. Click **Remove**; the analyzer is removed and the Welcome screen is again displayed.

5. Click **Exit** to close the dialog.

Changing an Analyzer Setup

Change the analyzer setup as follows:

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions** on page **4-4**.

2. From the Setup dialog, select **Change analyzer setup**, then click **Next**; the Change analyzer setup dialog is displayed.
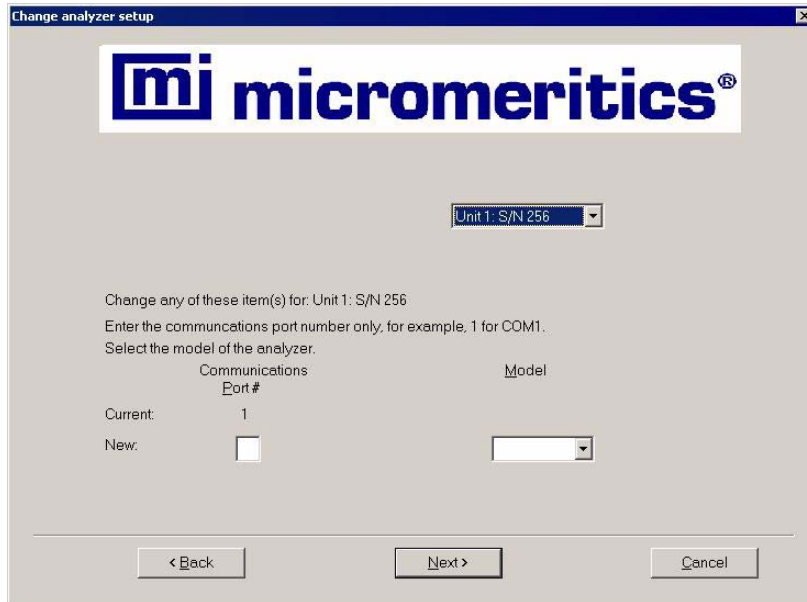
3. From the drop-down list, choose the analyzer you want to change.

4. Enter the new port number in the space provided.

5. Click **Finish;** the change is completed and the Welcome dialog is again displayed.

6. Click **Exit** to close the dialog.

Reinstalling the Calibration Files

You can reinstall the files containing an analyzer's factory calibration data as follows:

1. Start the Setup program. From the Setup dialog, select **Re-install calibration files for an analyzer**, then click **Next**; the Re-install calibration files dialog is displayed.

Not shown if only one analyzer is installed.
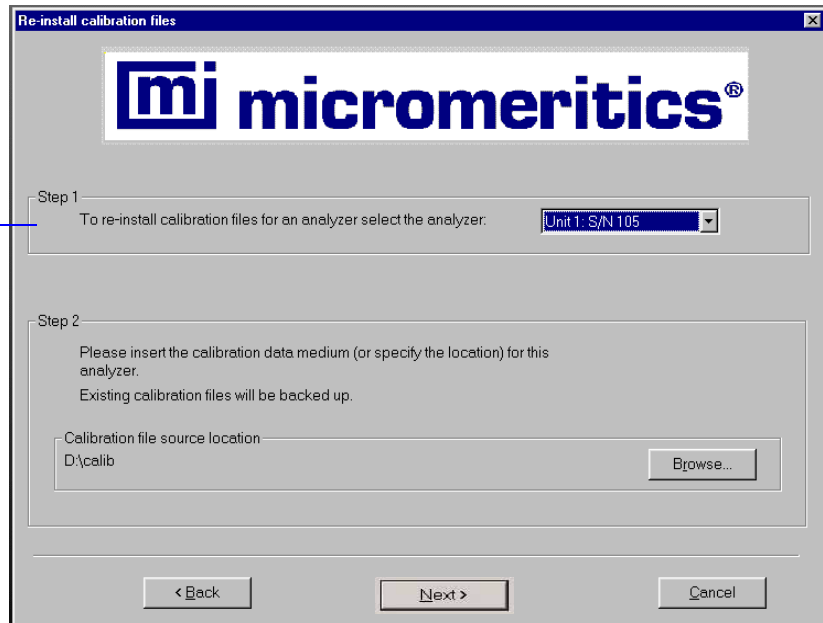
2. Select the analyzer whose calibration files you wish to reinstall from the drop-down list. If you have only one analyzer installed, a drop-down list is not included.

3. Insert the CD containing the calibration files; ensure that the CD drive is displayed as the calibration file source location. If not, click **Browse** and choose the CD drive.

4. Click **Finish;** the calibration files are reinstalled and the Welcome dialog is again displayed.

5. Click **Exit** to close the dialog.

Uninstalling the Analysis Program

When you uninstall the ASAP 2050 analysis program, the application removes the analysis program, status files, analyzer setup files, and resulting empty directories. It does not remove data files. Perform the following steps to uninstall the program:

1. Start the Setup program. From the Setup dialog, select **Uninstall**, then click **Next**; the Uninstall dialog is displayed.
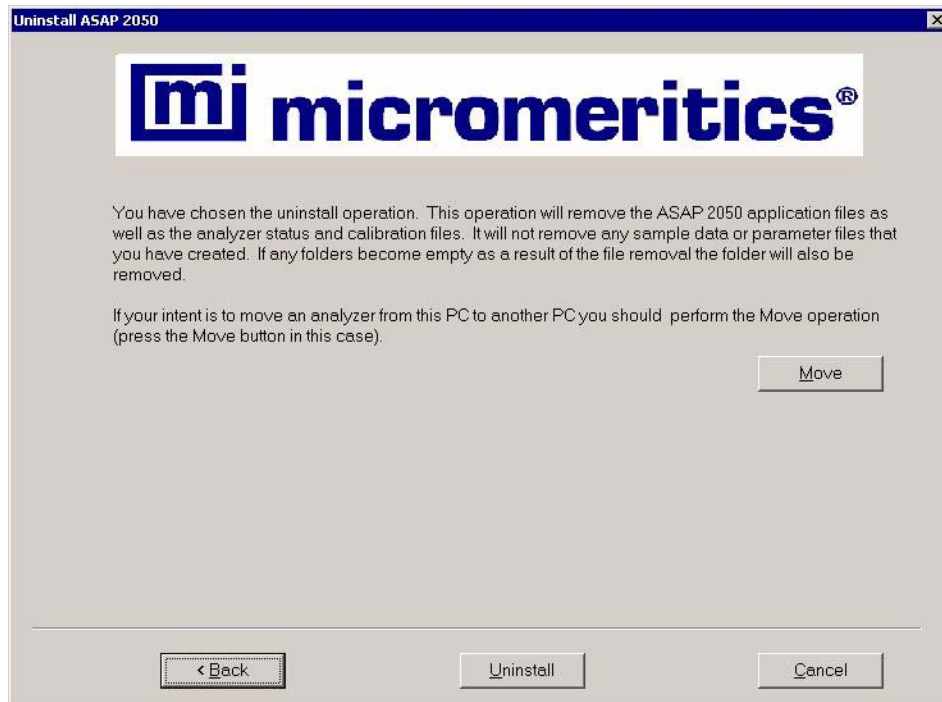
2. Click **Uninstall;** the Select Uninstall Method dialog is displayed.

3.   Choose one of the following:

   Automatic: click **Next**; the system uninstalls the analysis program automatically and the setup Welcome dialog redisplays.

   Custom: click **Next**; a series of dialogs is displayed, allowing you to choose the files you wish to uninstall. After all files are selected and uninstalled, the setup Welcome dialog redisplays.

4.   Click **Exit** to close the Welcome dialog.

5. FILE MENU

The File menu contains commands which allow you to manage sample and parameter files.

Description

Listed below are brief descriptions of the File menu commands. Detailed descriptions follow this section.

Open	Opens an existing sample information or parameter file. Page **5-2**.
Save	Saves the file in the active window. Page **5-81**.
Save As	Saves the file in the active window as a different name. You also can use this option to save a subset of the sample file as a parameter file. Page **5-81**.
Save All	Saves all open files. Page **5-83**.
Close	Closes the file in the active window. Page **5-83**.
Close All	Closes all open files. Page **5-83**.
Print	Prints the contents of a sample or parameter file. Page **5-84**.
List	Generates a list of certain information for sample or parameter files. Page **5-85**.

Export	Copies the isotherm data in a sample information file and exports it in ASCII format. Page **5-86**.
Exit	Exits the analysis program. Page **5-88**.

Open

Open allows you to create a new file or edit an existing one. The following file types are available:

- Sample information
- Sample tube criteria
- Degas conditions
- Analysis conditions
- Adsorptive properties
- Report options

Regardless of which file type you select, a dialog similar to the one shown here is displayed:
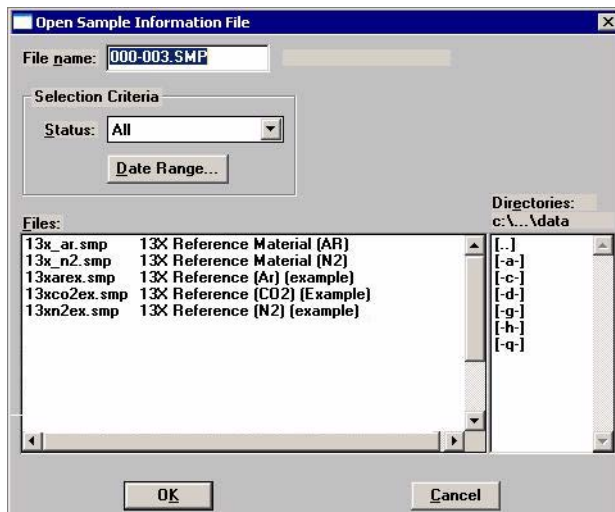
File name	For *sample information* files, this field contains the next sequenced file name (as specified in sample defaults) generated by the system. If this is a new file, you can use the name displayed or enter a different one.
	For *parameter* files, the file name displayed includes the wild card (*) and a default extension as follows:

File name
(continued)

*.DEG for degas conditions
*.STB for sample tube
*.ANC for analysis conditions
*.ADP for adsorptive properties
*.RPO for report options

If you are creating a new file, enter a name in the **File name** field. You can use up to 8 characters.

If you are opening an existing file, select the name from the **Files** window, then click **OK**

Date Range

Click this push button to display files created within a specified range of dates; the Select Dates dialog is displayed.

Refer to **Selecting Files**, page **2-11** for an explanation of the functions on this dialog.

Status

Displays for sample information files only. This drop-down list allows you to choose the types of sample files to display in the **Files** window. All files of the type you choose, within the range of dates, and in the current directory are displayed. Refer to **Table 2-2. File Status and Description**, page **2-12** for a description of the status types.

Directories

Displays the current directory. You can navigate to a different directory by clicking in the **Directories** list box or by entering the desired directory in the **File name** field.

Sample Information

Sample information files can be created and presented in the Advanced, Basic, or Restricted format. (Refer to **"Sample Information Files" on page 3-1**.)

Advanced Format

The Advanced format displays all parts of the sample file in a tabbed dialog. This format allows you to customize your sample file, moving easily among the parameters simply by clicking on the tabs; alternatively, you can use **Prev** and **Next**. Refer to **"Advanced Format" on page 3-6** for step-by-step instructions for creating a sample information file using the Advanced format.

These fields can be edited to display a different label if desired.

Sample   Contains the description of the current sample file. If this is a new file, this field contains the file description specified in Sample Defaults (refer to Advanced Format on page **8-7**). The above dialog shows the sample file number because the dollar ($) symbol was included when sample defaults were specified.

You can enter a new description or edit the existing one if desired.

Maximum number of characters: 50

Operator   Displays the operator and submitter names of the current
Submitter   sample file.

                                      If this is a new file, these fields contain the names specified in
                                      Sample Defaults.

                                      You can enter a different name or edit the current one if
                                      desired. You can use up to 40 characters.

                                      Either (or both) of these fields can be prevented from
                                      displaying on this dialog by selecting **Omit** in Sample
                                      Defaults. The labels also can be edited in **Sample Defaults**.

Mass   Enter the value recorded for *Mass of Sample, After Degas*
                                      from the Sample Data Worksheet.

                                      The value in this field is used in report calculations;
                                      therefore, it should be exact. To ensure accuracy, reweigh the
                                      sample after analysis. Enter the post-analysis value if
                                      different from the *After Degas* value before generating
                                      reports.

> **You should always use the same gas (typically nitrogen) for degassing and analysis. If different gases are used, the sample mass after degas and after analysis will not be equal. For example; if you use helium to backfill after degassing and nitrogen after analysis, you can get as much as a 0.02-gram difference.**

Density   If you plan to use a calculated free space, enter the sample's
                                      density in this field. A value in this field is ignored for
                                      measured or entered free spaces.

Type of Data   Displays the type of data for the current sample file.

                                      If this is a new file, choose whether you wish to have data
                                      collected automatically or if you plan to enter data collected
                                      from another source.

Comments   Allows you to enter pertinent information about the sample
                                      or analysis. The information you enter in this window is
                                      displayed in the report header. You may enter up to 500
                                      characters in this window.

Add Log Entry   Displays the Add Log Entry dialog, enabling you to enter
                                     comments to be included in the instrument log.

Replace All   Use this push button to replace all parameters of the current
                                     file with those from an existing one. You can edit the
                                     parameters after they have been copied into the current file;
                                     editing the current file does not affect the file from which
                                     they were copied.

Save   Saves all parameters of the current file; the dialog remains
                                     open.

Close   Closes the dialog. If the file contains unsaved changes, you
                                     are prompted to save before the dialog closes.

Basic   Switches the sample editor to the Basic format.

Basic Format

The Basic format displays all parts (parameters) of the file on a single dialog. With this format,
you can quickly create a sample information file using predefined parameter files. You can
also switch to the Advanced format if a specific parameter requires editing. Refer to **"Basic
and Restricted Formats" on page 3-8** for step-by-step instructions for creating a sample
information file using the Basic format.

Sample	Contains the description of the current sample file.
	If this is a new file, this field contains the next sequenced file description based on the format specified in Sample Defaults. The above dialog shows the sample file number because the dollar ($) symbol was included when sample defaults were specified.
	You can enter a new description or edit the existing one if desired.
	Maximum number of characters: 50
Mass	Allows you to enter the sample mass. If you are using the Sample Data worksheet, this is the value recorded for *Mass of Sample after Degas*.

Sample Tube
Degas Conditions
Analysis Conditions
Adsorptive Properties
Report Options
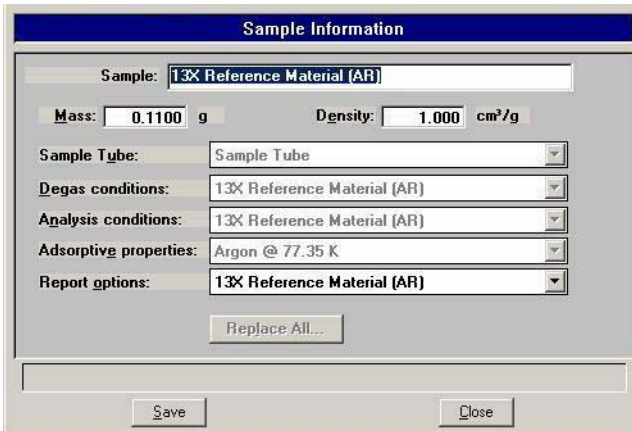
Each parameter field contains the description of the current parameter file.

If this is a new sample file, these fields contain the descriptions of the files chosen as the defaults.

Click on the down arrow to the right of each field to choose a different file. File parameters can be viewed or edited by switching to the Advanced format.

Replace All

Use this push button to replace all parameters of the current file with those copied from an existing one. A dialog is displayed allowing you to choose the desired file.

Save

Saves all parameters of the current file; the dialog remains open.

Close

Closes the dialog.

Advanced

Switches the sample editor to the Advanced format, allowing you to view or edit parameters of the current file.

Restricted Format

The Restricted format is used when analysis parameters must remain constant, for example, in the pharmaceutical industry where consistency and accuracy are crucial. A password is required to enter and exit this format. Refer to **Restricted**, page **8-3** for additional information on the Restricted format.

When you open an existing sample information file or create a new one using the Restricted format, all parts of the sample file are displayed in the same manner as the Basic format. Some menu functions, however, are disabled and you cannot switch to the Advanced format to edit file parameters.
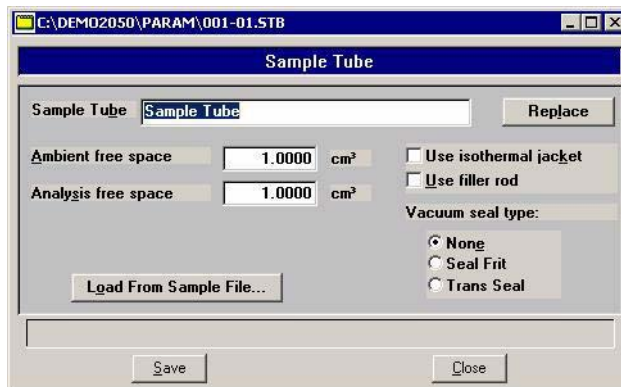
Sample Tube

The fields on this dialog are used for storing sample tube information. Before creating a sample tube file, you should perform a blank analysis (no sample) using the sample tube.

Sample Tube	Contains the description of the current file.
	If this is a new file, this field contains the name (description) you specified as the default. You can enter a new description or add to the existing one if desired.
	Maximum number of characters: 40

Replace   Use this push button to replace the values of the current file
                                        with those from an existing Sample Tube file. The Open
                                        Sample Tube dialog is displayed, allowing you to choose a
                                        file. After the values are copied into the current file, you can
                                        edit them as desired.

Ambient free space   Displays the ambient free space for the current file.

Analysis free space   Displays the analysis free space for the current file.

Use isothermal jacket   Select this option if an isothermal jacket is to be used in the
                                        analysis.

Use filler rod   Not used.

Vacuum seal type   If the sample is to be transferred under vacuum to the analy-
                                        sis port, select the one to be used. If not, leave **None** selected.

Load From Sample File   Click this push button to choose the sample file that was used
                                        in the blank analysis. The values for the Ambient and Analy-
                                        sis free spaces will be copied into the sample tube file.

Save   Saves all parameters of the current dialog.

Close   Closes the dialog. If the file contains unsaved changes, you
                                        are prompted to save before the dialog closes.

Degas Conditions

The fields on this dialog are used for specifying degas conditions.

Description	Contains the description of the current Degas Conditions file.
	If this is a new file, this field contains the name (description) you specified as the default. You can enter a new description or add to the existing one if desired.
	Maximum number of characters: 40
Replace	Use this push button to replace the parameters of the current file with those from an existing Degas Conditions file. The Open Degas Conditions File dialog is displayed, allowing you to choose a file. After the values are copied into the current file, you can edit them as desired.
Evacuation Phase group box	The options in this group box allow you to specify conditions for the evacuation phase of the degassing operation.
Temperature ramp rate	Allows you to specify the rate at which the temperature is to change while advancing to the target temperature during evacuation.
Target temperature	Allows you to specify a temperature at which the sample will be held for the remainder of the evacuation.

Evacuation rate	Enables you to specify an evacuation rate for initial evacuation.
Unrestricted evac. from	Enables you to specify a pressure at which unrestricted sample evacuation will begin.
Vacuum setpoint	Enables you to specify the vacuum level to be achieved before evacuation begins.
Evacuation time	Enables yo to specify how long the sample is to be evacuated prior to the second stage of heating.
Heating Phase group box	The options in this group box allow you to specify conditions for the heating phase of the degassing operation.
Ramp rate	Enables you to specify the rate at which the temperature will change after evacuation while advancing to the hold temperature.
Hold temp	Enables you to specify a temperature at which the sample is to be held during degassing.
Hold time	Enables you to specify how long the sample is to be held at the specified temperature before beginning to cool down.
Evacuation and Heating Phases	Enables you to specify the hold pressure.
Hold Pressure	If during the temperature ramp the pressure exceeds the value you enter in this field, the ramp will be suspended until the pressure returns to a safe level.
Backfill Sample Tube	Select this option to have the sample tube backfilled.
Save	Saves all parameters of the current dialog.
Close	Closes the dialog. If the file contains unsaved changes, you are prompted to save before the dialog closes.

Analysis Conditions

The Analysis Conditions dialog allows you to specify the data used to guide the analysis. You can create an Analysis Conditions file as an independent parameter file or include it as part of the sample information file.

Refer to **"Analysis Conditions" on page 3-11** for step-by-step instructions on creating an analysis conditions file.
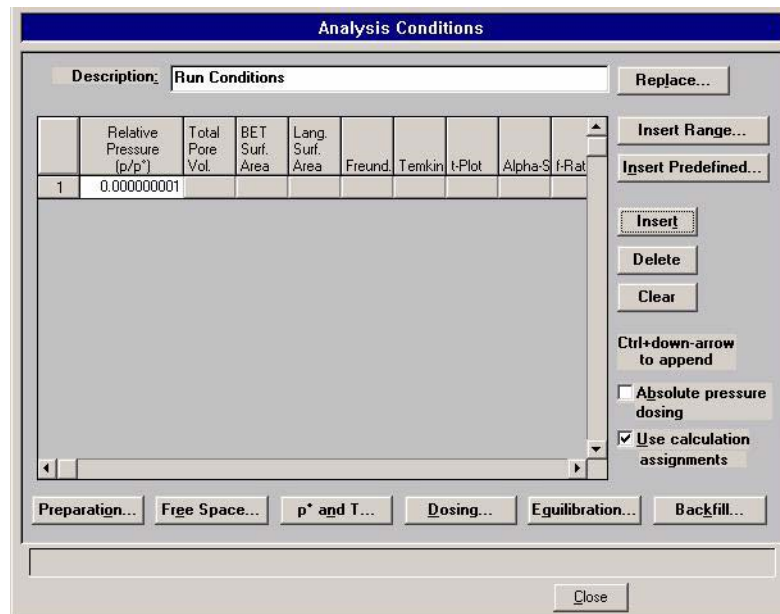
	Description	Contains the description of the current Analysis Conditions file.

Description   Contains the description of the current Analysis Conditions file.

If this is a new file, this field contains the name you specified as the default. You can enter a new description or add to the existing one if desired.

Maximum number of characters: 40

Replace   Use this push button to replace the parameters in the current file with those from an existing Analysis Conditions file. After you choose the file and the values are copied into the current file, you can edit them in any way desired. Editing the current file will not change anything in the file from which they were copied.

Pressure Table

A pressure table is a list of pressure points (with calculation assignments) at which data are to be collected. The pressures may span the entire range from the lowest relative value of 0.00000001 to the maximum value of 0.995 P/Po. In addition, one saturation point may be entered with relative pressure of 1.0.

Several analysis conditions files containing complete pressure tables are included with the ASAP 2050 software and can be found in the PARAMS subdirectory.

The pressure table for automatically collected data includes the pressure points for data collection and identifies calculations through which the data are processed.

Edit existing values by highlighting the field and entering the desired values. Use **Insert** to add rows for additional values. To append to the beginning or to the end of the table, use **Ctrl + Up Arrow** and **Ctrl + Down Arrow**.

The pressure table must contain one sequence of strictly ascending relative pressures, optionally followed by one sequence of strictly descending relative pressures. You can enter a maximum of one thousand pressure points.

To specify the calculations performed at a given relative pressure, click in the field of the desired calculation type; an **X** in the field indicates the calculation is selected.

Click **OK** to accept all entries in the pressure table and return to the Analysis Conditions dialog.

Insert Range   Displays the Insert Pressure Range dialog.
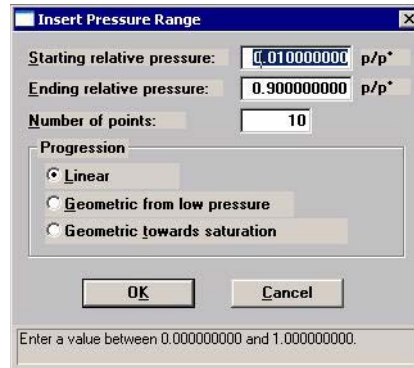
This dialog allows you to specify the starting pressure, the ending pressure, the number of points to insert within the specified range, and whether you wish to have linear or geometric progression.

Linear   Inserts evenly spaced points into your table.

Geometric from low pressure   Inserts geometrically spaced points from the low pressure range. For example, to insert 5 points with a 0.01 starting pressure and a 0.16 ending pressure, the following points are inserted into the table:

        0.01
        0.02
        0.04
        0.08
        0.16

Geometric from saturation   Inserts geometrically spaced points from the saturation pressure. For example, to insert 5 points with a 0.99 starting pressure and a 0.84 ending pressure, the following points are inserted into the table:

        0.99
        0.98
        0.96
        0.92
        0.84

Insert predefined   Displays the Insert Predefined Pressures dialog.
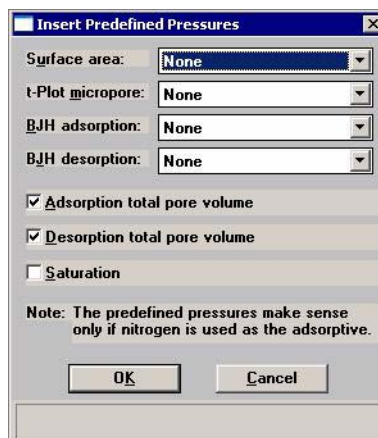
Choose predefined pressure points for surface area, t-Plot, and/or BJH adsorption/desorption.

Click on the down arrow for each field to choose the desired set of points. You can also specify Adsorption/ Desorption total pore volume and Saturation.

Insert   Inserts a row into the table above the selected line.

Delete   Deletes the selected row.

Clear   Removes all but one required entry from the table. The rows do not have to be selected. A warning message appears requesting confirmation before the table is cleared.

Absolute pressure
dosing   Select this option to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature.

Use calculation assignments

Select this option to assign the points to be collected for each type of report.

If this option is not selected, each report (with the exception of Langmuir and BET) uses a range of pressures as selected in the report options. The Langmuir and BET reports interpolate to entered relative pressures on the report options.

The outlier points can be selected so that they will not be reported.

Preparation

Displays the Analysis Preparation dialog.
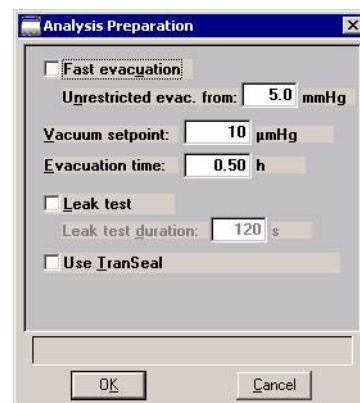
Fast evacuation

Select this option for samples (such as pellets) that do not fluidize or shed particles during evacuation.

Unrestricted evac. from

Enabled when Fast evacuation is not desired. This field allows you to enter the pressure at which unrestricted sample evacuation is to begin.

Vacuum setpoint

Allows you to specify the vacuum level to be achieved before evacuation begins.

Evacuation time

Enables you to enter the length of time for preliminary evacuation, which takes place prior to the free-space measurement.

Leak test   Enables the system to check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and you are notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists.

Leak test duration   Enables you to specify how long the pressure is to be monitored.

Use TranSeal   Not used.

Free Space   Allows you to specify free-space measurement conditions; the Free Space dialog is displayed.
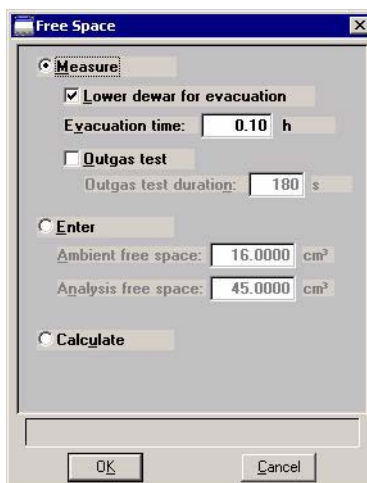
Measure   Select this option to have free space measured automatically by the system.

Typically, it is best to measure the free space when analyzing macro- and mesoporous samples, and for routine surface area measurements.

Select **Lower dewar for evacuation** to lower the dewar following free-space measurements.

Evacuation time enables you to specify how long evacuation is to take place following the measured free space.

Select **Outgas test** to check for system leaks or sample outgassing. After free space is measured, the Dewar is lowered and the sample evacuated for 30 minutes. The leak test is performed after evacuation. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the analysis stops and you are notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak occurs.

Outgas test duration indicates the number of seconds the leak test is performed.

Enter   Select this option to enter values for the ambient and analysis free spaces. You may need to use this option if the sample retains helium and measurement of very low micron range pressures are required.

> If you change the entered free-space value after the preliminary analysis stage is 50% complete, the change does not affect the analysis.

Ambient free space is the sample tube gas capacity measured at room temperature.

Analysis free space is the sample tube gas capacity measured with the Dewar raised.

Calculate   Select this option to have the free-space calculated using the mass, density, and sample tube parameters entered in the Sample Information and Sample Tube files. Calculated free space measurement takes less time than measured free space, so if you know the mass and density of the sample and the sample tube parameters, you may wish to use this choice of free space methods. If you do not know the parameters, you may wish to use *measured* free space.

Po and T   Displays the Po and Temperature Options dialog, allowing you to enter the ambient temperature and choose saturation pressure and analysis bath temperature.
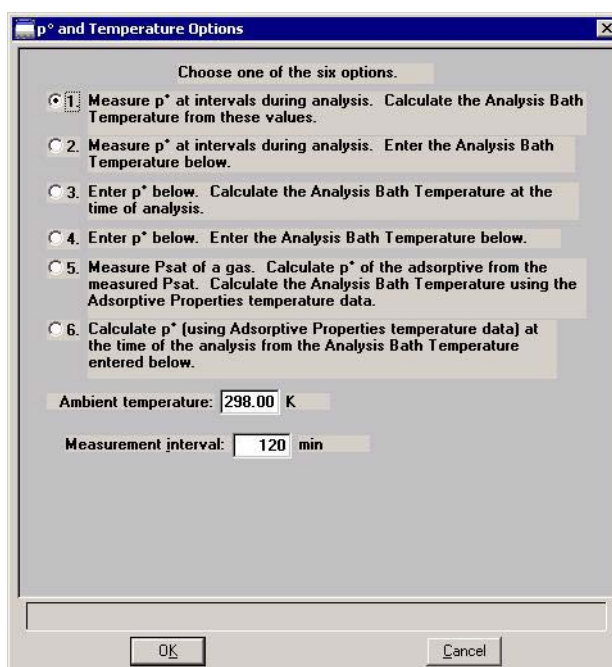
This dialog provides six options for obtaining the saturation pressure (Po) and analysis bath temperature. Clicking on each option prompts you for the entry of related parameters in the lower portion of the dialog.

Option	Prompts for...
1	a measurement interval (how often during analysis the Po is to be measured) Recommended for N_2, Ar

Option	Prompts for...
2	a measurement interval and the analysis bath temperature
3	a Po; the analysis bath temperature is calculated at the time of analysis

Saves time; useful for BET with N_2. |
| 4 | a Po and analysis bath temperature

an analysis bath temperature only (when **Absolute pressure dosing** is selected on the Analysis conditions dialog)

Saves time; useful for BET with N_2. |
| 5 | a psat gas other than the adsorptive; the temperature and pressure ranges for the chosen gas are displayed. Click Psat vs.T to edit the values if desired. Editing the values in the current table do not affect the ones of the original table.

May help save time. |
| 6 | an analysis bath temperature

May help save time. |

Dosing   Displays the Dosing Options dialog**.**
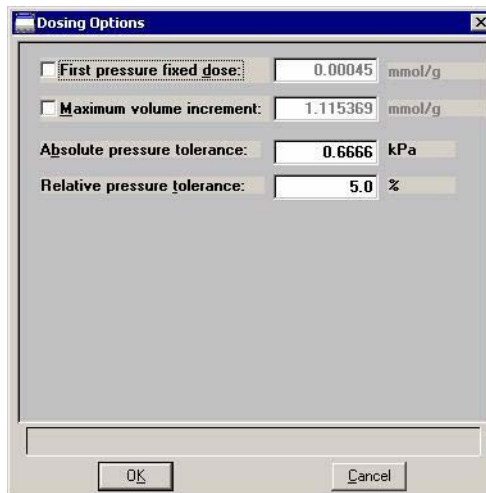
First Pressure Fixed   The most frequent use of this option is when you are
Dose   performing a standard nitrogen analysis of mesoporous
                                materials such as catalysts. If the first pressure table point is
                                low and you expect the gas uptake of the sample to be high,
                                this option can shorten the time required to reach the first
                                point on the pressure table.

                                The sample is dosed repeatedly at low pressures with a
                                specified amount of gas until the first pressure point is
                                reached. This initial dosing quickly meets the adsorptive
                                demand of the sample**.**

                                The first point on the pressure table is the threshold value,
                                triggering the transition from the **Fixed Dose Mode** to
                                Pressure Table Mode. When the first pressure table value is
                                reached, Fixed Dose Mode is disabled, and points are
                                equilibrated and recorded in accordance with the specified
                                pressure table.

                                In the field adjacent to this option, enter the amount of gas
                                that is to be added to the sample for each dose cycle.

Maximum volume increment

Select this option to determine when additional data points are collected between target pressures in regions of adsorption. When the maximum increment has been adsorbed since the last collected data point, another point is equilibrated and collected.

During desorption, this field is treated as a maximum volume "decrement" value**.**

Absolute and Relative pressure tolerance

The values entered in these fields are used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures the relative tolerance value is smaller, and at higher pressures the absolute tolerance value is smaller. The lesser (or more stringent) of these two criteria are used to determine the tolerance.

For example, with relative tolerance = 5% and absolute tolerance = 5 mmHg, the relative tolerance at 40 mmHg target pressure is 5% of 40 mmHg, or 2 mmHg; 2 mmHg is smaller than the absolute tolerance of 5 mmHg, so 2 mmHg is used. At 200 mmHg target pressure, the relative tolerance is 5% of 200 mmHg, or 10 mmHg; in this case, the absolute tolerance of 5 mmHg is smaller and is used.

In the above example, a minimum pressure of 40 - 2 = 38 mmHg must be attained to collect data for a target pressure of 40 mmHg. For a target of 200 mmHg, 200 - 5 = 195 mmHg must be attained.

Normally, surface area measurement points are widely spaced, and the resulting measurement is not very sensitive to the precise location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis.

Equilibration   Displays the Equilibration dialog.
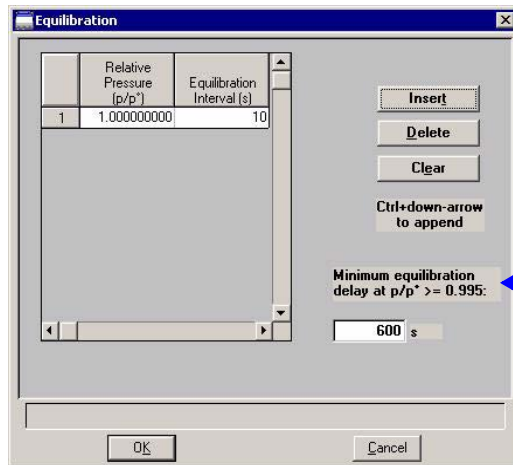
Does not display when **Absolute dosing** is selected.

Equilibration interval

The number of seconds between successive pressure readings during equilibration. Long equilibration intervals tend to lengthen analyses, however, they do improve data integrity. Short equilibration intervals produce a faster analysis but may reduce the accuracy of data.

Minimum equilibration delay at P/Po> = 0.995

The value entered in this field determines the minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995.

This field does not display if you select **Absolute pressure dosing** on the analysis conditions dialog.

Backfill

Displays the Sample Backfill Options dialog, allowing you to select options for backfilling the sample tube at the start and end of an analysis.
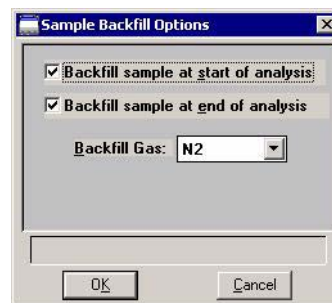
The options on this dialog are not available if the file status displayed in the Open Sample Information File dialog is Entered, Complete, or Analyzing.

Backfill sample at   Backfills the sample tube at the beginning of the analysis
start of analysis   with the specified backfill gas.

Backfill sample at   Backfills the sample tube at the end of the analysis with the
end of analysis   specified backfill gas. If the sample mass is determined after
                                the analysis, it is best to use nitrogen as the backfill gas.

> ⚠ **It is recommended that you backfill the sample tube at the start and at the end of the analysis. If you deselect either backfill option, proceed with caution as damage to the analyzer or sample tube may occur**.

Backfill Gas   Lists the available backfill gases. The gases in this list are
                                the ones for which Adsorptive Properties files have been
                                created; helium is also included.

Adsorptive Properties

This dialog allows you to specify the characteristics of the gases used in the ASAP 2050 system. An Adsorptive Properties file can be created as an independent parameter file or included as part of the sample information file.

Refer to **"Adsorptive Properties" on page 3-12** for step-by-step instructions on creating adsorptive properties files.

Adsorptive   If this is a new file, this field contains either the name of the
                                  default adsorptive gas or is blank. Enter the name of the
                                  desired adsorptive gas.

                                  Maximum number of characters: 40 alphanumeric

Replace   Click this push button to choose an Adsorptive Properties file
                                  containing parameters you wish to copy and use in the
                                  current file. After the values have been copied into the file,
                                  you can edit them as desired. It does not affect the values of
                                  the file from which they were copied.

Mnemonic   The mnemonic for the adsorptive gas.

                                  Maximum number of characters: 3 alphanumeric

Non-condensity   Select this option if this file is for a non-condensing gas
Adsorptive   (adsorptive). When you select this option, the **Density
                                  conversion factor** field and the **Psat vs. T** push button
                                  become disabled.

Maximum manifold   The maximum gas pressure allowed in the manifold.
pressure

Density conversion   This value is used to convert the gas volume at STP to liquid
factor   volume for the adsorptive. It is the ratio of gas density at STP
                                  to liquid density.

Therm. tran. hard-sphere   The thermal transpiration hard-sphere diameter.
diameter

Molecular cross-sectional   The molecular cross-sectional area.
area

Dosing Method   Select the manner in which the sample tube is dosed. **Normal**
                                  applies to gases under pressure and is used for standard 2050
                                  analyses.

Compressibility Factor Table Compressibility factors compensate for the forces of attraction between molecules in a real gas. Each gas compatible with the ASAP 2050 analyzer has a compressibility factor table. To select the table, click **Open**. The Open Compressibility Factor Table dialog is displayed.
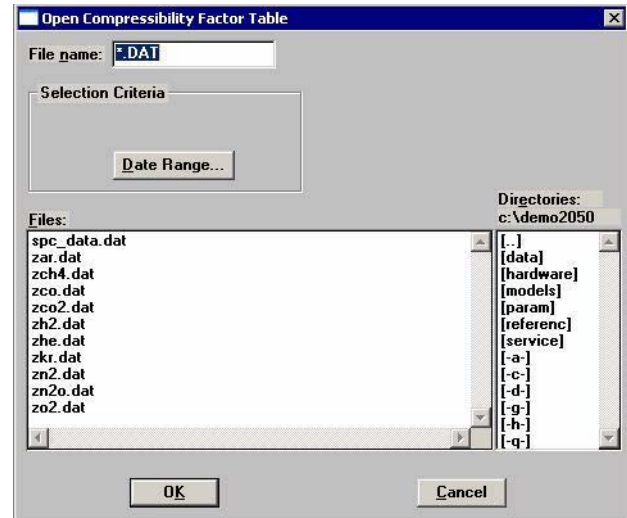
Click the file containing the mnemonic for the type of gas to be used for analysis. For example, select **zar.dat** for Argon.

Then click **OK** to close the dialog.
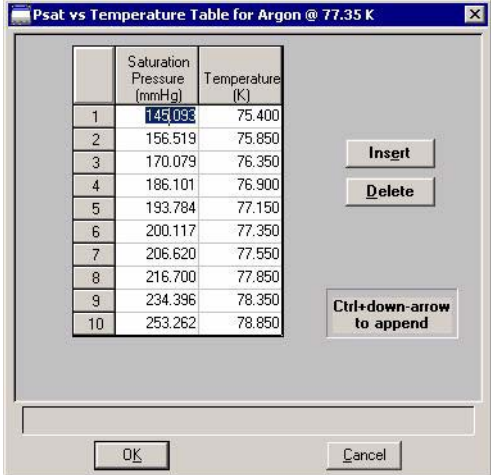
If you would like to save the compressibility factor table contained in a sample file, click the **Save** button located next to the Open button.
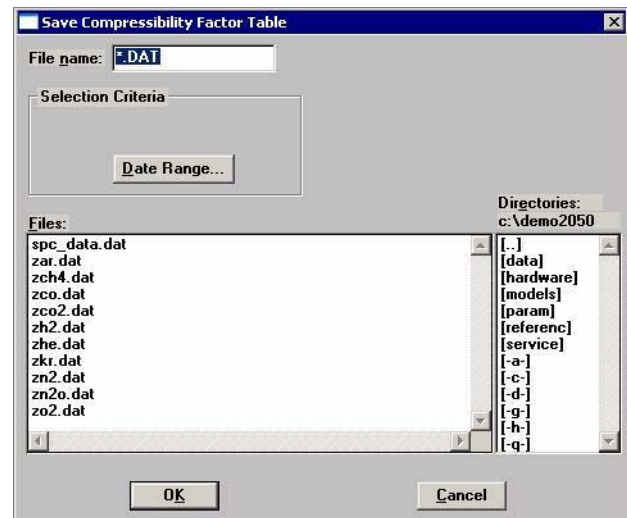
Enter a name in the File name field (make sure you use the .DAT extension), the click **OK**.

Psat vs. T   Click to view or edit the Psat vs. Temperature table for the
                                            adsorptive gas; this example shows Nitrogen.

This table contains saturation pressures and their correspond-
ing temperatures. As many as 10 entries may be included; at
least two entries are required.

You can edit the table by clicking on the desired saturation
pressure or temperature and entering a different value.

Use **Insert** to insert a new row above the selected line; the
cursor moves to the new line. Use **Delete** to delete the
selected row.

Report Options

This dialog allows you to specify report options. A report options file can be created as an independent parameter file or included as part of the sample information file.

Refer to **"Report Options" on page 3-13** for instructions on creating report options files.
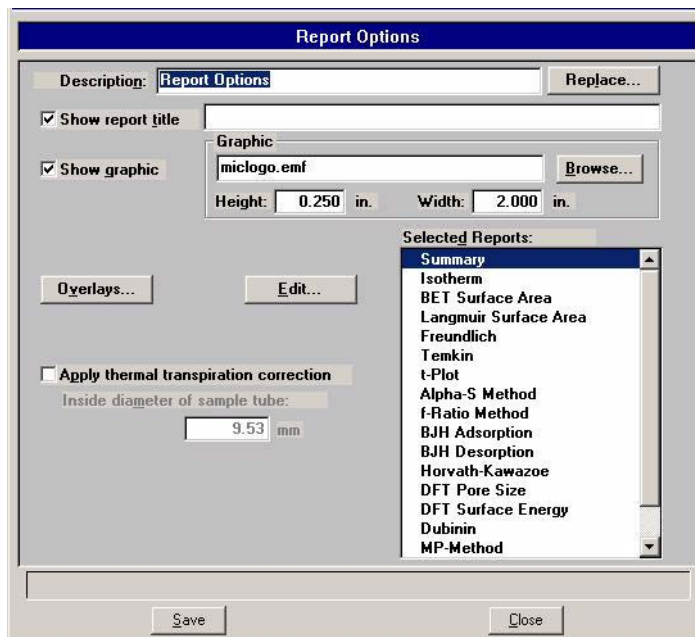
Description	Displays a description of the current Report Options file.
	If this is a new file, this field contains the description you specified as the default. You can enter a new description or add to the existing one if desired.
	Maximum number of characters: 40
Replace	Click this push button to choose an existing Report Options file containing parameters you wish to copy and use in the current file. After the values have been copied into the file, you can edit them as desired. It does not affect the values of the file from which they were copied.

Show report title   This option allows you to enter a title for your report.

If this is a new file, the title you specified as the default is displayed. Accept the default title or enter a different one.

Deselect this option to omit the report title.

Range: 40 characters

Show graphic   Select this option to have a graphic display above the report title.

The graphic can be in a bitmap (bmp) or an enhanced metafile (emf) format. For example, you may wish to display your company logo.

Click **Browse** to choose the graphic, then use the height and width fields to specify the size. The image can also be edited from the report window.

Overlays   Displays the Graph Overlay Samples dialog so that you can choose the sample files containing the data you wish to overlay onto a selected plot.
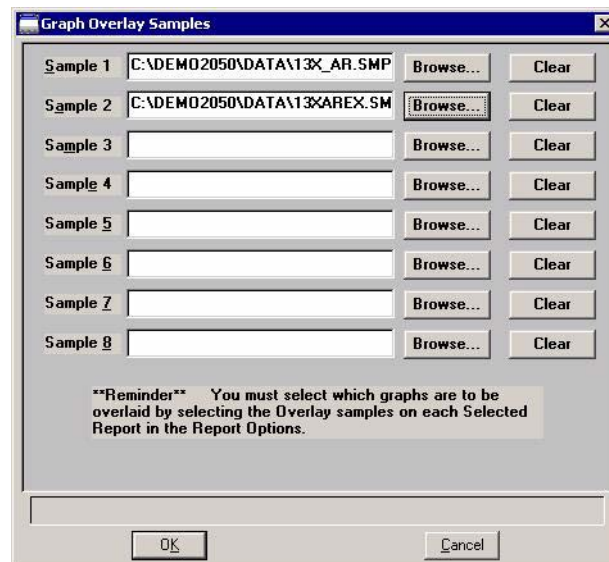
Click **Browse** to the right of the sample number field to choose the desired file. Use **Clear** to clear a field of its entry. You can select up to eight files.

After choosing the desired files, select the **Overlay samples** option for each report type you plan to overlay.

Edit   Allows you to edit parameters of the selected report. This
                                            push button is disabled for the Options report.

Apply thermal transpiration   Select this option to correct for the temperature-induced
correction   pressure difference between the manifold and the chilled
                                           sample tube. This option is most significant for pressures less
                                           than approximately 1.0 mmHg.

                                           Use this option when a cryogen liquid nitrogen or liquid
                                           Argon is used to control the bath temperature.

Inside diameter of   Enabled when you select **Apply Thermal transpiration**
sample tube   **correction**, so that you may enter the inside diameter of the
                                           sample tube.

Selected Reports   Contains a list of available reports. Choose reports by double-
                                           clicking on the desired report(s). Alternatively, you can
                                           highlight the desired report and press the Spacebar. A report
                                           is selected when it is preceded with a check mark. You may
                                           deselect reports in the same manner.

                                           The following reports are available:

                                           • Isotherm
                                           • BET Surface Area
                                           • Langmuir Surface Area
                                           • Freundlich
                                           • Temkin
                                           • t-Plot
                                           • Alpha-S Method
                                           • f-Ratio Method
                                           • BJH Adsorption
                                           • BJH Desorption
                                           • DFT Pore Size
                                           • DFT Surface Energy
                                           • Dubinin
                                           • Options
                                           • Summary
                                           • Sample Log

Isotherm Report Options

The isotherm report indicates adsorption (up to saturation pressure) and desorption (down from saturation pressure) of a gas by a solid held at constant temperature.
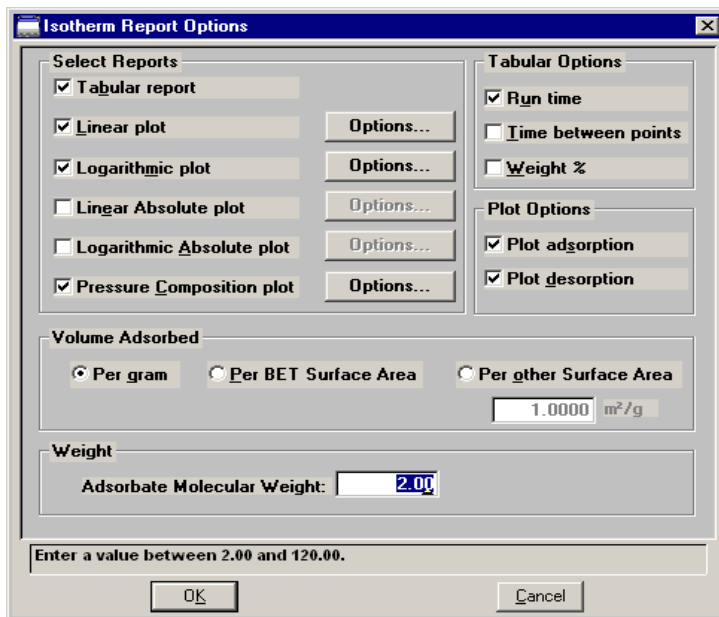
Select Reports   Lists the types of reports offered.

> *Choices: Tabular Report, Linear plot, Logarithmic plot,*
> *Linear Absolute plot, Logarithmic Absolute plot*
> *Pressure Composition plot*

When you select *Pressure Composition plot*, the **Adsorbate Molecular Weight** field is enabled so that you can enter a value for the adsorbate. This plot is useful for plotting pressure as a function of Weight % adsorbed; for example, H_2 adsorbed on carbon.

Options   Displays the related Plot Options dialog; this example shows
                                         the dialog for the Linear plot.
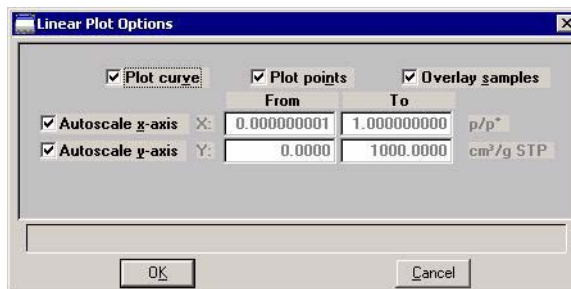
All plot dialogs contain the same options.

You can plot the graphs as a curve, points, or both.

Select **Overlay samples** to overlay data from the current plot
with data from other samples. The other sample files are
selected by clicking **Overlays** on the Report Options dialog.

Autoscale options enable you to have the X- and/or Y-axes
automatically scaled.

Linear X-axes begin at zero, and logarithmic X-axes begin at
an appropriate value. Y-axes begin at zero. The system uses
the highest values collected during analysis as the ending
points for axes ranges.

If you choose not to autoscale data, the **From** and **To** fields
are enabled, allowing you to specify a range. Data collected
outside these ranges are not included in the plot.

The value entered in the **To** field must be greater than the
value entered in the **From** field.

The X-axis fields show the relative pressure. The Y-axis
fields show the quantity of gas adsorbed

Tabular Options   Enables you to have **Run Time** and/or **Time Between Points**
                                         reported. Run time reports the time elapsed from the
                                         beginning of the analysis to the finish. Time between points
                                         reports the time relapsed between each point.

                                         Also enables you to report Weight % when plotting pressure
                                         composition.

Plot Options   Enables you to choose the type of isotherm you wish to plot. You can plot the adsorption and/or the desorption isotherm.

Volume Adsorbed   Enables you to choose the manner in which the volume adsorbed is reported. These data are reported by default as **Per Gram** (cm^3/g). However, you can choose to report data **Per BET Surface Area** (cm^3/m^2) or **Per Other Surface Area** (m^2/g). If the latter field is selected, a field is enabled allowing you to enter a value.

Weight   The **Adsorbate Molecular Weight** field is enabled when you choose *Pressure Composition plot*, allowing you to enter the molecular weight of the adsorbate.

                                    This weight appears on tabular reports.

BET/Langmuir Surface Area Report Options

- The BET calculation obtains the sample surface area value by determining the monolayer volume of adsorbed gas from the isotherm data.

- The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer.

Displays as Langmuir Surface Area Report Options if the Langmuir report is being edited.

The Langmuir and BET Surface Area dialogs include the same fields; the operating instructions for both are the same.

Tabular report	Select this option to have a tabular report of the plotted data.
BET transform plot	Generates a traditional BET (Langmuir) surface area plot that is used to determine monolayer volume and BET C constant.
BET Isotherm plot	Uses the BET (Langmuir) monolayer volume and constant to calculate an isotherm.
Overlay samples	Allows you to overlay data of the selected type from the current plot with data from other samples. The other samples are selected by clicking Overlays on the main Report Options screen.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axes scaled automatically.

Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points. If you choose not to autoscale data, the From and To fields are enabled, allowing you to enter the ranges.

From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot. The values entered in the **To** field must be greater than the value entered in the **From** field.

The X-Axis Range fields show the relative pressure.

The Y-Axis Range fields show the quantity of gas adsorbed.

Pressures

Displays the Report Pressure Table dialog so that you may edit or enter relative pressure points.
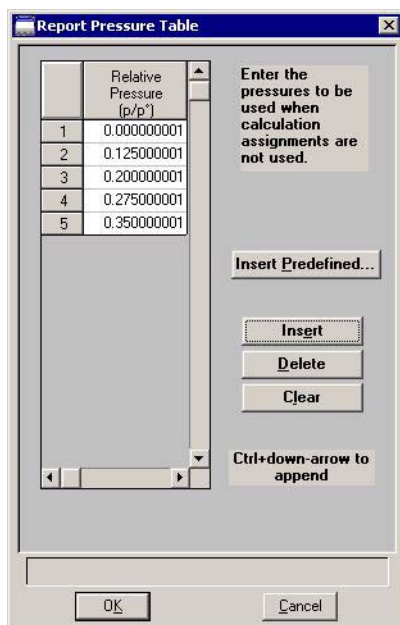
If **Use calculation assignments** is *not* selected on the Collected/Entered dialog, the isotherm is interpolated to these pressure points, and those interpolated values are used in the BET calculations.

If **Use calculation assignments** is selected, the collected data are used.

Insert predefined	Displays the Surface Area Report Pressure Selection dialog, allowing you to select predefined points.
	Choices: 1 Point, 3 Point, 5 Point, 5 Point Low Pressure
Insert	Inserts a row into the table above the selected line.
Delete	Deletes the selected row.
Clear	Removes all but one entry from the table; one entry is required. The rows do not have to be selected.
	A warning message requesting confirmation is displayed before the table is cleared.

Freundlich Isotherm

The Freundlich isotherm is an empirical isotherm that is used to model low-pressure adsorption data.

Specify monolayer capacity	In this field, enter the monolayer capacity of the sample.

Absolute pressure range   Allows you to enter a pressure range when calculation
                                 assignments are not requested.

Tabular report   Select this option to have a tabular report of the pressure
                                 points generated.

Transform plot   Plots the $\log(p)$ vs $\log(Q)$ in a straight line.

Isotherm plot   Plots the absolute pressure vs quantity adsorbed. Shows best
                                 fit line.

Overlay samples   Choose this option to overlay Freundlich isotherm data from
                                 the current file with the same type of data from other samples
                                 (files). The desired files are chosen by clicking Overlays on
                                 the Report Options dialog.

Autoscale x-axis   Select these options to have the X- and/or Y-axis scaled
Autoscale y-axis   automatically.

                                 Both X- and Y-axes begin at zero; the system uses the highest
                                 values collected during analysis as the ending points.
                                 If you choose not to autoscale data, the **From** and **To** fields
                                 are enabled, allowing you to enter the ranges.

                                 The X-axis shows the log of absolute pressure and the Y-axis
                                 shows the quantity of gas adsorbed.

From/To fields   Enabled when you choose not to autoscale data (deselect the
                                 Autoscale option), allowing you to specify the beginning and
                                 ending ranges of the X- and/or Y-axis. Data collected outside
                                 these ranges are not included in the plot.

Temkin Isotherm

The Temkin isotherm is used to model adsorption data where the heat of adsorption drops linearly with increasing coverage.

Specify monolayer capacity	In this field, enter the monolayer capacity of the sample.
Specify differential heat of adsorption at zero surface coverage	Enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all of Temkin constants.
Absolute pressure range	Allows you to enter a pressure range when calculation assignments are not requested.
Tabular report	Select this option to have a tabular report of the pressure points generated
Transform plot	Plots a linear form of the Temkin isotherm.

Temkin Isotherm plot   Overlays the Temkin isotherm with the analysis data.

Overlay samples   Choose this option to overlay Temkin isotherm data from the
                                 current file with the same type of data from other samples
                                 (files). The desired files are chosen by selecting **Overlays** on
                                 the Report Options dialog.

Autoscale x-axis   Select these options to have the X- and/or Y-axis scaled
Autoscale y-axis   automatically.

                                 Both X- and Y-axes begin at zero; the system uses the highest
                                 values collected during analysis as the ending points.

                                 The X-axis shows the natural log of absolute pressure and the
                                 Y-axis shows the quantity of gas adsorbed.

                                 If you choose not to autoscale data, the **From** and **To** fields
                                 are enabled, allowing you to enter the ranges.

**From/To fields**   Enabled when you choose not to autoscale data (deselect the
                                 Autoscale option), allowing you to specify the beginning and
                                 ending ranges of the X- and/or Y-axis. Data collected outside
                                 these ranges are not included in the plot.

t-Plot Report Options

The t-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area, the area external to micropores, is directly determined and often proves to be a valuable way of characterizing complex mixed materials.
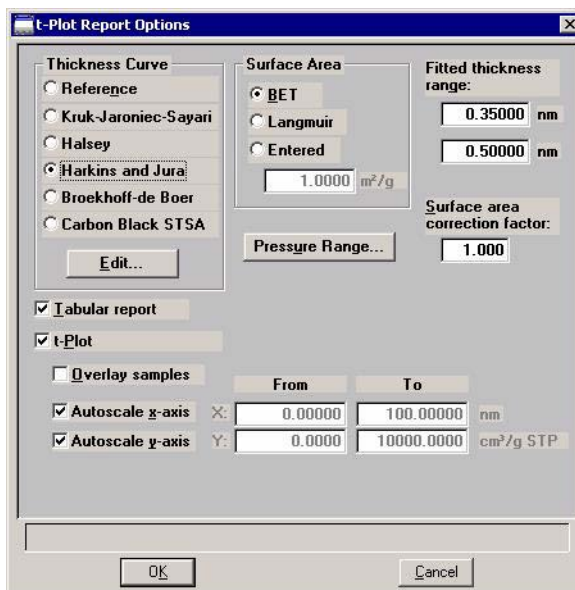
Thickness Curve	Presents the type of thickness curves available.

- Reference
- Kruk-Jaroniec-Sayari
- Halsey
- Harkins and Jura
- Broekhoff-de Boer
- Carbon Black STSA

You can also apply the Frenkel-Halsey-Hill thickness curve using the Halsey option, and entering the appropriate values in the equation. Use *Edit* to edit the values in the equation.

Edit	Displays the equation for the type of thickness curve selected so that you may view or edit the values.

Reference   Displays the Entered t-Curve dialog allowing you to define a
                                 t-curve by entering the relative pressure and thickness values.
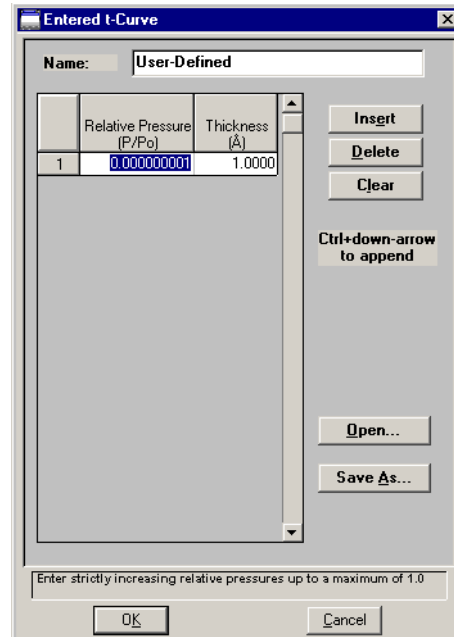
This table can also be created in another application if desired
and imported into this dialog using Open. When creating the
table in another application, the file must be saved as ASCII
text with a .THK extension. Use a two-column format with
the relative pressures in the first column and the thickness
values in the second column. Columns must be separated by a
space (or a tab).

Insert
Inserts a row above the selected row. A row cannot be
inserted if the value in the selected row is at its lowest value;
values must be strictly increasing. For example, in the table
shown above the default value is 0.000000001. Therefore,
you must use **Ctrl + Down Arrow** to insert rows.

Delete
Deletes the selected row.

Clear
Clears the table of all but one entry; one entry is required.

Open
Enables you to import the values from an existing t-curve.

Save As
Enables you to save the current table of values under a
different name.

Kruk-Jaroniec-Sayari   Displays the Kruk-Jaroniec-Sayari Thickness Equation
dialog.
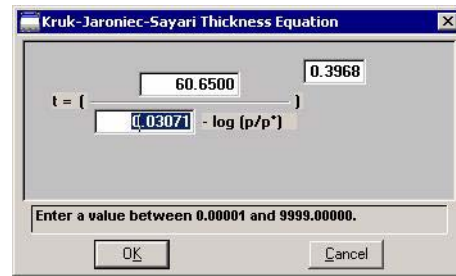
You can edit the values for the numerator, the first element of
the denominator, and the exponent. The range for a selected
field is shown in the information bar at the bottom of the
dialog.

Refer to Appendix C, Calculations, for more information.
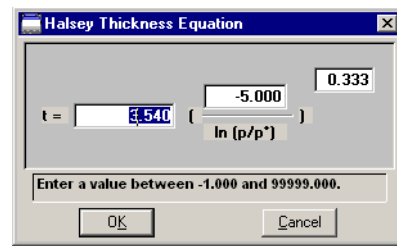
Halsey   Displays the Halsey Thickness Equation dialog.

You can edit the values for the multiplier, numerator, and
exponent. The range for a selected field is shown in the
information bar at the bottom of the dialog.

Refer to Appendix C for more information.

Harkins and Jura   Displays the Harkins and Jura Thickness Equation dialog.
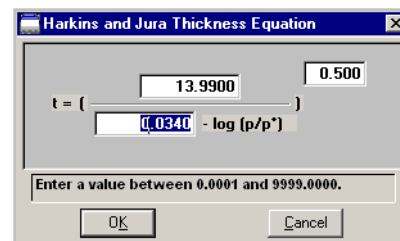
You can edit the values for the numerator, first element of the
denominator, and exponent. The range for a selected field is
shown in the information bar at the bottom of the dialog.

Refer to Appendix C for more information.

Broekhoff-de Boer   Displays the Broekhoff-de Boer Thickness Equation dialog.
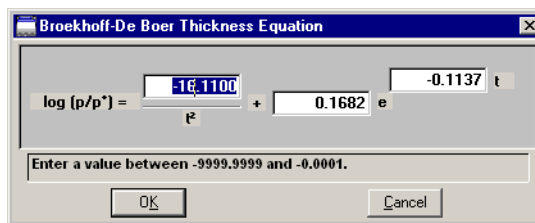
You can edit the values for the multiplier, numerator, and exponent. The range for a selected field is shown in the information bar at the bottom of the dialog.

Refer to Appendix C for more information.

Carbon Black STSA   Displays the STSA Thickness Equation dialog.
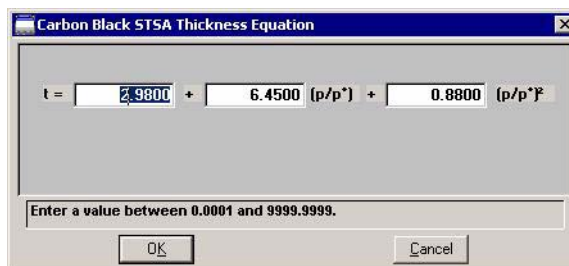
All coefficients can be edited. The range for a selected field is shown in the information bar at the bottom of the dialog.

Refer to Appendix C for more information.

Surface Area group box   Allows you to choose the surface area value used for thickness calculations. You can use BET (most commonly used), Langmuir, or enter one of preference.

Pressure Range   Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report**.**
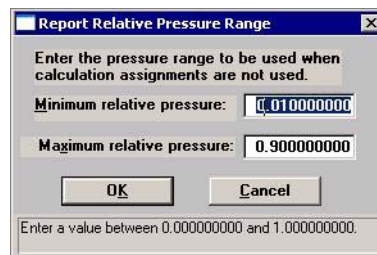
- • If **Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- • If **Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Surface area correction factor   This value corrects for surface areas that are not smooth and brings the values for BET surface area into accordance. For most samples, the default value of 1.000 is adequate

Fitted Thickness Range   Provides two fields, allowing you to enter the minimum and maximum thicknesses you wish to include in the thickness curve.

Tabular report   Select this option to generate a table of collected data.

t-Plot   Select this option to generate a graphical representation of the collected data.

Overlay samples   Allows you to overlay data from the current sample file with data from other sample file(s). The other sample file(s) are chosen by clicking Overlays on the main Report Options screen.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axes scaled automatically.

Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points.

If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis.

Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the relative pressure.

The Y-Axis Range fields show the quantity of gas adsorbed.

Alpha-S Plot
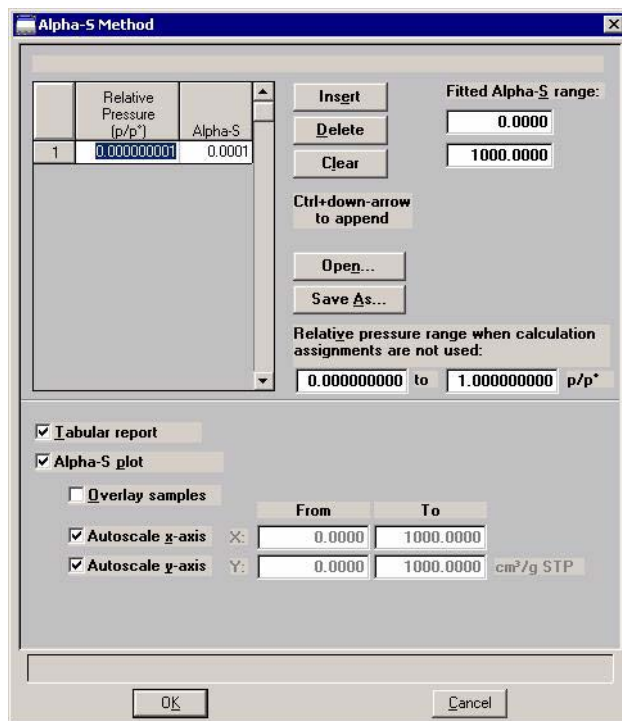
The Alpha-S plot converts the standard adsorption isotherm into a dimensionless isotherm using the quantity adsorbed at a relative pressure of 0.4.

Table   Provides two columns for entering the relative pressure (first column) and the alpha-s values (second column).

This table can also be created in another application if desired and imported into this dialog using Open. When creating the table in another application, the file must be saved as ASCII text with an ALS extension. Use a two-column format with the relative pressures in the first column and the alpha-s values in the second column. Columns must be separated by a space (or a tab).

Insert   Inserts a row above the selected row. A row cannot be inserted if the value in the selected row is at its lowest value; values must be strictly increasing. For example, in the table shown above, the default value is 0.000000001. Therefore you must use **Ctrl + Down** Arrow to insert rows.

Delete   Deletes the selected row.

Clear   Clears the table of all but one entry; one entry is required.

Open   Allows you to import the values from an existing alpha-s curve (ALS) into the table. One predefined curve is shipped with the analysis program. After the values are copied into the table, you may edit them if desired. Editing these values will not affect the file from which they were copied.

Save As   Enables you to save the current table of values under a file name.

Fitted Alpha-S range   Provides two fields for entering the minimum and maximum relative pressures from which the fit will be determined.

Relative pressure range   Provides two fields for entering a pressure range when calculation assignments are not used.

Tabular report   Select this option to generate collected data in a tabular manner.

Alpha-S plot   **S**elect this option to generate collected data graphically.

Overlay samples   Allows you to overlay data from the current sample file with data from other sample file(s). The other sample file(s) are chosen by clicking **Overlays** on the main Report Options screen.

Autoscale x-axis   Select these options to have the X- and/or Y-axes scaled
Autoscale y-axis   automatically.

                                    Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points.

                                    If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

From/To fields   Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis.

                                    Data collected outside these ranges are not included in the plot.

                                    The X-Axis Range fields show the relative pressure.

                                    The Y-Axis Range fields show the quantity of gas adsorbed.

f-Ratio Plot
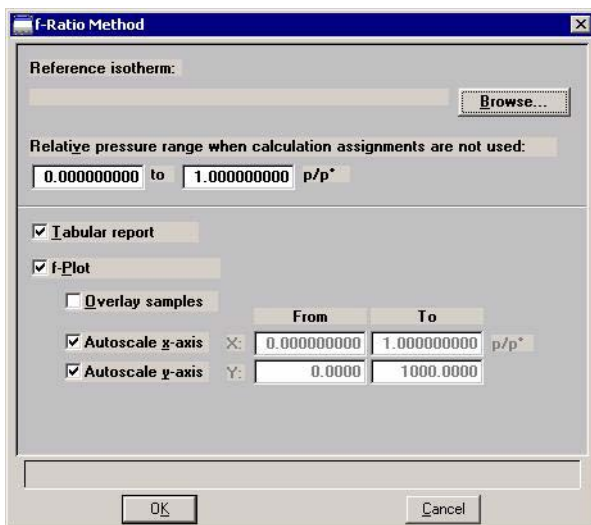
The f-Ratio report allows you to take the measured isotherm and normalize it using a reference isotherm.

Reference Isotherm	Displays the sample file you choose as a reference. You should always choose a file containing an isotherm measured from a non-porous sample of the same material as the current sample.
Browse	Click to choose the sample file you wish to use as a reference for the isotherm.
Relative pressure range	Allows you to enter a pressure range when calculations assignments are not used.
Tabular report	Choose this option to have a tabular report generated The tabular report contains four columns: relative pressure, quantity adsorbed for the isotherm of interest, quantity adsorbed for the reference isotherm, and the ratio of the quantities adsorbed.
f-Plot	Choose this option to have the data plotted as a graph.

Overlay samples   Allows you to overlay data from the current sample file with data from other sample file(s). The other sample file(s) are chosen by clicking Overlays on the main Report Options screen.

Autoscale x-axis   Select these options to have the X- and/or Y-axes scaled
Autoscale y-axis   automatically.

                                       Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points.

                                       If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

From/To fields   Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

BJH Adsorption/Desorption Report Options

This field does not display on the BJH Desorption dialog

```
The BJH Adsorption and Desorption dialogs include the same fields; the operating
instructions for both are the same.
```

The BJH calculation determines the mesopore volume/area distribution which accounts for both the change in adsorbate layer thickness and the liquid condensed in pore cores. You can generate BJH reports from adsorption and desorption data.

Thickness Curve Type   Presents the type of thickness curves available.

- Reference
- Kruk-Jaroniec-Sayari
- Halsey
- Harkins-Jura
- Broekhoff-de Boer
- Carbon Black STSA (ASTM D-6556-0lA)

You can also apply the Frenkel-Halsey-Hill thickness curve using the Halsey option, and entering the appropriate values in the equation. Use **Edit** to edit the values in the equation. Halsey is the default thickness curve and typically is used for BJH calculations.

> **If you select a thickness curve which is not a good match for the sample being analyzed, an incomplete pore distribution may be generated.**

Edit   Displays the equation for the type of thickness curve selected.

Refer to **t-Plot Report Options**, page **5-41** for information on the equations for thickness curves.

Minimum/Maximum BJH   Provides fields for entering the minimum and maximum size pore to be included in BJH reports.

The size can be reported in width (default), radius, or diameter. Select **Options** > **Units** to specify the desired unit.

Fraction of pores open at both ends

(not displayed on desorption dialog)

During calculations, the software assumes that all pores are closed at one end. Sometimes a percentage of pores may be open at both ends, causing disagreement in the adsorption and desorption data or in the values for total volume and total BJH pore volume. Enter a value in this field to compensate for this error.

This field does not display on the BJH Desorption Report Options dialog.

> **Due to normal hysteresis, data may disagree even after applying this corrective factor.**

Adsorptive

Displays the BJH Adsorptive Options dialog, allowing you to specify up to ten adsorptive/adsorbate property factor combinations.

The recommended adsorptives and their values are shown. You may specify up to six additional adsorptive/adsorbate property factor combinations.

The adsorbate property factor is the combination of constants in the numerator of the BJH equation which is specific to the type of gas used. Adsorbate property factors may be specified in angstroms or nanometers. Select **Options** > **Data Presentation** > **Units** to specify desired units.

Smooth differentials

Smooths all of the differential calculations, eliminating variations in the differential computation caused by noise in the input data.

Pressure Range	Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- **If Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- **If Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

BJH Correction	Enables you to choose the type of correction to apply to calculations. The type you choose will be displayed in the report header.
Standard	Uses original BJH models.
Kruk-Jaroniec-Sayari	Good for reference thickness curves.
Faas	Good for statistical thickness curves.
Selected Reports	Lists the available BJH reports.

- Tabular Report
- Cumulative Pore Volume
- dV/d* Pore Volume
- dV/dlog(*) Pore Volume
- Cumulative Pore Area
- dA/d* Pore Area
- dA/dlog(*) Pore Area

* = width, radius, or diameter

Choose a report by double-clicking on the report name or click the report name and press **Spacebar**. A report is selected when it is preceded with a check mark.

Edit	Displays an associated dialog for the selected report. Editing options for available reports are shown in subsequent sections.

> **You can measure pore width (W), pore radius (R), or pore diameter (D) for BJH reports. Select Options > Units to specify desired measurement.**

Tabular Report

The BJH Adsorption/Desorption tabular report allows you to specify the method of data reduction.
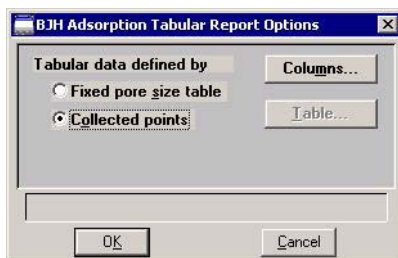
> **You can measure pore width, diameter, or radius; pore size can be expressed in angstroms or nanometers. Select Options > Units to specify desired units.**

Fixed pore size table   Select this option to specify exact pore sizes for which volume or area data are reported. Only the pore sizes within the specified range appear on the report. Click Table to enter (or edit) a fixed pore size table.

Collected points   Select this option to include the values collected by the system in the report calculations.

Columns

Displays the BJH Adsorption (or Desorption) Tabular Report column Options dialog, allowing you to select up to six columns of data for the tabular report.
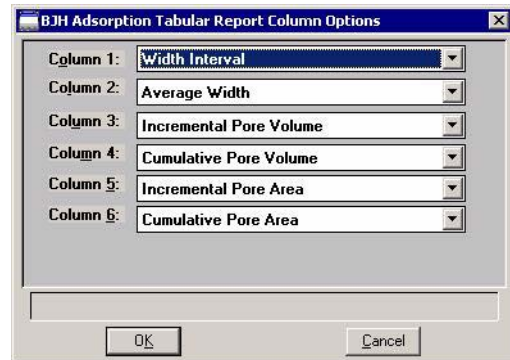
The default column title appears next to the column number. Each column includes a pull-down list of the data types to include in the report; the pore width, radius, or diameter can be measured (select **Options** > **Units** to choose the desired unit).

Table

Enabled when Fixed pore size table is selected for tabular data. Displays the BJH Adsorption (or Desorption) Fixed Pore Size Table dialog, allowing you to enter or edit a fixed pore size table**.**
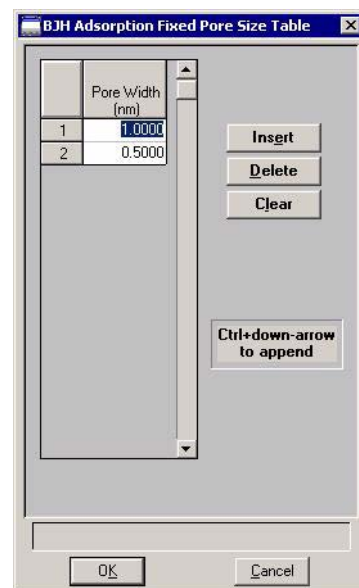
The table must contain a minimum of two points and may include as many as one thousand. Points must be strictly decreasing**.**

Insert

Inserts a row into the pressure table. The row is inserted
above the selected row and the cursor moves to the new row.

Delete

Deletes the selected row from the pressure table.

Clear

Removes all entries (except the two required ones) from the
table. A warning message requests confirmation before the
table is cleared.

Plot Options

Selecting a BJH plot option from the BJH Adsorption or Desorption Report Options dialog
enables you to specify the plotting method used for your report and to customize the plot.

> **You can measure pore width, diameter, or radius; pore size can be expressed in
> angstroms or nanometers. Select Options > Units to specify desired units.**

When you edit a BJH Adsorption or Desorption plot, a dialog similar to the following is
displayed.
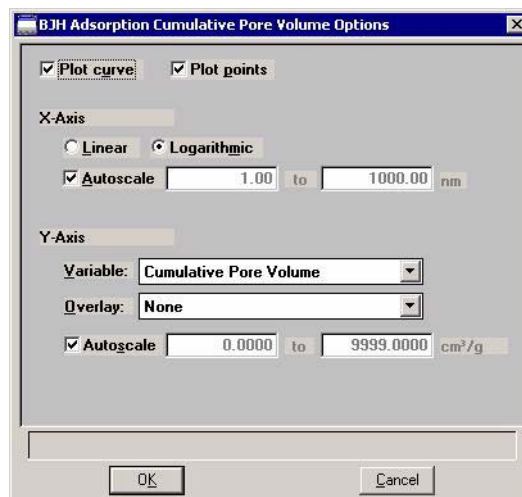
| **Plot Options** | Allows you to have data plotted as a curve, points, or both. |
| **X-Axis** | Lists the scale options for the x-axis |

Linear/Logarithmic	Choose whether you wish to have the X-axis on a logarithmic or linear scale. Unless you are performing micropore analyses, use the logarithmic option.
Autoscale	Choose this option to have the x-axis scaled automatically. Linear X-axes begin at zero, and logarithmic X-axes begin at an appropriate value. The system uses the highest values collected during analysis as the ending points.
	If you deselect this option, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside the specified ranges are not included in the plot**.**
	X-axis range fields show pore radius or diameter in angstroms or nanometers**.**
	Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.
Y-axis	Lists the options for the Y-axis.
Variable	Click on the down-arrow to choose a variable for the Y-axis.
Overlay	Displays a list of overlay choices. You can choose a different type of plot to overlay with the current plot. Or, you can choose **Samples** to overlay the current type of plot with the same type from other sample files. If you choose Samples, you select the other files using **Overlays** on the Report Options dialog.
Autoscale	Choose this option to have the y-axis scaled automatically. Y-axes begin at zero. The system uses the highest values collected during analysis as the ending points.
	If you deselect this option, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside the specified ranges are not included in the plot.
	Y-axis range fields show the quantity of gas adsorbed. Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Horvath-Kawazoe Report Options
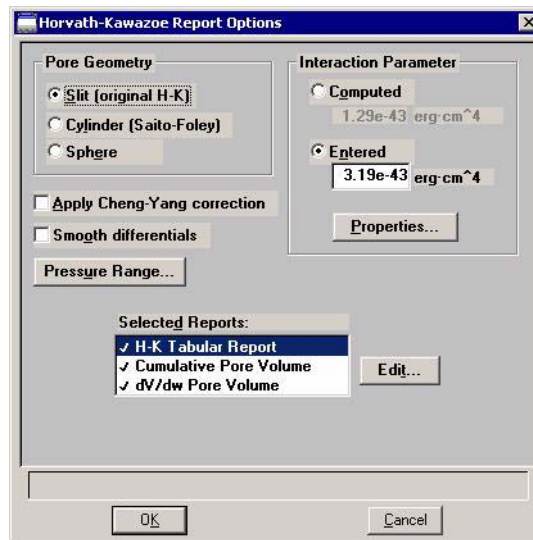
Pore Geometry	Select the option in this group box which best represents the physical geometry of the micropores in the sample material. Refer to **Appendix C** for additional information on calculations used for these parameters.
Apply Cheng/Yang correction	Allows you to apply the Cheng/Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation (see Appendix C).
Smooth differentials	Smooths the differential calculations, eliminating variations in the differential computation caused by noise in the input data.
Pressure Range	Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- If **Use calculation assignments** is not selected on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- If **Use calculation assignments** is selected, collected data points which are assigned to this report type are used.

Interaction Parameter	Select one of the options in this group box to determine which interaction parameter is used during report generation.
Computed	The interaction parameter is calculated using the parameters on the Horvath-Kawazoe Physical Properties dialog. You can click **Properties** to view or edit these parameters. Each time you change one of the parameters, the interaction parameter is recalculated. The formula for computing the interaction parameter is given in Appendix C.
Entered	The value you enter in the adjacent field will be used.
Properties	Use this push button to view or edit the constants (used during report generation) describing the physical properties of the adsorbent and adsorptive; the Horvath-Kawazoe Physical Properties dialog is displayed.

Adsorbent Group Box   Contains the parameters for the sample. If you select
                            Computed for the interaction parameter, all fields are
                            enabled and can be edited if desired. If you select **Entered**,
                            only the values in the **Diameter** and **Diameter at zero
                            energy** fields may be edited.

                            Description
                            The name of the adsorbent used in the analysis.

                            Click on the down arrow to make a new selection. Each time
                            you change the selection, the values for that adsorbent are
                            displayed in the appropriate fields.

                            Diameter
                            The diameter of the sample atom.

                            Diameter at zero energy
                            The diameter of an atom at zero interaction energy,
                            $(2/5)^{1/6}$ x diameter.

                            Polarizability
                            The polarizability of the adsorbent.

                            Magnetic susceptibility
                            The magnetic susceptibility of the adsorbent.

                            Density
                            The density per unit area of the adsorbent.

Adsorptive Group Box   Contains the parameters for the adsorptives (provided with
                            the software and/or user-defined). If you select **Computed**
                            for the interaction parameter, all fields are enabled and can be
                            edited if desired. If you select **Entered**, only the values in the
                            Diameter and **Diameter at zero energy** fields may be
                            edited.

                            Mnemonic
                            The mnemonic name of the adsorptive gas in use.

                            Click on the down arrow to make a new selection. Each time
                            you change the selection, the values for that adsorptive
                            display in the appropriate fields. If no parameters have been
                            defined, the default values are displayed.

                            Diameter
                            The diameter of the adsorptive atom.

Adsorptive Group Box *(continued)*	**Diameter at zero energy** The diameter of an atom at zero interaction energy, $(2/5)^{1/6}$ x diameter.

Polarizability
The polarizability of the adsorptive.

Magnetic susceptibility
The magnetic susceptibility of the adsorptive.

Density
The density per unit area of the adsorptive.

Selected Reports	Lists the available Horvath-Kawazoe reports. Choose a report by double-clicking on the report name or highlight the report name and press the **Spacebar**. A report is selected when it is preceded with a check mark. The following reports are available:

- Tabular Report
- Cumulative Pore Volume
- dV/dw Pore Volume

Edit	Allows you to edit the selected report.

Tabular Report Options

Displays the Horvath-Kawazoe Tabular Report Column Options dialog so that you can select the type of data to display in each column.
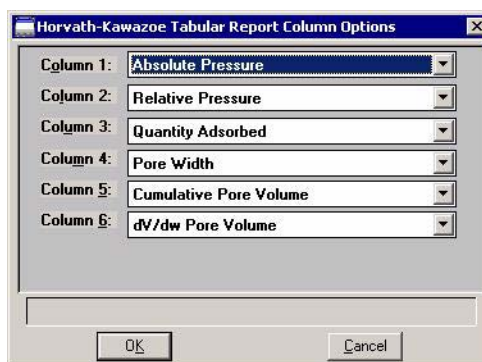
The default column title displays next to the column number. Each column includes a drop-down list of the types of data to include in the report.

Plot Options
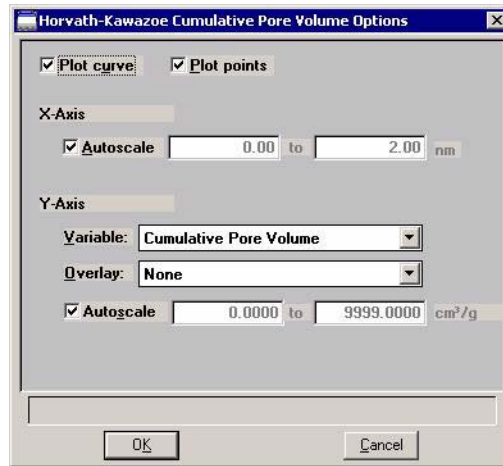
Selecting a Horvath-Kawazoe plot option from the Horvath-Kawazoe Report Options dialog enables you to specify the plotting method used for your report and to customize the plot. Plots for Cumulative Pore Volume and dV/dw Pore Volume are available.

When you select either of these plots from the Horvath-Kawazoe Report Options dialog, a dialog like the following is displayed.

Plot Options	Lists the data presentation styles available; you must choose to plot a curve, points, or both.
X-axis options	Choose **Autoscale** to have the x-axis scaled automatically. The X-axis begins at zero and the system uses the highest value collected during analysis as the ending point.
	If you deselect **Autoscale**, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.
	X-axis range fields show pore radius or diameter in angstroms or nanometers.
	Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-axis options

Click on the down-arrow at the Variable field to choose a variable for the Y-axis.

Click on the down-arrow at the **Overlay** field to overlay the plot with data from other samples.

Choose **Autoscale** to have the y-axis scaled automatically. Y-axes begin at zero. The system uses the highest value collected during analysis as the ending point.

If you deselect **Autoscale**, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

Y-axis range fields show the quantity of gas adsorbed Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

DFT Pore Size
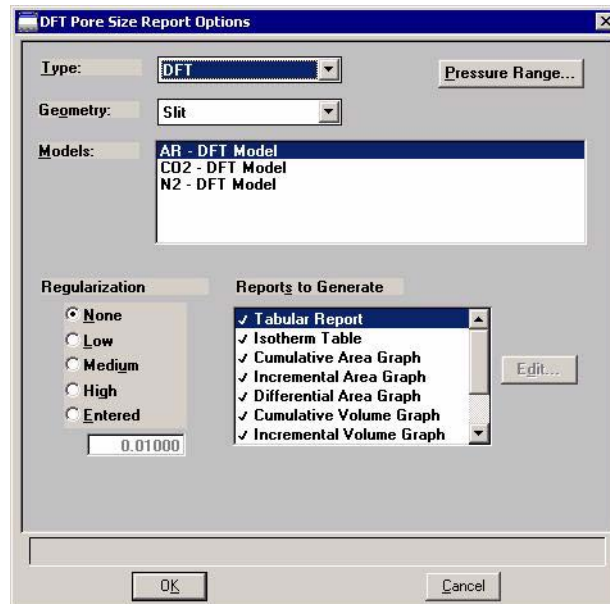
Type	Drop-down list containing the types of models available; DFT or Classical.
	DFT models are based on the density functional theory. Classical models are based on the Kelvin equation and thickness for determining the pore size distribution. Refer to Appendix F for a discussion on models.
Geometry	Drop-down list containing pore shapes available; Slit or Cylinder.
Models	Lists the models that meet the criteria specified and which match the adsorbate and temperature of the sample data.
	The models display in alphabetical order with the first one automatically selected; you may select any one desired.
	If the list is empty, there were no models that meet the selected criteria.

Pressure Range   Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- If **Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- If **Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Regularization   These choices enable you to choose the extent of smoothing you wish to have applied to your data, or your can choose **None**.

If you choose **Entered**, its field is enabled allowing you to enter a number giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.

Reports to Generate   Displays the types of reports available.

- Pore Size table
- Isotherm table
- Cumulative area graph
- Incremental area graph
- Differential area graph
- Log goodness of fit graph
- Goodness of fit graph

Graph details can be edited by selecting the **Edit** push button.

Edit   Use this push button to edit details of a selected graph; a
                                dialog like the following is displayed.

Displays the type of
graph you are
editing.
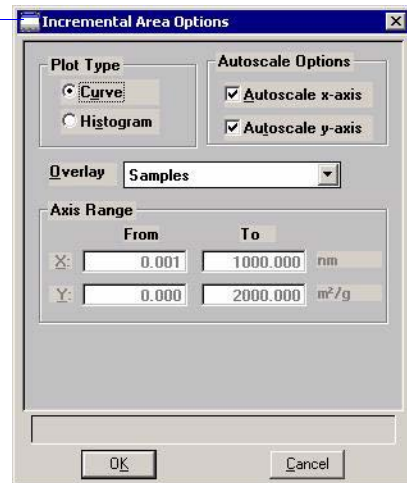
                                This push button is disabled for tables and Goodness of Fit
                                graphs.

Plot Type   Enables you to choose the manner in which you wish to
                                display plotted data.

Autoscale Options   Select these options to have the axes scaled automatically.

                                If you deselect one or both of these options, you must enter a
                                range in the related **Axis Range** fields which become enabled
                                automatically.

Overlay   This drop-down list contains the type of overlays available
                                for the current graph.

                                Choose **Samples** to overlay the same type of graph as the
                                current one from other data reductions. Then select **Overlays**
                                on the Report Options dialog to choose your files (explained
                                on page **5-29**).

Axis Range   The fields in this group box become enabled when you dese-
                                lect Autoscale, allowing you to specify beginning and ending
                                values for the X- and/or Y-axis. Data collected outside these
                                ranges are not included in the plot.

                                Valid ranges for a selected field are displayed in the informa-
                                tion bar across the lower portion of the dialog.

                                The X-axis displays the energy and the Y-axis displays the
                                area.

Surface Energy
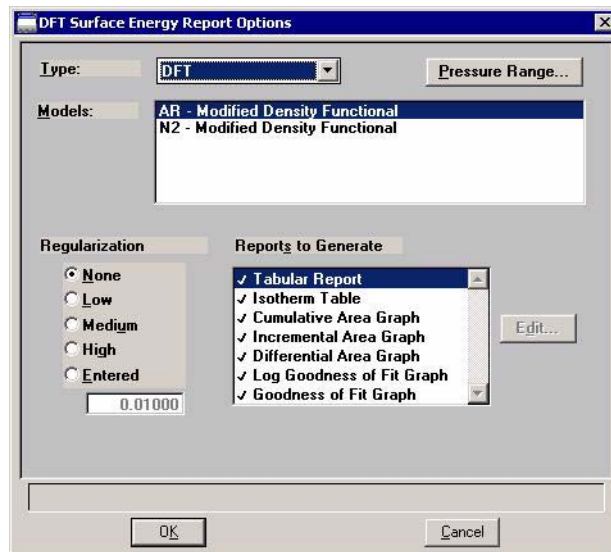
The fields on the Surface Energy dialog are identical to those on the DFT Pore Size dialog with the following exceptions:

Geometry drop-down list	Is not applicable to the Surface Energy report and, therefore, does not display on its dialog.
Reports to Generate	Displays the types of reports available.

- Surface energy table
- Isotherm table
- Cumulative area graph
- Incremental area graph
- Differential area graph
- Log goodness of fit graph
- Goodness of fit graph

Graph details can be edited by selecting the **Edit** push button.

Refer to the previous section for the DFT Pore Size report beginning on page **5-64** for information on the fields contained on this dialog.

Dubinin Report Options
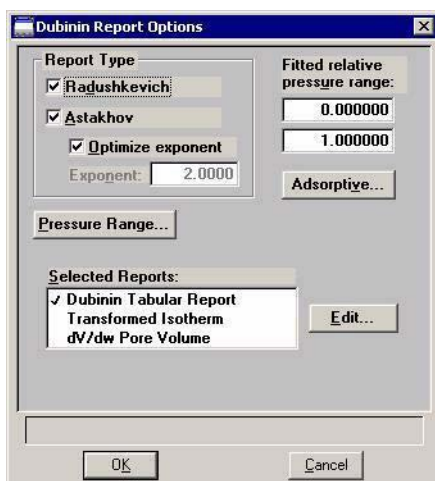
Report Type Allows you to specify the type of report you want. At least one type of report must be selected.

Select **Radushkevich** to generate the Radushkevich report.

Select **Astakhov** to generate the Astakhov report. With this choice, you may select **Optimize Astakhov exponent**. If you do not select this option, the **Exponent value** field is enabled so that you can enter a value.

Refer to Appendix C for more information.

Fitted relative pressure range Allows you to specify minimum and maximum limits on relative pressures included in the line fit. Data collected outside these limits are not included.

Adsorptive

Displays the Dubinin Adsorptive Options dialog allowing you to specify up to 8 Adsorptive/Affinity Coefficient (beta) combinations.
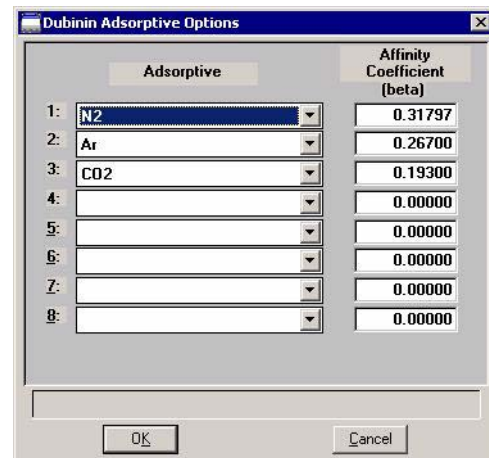
Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- **If Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- **If Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Selected Reports

Lists the available Dubinin reports. Choose a report by double-clicking on the report name or highlight the report name and press the **Spacebar**. A report is selected when it is preceded with a check mark.

The following reports are available:

- Tabular Report
- Transformed Isotherm
- dV/dw Pore Volume (Astakhov only)

Tabular Report

The Tabular report enables you to customize your report with one to six columns of data for Astakhov reports and one to five columns of data for Radushkevich reports. The Dubinin Tabular Report Column Options dialog is displayed.
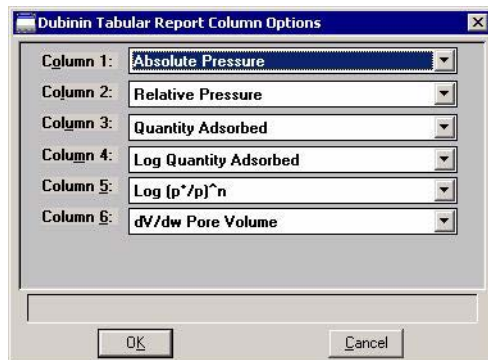
The default column title appears next to the column number. Each column includes a drop-down list of the data types to include in the report.

Transformed Isotherm

The Transformed Isotherm report allows you to restrict the line fit to a portion of the isotherm. The Dubinin Transformed Isotherm Plot Options dialog is displayed.
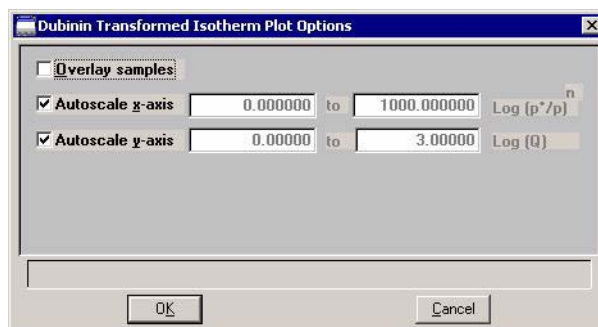
Overlay Samples	Select this option to overlay the current plot with data from other samples. Then click Overlays on the Report Options dialog to choose the sample files.
Autoscale x-axis **Autoscale y-axis**	Select these options to have the X- and/or Y-axes automatically scaled. Both X- and Y-axes begin at zero, and the system uses the highest values collected during analysis as the ending points for axes ranges.

Autoscale x-axis
Autoscale y-axis
(continued)

If you choose not to autoscale data for either (or both) axis, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the quantity of gas adsorbed at standard temperature and pressure.

The Y-Axis Range fields show the log of relative pressure.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Pore Volume

When you select the dV/dw Pore Volume plot from the Dubinin Report Options dialog, the Dubinin dV/dw Pore Volume Options dialog is displayed.

The Pore Volume report is unavailable if you choose Radushkevich as the type of report.
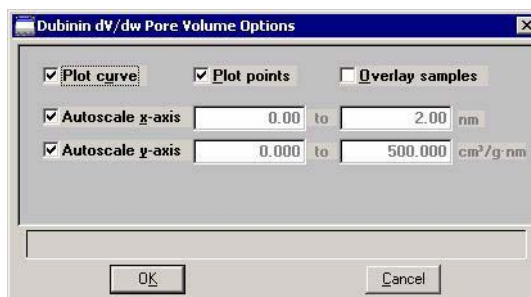
Plot options

You can plot data with a curve, points, or both.

Overlay Samples

Select this option to overlay data from other samples. Then click Overlays on the Report Options dialog to choose the files.

Autoscale x-axis   Select these options to have the X- and/or Y-axes
Autoscale y-axis   automatically scaled. Both X- and Y-axes begin at zero, and
                              the system uses the highest values collected during analysis
                              as the ending points for axes ranges.

                              If you choose not to autoscale data for either (or both) axis,
                              the adjacent fields are enabled so that you may enter a
                              beginning and ending value. Data collected outside these
                              ranges are not included in the plot.

                              The X-axis Range fields show pore width, radius, or
                              diameter in angstroms or nanometers.

                              The Y-axis Range fields show the quantity of gas adsorbed.

                              Data ranges are displayed in the information bar across the
                              bottom of the dialog when the cursor is in a numerical data
                              entry field.

MP-Method Report Options
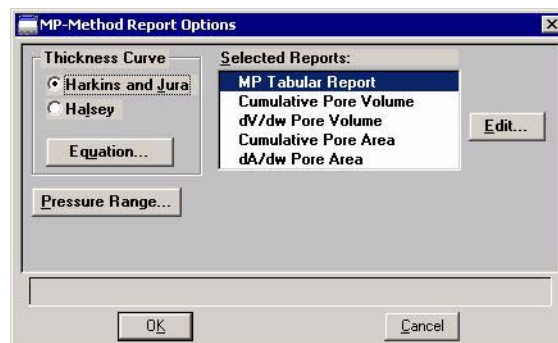
Thickness Curve group box   Select the type of thickness curve from this group box.
                                  You must choose either the Halsey equation or the
                                  Harkins and Jura equation. Use Equation to edit the
                                  values in the equation.

Equation   Displays the equation for the type of thickness curve
                                  selected

Harkins and Jura   Displays the Harkins and Jura Thickness Equation
                              dialog.
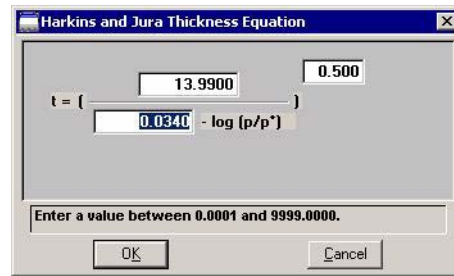
You can edit the values for the numerator, first element of
the denominator, and exponent. The range for a selected
field is shown in the information bar at the bottom of the
dialog.

Refer to Appendix C for more information.

Halsey   Displays the Halsey Thickness Equation dialog.
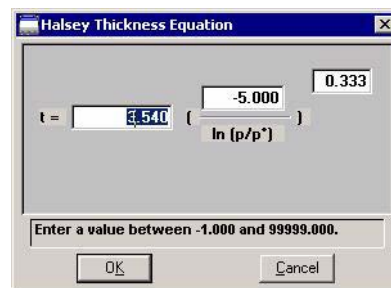
You can edit the values for the multiplier, numerator, and
exponent. The range for a selected field is shown in the
information bar at the bottom of the dialog.

Refer to Appendix C for more information.

Pressure Range   Displays the Report Relative Pressure Range dialog so
                                      that you may specify minimum and maximum relative
                                      pressures to use with this report.

    • **If Use calculation assignments is not selected** on
                                      the Collected/Entered dialog, all of the nonoutlier
                                      points of the collected data within the specified range
                                      are used for calculating the data for this report.

    • **If Use calculation assignments is selected**, col-
                                      lected data points which are assigned to this report
                                      type are used.

Selected Reports   Lists the available MP-Method reports. Choose a report
                                      by double-clicking on the report name or highlight the
                                      report name and press the Spacebar. A report is selected
                                      when it is preceded with a check mark.

                                      The following reports are available:

    • Tabular Report
    • Cumulative Pore Volume
    • dV/dw Pore Volume
    • Cumulative Pore Area
    • dA/dw Pore Area

Tabular Report

The Tabular report enables you to customize your report with one to six columns of data. (The data types in the first and second columns cannot be changed.) The MP-Method Tabular Report Column Options dialog is displayed.
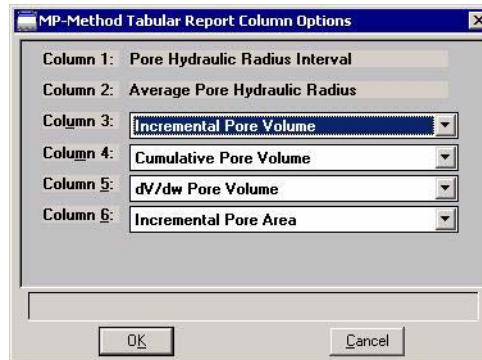
The default column title appears next to the column number for columns three through six. Each of these columns includes a drop-down list of the data types to include in the report. Columns 1 and 2 are fixed and cannot be edited.

> **The MP-Method reports hydraulic radius only. If you select Pore size in diameter from the Units dialog, the MP-Method still reports pore size in radius.**

Pore Volume/Pore Area Plot

MP-Method plot options enable you to specify the plotting method used for your report and to customize the plot. A dialog like the following is displayed. The fields on all dialogs are the same.

Plot options   You can plot data with a curve, points, or both.

X-axis options   Choose **Autoscale** to have the x-axis scaled automatically. The X-axis begins at zero and the system uses the highest value collected during analysis as the ending point.

                              If you deselect **Autoscale, t**he adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

                              X-axis range fields show pore radius or diameter in angstroms or nanometers.

                              Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-axis options   Allows you to choose Y-axis options.

Variable   Click on the down-arrow to choose a variable for the Y-axis.

Overlay   You can use the choices in this drop-down list to overlay plots. You can choose a different type of plot for the current sample to overlay with the listed **Variable**, or you can choose **Samples** to overlay the **Variable** plot with the same type of plot from other samples.

Autoscale   Choose this option to have the y-axis scaled automatically. Y-axes begin at zero. The system uses the highest value collected during analysis as the ending point.

                              If you deselect **Autoscale**, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

                              Y-axis range fields show the quantity of gas adsorbed

                              Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Options Report

The Options report is a subset of the contents report; it provides pertinent information for the following:

- Degas conditions
- Adsorptive properties
- Analysis conditions
- Sample tube criteria
- Free space
- Po and temperature
- Equilibration
- Isotherm collection

Summary Report

The Summary report provides a condensed listing of analysis statistics and data results. The Summary Report Options dialog allows you to choose the type of information to include in the report.

Sample Log Report

The Sample Log report displays the following:

- Manual control operations performed during analysis
- Information entered using **Add Log Entry** on the sample file editor
- Warnings and/or errors which occurred during analysis

Collected/Entered Data

If you choose **Manually entered** as the type of data in the Sample Information dialog, an **Entered** tab is added allowing you to enter the data.

If you choose **Automatically collected** as the type of data in the Sample Information dialog, a **Collected Data** tab is added when analysis is complete. The Collected Data dialog will contain the data points collected during analysis and the specified calculation assignments.

This example shows the Collected Data dialog from a completed sample information file; the Entered dialog is identical except the data is entered and the tab displays as Entered Data.

Pressure table	For **collected data**, columns for the following are displayed**:**

- absolute pressure

- relative pressure

- quantity adsorbed

- outliers (if **Use calculation assignments** is not selected)

- calculation assignments for each requested report option (if **Use calculation assignments** is selected)

For **entered data**, columns for the following are displayed:

- absolute or relative pressure (depending on the selection in the Pressures group box)

- quantity adsorbed

Insert	Enabled for entered data; inserts a row into the pressure table.
Delete	Enabled for entered data; deletes the selected row.
Clear	Enabled for entered data; clears the entire table of all entries except one (one is required).
Use calculation assignments	If selected, allows you to assign the points to be collected for each report type.

If deselected, each report (with the exception of Langmuir and BET) uses a range of pressures as selected in Report Options. The Langmuir and BET reports interpolate to entered relative pressures on Report Options.

The outlier points can be selected so that they will not be reported.

Po and T   Displays the Po and T dialog, allowing you to edit the
                                saturation pressure and temperature. Only the applicable
                                options are enabled.

Free Space   Displays the Free Space dialog, allowing you to edit the
                                ambient and analysis free spaces. This dialog also includes
                                the plotted isotherm. The isotherm is redrawn each time you
                                edit the values in either field.

                                Also available for the isotherm is a shortcut menu containing
                                options allowing you to edit the curve and axes. You also can
                                zoom in for finer detail. Refer to **Onscreen Reports**, page **7-
                                16** for the options available.
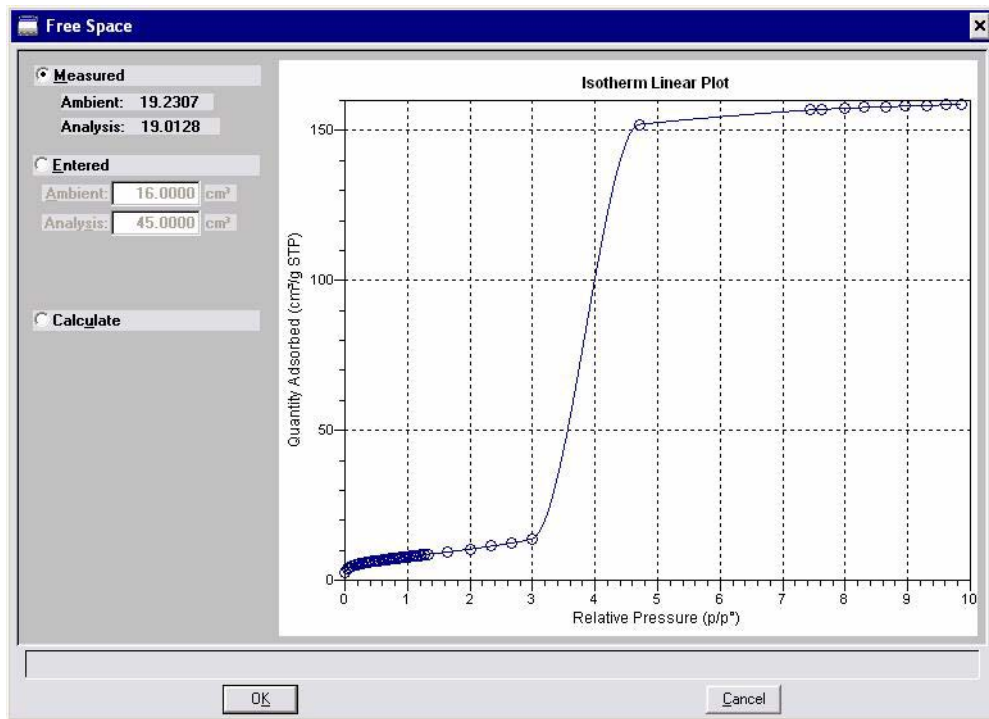
Save

Save enables you to save any changes you have made to the file in the active window. The file is saved under its current name.

Save As

Save As enables you to:

*   save a sample or parameter file in the active window under a different name. This option is useful for making a duplicate copy of a file that you can modify as desired without changing the original one. The original file remains open when you use this function, so be sure to open the new file before making any changes.

*   save a subset (parameter) of the sample file in the active window as a standalone parameter file. For example, select Analysis Conditions from the Save As menu to create a standalone parameter file of the analysis conditions portion of the active sample file.

*   save as an ASCII file the relative pressures and corresponding thicknesses (t-Curve) from the collected data. These data are derived by dividing the condensed volume of adsorptive by the selected surface area. The density conversion factor in the adsorptive properties file is used to convert quantity adsorbed to volume of condensed adsorptive.

*   save as an ASCII file the relative pressures and resulting quantities adsorbed (Alpha-S) from the collected data. These data are derived by taking the isotherm and dividing it by the quantity adsorbed at 0.4 relative pressure.

Sample and Parameter Files

A dialog similar to the one shown below is displayed when you select Sample Information, Sample Tube, Degas Conditions, Analysis Conditions, Adsorptive Properties, or Report Options.

Indicates the type of file being saved ————
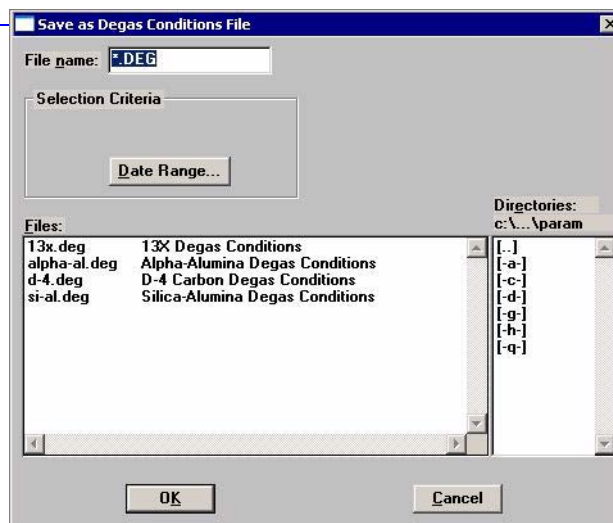
Enter a file name (up to eight characters) in the **File name** field; the appropriate extension is appended automatically when you click **OK.** The new file is saved as specified, but does not remain in the active window. Be sure to open the new file before making any changes to the file.

t-Curve and Alpha-S Files

The same type of dialog shown above is also displayed when you select t-Curve or Alpha-S. However, before you receive this dialog for t-Curve, the Save As t-Curve dialog is displayed. This dialog allows you to choose the surface area for the sample.
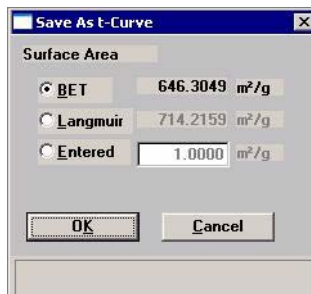
Save All

Save All enables you to save all open files under their current names. This option provides a faster way to save *all* open files at one time and avoids having to perform a **Save** operation on each individual file.

A dialog prompting for a reason for the current change version of each file is displayed.

Close

Close enables you to close the file in the active window. If the file contains changes that have not been saved, the following message is displayed:

(file name) has been changed. Save changes before closing?

Yes   **No**   **Cancel**

Click **Yes**; a dialog prompting for a reason for the change is displayed before the file closes.
Click **No** to close the file without saving the changes.
Click **Cancel** to return to the active file.

Close All

Close All enables you to close *all* open files under their current names. The following message displays for every file containing changes that have not been saved:

(file name) has been changed. Save changes before closing?

Yes   **No**   **Cancel**

Click **Yes**; a dialog prompting for a reason for the change is displayed before the file closes.
Click **No** to close the file without saving the changes.
Click **Cancel** to return to the active file.

Print

Print enables you to print the contents of one or more files to the screen, a printer, or a file. For example, if you choose Analysis Conditions, you will receive the parameters used for all analysis conditions associated with the file(s). The print dialog is common to all file types.

Select the desired file type from the drop-down menu; a dialog similar to the following is displayed:

Indicates the type of report you are printing
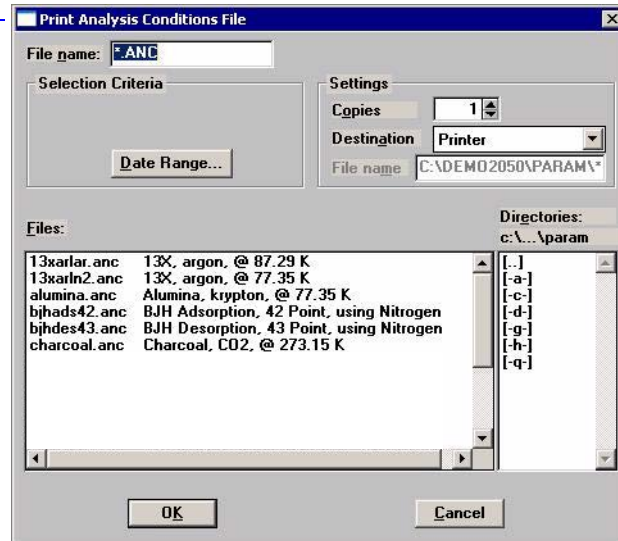
File name	The name of the file you choose to print is copied to this field. If you choose multiple files, the name of the last one selected is displayed.
Copies	Enabled when Printer is selected as the print destination. You may print up to four copies
Destination	Select the destination. You may print to a printer, the screen, or a file.

File name   Enabled when you select File as the destination. A default
                                           file name (the same name as the sample file) appears if you
                                           choose only one file; you can use the default name or enter
                                           another one.

                                           If you select multiple files, the default displays as *.RPT;
                                           each file will be exported as its original file name with the
                                           RPT extension.

Refer to **"Selecting Files" on page 2-11** for a description of the other fields on this dialog and
the **Date Range** push button.

List

List enables you to generate a listing of the following information on a selected sample or
parameter file.

- File name
- Date the file was created or last edited
- Time the file was created or last edited
- File identification
- File status

Regardless of the type of file you choose, the dialogs are similar; the options presented in the
header vary slightly. The types of dialogs displayed are identical to the ones displayed for the
Print function; you simply obtain different types of information. This example shows a dialog
for sample files.
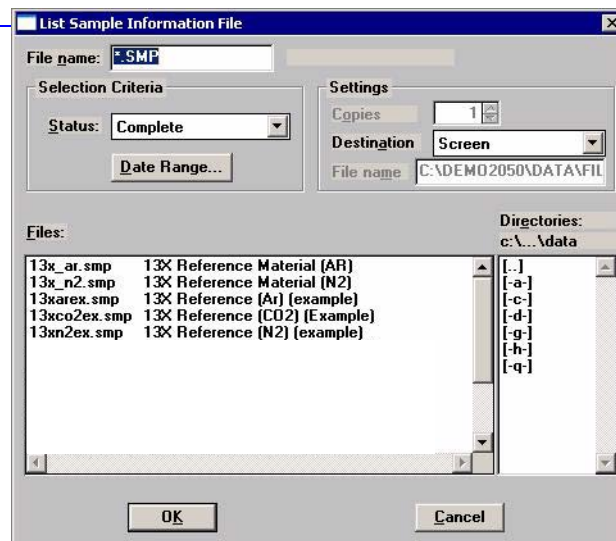
Refer to **Print**, page **5-84** for an explanation of the fields on this dialog.

You may request a list of multiple files by holding down **Ctrl** while selecting files. If no files
are selected, a list is generated for *all* files.

Export

Export copies the isotherm data in the sample information file and reformats it in a text format acceptable to other programs, such as spreadsheets. The output file consists of 4 columns containing the absolute pressure, relative pressure, specific quantity adsorbed, and the elapsed time.
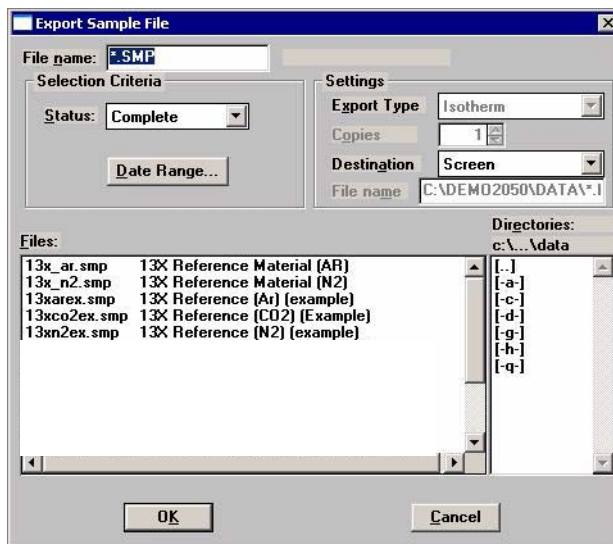
File name	The name of the file you choose to export is copied to this field. If you choose multiple files, the name of the last one selected is displayed**.**
Settings	This group box enables you choose print options.
Destination	Select **Destination** to export files to a printer, the screen, or to a file. The **Copies** field is enabled if you choose printer, allowing you to print up to four copies.

The **File name** field is enabled if you select File for export destination. For the name, you can:

- Accept the default value where all selected files you choose to export will have the same name as those in the sample information file with an .ISO extension.

  For example, if you select the following files to export, names will be assigned as indicated:

Selected Files	Export Names
C:\DATA1\SAMP1.SMP	C:\DATA1\SAMP1.ISO
C:\DATA1\SAMP2.SMP	C:\DATA1\SAMP2.ISO
C:\DATA1\SAMP3.SMP	C:\DATA1\SAMP3.ISO

- Enter a name for the file (full path name). The extension does not have to be .ISO. If exporting more than one file, a wildcard character should be entered.

  For example, if you use a .TXT extension, enter C:\DATA2*.TXT in the File name field.

Selected Files	Export Names
C:\DATA1\SAMP1.SMP	C:\DATA2\SAMP1.TXT
C:\DATA1\SAMP2.SMP	C:\DATA2\SAMP2.TXT
C:\DATA1\SAMP3.SMP	C:\DATA2\SAMP3.TXT

Exit

Exit enables you to exit (close) the ASAP 2050 program.

• If an analysis is in progress, the following message is displayed:

> **CAUTION: Sample analysis is in progress. If you exit
> the program analysis will continue to completion, but
> the data are not saved to disk until the program is
> restarted. Do you want to exit the program?**
>
> **Yes   No**

Click **Yes** to close the ASAP 2050 program. The analysis continues and data are collected. Click **No** to allow the program to remain active.

> **Although data are stored in the analyzer when you exit the program during analysis, they are not saved in the file until the program is restarted. At that time the data are saved automatically. If a power failure occurs in the interim and you do not have an Uninterruptible Power Supply (UPS) attached, loss of data will result.**

• If a report is in progress, you are cautioned that reports are being generated, and given the opportunity to either cancel the print job or allow the reports to print before exiting.

> **Observe the Status area of the instrument schematic; it indicates when an analysis is complete.**

6. UNIT MENU

The Unit menu contains the options for the operations that can be performed with the ASAP 2050 analyzer. The main menu will contain a Unit menu for each attached analyzer. For example, if you have two attached analyzers, the main menu contains two Unit menus. The appropriate unit number and serial number display in the title bar of the operational windows. The status windows also display in different colors. This feature is especially useful when you have more than one analyzer attached to the same computer.

> **The Unit menu does not display on the menu bar if the analysis program is being used for offline data manipulation on a computer other than the one controlling the analyzer.**

Description
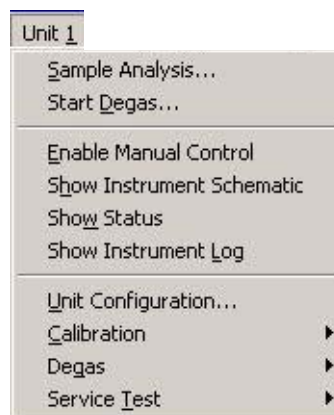
Listed below are brief descriptions of the Unit menu options. Detailed descriptions are found in subsequent sections.

Sample Analysis   Starts an analysis. This option is disabled if analyses are in progress. Page **6-3**.

Start Degas   Allows you to start the degassing operation on one or two samples. Page **6-7**.

Enable Manual Control   Allows you to control the system manually. Page **6-8**.

Show Instrument Schematic Displays a schematic of the analyzer components. Page **6-11**.

Show Status   Shows the status window of the operation in progress.
                                    Page **6-12**.

Show Instrument Log   Displays a log of recent analyses, calibrations, and error mes-
                                    sages. Page **6-13**.

Unit Configuration   Displays the configuration of the analyzer. Page **6-15**.

Calibration   Enables you to calibrate certain components of the analyzer.
                                    Page **6-17**.

Degas   Allows you to perform sample degassing operations.
                                    Page **6-22**.

Service Test   Enables you to perform certain troubleshooting procedures.
                                    This option is enabled only with the direction of a Micromer-
                                    itics service representative. Page **6-27**.

Sample Analysis

When you select this option from the Unit menu, the Sample Analysis dialog is displayed with all fields disabled (grayed) and the Start Analysis dialog positioned on top. This allows you to select a sample file for your analysis or to create a new one.

After a sample file has been designated, the Analysis dialog is displayed. The fields now contain the values for the selected file or, if creating a new file, the specified defaults.

View   Allows you to view one of the following in the current window:

- the current operation
- the instrument schematic
- the instrument log

Refer to **Show Instrument Schematic** on page **6-11** and **Show Instrument Log** on page **6-13**.

Browse   Displays the Start Analysis dialog allowing you to choose a different sample file for the current analysis.

Sample   Displays an identification of the sample file.

Mass   Enables you to enter the sample's mass.

Density   Enables you to enter the density of the sample.

Sample Tube   Displays the name of the Sample Tube file for the current
                                   sample file.

                                   If this is a new file, this field contains the file you specified as
                                   the default.

                                   The drop-down list contains a list of predefined parameter
                                   files that were saved to the Parameter Files Directory (refer to
                                   Parameter Files Directory on page **8-11**), as well as those
                                   included with the analysis program. You may choose a dif-
                                   ferent file from this list if desired.

Analysis Conditions   Displays the name of the Analysis Conditions file for the cur-
                                   rent sample file.

                                   If this is a new file, this field contains the file you specified as
                                   the default.

                                   The drop-down list contains a list of predefined parameter
                                   files that were saved to the Parameter Files Directory (refer to
                                   Parameter Files Directory on page **8-11**), as well as those
                                   included with the analysis program. You may choose a dif-
                                   ferent file from this list if desired.

Adsorptive Properties   Displays the name of the Adsorptive Properties file for the
                                   current sample file.

                                   If this is a new file, this field contains the file you specified as
                                   the default.

                                   The drop-down list contains a list of predefined parameter
                                   files that were saved to the Parameter Files Directory (refer to
                                   Parameter Files Directory on page **8-11**), as well as those
                                   shipped with the analysis program. You may choose a differ-
                                   ent file from this list if desired.

Report Options   Displays the name of the Report Options file for the current sample file.

If this is a new file, this field contains the file you specified as the default.

The drop-down list contains a list of predefined parameter files that were saved to the Parameter Files Directory (refer to **Parameter Files Directory** on page **8-11**), as well as those shipped with the analysis program. You may choose a different file from this list if desired.

Po   Enabled if you have chosen to enter the Po measurement. Enter (or confirm) the saturation pressure of the adsorptive, or edit the current one if desired.

Bath temperature   Enabled if you have chosen to enter the bath temperature (Po and T option on the Analysis Conditions dialog). Enter (or confirm) the temperature for the analysis bath.

Ambient Temperature   Enter the ambient temperature.

Ambient and Analysis Free space   Enabled if you have chosen to enter the free space values (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the appropriate values.

Report After Analysis   Displays the Report Settings dialog so that you may specify report output options. If you choose Screen, reports have many options for being customized and manipulated. (Refer to **Onscreen Reports** on page **7-16** for details about onscreen reports.)
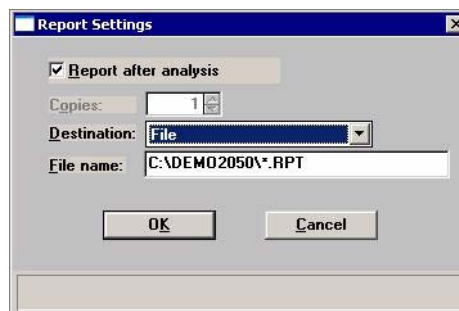
Start   Begins the analysis; after data start to collect, an analyzing view of the Analysis dialog is displayed.
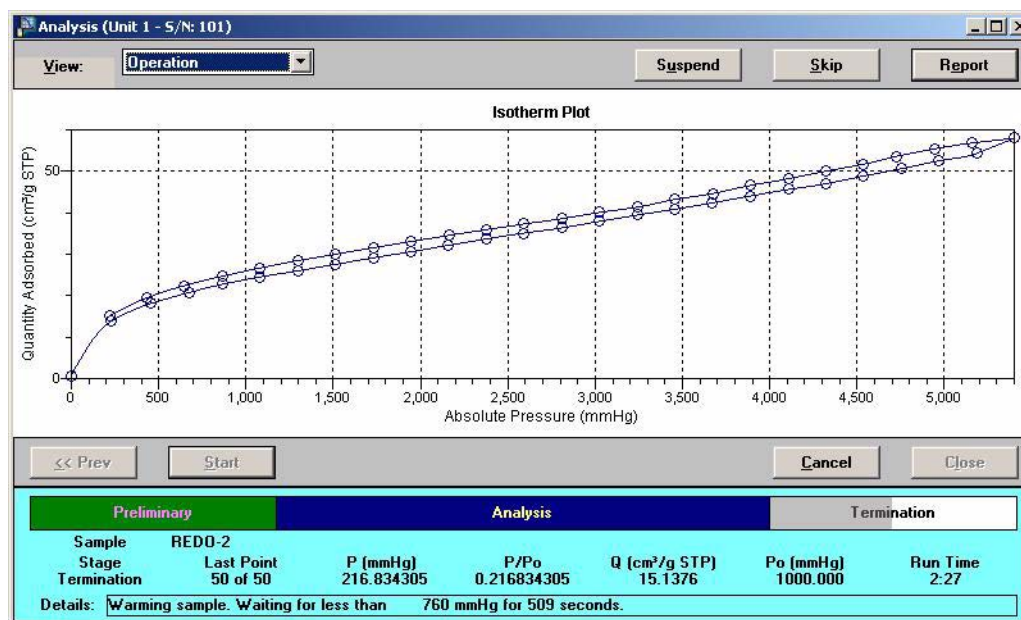
| Suspend | Suspends the analysis. |

| Skip | Skips the current step of the analysis. |

> ⚠️ **Use the Skip command with caution; the ASAP performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.**

| Resume | Resumes a suspended analysis. |

| Cancel | Cancels the analysis. |

| Start | On this view of the analysis dialog, this push button changes to **Next** when the analysis is finished. |

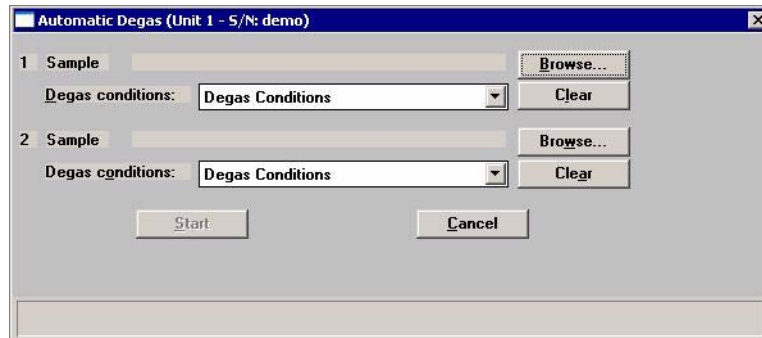| Next | Enabled at the completion of the analysis. Returns you to the first view of the Analysis dialog so that you may schedule another analysis. |

| Close | Enabled at the completion of analysis. Closes the dialog. |

Start Degas

This option allows you to choose the file and start degas operations on one or two samples; the Automatic Degas dialog is displayed.

|	
Sample	Click **Browse** to the right of this field to select a sample file that will be used with the current sample.
Degas Conditions	Displays the Degas Conditions file designated as the default. After you choose a sample file, this field displays the file associated with the sample file.
	You can choose a different Degas Conditions file from the drop-down list if desired. If you choose a different file, the values in the current file will be overwritten with the values of the new one.
Clear	Clears the sample file from the port assignment. Click **Browse** to choose a different file.
Start	Starts the degassing operation
Cancel	Closes the dialog, cancelling the degassing operation.

Enable Manual Control

Select this option to control certain components of your system manually. If the instrument schematic is not displayed, select **Instrument Schematic** from the **View** drop-down list or from the Unit menu**.**
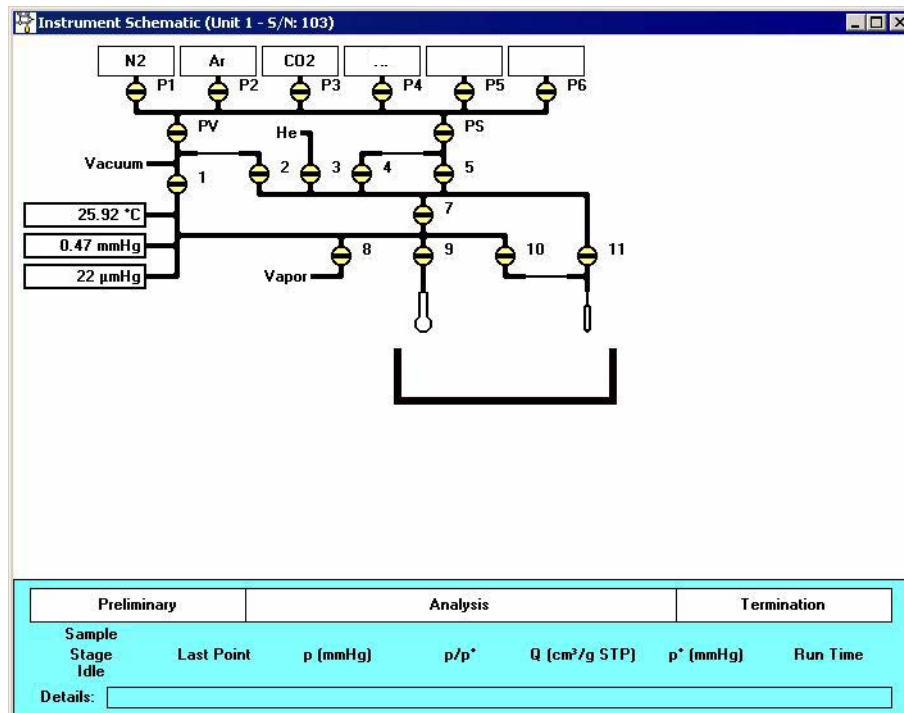
When manual control is enabled, the valve symbols change color on the monitor screen to indicate their status.

> Green = open
> Yellow = closed

Use the mouse pointer to select a component. A component is selected when it is surrounded by a thin line. Each component has a shortcut menu displaying the operations available for that particular component. These menus may be accessed by right-clicking on the desired component, or by using the shortcut keys **Shift + F9**.

You can open and close valves, and raise or lower the elevator by using one of the following methods:

- right-click on the valve or elevator symbol to access the shortcut menu and select the appropriate action

- double-click on the valve or elevator symbol

- select the valve or elevator symbol and press the **Spacebar**

Valves

Open   Closed

System valves and their functions are listed in Table 6-1.

Actions: Open, Close, Pulse

Pulse briefly opens and closes (or vice versa) the valve.

Table 6-1. System Valves

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Free-space measurement gas (helium)
4	Restricted analysis gas
5	Unrestricted analysis gas
7	Lower manifold isolation
8	Vapor
9	Sample port
10	Restricted Psat tube port
11	Unrestricted Psat tube port
P1 through P6	Gas inlet port valves
PS	Supply valve for physisorption gases
PV	Vacuum valve for physisorption gases

Dewar positions
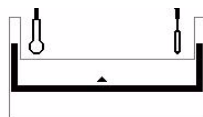
The Dewar can be raised, lowered, or stopped.

Indicates the Dewar is being raised; note the position of the arrow in the center of the Dewar symbol.
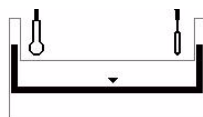
Indicates the Dewar is being lowered; note the position of the arrow in the center of the Dewar symbol.

Indicates the Dewar is stopped in the raised position.

Indicates the Dewar is stopped in the lowered position.

The remainder of the components are displayed for informational purposes only; they cannot be controlled manually.

Represents the sample tube.

Represents the Psat tube.

Temperature   25.92 °C   This group of rectangles on the left side of the schematic dis-
                                             plays the temperature, the transducer, and the vacuum gauge
Transducer   0.47 mmHg   pressure.

Vacuum Gauge   22 µmHg

Show Instrument Schematic

Select this option to display a schematic of the ASAP 2050 analyzer. The schematic is a graphical representation of the plumbing system including system valves, the analysis station, and the Dewar position. The valves and Dewar can be controlled from the schematic when Manual control is enabled. Refer to **Enable Manual control** on page **6-8** for an explanation of the components displayed on the instrument schematic.
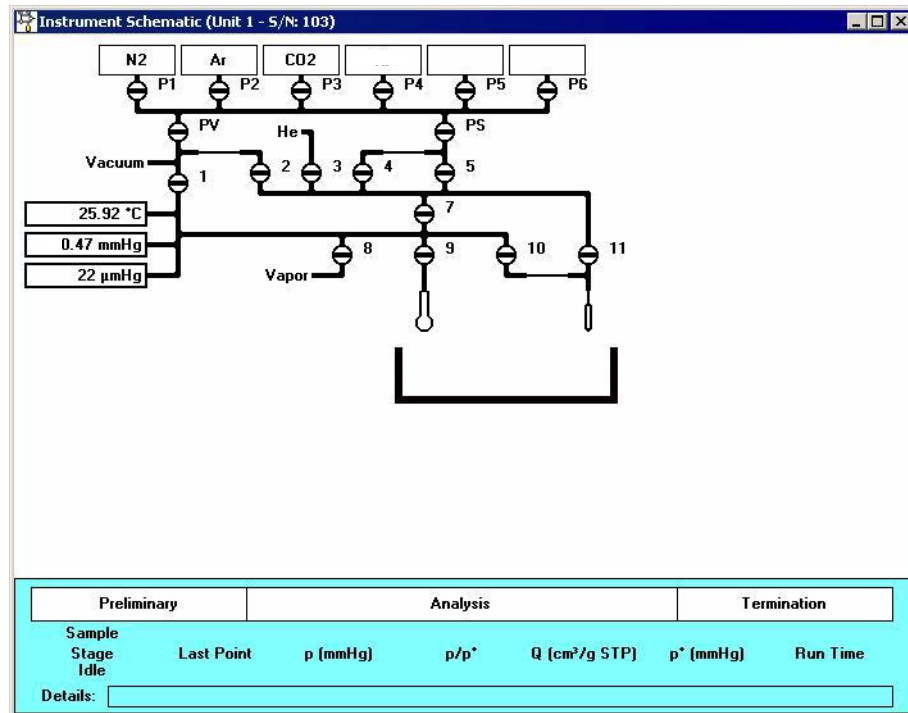
The state of the valves can be determined quickly by color representation even when manual control is not enabled:

- Green = open
- White = closed

If you wish to change the state of a valve, you must enable manual control.

Show Status

A status window is shown across the bottom of all operational dialogs as well as the instrument schematic. The Show Status option enables you to show only the status window. This frees up your computer screen allowing you to perform other tasks (such as creating or editing sample files), while still monitoring the progress of your analysis.

If you have multiple instruments attached to your computer, the status bar for each instrument is displayed in a different color.

Analysis status bar   Shows the progression of the analysis. This bar contains three stages:

- **Preliminary**: ambient and analysis free spaces are determined; displays green during progression

- **Analysis**: data are collected; displays blue during progression

- **Termination**: the Dewar lowers, and the sample tube is warmed and backfilled; displays gray during progression

All stages are displayed in red if the analysis has been suspended.

Analysis details   The following analysis details are displayed:

Sample:	Sample file number
Stage:	Stage of the analysis (as in status bar)
Last Point:	Last point and number of points requested
P:	Absolute pressure of last point
P/Po:	Relative pressure of last point
V(cm^3/g):	Volume adsorbed
Po:	Absolute pressure of last measured or calculated saturation pressure (Po)
Run Time:	Elapsed time since the start of analysis

Step details   Provides details of the current step of the analysis.

Show Instrument Log

Displays a log of recent analyses, calibrations, and errors or messages.
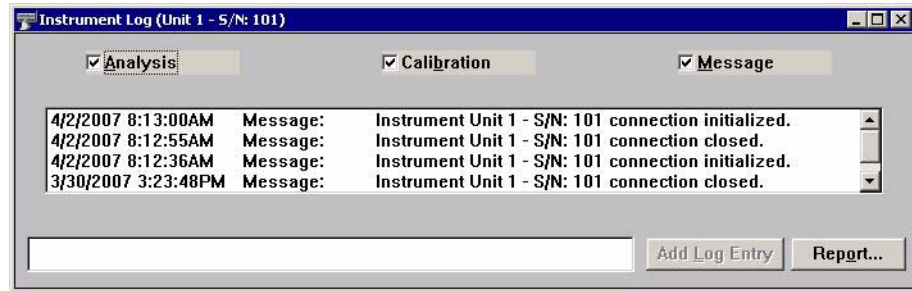
By default, this information is logged for a 60-day period for analyses and messages, and a 90-day period for calibrations. You may change the time for which this information is retained in the Unit section of the WIN2050.INI file. Simply replace the default value with the desired number.

Analysis **Calibration** **Messages**	These options allow you to choose which entries are displayed in the window. For example, select the **Calibration** check box to display only calibration information.
Add Log Entry	Enables you to make an entry in the instrument log that cannot be recorded automatically through the application software. For example, you may change the port filter. The field adjacent to the push button allows you to enter the operation; the push button is enabled when you make an entry in the field, allowing you to add the entry.

Report

Displays the Log Report Settings dialog allowing you to generate the log contents to a specified destination.

Use the **Start Date** field to specify the date at which to start the printout. This date does not limit the entries that display in the window, it is only pertinent to the entries that will be printed on the report.

You can specify a date using one of the following methods:

• Highlight the field (or press **F2** to clear the field) and type in the desired date.

• Double-click in the field (or press **F4**) to display a calendar to choose a date.

• Press **F3** to insert the current date.

Enter the number of copies desired in the **Copies** field; you may print up to 4. This field is disabled if you are printing to a File or to the Screen.

Choose the report destination from the drop-down list in the **Destination** field. If you choose File, the **File name** field is enabled, allowing you to enter a name for the printed file (or you may accept the default).

Unit Configuration

Select this option to view the current calibration settings, the date on which calibration was performed, and the software and hardware configuration of your system.
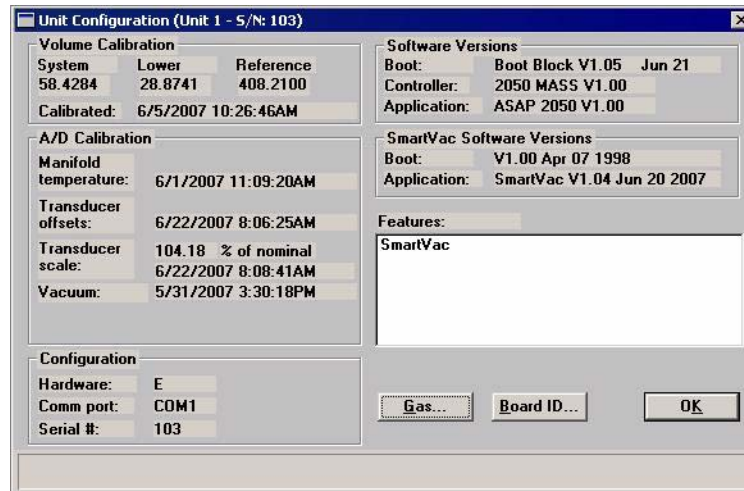
Volume Calibration	Displays the system, lower, and reference volumes from the most recent calibration. It also displays the date and time the calibration was completed.

A/D Calibration	Displays the following:

- The date and time the manifold temperature was last calibrated.

- The date and time the transducer offsets were last calibrated.

- The percent of nominal transducer scale in use, and the date and time the transducer scale was last calibrated.

- The date and time the vacuum gauge was calibrated.

Configuration	Displays the following:

- The hardware code specifying the hardware components present in your analyzer. This code is provided so the Service Department may quickly identify your hardware configuration and provide prompt assistance.

Configuration
(continued)

- The identification number of the port being used to control the analyzer.

- The serial number of the attached analyzer (active unit).

Software Versions

The software versions of the MIC BIOS, Controller, and ASAP 2050 System software.
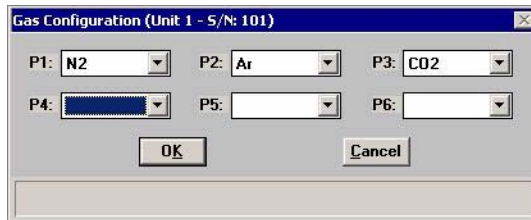
SmartVacSoftware Versions

The software versions of the MIC BIOS and degas system software.

Features

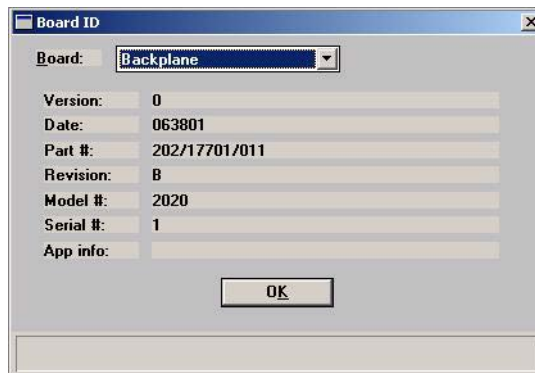The options currently enabled on your system.

Gas

Displays the Gas Configuration dialog.

This dialog allows you to choose the gases attached to each port of the analyzer.

Board ID

Displays the Board ID dialog so that you can view the statistics of the board contained in the requested slot of the card cage in the instrument.
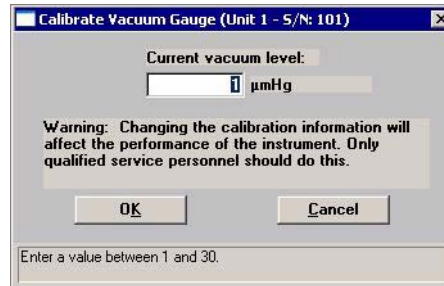
Calibration

This option allows you to calibrate the vacuum gauge, pressure gauge, pressure scale, manifold temperature, and system volume of the analysis system. You can also save the current calibrations to a file or load a different calibration file.

Vacuum Gauge

Displays the Calibration Vacuum Gauge dialog containing the current vacuum level.
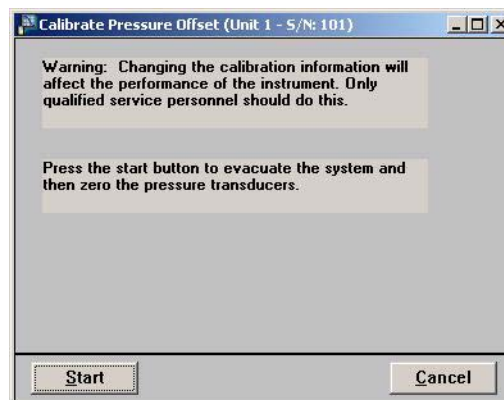
This dialog also allows you to enter a new value (if necessary) after calibration using an external reference gauge.
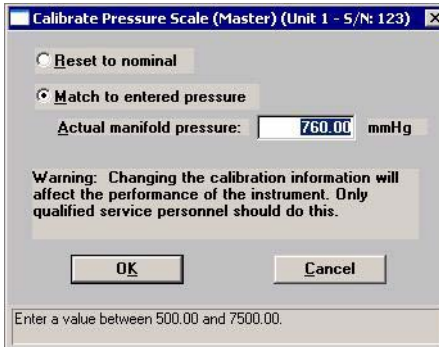
Pressure Gauge

Displays the Calibrate Pressure Offset dialog.

This dialog allows you to evacuate the manifold and zero all pressure transducers in the system.

Pressure Scale

Use this option to adjust the scale of the transducer to match a reference standard; the Calibrate Pressure Scale dialog is displayed.

Reset to nominal   Restores factory calibration settings for the transducer.

Match to entered pressure   Select this option to enter a value obtained using a reference
                                  pressure gauge to determine the actual manifold pressure.

Temperature

Use this option to adjust the offset of the manifold temperature sensor to agree with a value obtained using a calibration device. The Calibrate Manifold Temperature dialog is displayed so that you may enter the measured value.
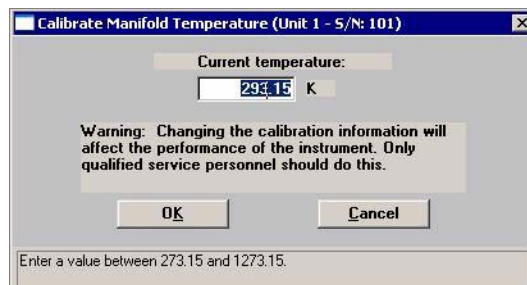
Volume

Use this option to enter a predetermined system volume in the event of data loss.

Displays as Next if you choose Measured.

The system volume typically is calibrated after servicing valves, transducers, or any part of the analysis plumbing. The ASAP 2050 must be idle to calibrate system vol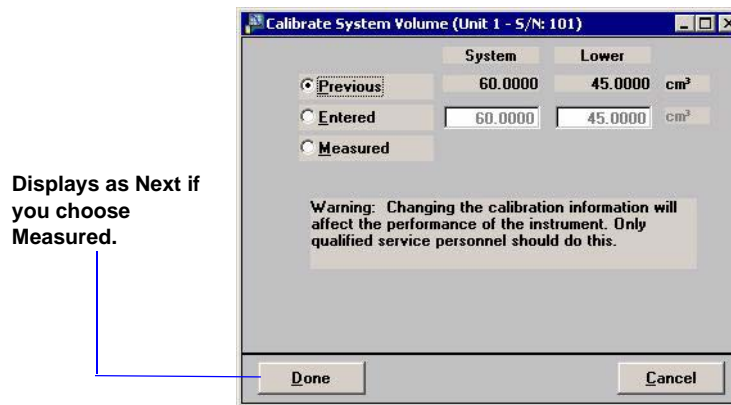ume. Before system volume is calibrated, the manifold temperature sensor and the master pressure gauge must be calibrated.

Previous   Retains the values currently in use.

Entered   Allows you to enter values to use.

Measured   This field is used by authorized service personnel when measuring the system and lower manifold volumes. A special Service Kit is required to perform these procedures.

Done   Closes the dialog (displays for the Previous and Entered selections).

Next   Appears only when **Measured** was selected; displays a dia-
                                        log that enables service personnel to enter measured volumes.
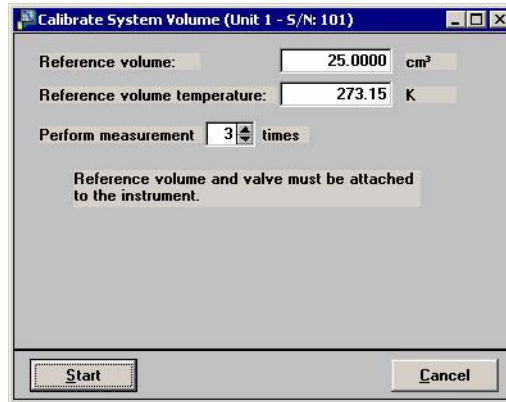
Reference Volume   Enter the number of cubic centimeters for the reference vol-
                                        ume you are using in this calibration.

Reference Volume Temp.   Enter the temperature of the reference volume. Since the ref-
                                        erence volume should be submerged in an ice bath, this value
                                        should remain at 0.0°C for best accuracy.

Perform measurement   Choose the number of measurements you wish to perform;
                                        you may choose up to 9.

Start   Begins the calibration; a status view is displayed. After the
                                        calibration has finished, a confirmation view of the Calibrate
                                        System Volume dialog is displayed.
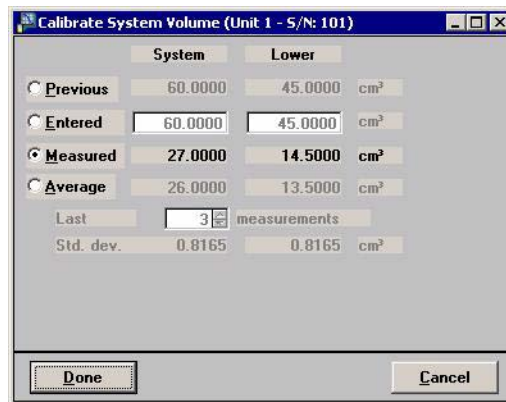
This dialog allows you to choose the volume you wish to use.

Previous
The system volume and lower manifold volume in use before this measurement.

Entered
An entered measurement.

Measured
The last measured system and lower manifold volumes; for example if you choose 3 measurements, this value is that of the last (third) measurement.

Average
The average of the series of measurements; for example if you choose 3 measurements, this value is the average of the three measurements.

Click **Done** to close the dialog.

Save to File

Use this option to save the current calibration settings to a file; the Select Calibration File dialog is displayed
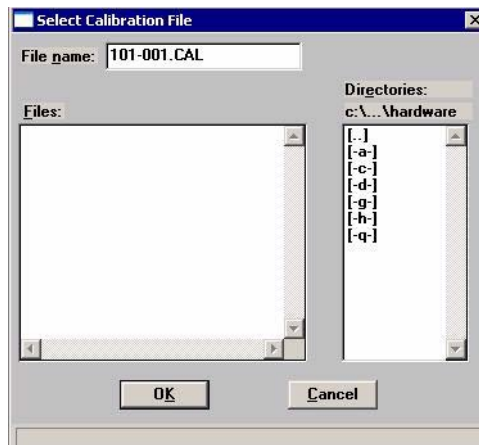
Load from File

File name   Defaults to the next sequence number for calibration files. The sequence number consists of: xxx-yyy.CAL, where xxx = serial number of unit and yyy = sequence number. For example, the number listed in the above dialog represents the first calibration file for unit serial number 101.

Displays the Select Calibration File dialog (shown above) so that you can load a previously saved calibration file.

Degas

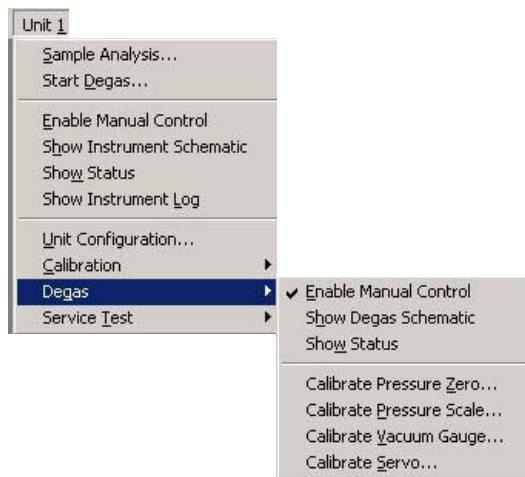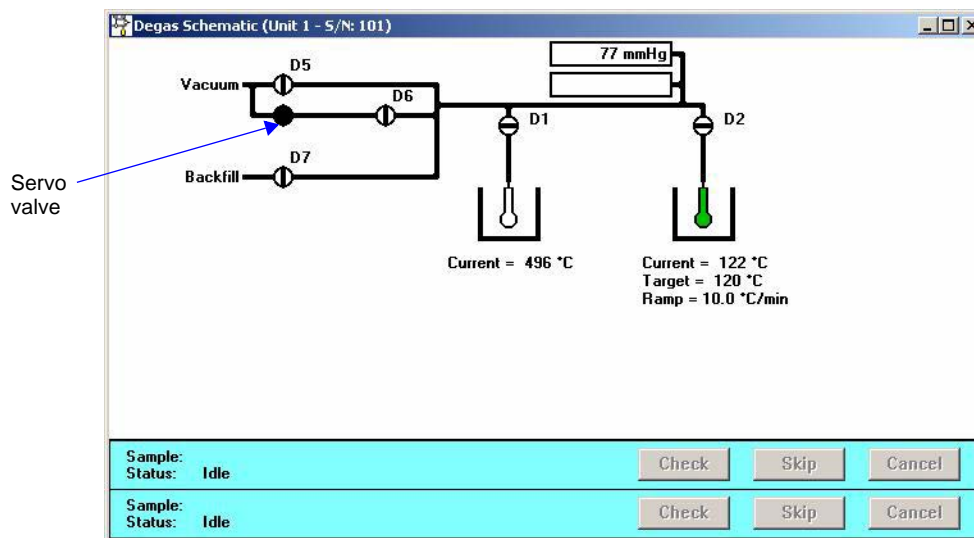This option allows you to select options for the degas system.

Enable Manual Control

Select this option to control manually certain components of the degas operation. If the degas schematic is not displayed, select **Show Instrument Schematic** from the Degas menu.

Valves D1, D2, D5, D6, D7   Degas valves and their functions are listed in Table 6-2.

Actions: Open, Close

Table 6-2. Degasser Valves

Valve	Description
D1 and D2	Sample port valves
D5	Vacuum valve
D6	Servo isolation valve
D7	Gas inlet port valve

Servo Valve   *Actions: Set, Close*

Set displays the Servo Value Settings dialog so that you can enter a target pressure for sample evacuation. This feature enables you to use the software to bring down the sample pressure slowly and incrementally.

Close closes the servo valve.

Heaters   *Actions: Set, Disable*

Set displays the Port [n] Heater Settings dialog so that you can specify a ramp rate and target temperature. After you click and close the dialog, the settings are displayed directly below the heater symbol.

Disable cancels the settings.

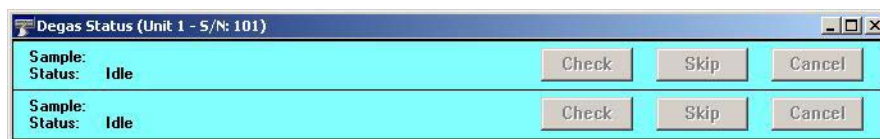**Status window**   Displays the status of the current degassing operation(s).

Show Instrument Schematic

Displays the schematic (shown in the previous section) for the Degas system.

Show Status

This option allows you to monitor the degassing operation.

Sample   Displays the sample file being used with the degassing oper-
                            ation for each port.

Status   Displays the current stage of the degassing operation for
                            each port.

Check   Allows you to check the outgassing rate of the sample on the
                            related port; the following actions occur:

                            • current degassing step is suspended (on both ports).
                              Degassing can be checked after the vacuum setpoint has
                              been attained, or during a temperature ramp and hold. If
                              you choose this option during any other step, a message
                              indicating the degas system is not in a valid state is dis-
                              played.

                            • vacuum valves are closed and the vacuum level moni-
                              tored.

                            • the Status window will indicate that the degassing opera-
                              tion is being checked and will display the outgassing rate
                              as it becomes available.

                            During the degas check, this push button changes to **Con-
                            tinue**. When **Continue** is clicked, the valves open, the tem-
                            perature ramp or hold continues, and the degassing operation
                            resumes. If your outgassing rate indicated that the sample
                            has been rid of contaminants (minimal pressure increases),
                            you can click **Skip** to advance to the next state of the degas-
                            sing operation. For example, if you check degassing after the
                            setpoint is attained, **Skip** advances you to the ramping stage.

Skip   Skips the current stage of the degassing operation for the
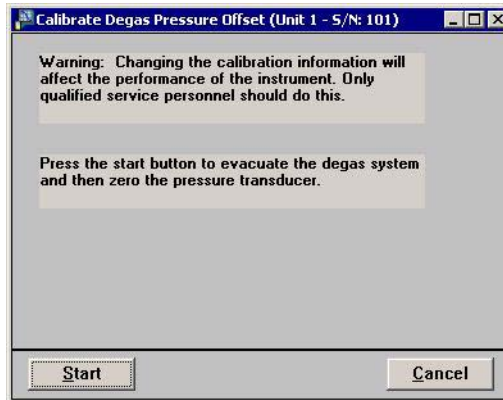   port associated with this push button.

Cancel	Cancels the degassing operation for the port associated with this push button.

Calibrate Pressure Zero

Use this option to evacuate the manifold and zero the transducer
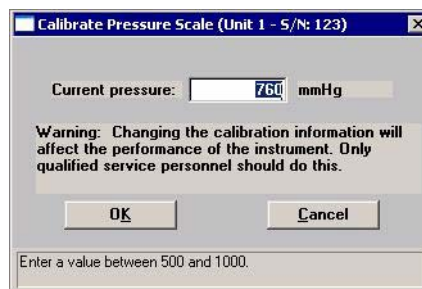

Start	Begins the calibration.
	Status messages are displayed during this procedure, then the dialog closes automatically.

Calibrate Pressure Scale

Use this option to adjust the scale of the pressure transducer.
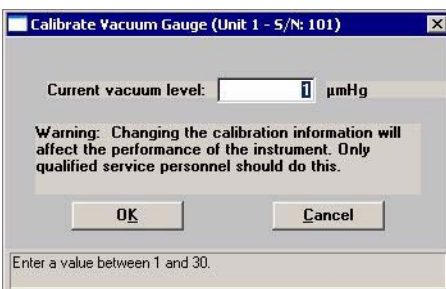

Current Pressure	Enter the current pressure of the degas manifold.

Calibrate Vacuum Gauge

Displays the Calibrate Vacuum Gauge dialog containing the current vacuum level.

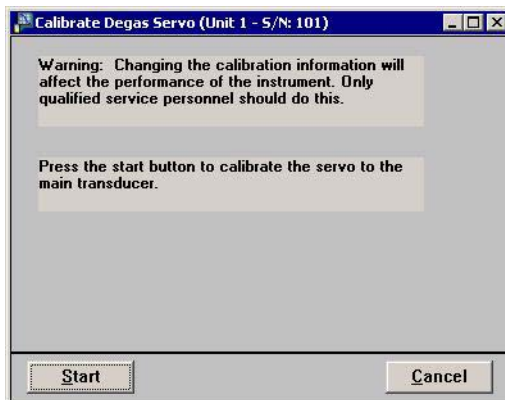This dialog also allows you to enter a new calibration value (if necessary) obtained using an external reference gauge.

Calibrate Servo

Use this option to calibrate the servo valve to the manifold pressure transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The Calibrate Degas Servo dialog is displayed.

Start   Begins the calibration. The servo valve is used to evacuate and equilibrate the manifold. The pressure transducer readings are used to calibrate the servo set point. Status messages are displayed during this procedure, then the dialog closes automatically.

Service Test

Various service tests are included in the ASAP 2050 operating program. These tests can be enabled and performed only with the assistance of a trained Micromeritics service representative. These tests are designed to provide your service representative with instrument readouts, as well as to assist him in troubleshooting potential problems and, perhaps, eliminating unnecessary repair services. This service strategy allows you to conduct expert tests in less time than it would take to be trained in servicing the instrument properly.

7. REPORTS MENU

This chapter describes the commands on the Reports menu.

Reports can be generated for data:

- collected on a sample that has completed analysis

- collected on a sample that is currently being analyzed (includes only the information col- lected up to the time of the report)

- that is manually entered

Description

Listed below are brief descriptions of the commands contained on the Reports menu. Detailed descriptions follow this section.

|	
Start Report	Allows you to generate a report on a completed sample analysis or on the data collected thus far for an analysis in progress. Page **7-2**.
Close Reports	Closes all open report windows. Page **7-4**.
Open Report	Enables you to open a report that was saved from the report window. Page **7-4**.
SPC Report Options	Allows you to specify the sample data to be included in SPC reports. Page **7-5**.
Regression Report	Allows you to generate a regression report. Page **7-6**.

Control Chart	Allows you to generate a control chart report. Page **7-10**.
Heat of Adsorption	Allows you to generate a Heat of Adsorption report. Page **7-13**.
Rate of Adsorption	Not used.

Start Report

Select this option to generate a report on a sample analysis. Select **Start report** from the **Reports** menu to display the Start Report dialog.
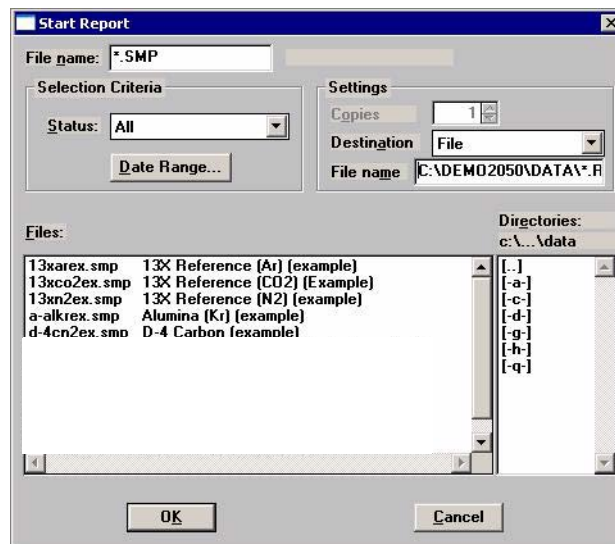
File name	The name you select from the Files list box is copied to this field. If you select multiple files, only the last one selected is displayed. If you have a sample file open when you select this option, its name is displayed.
Status	This drop-down list determines what type of sample files are displayed in the **Files** list window in the specified directory for all dates, or within the specified range of dates (using push button).
	Choices:All, Analyzing, Complete, Entered
Status *(continued)*	Refer to **"Table 2-2. File Status and Description" on page 2-12** for a description of the status types.

Date Range   Displays the Select Dates dialog so that you may specify a
                            range of dates. Refer to **"Selecting Files" on page 2-11** for a
                            description of this push button.

Copies   Enabled when the Printer destination is chosen. This option
                            allows you to print up to four copies of the selected report(s).

Destination   Displays a drop-down list of output destinations.

                            Choices:File, Printer, Screen

                            If you select **Printer**, requested reports are printed to the
                            selected printer.

                            If you select **Screen**, many options are available for
                            manipulating and customizing reports. Refer to Printed
                            reports on page **7-16.**

                            If you select **File**, the tabular reports of the requested file are
                            converted to a text file which can be viewed with a text editor
                            or other text file manipulation tool. Graphical data cannot be
                            generated to a File.

File name   Enabled when you select File as the destination. Allows you
                            to enter a name, or you may accept the default.

Files list box   Displays a list of the available sample files for the choice
                            shown in the **Status** field and within the range of dates
                            specified in the Dates dialog.

Directories   Displays a list of available drives and directories. The drive
                            and directory last accessed is displayed immediately above
                            the Directories list box.

                            If you choose a single sample file, the Reports to Generate
                            dialog is displayed. The reports selected are the ones you
                            specified in the sample file. This dialog allows you to
                            deselect reports or select additional ones.

Double-click on the report(s) you wish to select (or deselect); alternatively, you can highlight the report and press **Space-bar**. A check mark is placed before each selected report. After you make your report selection and click **OK**, the requested reports are sent to the specified destination.

If you choose more than one file, this dialog is not displayed. The reports specified in each sample file will be generated.

Close Reports

This option enables you to close all open report windows at one time. This avoids having to select close on each report window.
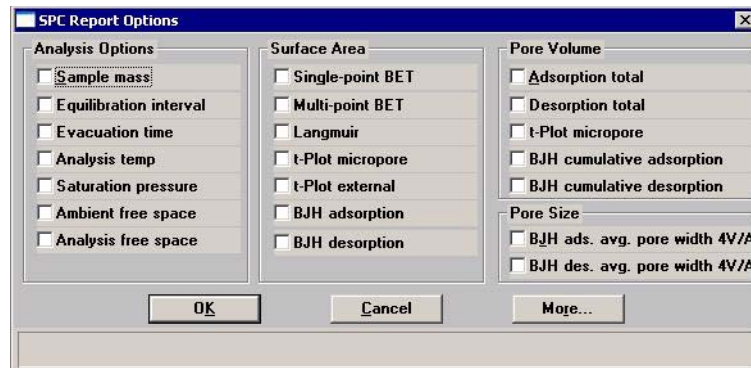
Open Report

Enables you to open a report that you saved from the report window.

SPC Report Options

When you select SPC Report Options, the SPC Report Options dialog is displayed.

The SPC Report Options dialog lists the variables that are most frequently used for SPC reporting. You can check as many as desired. All variables selected are computed for *each* sample file used in an SPC report.

Click **More** for additional SPC calculations for the detailed reports.
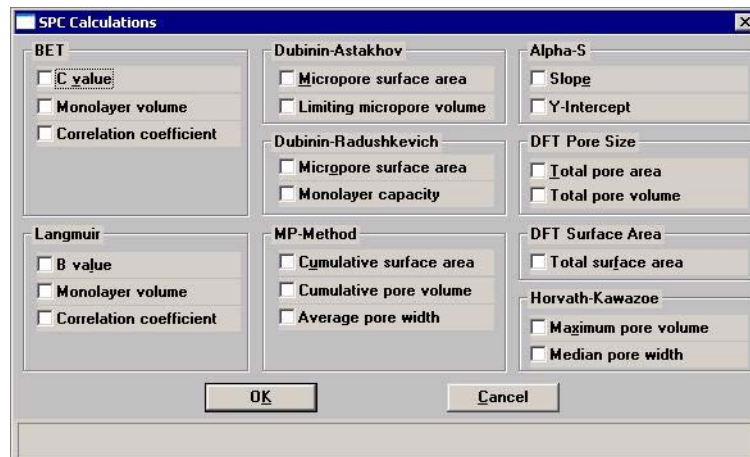
Regression Report

Select this option to generate a regression report. The regression report is used to determine the interdependency between two variables. Up to three dependent variables (Y-axis) may be plotted against a single independent variable (X-axis). The degree of correlation between the variables also is reported. The graphs for the regression report are scaled so that all three fit on a single page. If you choose less than three, the graphs are scaled to fill most of the page.

Show report title	Select this option to have a title display on your report. Accept the default or enter a new title. You can enter up to 40 alphanumeric characters.	

Deselect this option to omit the report title.

Show graphic   Select this option to have a graphic display above your report title. For example, you may wish to display your company logo. The graphic must be a bitmap (bmp) or enhanced metafile (emf).

Click **Browse** to choose the file, then use the **Height** and **Width** fields to specify a size. This image can be edited in the report window (when printed to the screen), or removed if desired.

|	
X- and Y-Axes Variable fields	Allows you to designate the X- and Y-axes variables. Click on the down arrow to display a list of variables. The variables in this list are the ones you specified in the **SPC report options**.
	With this option, you can plot the regression of up to three Y-axis variables against the X-axis variable. The X-axis specifies the independent variable for the regression, while the Y-axes provide the dependent variables.
Axis Range	Enables you to specify the beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot. These fields are disabled if you choose **Autoscale**.
Autoscale	Allows you to have the X- and/or Y-axes scaled automatically. When scaled automatically, both axes begin at zero. The analysis program uses the highest values collected during analysis as the ending points.
Recalculate archived SPC results	Select this option to have archived SPC values recalculated. This ensures that any changes made to the SPC Report Options are included in the new report; however, it does lengthen the time required to generate the report.
Tabular report	Enables you to generate tabular, as well as graphical, data of the included samples. A tabular report contains the numeric values contributed by each sample.
Label data	Allows you to label the points on the plot to correspond with the values in the sample files.
Samples	Displays the Regression Report Sample Selection dialog, allowing you to choose the sample files you wish to have reported.
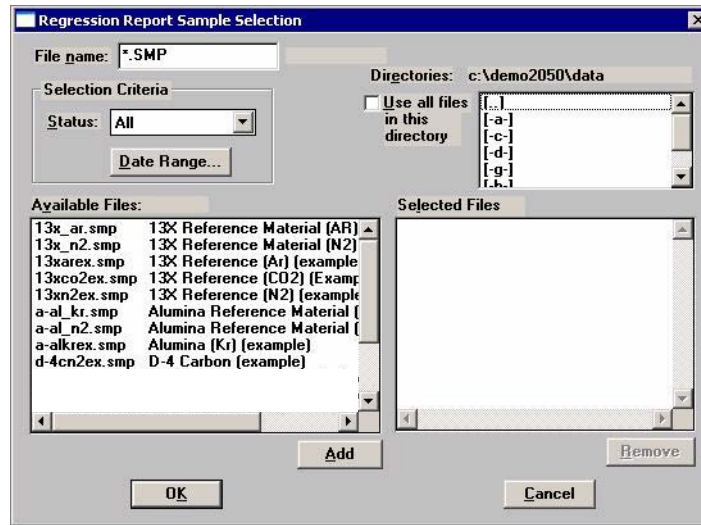
File name	Use this field to limit the files displayed in the Available Files pane. For example; enter **g*.smp** to display only the files beginning with an **g**.
Status	This drop-down list determines the type of sample files that display in the **Available Files** pane in the selected directory for all dates, or within the specified range of dates (using **Date Range** push button). Refer to **"Table 2-2. File Status and Description" on page 2-12** for an explanation of the Status types.
Date Range	Displays the Select Dates dialog so that you can specify a range of dates. Refer to **"Selecting Files" on page 2-11** for an explanation of this dialog.
Use all files in this directory	Select this option to include all files from the selected directory in the report.
Directories	Lists the current directory. Use the directory window to navigate to a different directory.
Add	Moves the selected file in the **Available Files** pane to the **Selected Files** pane. Alternatively, you can simply double-click on the desired file(s). You can select multiple files by holding down **Ctrl** while making your selections. You can include up to 200 sample files.

Remove	Removes the selected file from the **Selected Files** pane and places it back into the **Available Files** pane.
Save As Default	Saves the current definition of the report as the default.
Report Settings	The options in this group box enable you to choose output criteria.
	The **Copies** field is enabled when you choose **Printer** as the destination, enabling you to print up to four copies of the report.
Report	Generates the report.

Control Chart

This option enables you to generate a control chart report which plots the changes in a statistic.

|	
Show report title	Select this option to have a title display on your report. Accept the default or enter a new title. You can enter up to 40 alphanumeric characters. Deselect this option to omit the report title.
Show graphic	Select this option to have a graphic display above your report title. For example, you may wish to display your company logo. The graphic must be a bitmap (bmp) or enhanced metafile (emf). Click **Browse** to choose the file, then use the **Height** and **Width** fields to specify a size. This image can be edited in the report window (when printed to the screen), or removed if desired.
X-axis Order By	Enables you to choose the order in which X-axis statistics are placed. You can have them placed by **Time**, **File name**, **Date**, **Minutes**, or **Days**.

Graph [n]   Displays the Control Chart Graph [n] Options dialog, allowing you to define the Y-axis of each graph.
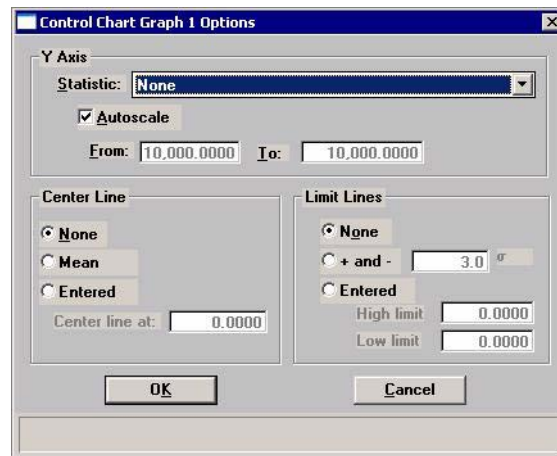
Statistic   This drop-down list displays the SPC variables selected on the SPC Report Options dialog. The variable you choose will be plotted against time.

Autoscale   Allows you to have the Y-axis scaled automatically. If you wish to specify a range, deselect this option and enter a range in the **From** and **To** fields.

Center Line   Displays placement options for the variable's optional value. Choose *Entered* to specify placement of the line.

Limit Lines   Displays the options available for limiting lines. You can have the lines placed at some multiple of the standard deviation or at specified positions (*Entered*).

                                 When you select *Entered*, the **High limit** and **Low limit** fields are enabled, allowing you to enter appropriate values.

Tabular report   Allows you to generate tabular, as well as graphical, data of the included samples. A tabular report contains the numeric values contributed by each sample.

Recalculate archived SPC results   Select this option to have archived SPC values recalculated. This ensures that any changes made to the SPC Report Options are included in the new report. It also lengthens the time required to generate the report.

Samples
Displays the Control Chart Sample Selection dialog, allowing you to choose the sample files on which you wish to report.
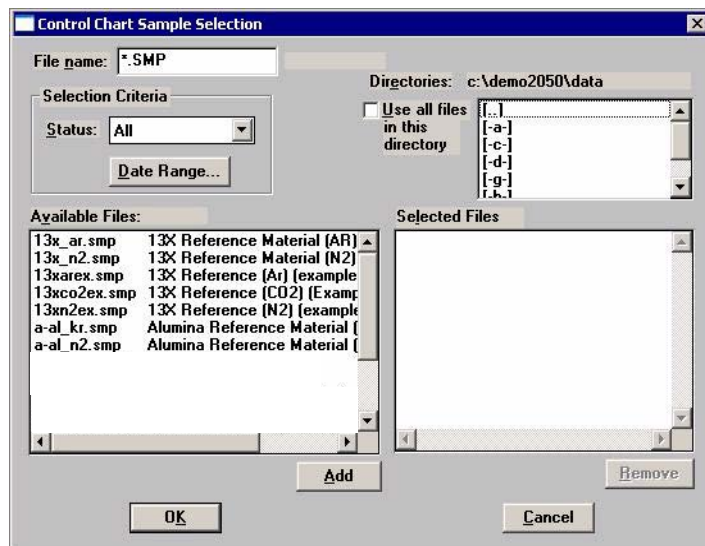
This dialog functions in the same manner as the Regression Report Sample Selection Dialog explained on page **7-7**.

Save as Default
Saves the current definition of the report as the default.

Report Settings
The options in this group box enable you to choose output criteria.

The **Copies** field is enabled when you choose **Printer** as the destination, enabling you to print up to four copies of the report.

Report
Generates the report.

Heat of Adsorption

The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity, providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms are obtained on the same sample using the same adsorptive but at different temperatures to obtain the heat of adsorption.

This option allows you to choose the sample files, define the quantities, and generate a Heat of Adsorption report; the Heat of Adsorption dialog is displayed.
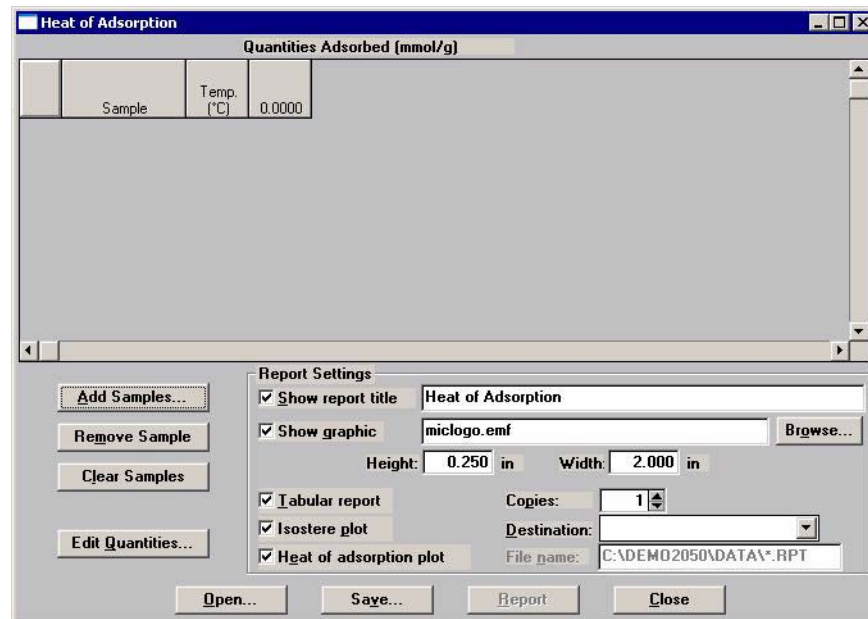
| **Table**	Contains the files you choose; also lists the quantity adsorbed.
Add Samples	Displays the Select Samples dialog so that you can choose desired files.
Remove Samples	Removes the selected sample from the list. If no samples are selected, the last one is removed.
Clear Samples	Removes all samples from the list.

Edit Quantities

Displays the Edit Quantities Adsorbed dialog so that you can specify the range of surface coverage to include in the heat of adsorption report.
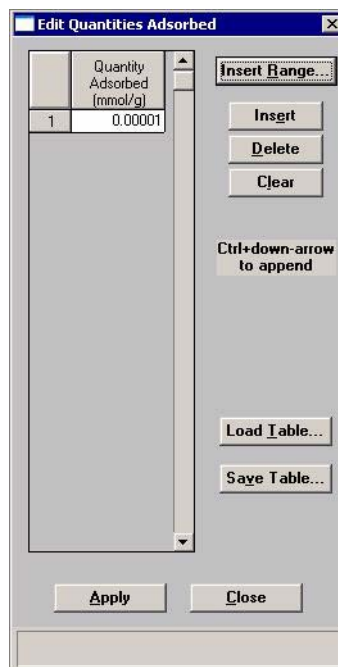
Quantity Adsorbed table

Allows you to enter the points.

Insert Range

Displays the Insert Quantity Range dialog.
This dialog allows you to specify the starting and ending quantities adsorbed, as well as the number of points to insert within the specified range.
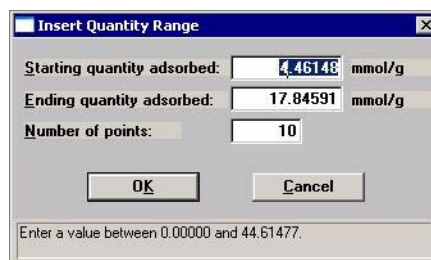
Save Table

Allows you to save the current table as a file (QNT extension).

Load Table

Allows you to import a previously saved table.

Insert

Inserts a row above the selected row.

Delete	Deletes the selected row.
Clear	Clears the entire table of all entries except one; one is required.
Report Settings	The options in this group box allow you to choose report criteria.
Show report title	Choose this option to have a title display in the header of your report, then use the adjacent field to enter the title.
Show graphic	This option allows you to have a graphic appear in your title; for example, you may wish to show your company logo. You can use a bitmap or an enhanced metafile. Use the Height and Width fields to define a size.
Copies	Enabled when you choose Printer as the destination, allowing you to print up to four copies of your report(s).
Destination	Enabled when you choose Printer as the destination, allowing your to print up to four copies of your report(s).
File name	Enabled when you choose File as the destination, allowing you to enter a name (or you can accept the default).
Report	Generates the report.
Tabular report	Select this option to have data generated in a tabular format.
Isostere plot	Select this option to generate a graph showing quantities of gas adsorbed vs. the temperature.
Heat of adsorption plot	Select this option to generate the Heat of Adsorption data in a graphical format.

Printed Reports

Header

All printed reports (either to the screen or to a printer) contain a header displaying file statistics.

- Tabular and graphical reports contain sample and instrument statistics such as when the file was created and by whom, date and time of analysis, analysis conditions, and so forth.

  The headers for these reports also contain notes of any changes to the sample file that occur after analysis.

- Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.
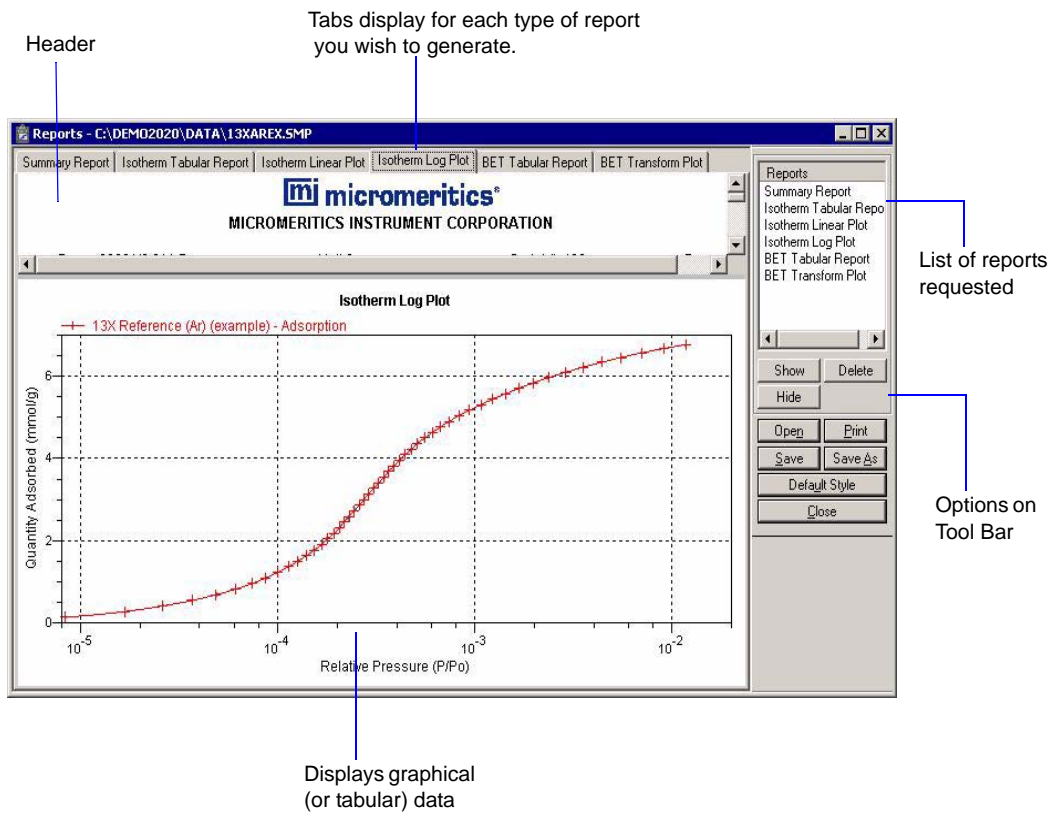
Onscreen Reports

The report window containing onscreen reports provides many options for customizing and manipulating reports:

- a tool bar
- shortcut menus
- zoom feature
- axis cross-hairs

When reports are printed to the screen, they are printed in a window like the one shown below. Each requested report is listed in the Reports window on the tool bar; they are also indicated by selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports window and click **Show**.

Tabs display for each type of report
you wish to generate.

Header

List of reports
requested

Options on
Tool Bar

Displays graphical
(or tabular) data

Tool Bar


Reports   Contains a list of all requested reports.

Show   Shows the selected report in the report window. If the report
                               has been hidden, it and its associated tab will become visible.

Delete   Deletes the selected report. A deletion confirmation dialog is
                                     displayed since this function cannot be undone. The deleted
                                     report(s) will have to be regenerated if deleted in error.

Hide   Hides (removes) the selected report from the report window.
                                     The report's associated tab is also removed.

Open   Allows you to open a previously saved report file.

Print   Displays a print dialog so that you can choose an appropriate
                                     printer for report output. A list of available reports is
                                     displayed in the window on the right side of the dialog.
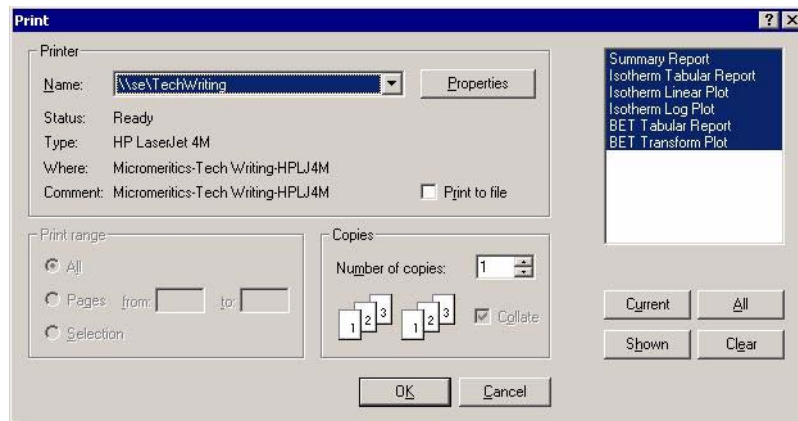
For convenience in selecting which reports to print, push
buttons are provided beneath the report window. Or, you can
make your selection by clicking on the desired reports.

Current selects the report displayed in the report window.

Shown selects only the shown reports; any non-highlighted
reports indicate they are hidden. You can still select hidden
reports from this window to print.

All selects all reports, including those that may have been
hidden.

Clear clears all selections.

Save   Saves all reports of the currently open file in a report format using the same name as the sample file, only with an **rep** extension. If you wish to specify a name and/or specific reports to save, use the **Save As** push button.

Save As   Saves all or specified reports from the currently open file. The push buttons displayed on this dialog perform in the same manner as the print dialog (explained above).

                            Reports can be saved in three different formats:

                            Report system (.rep)*: Saved in a format which allows you to reopen the file using the push button on the Report window tool bar.

                            Spreadsheet (*.xls): Saved in a format which can be imported into most spreadsheet programs.

                            Ascii Text (.txt)*: Saved in ASCII text which can be imported into programs accepting this type of file.

Default Style   Displays the Default Style dialog so that you can specify default parameters for report fonts and curve properties.
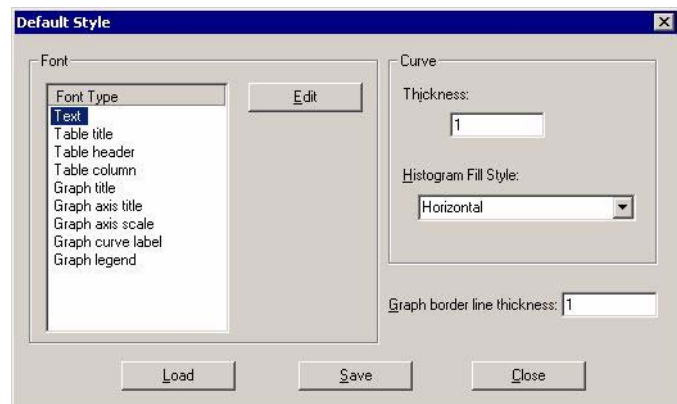
Fonts   Contains a list of report elements for which the font can be edited. Simply highlight the desired element and click **Edit**; a font dialog is displayed so that you can specify the desired font and attributes.

Curve   The items in this group box enable you to specify a thickness for report curves and, when using histograms, the type of fill to apply.

Graph border line thickness	Enables you to specify a thickness for the border of the graph.
Load	Loads the last *saved* defaults.
Save	Saves the changes as the defaults. If you do not click **Save**, the changes will apply to the current report set only. The next reports will revert to the defaults.
Close	Closes the dialog and applies the changes. If you clicked **Save**, the changes become the defaults. If you did not click **Save**, the changes apply to the current report only.
Close	Closes the report window.

Shortcut Menus

Shortcut menus are accessed when you right-click on the tabular or graphical portion of a report.

Tabular Reports

```
Resize column
Rename column

Move column            ▶
Align column           ▶

Hide column            ▶
Show column            ▶

Column font
Header font

Edit title...

Copy table as text
```

Resize column   Displays a dialog so that you can specify the width of the selected column (in inches).

Rename column   Displays a dialog so that you can edit the name of the selected column. Use Ctrl + Enter to insert line feeds.

Move column   Allows you to move the location of the selected column to the left or to the right.

Align column   Enables you to right-align, left-align, or center the data in the selected column.

Hide column   Displays a list of the columns that have not been hidden, enabling you to select the one you wish to hide.

Show column   Displays a list of all hidden columns, enabling you to select the one you wish to have shown again.

Column font   Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.

Header font   Displays a Font dialog, allowing you to change font attributes for column headers in the current report.

Edit title   Allows you to edit the table title and font.

Copy table as text   Enables you to copy the entire table (column headers and data) and then insert it into another program. Columns are tab-delimited, allowing easy alignment

<u>**Graphs**</u>

Autoscale   Autoscales all axes of the graph. This function is useful for returning to a full view after having zoomed in.

Redraw   Sets axis boundaries to its original view. You can also use this function to remove cross-hairs.

Show curve   Shows any curve(s) that has been hidden. This option is disabled (grayed) if no curves have been hidden.

Hide curve   Hides (removes from view) any unwanted curve(s).

Edit curve   Displays the Curve Properties dialog, allowing you to edit
                                  curve properties.
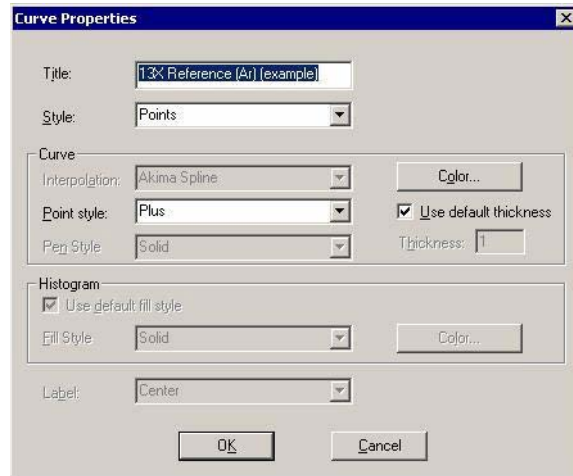
Title   Displays the title of the curve you are editing. You can edit
                                  the title if desired.

Style   This drop-down list contains the styles in which collected
                                  data can be displayed.

                                  Choices:Curve, Histogram, Points, Curve and Points

Curve group box   Contains options for curves and points. You can edit the
                                  curve interpolation, the style of curve and/or points, as well
                                  as the pen color. You can also select to use the default curve
                                  thickness or specify a different thickness.

                                  The options in this group box are disabled if Histogram is
                                  chosen in the Style drop-down list.

Histogram group box   The options in this group box are enabled when you choose
                                  Histogram as the style for collected data. The choices in the
                                  drop-down list allow you to choose the type of fill for the
                                  Histogram, then you can click Color to select the fill color.

Edit axis   Displays the Axis Properties dialog, allowing you to edit axis properties.
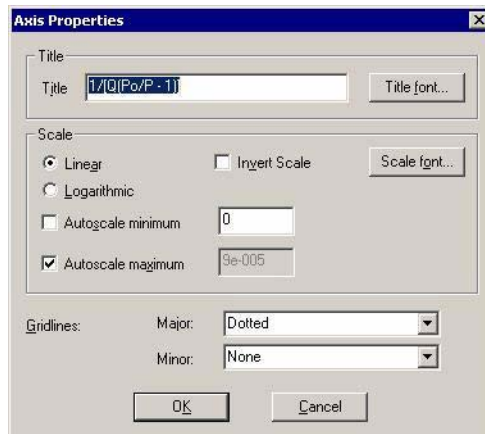
Edit legend   Displays the Legend Properties dialog, allowing you to edit the placement of the legend. You can also edit the font if desired.

Edit title   Displays the Title Properties dialog, allowing you to edit the current graph's title and font.
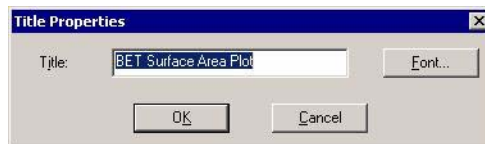
Copy as metafile   Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

Copy as text   Copies the data used to generate the graph as a series of tab-delimited columns of text.

Zoom Feature

A zoom feature is included with the report system so that you can zoom in to examine fine details. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a box) across the area you wish to view; then release the button. The enlarged area immediately fills the graph area. Right-click in the graph area and choose **Autoscale** from the shortcut menu to return to the normal view

Axis Cross Hair

A cross-hair function is available so that you can view axis coordinates. To use this feature, simply left-click in the desired area of the graph.
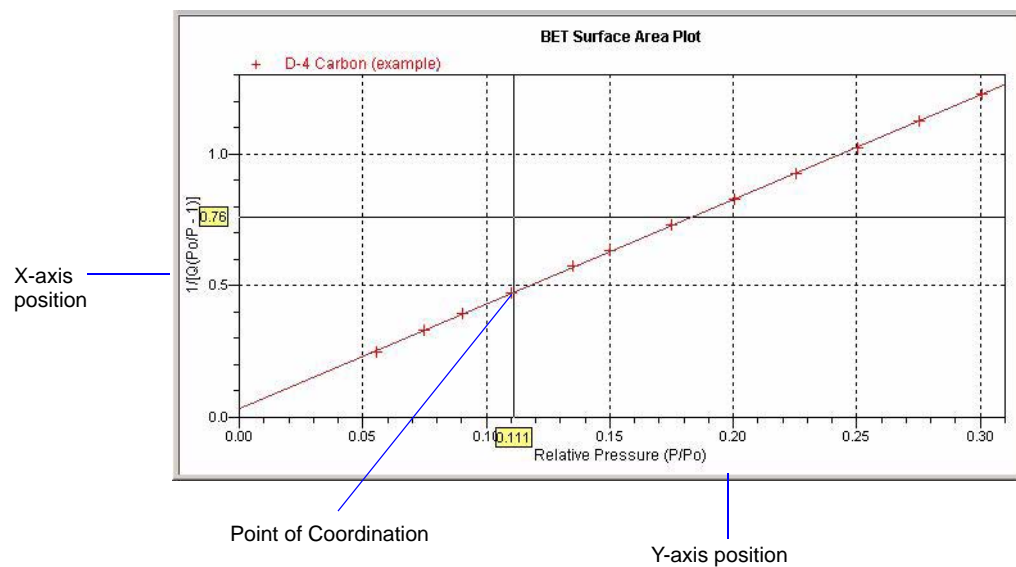
Right-click in the graph area and choose **Autoscale** or **Redraw** from the shortcut menu to remove cross-hair lines and return to the normal view. Alternatively, you can click outside of the graph area.

Sample Reports

The remainder of this chapter contains samples of some of the reports that can be generated by the ASAP 2050 program. Most of the reports can be varied through entries on the report options screens.

Summary Report

---

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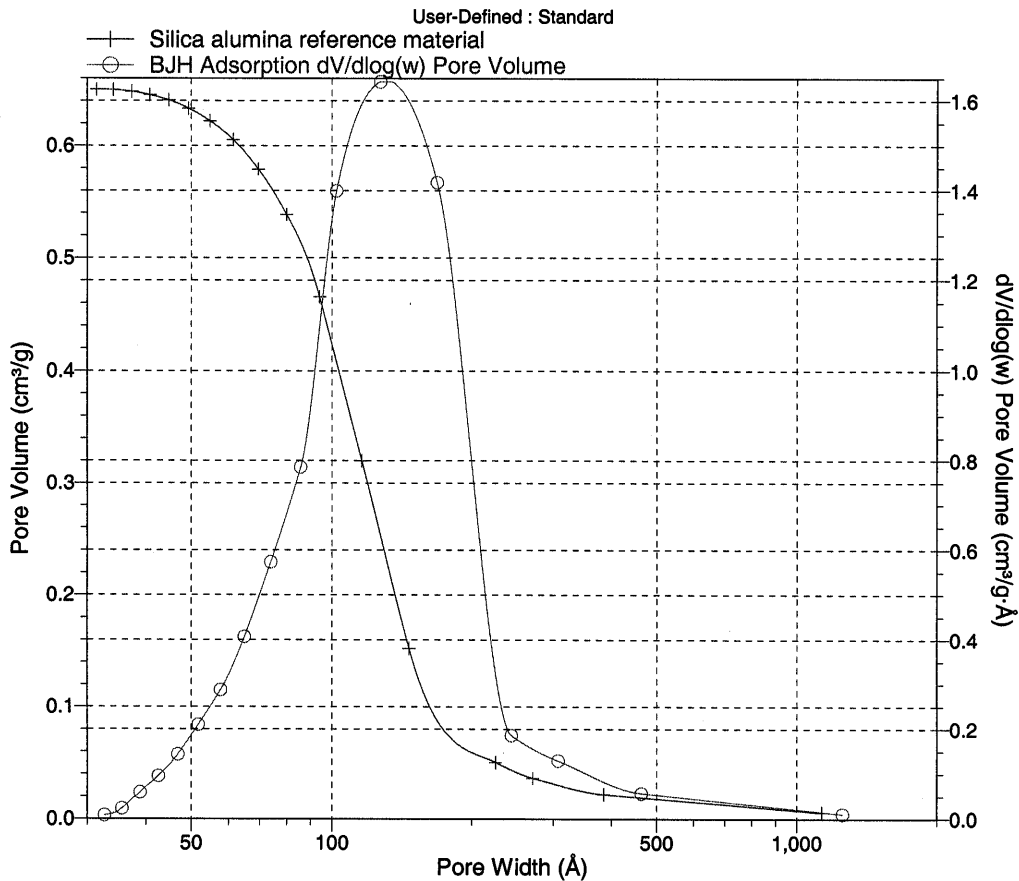2050 'V X.XX   Unit 1   Serial #: 102   Page 1

Sample: Silica alumina reference material
Operator: JCH
Submitter: Unit 102
File: Y:\...\HANKH\SIAL_01.SMP

Started: 7/30/2007 4:10:03PM   Analysis Adsorptive: N2
Completed: 7/31/2007 1:04:49AM   Analysis Bath Temp.: 77.300 K
Report Time: 8/27/2007 2:30:39PM   Thermal Correction: No
Sample Mass: 0.2235 g   Ambient Free Space: 16.3383 cm³ Measured
Analysis Free Space: 49.4051 cm³   Equilibration Interval: 5 s
Ambient Temperature: 295.15 K   Automatic Degas: Yes

Comments: Silica Alumina STD

Summary Report

Surface Area
Single point surface area at p/p° = 0.298614971: 205.4358 m²/g

BET Surface Area: 209.9056 m²/g

BJH Adsorption cumulative surface area of pores
between 17.000 Å and 3000.000 Å width: 247.929 m²/g

BJH Desorption cumulative surface area of pores
between 17.000 Å and 3000.000 Å width: 323.1344 m²/g

Pore Volume
Single point adsorption total pore volume of pores
less than 4530.409 Å width at p/p° = 0.995803560: 0.600783 cm³/g

BJH Adsorption cumulative volume of pores
between 17.000 Å and 3000.000 Å width: 0.650128 cm³/g

BJH Desorption cumulative volume of pores
between 17.000 Å and 3000.000 Å width: 0.603143 cm³/g

Pore Size
Adsorption average pore width (4V/A by BET): 114.4863 Å

BJH Adsorption average pore width (4V/A): 104.889 Å

BJH Desorption average pore width (4V/A): 74.662 Å

BJH Adsorption Cumulative Pore Volume

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2050   V X.XX   Unit 1   Serial #: 102   Page 9

Sample: Silica alumina reference material
Operator: JCH
Submitter: Unit 102
File: Y:\...\HANKH\SIAL_01.SMP

Started: 7/30/2007 4:10:03PM
Completed: 7/31/2007 1:04:49AM
Report Time: 8/27/2007 2:30:39PM
Sample Mass: 0.2235 g
Analysis Free Space: 49.4051 cm³
Ambient Temperature: 295.15 K

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.300 K
Thermal Correction: No
Ambient Free Space: 16.3383 cm³ Measured
Equilibration Interval: 5 s
Automatic Degas: Yes

Comments: Silica Alumina STD

BJH Adsorption Cumulative Pore Volume

User-Defined : Standard

Isotherm Pressure Composition

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2050 V X.XX   Unit 1   Serial #: 102   Page 6

Sample: LaNi5 First Run (2.5g)
Operator: JE
Submitter: JK
File: Y:\24ZEH5~2\DATA\LANI5F.SMP

Started: 7/10/2007 2:20:01PM	Analysis Adsorptive: H2
Completed: 7/11/2007 6:56:42PM	Analysis Bath Temp.: 298.000 K
Report Time: 8/27/2007 2:28:46PM	Thermal Correction: No
Sample Mass: 2.5013 g	Ambient Free Space: 19.1798 cm³ Measured
Analysis Free Space: 19.2126 cm³	Equilibration Interval: 5 s
Ambient Temperature: 295.15 K	Automatic Degas: No

Isotherm Pressure Composition

Isotherm Linear Absolute Plot

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2050 V X.XX   Unit 1   Serial #: 102   Page 3

Sample: Li - X N2
Operator: JCH
Submitter: Unit 102
File: Y:\...\DATA\13X_N201.SMP

Started: 8/7/2007 8:18:38AM   Analysis Adsorptive: N2
Completed: 8/7/2007 3:39:53PM   Analysis Bath Temp.: 298.000 K
Report Time: 8/27/2007 2:16:43PM   Thermal Correction: No
Sample Mass: 0.5991 g   Ambient Free Space: 19.1665 cm³ Measured
Analysis Free Space: 19.3325 cm³   Equilibration Interval: 10 s
Ambient Temperature: 295.15 K   Automatic Degas: No

Comments: Water bath used

Isotherm Linear Absolute Plot

13X_N201 : Li - X N2
13X_O201 : Li - X O2

Isotherm Linear Absolute Plot

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Demo 2050 V1.00 E   Unit 1   Serial #: 102   Page 5

Sample: Silica alumina reference material
Operator: JCH
Submitter: Unit 102
File: Y:\...\HANKH\SIAL_01.SMP

Started: 7/30/2007 4:10:03PM	Analysis Adsorptive: N2
Completed: 7/31/2007 1:04:49AM	Analysis Bath Temp.: 77.300 K
Report Time: 8/27/2007 2:30:39PM	Thermal Correction: No
Sample Mass: 0.2235 g	Ambient Free Space: 16.3383 cm³ Measured
Analysis Free Space: 49.4051 cm³	Equilibration Interval: 5 s
Ambient Temperature: 295.15 K	Automatic Degas: Yes

Comments: Silica Alumina STD

Isotherm Linear Absolute Plot

—+— Silica alumina reference material - Adsorption
—⊖— Silica alumina reference material - Desorption
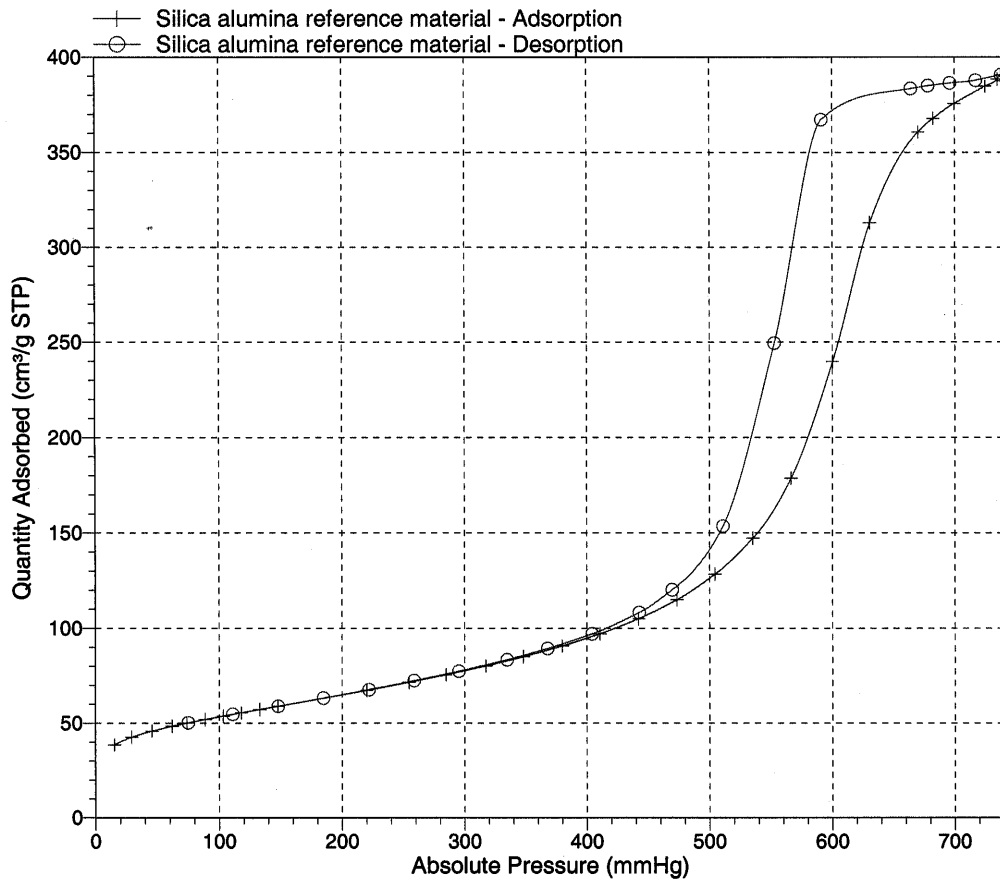
8. OPTIONS MENU

The commands on the Options menu enable you to configure the system to your laboratory's requirements and establish sample defaults.

Description
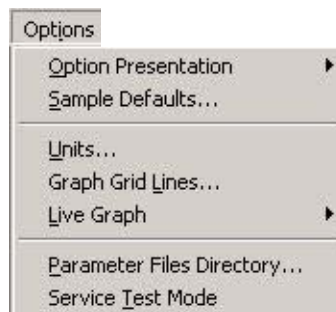
Option Presentation   Allows you to display the sample file dialog in Advanced, Basic, or Restricted mode. Page **8-2**.

Sample Defaults   Enables you to specify default values for sample and parameter files. Page **8-4**.

Units   Enables you to choose the way in which data are presented on the screen and in printed reports. Page **8-10**.

Graph Grid Lines   Enables you to choose the type(s) of grid lines to show on your report. Page **8-10**.

Live Graph   Enables you to choose the variable for the X-axis during data collection. Page **8-11**.

Parameter Files Directory   Allows you to specify a location for the parameter files used in the Basic sample file editor. Page **8-11**.

Service Test Mode   Enables you to perform certain troubleshooting procedures. This option is available only under the direction of a Micromeritics service representative. Page **8-12**.

Option Presentation

The sample editing dialogs for the ASAP 2050 analysis program may be presented in three modes: Restricted, Basic, and Advanced. Each format displays sample information dialogs differently.

- **Advanced:** displays all parts of the sample information file in a tabbed dialog as in an index card file. You just click on the tab of the desired parameter. This format also allows you to switch to the Basic mode.

- **Basic**: displays all parts of the sample file as a single dialog. in which you select from pre-defined parameter files. This format also allows you to switch to the Advanced mode if editing of parameters is desired.

- **Restricted:** displays in the same manner as the Basic format. Certain menu options, how-ever, become disabled and you cannot switch to the Advanced mode. You also must enter a password to access and exit this format.

Advanced

The Advanced format presents all parts of the sample information file in a tabbed dialog. Each tab opens its associated dialog. For example, if you are using the Advanced format and you open or create a sample information file, the following dialog is displayed.
.

The Advanced format can be used to edit sample parameters and customize files.

Basic

The Basic format presents the sample information file and its parameter files as a single dialog. You also have access to all menu options. For example, if you are using the Basic format and open or create a sample information file, the dialog is displayed in this manner.
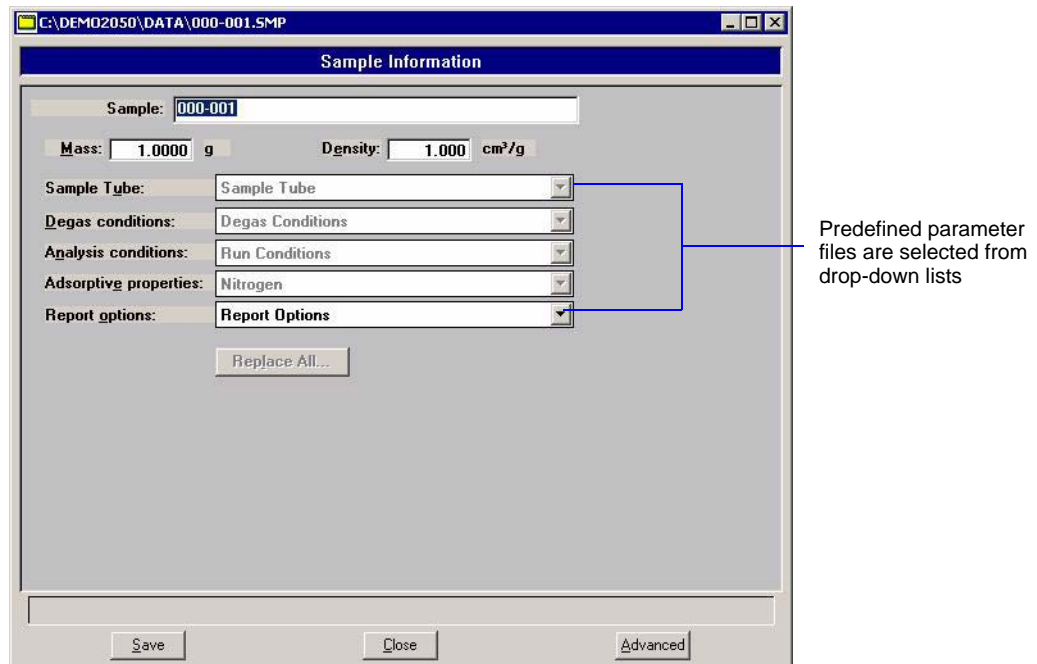
The Basic format is used to quickly create sample information files using previously defined parameter files. You also can switch easily to the Advanced format to view or edit details of parameter files.

Restricted

The Restricted format is identical to the Basic format, except that certain options are disabled. You also cannot switch to the Advanced format. This format is password-protected and typically is used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.

When you select Restricted, a dialog requesting a password is displayed.

You can enter any password (up to 31 characters) to enable the Restricted mode. You must enter the same password to exit the Restricted mode. For example, if you enter "password" to

enable the Restricted format, then you must enter "password" to exit. If you forget the password, open the application INI file and navigate to the Private section. The current password is shown immediately following "OptionPresentationPassword." Make a note of the password, exit the INI file, and enter the password where requested. Deleting the password from the INI file will not disable the Restricted mode; you must enter the password using the Password dialog to exit the Restricted format.

Sample Defaults

Sample defaults are the values you see in the sample information editors when you create a new sample file. This option allows you to specify the default values. This feature makes it easy for you to apply the same conditions to many samples.
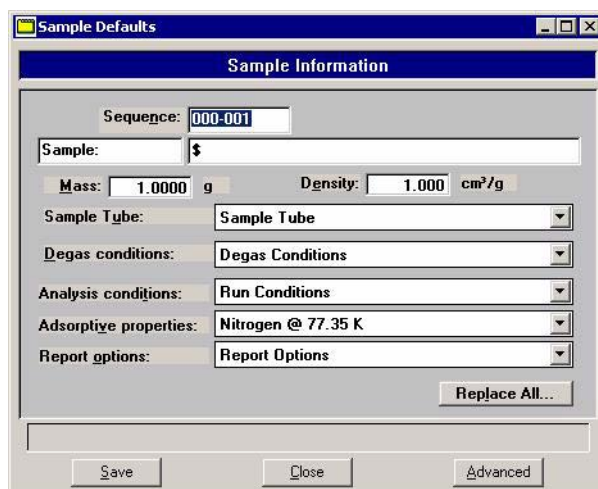
For efficiency, it is best to specify defaults for materials you most commonly analyze. You can always edit the values in the sample file when it is created. Sample defaults can be specified using the Basic or Advanced format.

The analysis program contains one complete default sample information file. When you select **Open, Sample information** from the Main Menu, a new file with these default values is generated. Default files for degas conditions, analysis conditions, adsorptive properties, and report options also are included. **Appendix F, Default Files and System Files** lists details on system default files.

Sample defaults can be specified using the Advanced or Basic format. The Advanced or Basic format is chosen by selecting **Options > Option presentation** from the main menu.

Basic Format

When you select Sample Defaults using the Basic format, the Basic Sample Defaults dialog is displayed.

Sequence   Allows you to specify a default sequence for the sample file
                      name. The number you specify is incrementally sequenced
                      each time you create a sample file. It is the number that
                      appears in the **File name** field when you select **File > Open
                      > Sample information**.

                      Use numbers, letters, or other printable characters, such as
                      dashes. At least three numbers must be included.

                      • Use up to eight characters.
                      • Do not use characters such as * or ?.

Sample   Allows you to enter an additional identification that provides
                      more information than the sample file name itself.

                      The field on the left allows you to specify a name for the
                      identification label. For example, you may prefer to use Test
                      or Material.

                      The field on the right allows you to specify a format for the
                      sample identification.

                      Use numbers, letters, or other printable characters, such as
                      dashes.

                      • Use up to 20 characters for the prompt.

                      • Use up to 43 characters (including the $ symbol).

                      • Include the automatically generated file name as part of
                        the identification by using the $ symbol where you want
                        the sequence number to appear.

                      For example, if the sequence number is 000-001, enter
                      the identification as follows:

                             Lab #25 - $

                      The resulting sample identification for the first sample
                      information file would be:

   **Lab #25 - 000-001**

                      for the second file:

                             Lab #25 - 000-002, and so on.

Mass   Enter a value that represents the approximate mass of your
                                 most commonly analyzed material. You can enter a more
                                 accurate value in the sample file when the analysis is
                                 completed.

Density   The sample density is used when using a calculated free
                                 space. The default value is appropriate unless you typically
                                 use a calculated free space. This field can always be edited
                                 in the sample file.

Sample Tube   Each parameter has a drop-down list containing files
Degas Conditions   included with the software, as well as any you may have
Analysis Conditions   created and saved to the Parameter Files Directory.
Adsorptive Properties
Report Options
                                 Choose from the list provided for each parameter the file you
                                 wish to use as the default.

Replace All   Use this push button to replace the current default values
                                 with those from an existing sample file.

Save   Saves the current definition as the defaults

Close   Closes the dialog.

Advanced   Switches the sample editor to the Advanced format.

Advanced Format

When you select Sample Defaults while using the Advanced format, the Advanced Sample Defaults dialog is displayed.

> **The defaults you specify for the parameters in the Advanced format display as the defaults for newly created standalone parameter files**

Sequence   Allows you to specify a default sequence for the sample file name. The number you specify is incrementally sequenced each time you create a sample file. It is the number that appears in the **File name** field when you select **File > Open > Sample information**.

- Use numbers, letters, or other printable characters, such as dashes. At least three numbers must be included.

- Use up to eight characters.

- Do not use characters such as * or ?.

Sample   Allows you to enter an additional identification that provides
                               more information than the sample file name itself.

                               The field on the left allows you to specify a name for the
                               identification label. For example, you may prefer to use *Test*
                               or *Material*.

                               The field on the right allows you to specify a format for the
                               sample identification.

                               • Use numbers, letters, or other printable characters, such
                                 as dashes.

                               • Use up to 20 characters for the prompt.

                               • Use up to 43 characters (including the $ symbol).

                               • Include the automatically generated file name as part of
                                 the identification by using the $ symbol where you want
                                 the sequence number to appear.

                               For example, if the sequence number is 000-001, enter
                               the identification as follows:

                                     Lab #25 - $

                               The resulting sample identification for the first sample
                               information file would be:

                                     Lab #25 - 000-001

                               for the second file:

                                     Lab #25 - 000-002, and so on.

Operator   These fields contain the name (or other identification) of the
Submitter   operator and submitter.

                          The fields on the left can be edited to display a different label
                          if desired.

                          The fields on the right allow you to specify default names or
                          titles.

                          •   Include the automatically generated file number as part
                              of the identification by using the symbol $ where you
                              want the sequence number to appear. Refer to the exam-
                              ple in the previous section.

                          •   Use up to 20 characters for the prompt.

                          •   Use up to 43 characters (including the $ symbol).

                          •   Omit either (or both) item entirely from the sample infor-
                              mation file by selecting **Omit**.

Mass   Enter a value that represents the approximate mass of your
                          most commonly analyzed material. You can enter a more
                          accurate value in the sample file when the analysis is
                          completed.

Type of Data   Choose whether you wish to enter data or have it collected
                          automatically by the system.

Comments   Use this window to enter sample characteristics, analysis
                          conditions, etc. Anything you type in this window is printed
                          in the report header.

Replace All   Use this push button to replace the current default values with
                          those from an existing sample file.

Save   Saves the current definition as the defaults

Close   Closes the dialog.

Basic   Switches the sample editor to the Basic format.

Units

This menu command displays the Unit Selections dialog which allows you to choose the manner in which to display data on the dialogs and reports.
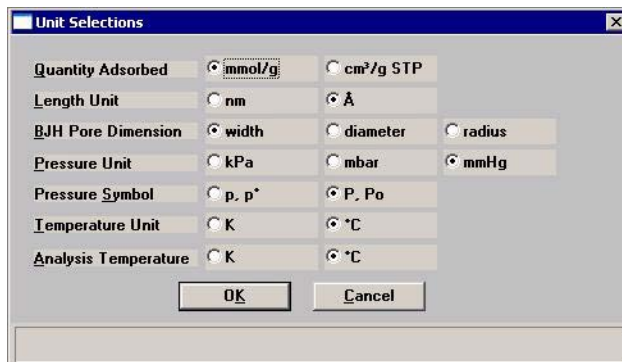
Graph Grid Lines

Graph Grid Lines enables you to choose the type(s) of grid lines to show on your reports; the Graph Grid Lines dialog is displayed.
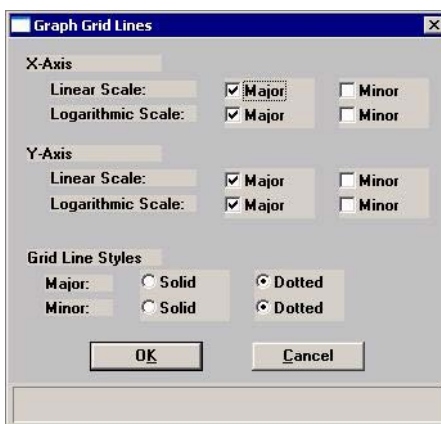
X-Axis **Y-Axis**	Enables you to choose **Major** and/or **Minor** lines to display in printed reports for the Logarithmic and Linear scales.
	If you deselect these items (remove the check marks), your report will not display grid lines.
Grid Line Style	Allows you to choose the type of grid line to display if grid lines are being shown.

Live Graph
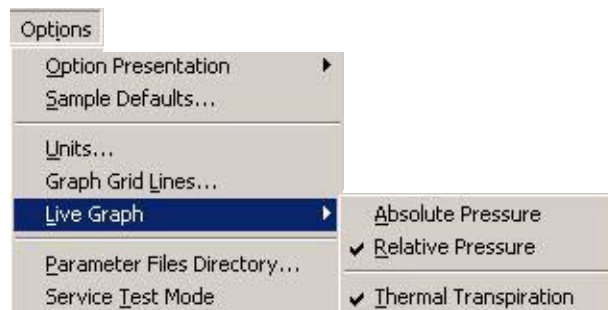
This option enables you to choose the variable for the x-axis during data collection. You can also choose to correct for thermal transpiration (refer to page **5-31** for additional information on thermal transpiration).

Parameter Files Directory

This option allows you to specify a location for the predefined Analysis conditions, Degas conditions, Adsorptive properties, and Report options files displayed in the drop-down lists on the Basic Sample Information dialog. The current directory is displayed above the drives/ directory window.
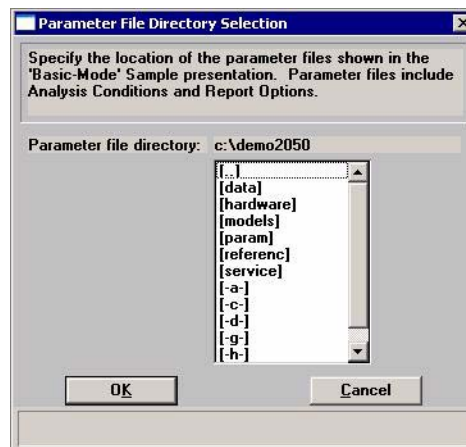
The default directory is params and includes several parameter files supplied with the analysis program. If you specify a different directory, these files will not be included in the drop-down lists unless you copy (or move) them to the new directory.

If you wish to continue using the params directory for parameter files, be sure to save any files you create to this directory(s).

Service Test Mode

Various service tests are included in the ASAP 2050 operating program. These tests can be performed only with the assistance of a trained Micromeritics service representative. When you select **Service Test Mode** from the Options menu, a dialog prompting you to enter a password is displayed. This password is coded to change on a regular basis and is known only by your service representative. You will not be able to perform these tests without his guidance.

9. TROUBLESHOOTING AND MAINTENANCE

The ASAP 2050 system has been designed to provide efficient and continuous service. However, to get the best results over the longest period of time, certain maintenance procedures must be followed. This chapter includes troubleshooting information, recommended preventive maintenance procedures, and routine maintenance procedures.

Troubleshooting

Most operational problems are caused by the following:

- Leaks (commonly around the sample tube O-ring at the analysis port)
- Sample weighing errors
- Use of too much analysis bath fluid in the Dewar at the start of an analysis
- Entry of incorrect system volume for analysis

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

What Happened	Why	What To Do
Analysis Dewar cannot be raised (or lowered).	Elevator that moves Dewar stuck in up position, down position, or somewhere in between.	Check for possible obstruction to elevator movement.
Vacuum pump system makes loud noise continuously.	Sample tube connector nut is loose.	Turn the connector nut clockwise to tighten.

What Happened	Why	What To Do
Vacuum pump system makes loud noise continuously. *(continued)*	Sample tube fitting is loose.	Tighten the fitting securely with a wrench.
	Sample tube O-ring is worn or cracked.	Replace the sample tube O-ring. Refer to **"Replacing a Sample Tube O-ring" on page 9-8**.
	No sample tube is loaded on a selected port.	Make sure the port valve is closed. Install a plug or empty sample tube on the port.
	A gas inlet valve is open while the vacuum valve is open.	Enable manual control, then use the instrument schematic to close the gas inlet valve. Refer to **"Enable Manual Control" on page 6-24**.
Vacuum gauge shows reading above 20 μmHg, even after extended pumping through unrestricted valve with analysis or degas ports closed.	Vacuum pump oil is low, causing ineffective evacuation.	Add or change vacuum pump oil. Be sure to add oil to proper level according to indicator window on the pump. Refer to **"Inspecting and Changing Vacuum Pump Fluid" on page 9-9**.
	No power to the vacuum pump.	Check the pump power plug, power switch, and line circuit breaker.
	Filter in the port being used is dirty.	Replace the port filter. Refer to **"Replacing the Port Filters" on page 9-5**.
	Alumina in the oil vapor trap is holding moisture because it was not sufficiently dried before being added to the alumina trap.	Replace or dry the alumina. Refer to **"Replacing the Alumina in the Oil Vapor Traps" on page 9-15**.

What Happened	Why	What To Do
Analysis valves cannot be operated.	Circuit was opened by the circuit breaker.	Push in the breaker button, located on the side of the instrument near the power entrance. If it will not stay in, contact appropriate service personnel. Breaker button
	Cable from the computer to the analyzer is loose.	Make sure the cable is connected properly to the computer and the analyzer.

Preventive Maintenance

The table below lists the preventive maintenance procedures you should complete to keep your analyzer operating at peak performance. Instructions for each procedure follow the table. Micromeritics also recommends that you have preventive maintenance procedures and calibration performed by one of our service representatives every 12 months.

Maintenance Required	Frequency
Check and clean analysis port Dewars, **page 9-5**	Weekly
Replace port filters, **page 9-5**	Every 30 days
Clean the outside of the analyzer, **page 9-8**	As required
Replace sample tube O-ring, **page 9-8**	As required or every 3 months
Inspect and change vacuum pump fluid, **page 9-9**	As required or every 3 months
Replace alumina in oil vapor traps, **page 9-15**	As required or every 3 months
Clean cold trap glass tube, **page -21**	As required or every 3 months
Lubricate elevator screw, **page 9-24**	As required or every 6 months
Check outgassing rate and test for leaks, **Appendix D**	As required or every 6 months
Replace vacuum pump exhaust filter, **page 9-25**	As required or every 12 months
Zero transducer pressure gauge, **page 9-27**	As required or every 12 months
Calibrate transducer pressure scale, **page 9-28**	As required; should be performed by qualified service personnel
Calibrate manifold temperature sensor, **page 9-29**	As required; should be performed by qualified service personnel
Calibrate system volume, **page 6-19**	As required; should be performed by qualified service personnel

Checking and Cleaning the Analysis Port Dewars

When handling Dewars, be sure to observe the Dewar precautions described on page 3-24.

Ice and suspended frost particles may accumulate in the bottom of an analysis port Dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tube and the bottom of the Dewar, causing the Dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tube. This causes the sample temperature to be slightly higher which, in turn, can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure.

To ensure problems do not develop due to ice accumulation, check the Dewar after each use. Clean Dewars on a weekly basis as follows:

1. Remove the Dewar and pour the liquid nitrogen from the Dewar into an appropriate cryogenic container.

Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.

2. Rinse the Dewar with warm water to melt any ice accumulation which may remain in the Dewar, then dry thoroughly.

Replacing the Port Filters

A porous metal filter is located in each analysis and degas port. If a degas port is not plugged with either a sample tube or a port plug then the filter will absorb atmospheric gases, resulting in extended degassing time for samples on that port.

Using a contaminated filter on an analysis port may extend the time required to achieve a vacuum at that port. More importantly, the contaminant may adsorb or desorb during analysis, affecting the analysis results. A contaminated filter on an analysis port may cause a leak test to fail (if the contaminant outgasses) or cause a free-space reading to be much lower than normal.

Analysis Ports

> ✏️ **Before removing and installing a port filter, ensure that the port valve is closed. Observe the analysis system schematic to verify valve status.**

1. Using a wrench, remove the analysis port fitting from the analyzer.

Analysis port fitting →

2.   Remove the filter and O-ring.
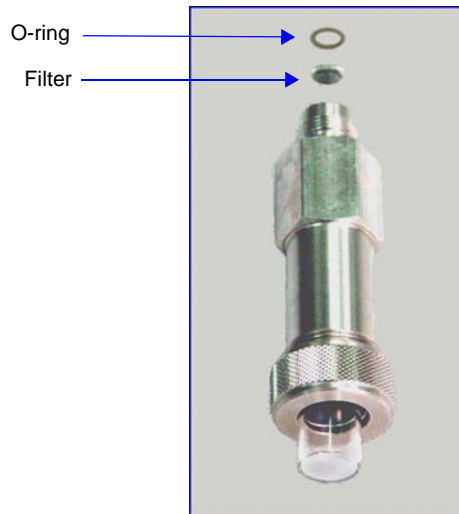
O-ring →
Filter →

3. Using latex gloves, replace with a new filter and O-ring.

> ✏️ **To avoid analysis problems, the new filter and O-ring must be clean. Do not touch them with bare hands.**

4. Reinstall the analysis port fitting; securely tighten with a wrench to prevent leaks during evacuation.

Degas Ports

Before removing and installing a port filter, ensure that the port valve is closed. Observe the degas system schematic to verify valve status.

1. Using a wrench, remove the degas port fitting.

Degas port fitting

2. Remove the O-ring and filter.
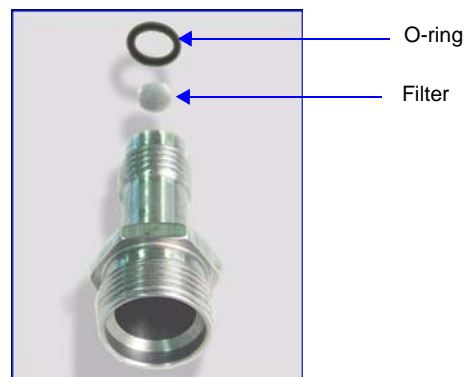
O-ring

Filter

3. Replace the filter and the O-ring.

To avoid analysis problems, the new filter and O-ring must be clean. Do not touch them with bare hands.

4. Reinstall the degas port fitting; securely tighten with a wrench to prevent leaks during evacuation.

Cleaning the Analyzer

The ASAP 2050 should be cleaned as required.

Clean the outside casing of the analyzer (including the shields) with a clean cloth dampened with isopropyl alcohol (IPA), a mild detergent in water, or a 3% hydrogen peroxide solution. It is not necessary to remove any switches or screws to clean the outside casing.

> **Use a mild detergent in water or isopropyl alcohol to clean the shields. Avoid using acetone because it will damage the surface of the shields.**

Replacing a Sample Tube O-ring

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced.

1. Remove the O-ring from the top of the sample tube.

O-ring

2. Slide a new O-ring over the sample tube.

Inspecting and Changing Vacuum Pump Fluid

The fluid in the vacuum pump should be changed every three months or when the efficiency of the vacuum pump declines (requiring increased time to reach vacuum levels). The fluid can first be inspected to determine if a change is necessary.

Use oil supplied by Micromeritics, or refer to the vacuum pump manual for other acceptable oils.

In order to use oxygen with the ASAP 2050, your 2050 analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin® (or a suitable equivalent) pump fluid. Failure to use the proper vacuum system could result in hazardous conditions including fire and personal injury.

Inspecting Fluid

Make sure the pump is running when inspecting fluid.

1. Choose one of the following to view the vacuum pump sight window.

   a. Remove the four screws located along the sides of the vacuum pump panel, then remove the panel.

   b. Remove (or fold over to the right) the protective rubber mat on the lower front panel, then lift off the metal cover; this exposes the vacuum pump sight window.

2. View the vacuum pump fluid through the sight window. Fluid in good condition is clean, clear or light in color, and transparent. If the color of the fluid is darkened, the fluid should be changed.

If the fluid is just above the lower level indicator mark, fluid should be added.

Oil-level window

Level indicator mark
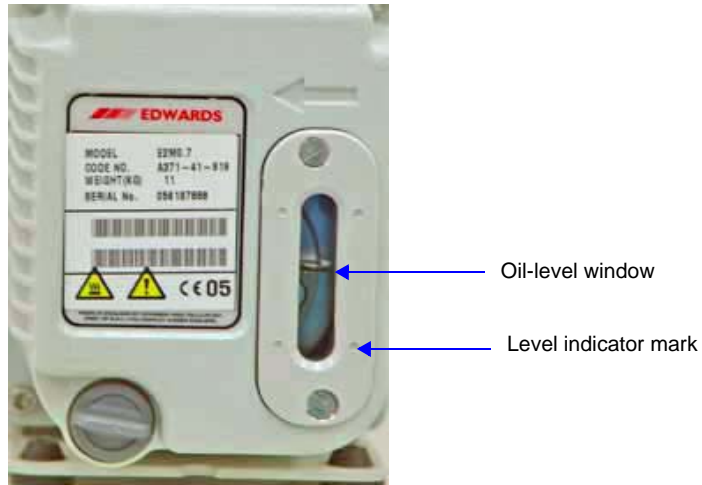
Adding Fluid

1. If the front panel has not been removed, remove the four screws located along the sides of the vacuum pump panel, then remove the panel.

2. Remove the oil-fill plug from the vacuum pump.

Oil-fill port

> ⚠ **Adding fluid above the midway position on the fluid level mark may cause fluid to splash into the vacuum hoses and leak from the vacuum pump.**

3. Add fresh fluid to the oil-fill port until the level is midway between the indicator marks in the pump sight window.

Level indicator marks
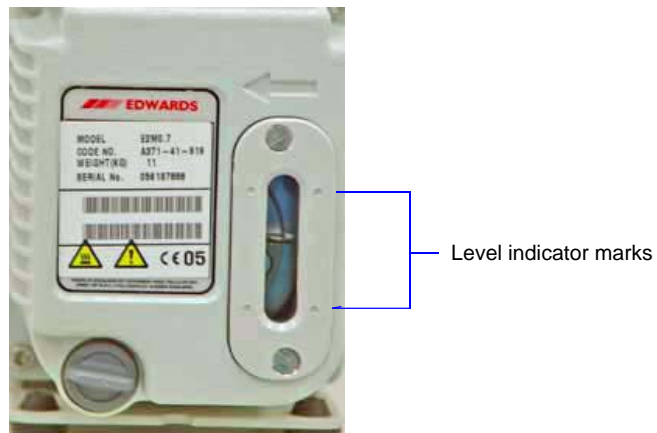
4. Check the washer or O-ring used at the oil-fill port; replace if necessary.

5. Replace the oil-fill plug.

Changing Fluid

> **Always drain the vacuum pump while the pump is warm and disconnected from the power source.**

1. Place the analyzer ON/OFF switch in the OFF position.

2. If the vacuum pump panel is not already removed, remove the four screws located along the sides of the panel, then remove the panel.

3. Disconnect the vacuum pump from the power source.

4. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.

Disconnect hose

Clamp wing-nut

Oil-vapor trap
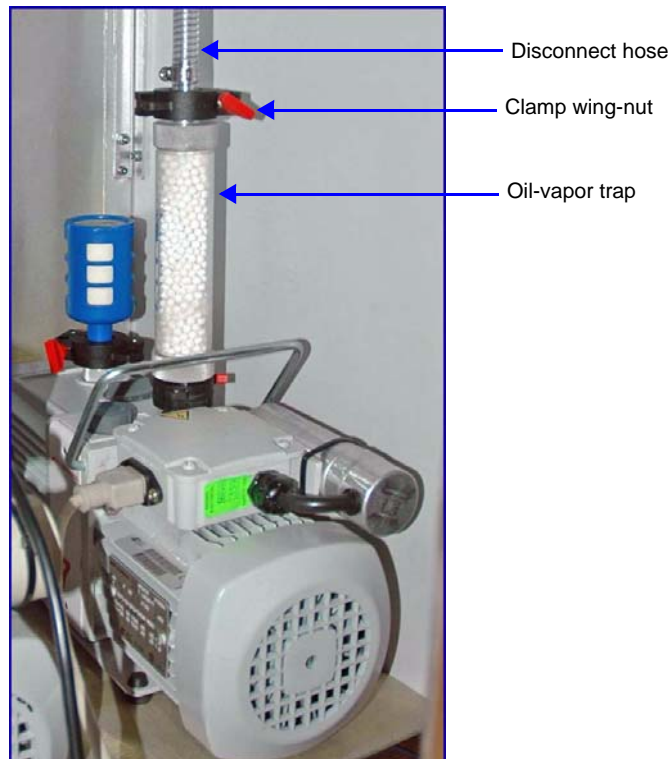
5. Grasp the handle on top of the vacuum pump and lift it out of the analyzer; place the vacuum pump on a work table.

6. Drain the used oil:

    a. Place a waste container under the drain spout.

    b. Remove the plug from the drain spout.

Drain plug

    c. Allow the oil to drain into the waste container.

    d. Replace the drain plug.

7. Remove the oil-fill plug.

Oil-fill port

> **Adding fluid above the midway position on the fluid level indicator marks may cause fluid to splash into the vacuum hoses and leak from the vacuum pump.**

8. Add fresh fluid to the oil-fill port until the level until the level is midway between the two indicator marks in the pump sight window.

9. Check the washer or O-ring used at the oil-fill port; replace if necessary.

10. Replace the oil-fill plug.

11. If the fluid was badly contaminated (dark brown color), flush the pump with fresh fluid as follows:

    a. Cover the top of the oil vapor trap.

    b. Plug the pump power cord into a power source and run for about 2 minutes.

    c. Unplug the power cord.

    d. Remove the plug from the drain spout.

    e. Allow the oil to drain into the waste container.

    f. Reinstall the drain plug.

    g. Add fresh fluid to the oil-fill port until the level is midway between the two indicator marks.

    h. Reinstall the oil-fill plug.

    i. Repeat steps **a** through **h** if necessary.

12. Check the alumina in the oil vapor trap and replace if necessary (refer to **"Replacing the Alumina in the Oil Vapor Traps" on page 9-15**).

13. Place the vacuum pump back onto the drip tray in the analyzer.

14. Reconnect the vacuum pump hose.

15. Reconnect the power cord to the power source.

16. Reinstall the front analyzer panel.

17. The pump must run a few hours (or overnight) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

Replacing the Alumina in the Oil Vapor Traps

The activated alumina in the oil vapor traps becomes saturated during use. The alumina should be replaced if either of the following conditions exist:

• The alumina has been used for a three-month period.
• Most of the alumina pellets are no longer white.

Oil vapor trap
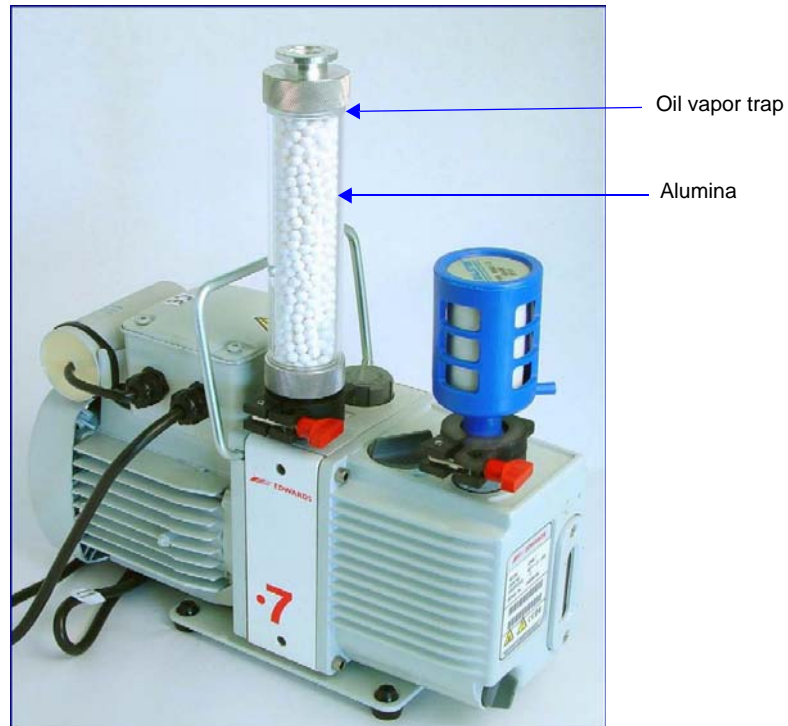
Alumina

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire or an explosion.

1. Prepare fresh alumina as follows:

   a. Heat an oven between 250 °C and 350 °C.

   b. Pour about 180 grams of alumina into a glass or metal container for each trap (approximately 250 mL if a graduated beaker is used). Place the container in an oven.

   c. Bake the alumina for two hours; turn off the oven.

   d. Allow the alumina to cool in the oven before pouring it into the traps.

2. Select **Unit [n] > Enable Manual Control**. If the analysis schematic is not displayed, select **Unit [n] Show Instrument Schematic** to display the schematic in manual mode.
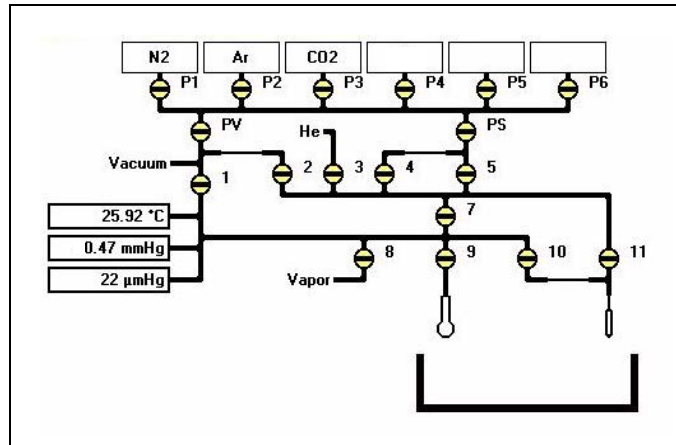
3. Open the analysis vacuum valve: Valve **1**. Make sure all other valves are closed.

4. Select **Unit [n] > Degas > Enable Manual Control**. If the degas schematic is not displayed, select **Unit [n] > Degas > Show Instrument Schematic** to display the schematic in manual mode.
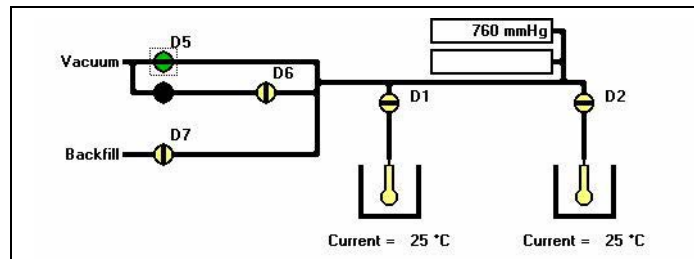
5. Open the degas vacuum valve: **D5**.

6. When the pressure stabilizes around atmospheric pressure, close all valves.

7. If the vacuum pump panel is not already removed, remove the four screws located along the sides of the panel, then remove the panel.

8. Unplug the pump power cord.

9. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.

Disconnect hose

Clamp wing-nut
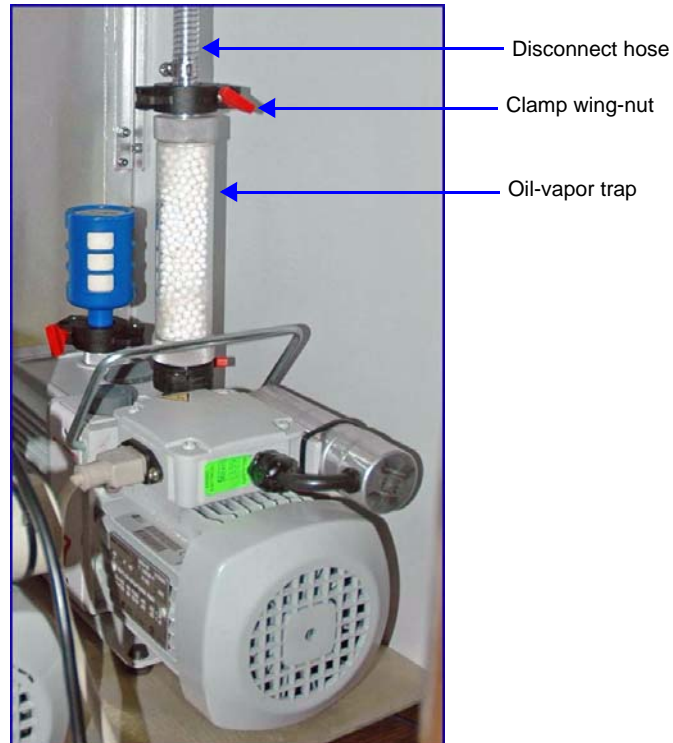
Oil-vapor trap

10. Remove the pump from the analyzer for easier access, if you wish.

11. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Swing the clamp open and remove it.

Oil vapor trap

End fitting

Wing nut
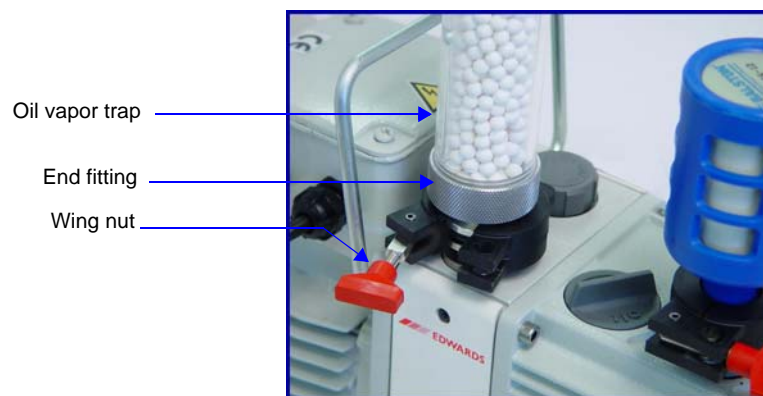
12. Remove one end fitting from the trap body.

13. Remove the used alumina from the trap body and dispose of it in an appropriate manner.

14. If the trap body interior appears oily or dirty, wash it with isopropyl alcohol or ethyl alcohol and dry it thoroughly.

> **Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.**

> **Ensure adequate ventilation when using solvents for cleaning purposes.**

15. Pour the new activated alumina pellets into the trap until they are level with the top of the trap body.

End fitting   Centering ring

                                                              O-ring

16. Insert the O-ring into the end fitting, screw the end fitting back onto the trap, then tighten securely by hand.

17. Make sure the centering ring is in place on the intake port.

18. Place the trap on the centering ring.

Oil vapor trap ———————→

Centering ring ————————→
Intake port —————————→
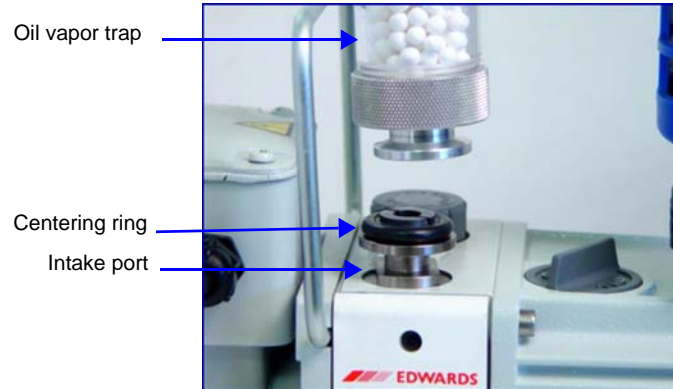
19. Open the clamp and place it around the flange of the intake port and the flange of the trap. Swing the clamp fastening screw toward the intake port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

20. If you removed the pump from the analyzer, place it onto the drip tray in the analyzer.

21. Reconnect the hose from the analyzer to the oil vapor trap using the clamp and wing nut.

Tighten clamp to attach trap to hose

22. Plug the pump power cord into the power source.

23. Reinstall the front panel on the analyzer.

Cleaning the Cold Trap Tubes

Oil vapor from the vacuum pumps accumulates in the cold traps. The tubes on the cold trap ports should be cleaned as follows:

1. Remove the vacuum pump panel.

2. Disconnect both vacuum pump power cords from their power sources.

3. Select **Unit [n] > Instrument Schematic**; then select **Unit [n] > Enable Manual Control**.
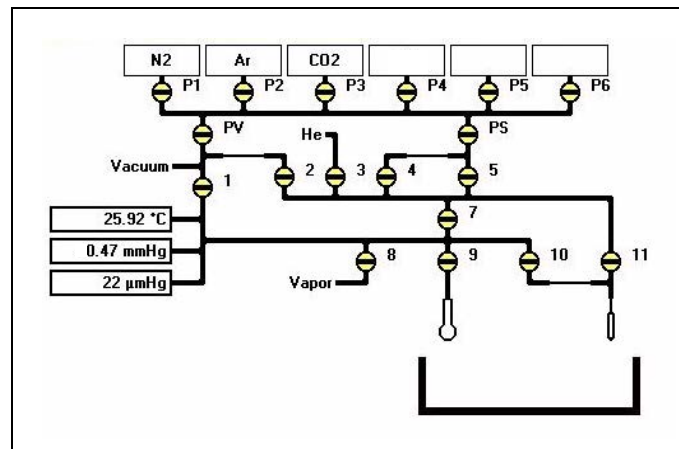
4. Open analysis valves in the following order: **P1**, **PS**, **5**, **7**, and **1**. Allow the pressure to stabilize until around atmospheric. Then, close the valves in the same order as they were opened. This step fills the analysis vacuum section before disconnecting.

5. Select **Unit [n] > Degas > Degas Schematic**; then select **Unit [n] > Degas > Enable Manual Control**.
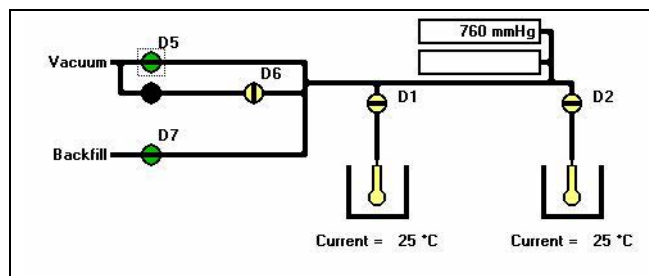
6. Open degas valves in the following order: **D5** and **D7**. Allow the pressure to stabilize until around atmospheric. Then, close valve **D7** and **D5**. This step fills the degas vacuum section before disconnecting.

7. While holding the tube, remove the connector nut from the degas cold trap port.

8. Carefully slide the glass tube down over the metal tube and remove the glass tube.

The connector nuts must be reinstalled on the port from which they were removed. They are not interchangeable.

9. Remove the connector nut from the other cold trap port and remove the metal tube.

10. Inspect the O-rings. If an O-ring is cracked or worn, it should be replaced.

11. Rinse the tubes with acetone; then dry them.

12. Slide the O-rings over the top of the tubes.

O-ring ——————

—————— O-ring

Connector nut ——————
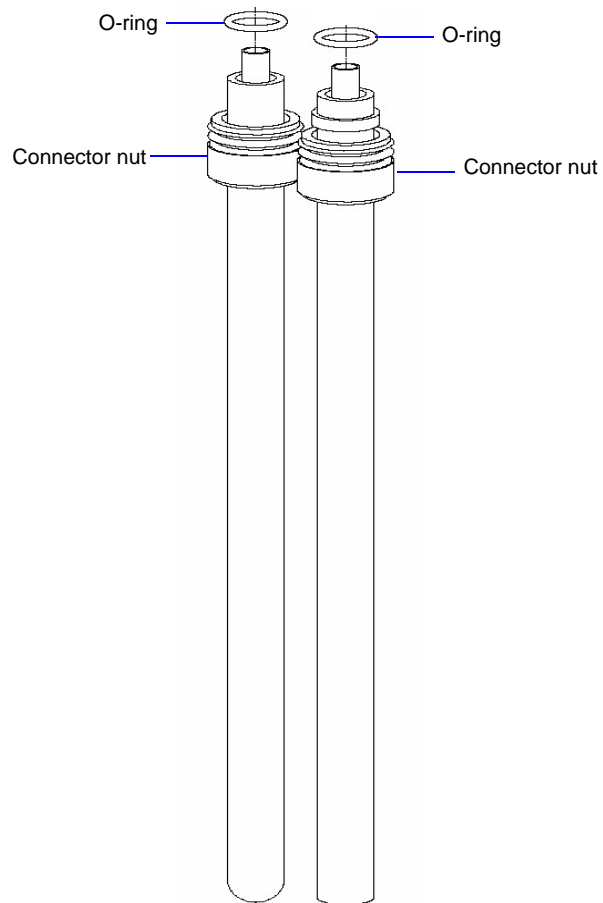
—————— Connector nut

13. Reinstall the cold trap tubes and tighten the connector nuts. Make sure you use the correct connector nut on each port.

14. Reconnect the vacuum pump power cords.

15. Replace the vacuum pump access panel.

Lubricating the Elevator Screw

1. Remove the analyzer's back panel by loosening, then removing the retaining screws.

2. Apply a light coat of lithium grease to the elevator screw.

Elevator screw
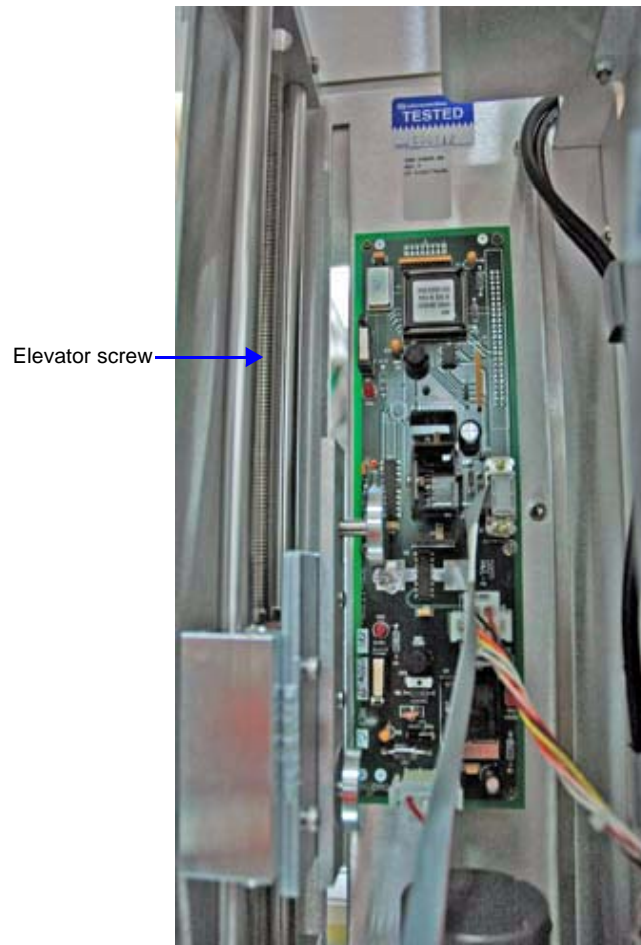
3. Replace the back panel.

Checking Outgassing Rate and Testing the Analyzer for Leaks

Refer to Appendix D **TESTING THE ANALYZER FOR LEAKS** for instructions on checking ASAP 2050 outgassing rates and testing for leaks.

Replacing the Vacuum Pump Exhaust Filter

The gases used by the ASAP 2050 are exhausted by vacuum pumps. An exhaust filter is installed on the exhaust port of each vacuum pump. The filter minimizes the release of oil vapor and should be replaced when it becomes so saturated with oil that it is ineffective.

> ⚠ **Exhaust filters are used on the vacuum pump to minimize the release of oil vapors. The gases used are diluted substantially upon being released from the vacuum pumps. However, it may be desirable in some locations to provide a fume hood for added protection from hazardous gases and vapors released into the work area.**

1. Remove the four screws located along the sides of the vacuum pump panel, then remove the panel.

2. If you wish to remove the vacuum pump from the analyzer, refer to steps 1 through 5 under **"Adding Fluid" on page 9-10**.

Vacuum pump exhaust filter

3. Loosen the wing nut of the clamp at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.
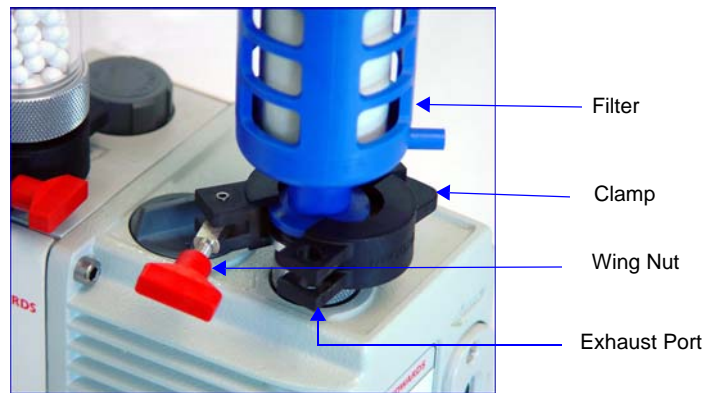
4. Discard the oil filter.

5. Retain the centering ring, clamp, and wing nut.

6. Remove the dust cover from the new filter.

7. Make sure the centering ring is on the exhaust port.

8. Place the new filter on the centering ring.

9. Open the clamp and place it around the flange of the exhaust port and the flange of the exhaust filter. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

10. If you removed the vacuum pump from the analyzer, place it back onto the drip tray in the analyzer, reconnect the hose, then reinstall the vacuum pump panel.

Calibrating the Pressure Gauge

When operating the system in manual mode *Calibration, pressure gauge* enables you to evacuate the manifold and zero the transducer.

The system automatically zeros the transducer before an analysis begins. Therefore, you do not need to use this feature unless you are operating the system in manual mode. Perform the following steps to calibrate the pressure gauge:

1. Select **Unit** > **Calibration** > **Pressure gauge**; the Calibrate Pressure Offset dialog is displayed.
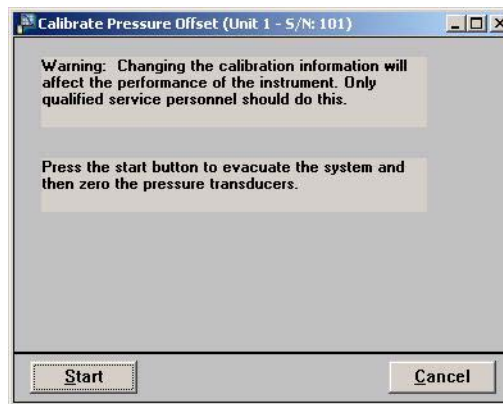
.
2. Click **Start** to evacuate the manifold and zero the pressure gauge(s).

3. When the calibration is complete, click **OK** to close the dialog.

> **You may cancel a calibration at any time by selecting Cancel. If you cancel a calibration, no new calibration data are saved.**

Calibrating the Pressure Scale

Calibration, Pressure scale allows the software to adjust the gain of the selected transducer to match a reference standard.

1. Attach a reference pressure gauge to the sample port of the analyzer.

2. With manual control enabled:

    a. Dose the manifold with helium or nitrogen to 5200 to 7500 mmHg.

    b. Open the sample port to expose the reference pressure gauge to the same pressure as the analyzer's transducer.

3. Select **Unit** > **Calibration** > **Pressure scale**; the Calibrate Pressure Scale dialog is displayed.
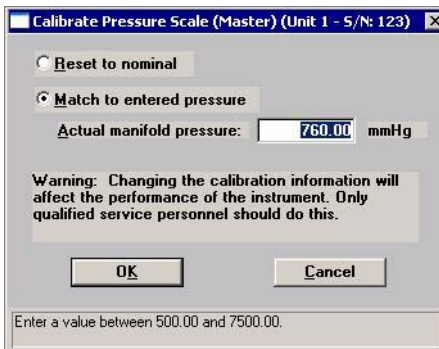
4. Select *Match to entered pressure*.

5. Enter the pressure reported by the reference pressure gauge, then click **OK**.

6. Close the sample port and evacuate the manifold.

7. Remove the reference pressure gauge.

Calibrating the Manifold Temperature Sensor

You should calibrate the manifold temperature on an annual basis. This allows you to correct for any changes in the manifold temperature sensor that occur with time.

You will need a small diameter probe, such as a 1/16-in. (1.5-mm) thermocouple to calibrate the manifold temperature sensor.

1. Insert the thermocouple probe into the opening located to the left of the analysis port.
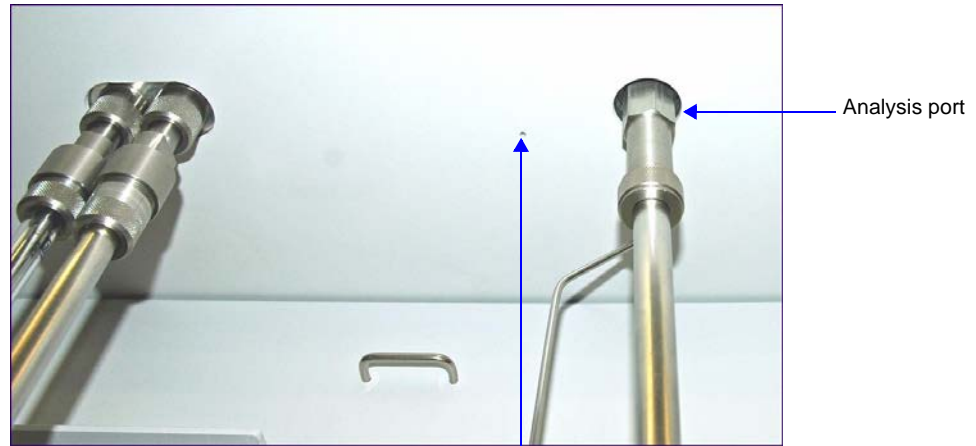
Analysis port

Manifold Probe Hole (to the left of the analysis port)

2. Move the thermocouple back and forth until you locate the small opening on the underside of the manifold. Push the thermocouple into the opening until the thermocouple stops.

3. Allow the temperature gauge reading to stabilize.

4. Select **Unit** > **Calibration > Temperature**; the Calibrate Manifold Temperature dialog is displayed.
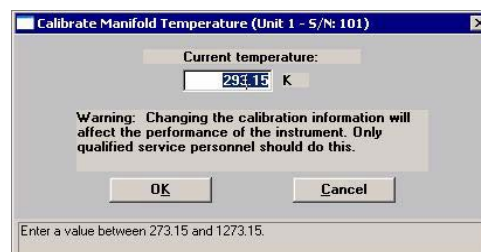
5. Enter the manifold temperature indicated on the reference temperature gauge. Click **OK** to store the new value.

6. Remove the thermocouple probe.

Routine Maintenance

The procedures in this section are performed as needed.

Changing Gases

Guidelines for Connecting Gases to the Analyzer

Use these guidelines when installing regulators and gas lines:

- Ensure that all gases needed are available and near the instrument. If the gas bottles are not located near the instrument, you will have to use gas line extenders, which may degrade gas quality and reduce pressure.

Retaining strap

- Use a retaining strap (or other appropriate tether) to secure the gas bottle.

- Carefully route the gas lines from the bottle to the analyzer, avoiding overlapping or entangling gas lines. This will ensure the correct gas pressure and make maintenance easier.

- Label both ends of the gas lines before connecting for ease in identification and maintenance.

- Ensure that all gas bottles are closed before connecting them to the analyzer.

The instructions below 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.

In order to use oxygen with the ASAP 2050, your 2050 analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin® (or a suitable equivalent) pump fluid. Failure to use the proper vacuum system could result in hazardous conditions including fire and personal injury.

Connecting a Gas Regulator to the Gas Supply

Dual-stage regulators must be used for all gas supplies.

These instructions apply to connecting inert gases only. When connecting hazardous gases, follow the safety procedures used by your lab.

1. Disconnect the gas bottle you are replacing:

    a. Turn the gas bottle Shut-Off valve clockwise to close.

b. Disconnect the gas line from the regulator and allow the regulator line to purge. You will hear gas vent.
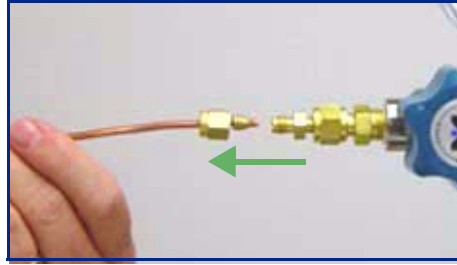
It is not necessary to disconnect the gas line from the instrument.

2. Turn the regulator Shut-Off valve counterclockwise to open (more gas will vent).

3. Turn the Pressure Control knob clockwise to open and vent any remaining gas; both gauges should then read approximately zero.

4. Close the Pressure Control knob.

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

6. Replace the protective cap, disconnect the retaining strap, and move the gas bottle to an appropriate location.

Depleted Bottle

Use caution when transporting gas bottles.

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

    Place the protective cap in a secure location. You will need it to recap the gas bottle when it is depleted and replaced.

New Bottle
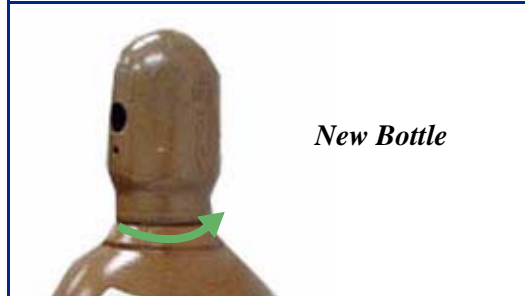
8. Attach the gas regulator to the connector on the gas bottle. Hand-tighten the nut. Use an appropriate wrench to tighten it an additional 3/4 turn.

> **Do not overtighten the fittings. Doing so could cause a leak.**

9. Check for leaks in the high-pressure side of the regulator and in the connector.
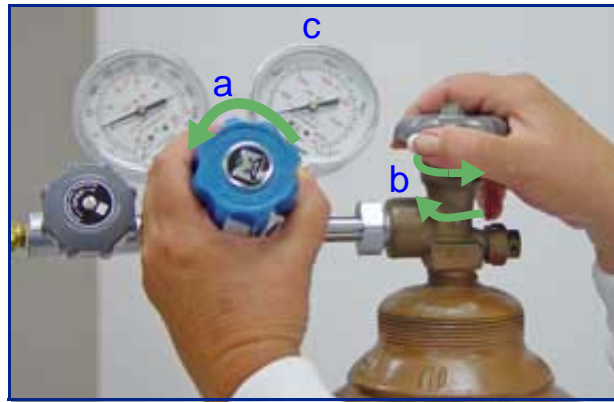
a. Turn the regulator control knob fully counterclockwise.

b. Slowly open the gas bottle valve, then quickly close it.

c. Observe the pressure on the high-pressure gauge for approximately one minute.

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

   If the pressure does not remain stable, remove the regulator and clean all contacts at the regulator connection. Then reinstall the regulator.

10. Perform the following steps to purge air from the lines:

a. Open the gas bottle valve to pressurize the regulator, then close the valve.

b. Adjust the Pressure Control knob to approximately 5 psi (34 kPa).
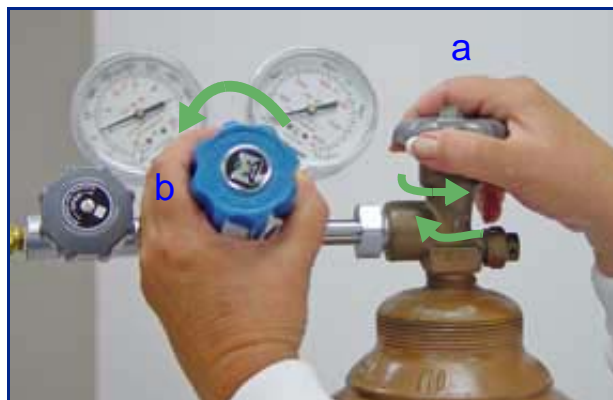
    c. Turn the regulator shut-off valve counterclockwise to open.

    d. Allow gas to flow until both gauges read approximately zero.

    e. Close the regulator shut-off valve to stop gas flow.
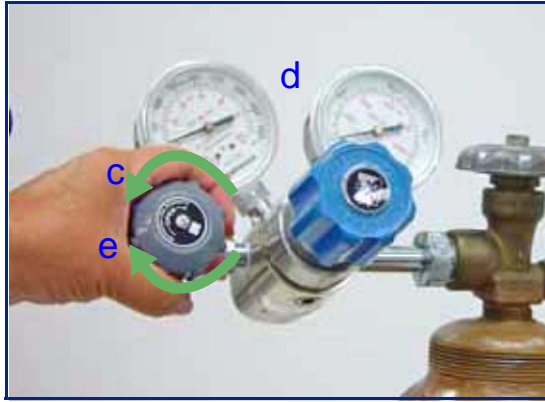
11. Reconnect the gas line to the regulator.
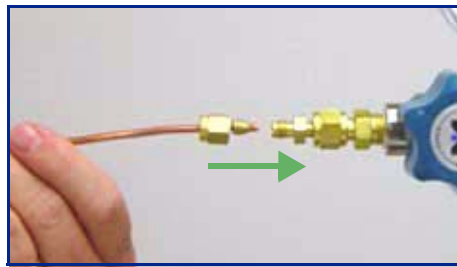
12. Use two 7/16-in (11-mm) wrenches to tighten the gas line connection; one to hold the fitting steady and one to tighten the connector nut.

13. Set the instrument pressure:

    a.   Turn the regulator control knob clockwise until the low-pressure gauge indicates 150 psig (1034 kPag)

    b.   Open the regulator shut-off valve.

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

    d.   Close the regulator shut-off valve.

    e.   Close the gas bottle valve.
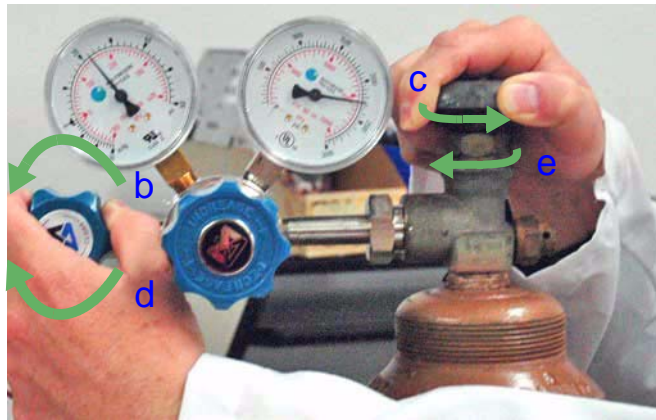
14. If you disconnected the gas line from the instrument, proceed to **Connecting the Gas Delivery Tubing to the Analyzer** in the next section.

15. If you changed to a different type of gas, you must specify the change in the analysis program; refer to **Specifying Gas Ports**.

Connecting the Gas Delivery Tubing to the Analyzer

The gas inlet ports are located on the right side panel of the analyzer. The ports are labeled **Physi 1** through **6, Degas Backfill** and **Freespace Helium**.
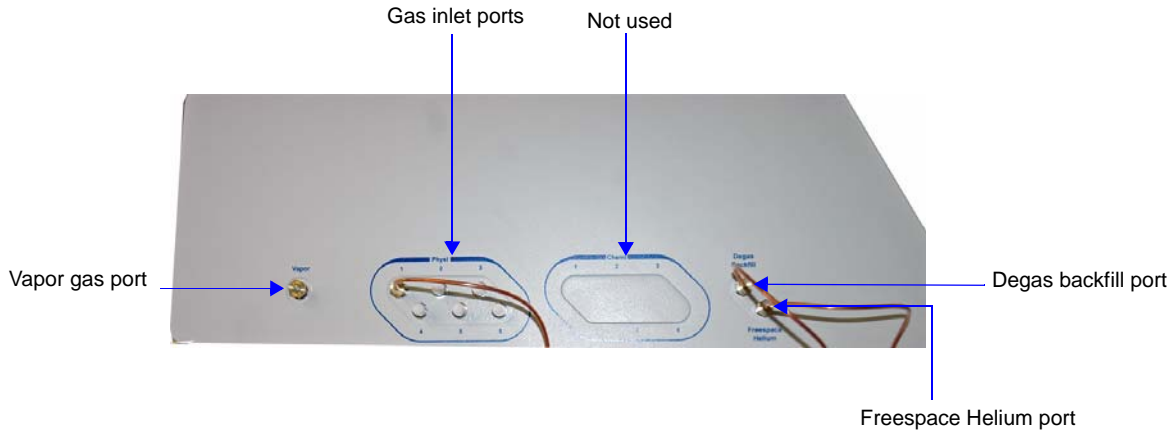
To change the gas attached to one of the ports:

1. Make sure the port valve is closed (refer to **"Enable Manual Control" on page 6-24**).

2. Detach the gas line from the desired port.

3. Attach the new gas line to the port.

4. Make sure any unused gas inlet ports are plugged.

5. Record the gas installed in the port in the analysis program as described in the following section.

Specifying Gas Ports

When you change the gas attached to a port on the analyzer, you must enter the change in the analysis program as follows.

1. Select **Unit 1 > Unit Configuration** from the main menu. The Unit Configuration dialog is displayed.

2. Click **Gas**. The Gas Configuration dialog is displayed.
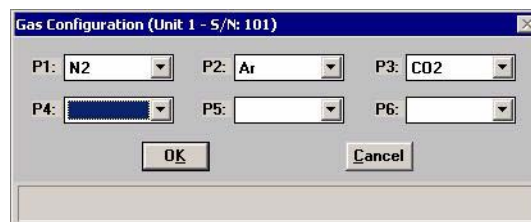
3. Click on the down-arrow at the port to which you attached a gas line and choose the appropriate gas.

4. Click **OK** to close the dialog, and then click **OK** again to close the Unit Configuration dialog.

Performing a Reference Material Analysis

When you have doubts about the integrity of analysis data or when you would just like to ensure the highest quality data, perform a reference analysis. Reference Material Kits are available from Micromeritics (refer to Chapter 10, **ORDERING INFORMATION**). Each kit contains reference material and an instruction booklet. Follow the instructions in the booklet to perform the analysis and review the results.

10. ORDERING INFORMATION

The ASAP 2050 system components and accessories can be ordered using one of the following methods:

- Call our Customer Service Department at (770) 662-3636
- Access our web site at www.micromeritics.com
- Contact your local sales representative

When ordering, please use the information provided in this chapter to place your order:

Analysis System Components and Accessories

Part Number	Item and Description
Peripheral Accessories	
060-00030-00	FlowPrep 060, degasses up to six samples at up to 400 ℃ with flowing gas.
061-00030-00	VacPrep 061, degasses up to six samples at up to 400 ℃ with either flowing gas or by evacuation (evacuation requires a vacuum pump).
021-00000-00	Model 021 LN_2 transfer system, for easy filling of sample Dewars; includes 47-L Dewar, mobile platform, and pump with 100-cm discharge line.
202-33050-00	Water vapor option; allows water vapor adsorption studies.
System Components	
202-25849-00	Dewar, 3 liter, cold trap
202-25850-00	Dewar, 3 liter, analysis
200-25928-00	Dewar, 1 liter, analysis (wide mouth)
202-33053-00	Stainless Steel Dewar Kit, 4 liter, wide mouth, includes analysis Dewar, cold trap Dewar, and elevator adapter
240-25901-00	Dewar depth gauge
202-31708-00	Dewar cover, cold trap
202-31707-00	Dewar cover, analysis
200-25929-00	Dewar stopper, sample port, 1-liter Dewar
004-25103-00	Ferrule, front, Teflon 1/4 in.

Part Number	Item and Description
004-25104-00	Ferrule, rear, Nylon 1/4 in.
290-25846-00	Copper tube, for gas inlet, 1/8-in. diameter, 6 ft (1.8 m) long.
290-25846-01	Same as 290-25846-00, but uses 16-ft. (5m) tubing
004-25549-00	Reducer, 1/8-in. tube x 1/4-in. tube, accepts 1/8-in. tube, connects to 1/4-in. swage fittings
004-62230-35	Gas pressure regulator, CGA 350 fitting (CO, H_2), 30 psig
004-62230-54	Gas pressure regulator, CGA 540 fitting (O_2), 30 psig
004-62230-326	Gas pressure regulator, CGA 326 fitting (N_2O), 30 psig
004-62230-705	Gas pressure regulator, CGA 705 fitting (NH_3), 30 psig
004-62230-58	Gas pressure regulator, CGA 580 fitting (He, N_2, Ar), 30 psig
004-62230-32	Gas pressure regulator, CGA 320 fitting (CO_2), 30 psig
004-62250-320	Gas pressure regulator, CGA 320 fitting (CO_2), 250 psig
004-62250-350	Gas pressure regulator, CGA 350 fitting (CO, H_2), 250 psig
004-62250-540	Gas pressure regulator, CGA 540 fitting (O_2), 250 psig
004-62250-580	Gas pressure regulator, CGA 580 fitting (He, N_2, Ar), 250 psig
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator, includes fittings and instructions
004-33602-00	Pressure Relief Kit, 35 psig, prevents excessive gas pressure in the event of regulator failure (not to be used with noxious gases or with 150 psig gas lines)
003-26046-00	Heating mantle with type K thermocouple, 450 ℃ maximum, 24 V, with elastic hanger.
230-25808-00	Heating mantle clip
205-42801-00	Operator's manual
004-25006-00	O-ring, size 006, Buna-N, for Po tube
004-16843-00	Reference material, 13x molecular sieve
004-16821-00	Reference material, Surface Area ~215 m^2/g, PV ~0.6cc/g
004-54609-00	Sample tube brush
240-25853-00	Sample tube funnel
240-14855-00	Sample tube rack

Part Number	Item and Description
004-54618-00	Tool, for removing sample tube O-ring
200-25840-00	Saturation pressure tube, includes isothermal jacket
250-25608-00	Valve gasket, Kel-F, for analysis manifold
004-25459-00	Valve plunger, Kalrez seal, for analysis manifold
250-25602-00	Valve spring, for Kalrez plunger, for analysis manifold
Vacuum System Components	
062-00001-11	Vacuum pump, oil-sealed, with built-in anti-suckback valve, 110/120 VAC; includes hose kit and alumina oil vapor trap
062-00001-23	Vacuum pump, oil-sealed, with built-in anti-suckback valve, 220/240 VAC; includes hose kit and alumina oil vapor trap
004-16003-01	Vacuum pump oil, 1 liter (not for use with oxygen-compatible pump)
062-33002-00	Activated alumina oil vapor trap, for one vacuum pump
062-00002-11	Vacuum pump, Inert fluid for oxygen use, 110/120 VAC
062-00002-23	Vacuum pump, Inert fluid for oxygen use, 220/240 VAC
004-16009-00	Fomblin® YL-VAC 14/6 inert fluid for vacum pumps that are pre-conditioned for oxygen use
200-25879-00	Vacuum pump oil funnel
004-27040-00	Vacuum pump exhaust filter
004-16830-00	Activated alumina, 500 grams, for oil vapor trap
004-25652-00	O-ring, size 217, for oil vapor trap
Software	
202-33025-00	Isotherm Cycling; allows you perform up to 500 adsorption/ desorption cycles over a user-defined range. Includes software and operating instructions.
205-33001-00	Software package, ASAP 2050; includes current version of software and operator's manual.

Sample Tubes and Components
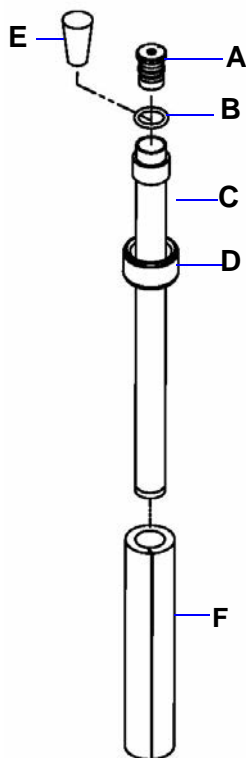
Part Number	Item and Description
Sample Tubes and Components	
205-25802-01 A	Seal Frit with built-in check valve for air-sensitive samples
004-25474-00 B	O-ring, size 013, Kalrez for sample tube
205-25807-00 C	Sample tube
300-25824-00 D	Nut, sample tube
240-32000-00 E	Stopper, for sample tube
205-25815-00 F	Isothermal jacket, for sample tube

Cold Trap Tubes and Components
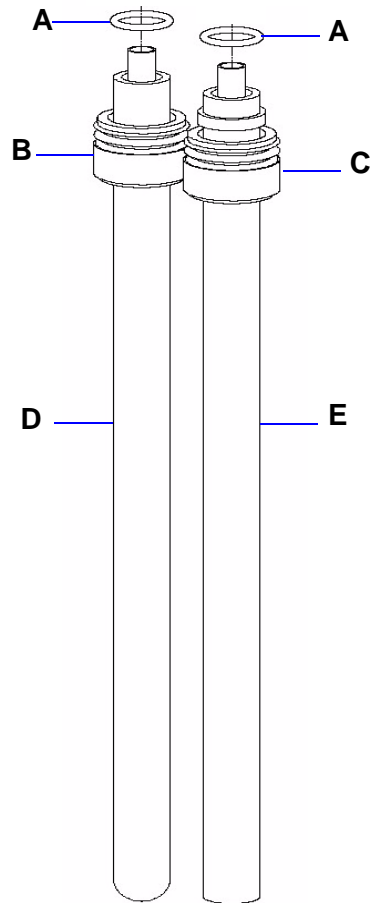
Part Number		Item and Description
		Cold Trap Tube and Components
004-25469-00	A	O-ring, Buna-N, size 014, for glass or stainless-steel cold trap
004-25979-00	B	Nut, for glass cold trap
205-25808-00	C	Nut, for stainless-steel cold trap
004-61063-00	D	Tube, 1/2-in. OD stem, cold trap, glass
205-25806-00	E	Tube, 1/2-in. OD stem, cold trap, stainless-steel

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A. ASAP SERIES SAMPLE DATA WORKSHEET

This form is provided to assist you in obtaining an accurate sample mass for report calculations. You may use the *After Degas* value (Step 5) or the *After Analysis* value (Step 7), provided they are close to the same.

Sample tube:_____   **Sample:** _____

Before Degas:

1.   Mass of empty sample tube   _____ (g)

2.   Mass of sample tube plus sample   _____ (g)

3.   Mass of sample (Step 2) – (Step 1)   _____ (g)

After Degas:

4.   Mass of sample tube plus sample   _____ (g)

5.   Mass of sample (Step 4) – (Step 1)   _____ (g)

After Analysis:

6.   Mass of sample tube plus sample (g)   _____ (g)

7.   Mass of sample (Step 6) – (Step 1)   _____ (g)

Compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5). These two values should be close in range. If a significant difference is noted, analysis problems may exist or the sample may have been improperly degassed.

B. ERROR MESSAGES

Error messages are listed numerically. If the Action response instructs you to contact your service representative, record the error message and make backup copies of any files involved in the operation.

> **The 1000-series error messages (used primarily for software testing) are not included in this appendix. These errors should not occur during normal operation. If you receive a 1000 series error message or an error message not listed in this appendix: record the error message, make backup copies of any files involved, then contact your service representative.**

2200 and 2400 Series

2201- Cannot execute report subsystem.

Cause: Start Report failed to execute the report subsystem (which is a separate process).

Action: Restart the computer. If the problem persists, reinstall the application (this will not affect any of your sample files). If the problem continues, contact a Micromeritics service representative.

2401- FATAL ERROR: (error message).

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2430- Error accessing file (file name), error code = (number).

Cause A: A computer or network problem occurred.

Action A: Check the performance of your computer devices or network.

Cause B: A software error occurred when the file was accessed.

Action B: Contact your service representative.

2431-   Error writing file (file name), error code = (number).

Cause: The hard disk does not have enough space left to perform the opera-
                tion.

Action: Copy files not used regularly from the hard disk to a diskette, CD, or
                network directory, delete them from the hard disk, and then try the
                operation again.

2432-   Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error
                message.

**2433-   New entries have been found in this directory. Refresh the directory
information?**

Cause: Several ASAP 2050 files (sample information, analysis conditions,
                adsorptive properties, or report options) have been added to this direc-
                tory by some function other than the ASAP 2050 program.

Action: Select **Yes** to update the directory information with data from each new
                file. This operation may take a minute.

                Select **No** if you do not want to spend the time updating the directory
                information. This option may be feasible if a large number of files have
                been copied into the directory and you know the name of the file you
                wish to access.

2434-   File (file name) — Subset # (number) wrote wrong amount of data.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error
                message.

2436-   Path specification (path name) is invalid.

    Cause: You entered an invalid path name and/or extension.

    Action: Type a valid path name (including the proper extension) and press **Enter**.

2437-   File (file name) does not exist.

    Cause: You entered a file specification that does not exist.

    Action: Enter an existing file specification or select a file name from the list box.

2438-   Disk drive (letter): is inaccessible.

    Cause: You selected a disk drive that is not presently accessible.

    Action: Ensure that the disk is not write-protected.

2439-   Could not register file.
2440-   Subset not found.
2441-   Seek within file failed.
2442-   Bad header in subset file.
2443-   Subset owner denied access.
2444-   Not a valid file format.
2445-   Subset wrote the wrong amount of data.
2446-   Error reading data.
2447-   Error writing data.

    Cause: An unexpected error occurred when you tried to access a data file.

    Action: Contact your service representative.

2448-   File directory (path name) is invalid. Resetting to the installation directory.

    Cause: A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

    Action: The installation directory will be substituted. The next time you open a file, use the Directories: list to move to the correct directory.

2449-   This field does not contain a valid file specification.

Cause: You entered an invalid file name.

Action: See the description of file naming conventions in your DOS or Windows manual and re-enter the name.

2450-   Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: You are in the process of initiating an automatic analysis (an analysis in which sample files are created using the defaults) while editing the defaults.

Action: Finish your edit session of the defaults and close the dialog. Then restart the automatic analysis.

2454-   Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

2455-   Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: You selected too many files for this operation.

Action: Select only the number of files specified in the message.

2456-   Insufficient file handles available. Application cannot continue.

Cause: You have more than 50 files open at the same time.

Action: Refer to the manual for your operating system and set the limit for open files to 50 or greater.

2457-   Results cannot be displayed. More than (number) windows are currently displaying or printing results.

Cause: You have too many windows open in the application.

Action: Close some of the open windows.

2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.

> Cause: You attempted to exit the application while the analyzer is performing a critical operation. This operation must be completed before the application can be stopped.

> Action: Wait a few minutes before attempting to exit the application again.

2459- An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit?

> Cause: You attempted to exit the application while the analyzer was performing an operation.

> Action: Rather than exiting the application you may choose to minimize it.

2460- Fatal Communications error on (unit nn).

> Cause: Repeated attempts to maintain communication with the analyzer have failed.

> Action: Check the communications cable connecting the analyzer to the computer. Confirm that the analyzer is turned on and that the status light is not blinking.

> If these checks are okay, exit the analysis program and then restart the application. If this error persists, contact your service representative.

2461- No instruments are in operation. This application will unconditionally terminate.

> Cause: At least one analyzer must be active for the application to operate. The initialization of the analyzers configured with the Setup program has failed. The application stops.

> Action A: Usually this message is preceded by another message giving the reason for the analyzer's failure to initialize. See the instructions for that message.

Action B: Check the cable connection between the analyzer and the computer. Verify that the analyzer has the power switch in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the analyzer, contact your service representative.

2471- Unit (number) - S/N; (number) has an invalid communications port specified. It cannot be initialized.

Cause: The communications port specified for this analyzer during installation (or configuration) is invalid.

Action: Use the setup program to change the analyzer configuration.

2474- Unit (number) Communications port COM (port number) specified in the program control files is already in use. Unit cannot initialize.

Cause: The communications port assigned to the indicated unit is in use by another program.

Action: End the program using the busy port or use the Setup program to change the communications port assigned to this program.

2475- Unit (number) Communications port COM (port number) specified in the program control files cannot be accessed. Unit cannot initialize.

Cause: The operating system has prevented the application from accessing the communications port.

Action: Review the hardware configuration of the computer, ensure that no other application is using the port. Contact your service representative if you continue to receive this error message.

2476- Unit startup failed. Cannot initialize the communications port.

Cause: The communications port specified during installation is invalid.

Action: Use the setup program to change the analyzer configuration. Contact your service representative if you continue to receive this error message.

2477-   Unit (number); Serial (number) did not properly initialize.

Cause: The software was unable to initialize the analyzer.

Action A: Run the Setup program and ensure that a valid port is specified; if not, specify a valid one when prompted.

Action B: Reinstall the software, then restart application.

Action C: Contact your Micromeritics service representative if you continue to get this message.

2478-   Error copying sequential data segment.

Cause: An internal processing and/or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors; for example, you may wish to use a utility such as ScanDisk. Contact your service representative if you continue to receive this error message.

2479-   Unit (number); Serial (number) The instrument is busy performing an operation of which this application is unaware. Do you want to cancel? (Yes, No)

Cause: During initialization of the application, the status of the analyzer was found to be in a different state than expected.

Action: Click **Yes** to cancel the operation in process, allowing the analyzer to reset and continue with initialization.

Click **No** to cancel the initialization process.

If you continue to get this message, verify that files in the application directory structure are not being changed or removed.

2480-   File (file name) cannot be analyzed. It is currently being edited.

Cause: You attempted an analysis using a sample file that is being edited.

Action: Save the changes and close the dialog box.

2481-   Error accessing the sample information file (file name).

    Cause A: You attempted to open a file that is already open, possibly minimized.

    Action A: View the minimized icons, locate and maximize the file.

    Cause B: A computer or network problem occurred.

    Action B: Check the performance of your computer devices or network.

    Cause C: A software error occurred when the file was accessed.

    Action C: Contact your service representative.

2482-   File cannot be opened for writing. It is already in use.

    Cause: You are attempting to open a file that is currently being used (either by this application or another).

    Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all windows, one of which may contain this file).

**2483- An analysis cannot be performed on (file name). It is open for editing and con-
tains errors.**

    Cause: You attempted to use a sample file containing errors that is currently open.

    Action: Go to the window containing the file, correct the errors, and save it.

**2484-   The edit session for (file name) must be saved before the analysis. Save
changes and continue with the analysis.**

    Cause: You attempted an analysis using a sample file that contains unsaved changes.

Action: Select **Yes** to save the changes and continue with the analysis.

Select **No** to abort the analysis and return to the sample file.

2485-   The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected service test file has a status other than **No Analysis**.

Action: Select a different service test file or create a new one and use **Replace All** to copy parameters from the file you originally selected.

2486-   Cannot construct (name) report type. Program will terminate.
2487-   Cannot start report generator. Error code (number). Program will terminate.

Cause A: You may not have full rights to the application's folders and files.

Action A: Contact your system administrator and have him grant you full rights.

Cause B: An internal processing and/or hardware error has occurred.

Action B: Contact your service representative if you continue to receive this error message.

2488-   File (file name) cannot be opened for editing. It is already in use.

Cause: The file you specified is being used in another edit operation.

Action: Check the Windows list to locate the other edit session.

2490-   No '.INI' file present. Application will terminate.

Cause: The ASCII (.INI) file containing initialization and system options information cannot be found. The .INI file may have become corrupted. The application cannot operate without this file.

Action: Use the Setup CD to uninstall the ASAP 2050 application. When you uninstall the application, only the application files are deleted; data files remain intact.

After the uninstall operation is complete, reinstall the ASAP 2050 application.

2491-   Highlighted fields contain errors. Please correct the errors before closing dialog box.

Cause: The highlighted fields contain invalid entries. You will not be able to close the dialog box until you correct the errors.

Action: Check the entries, correct the errors, and close the dialog box

2492-   This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493-   An entry is required for this field.

Cause: This field requires a valid entry for you to proceed.

Action: Enter or select an appropriate value.

2494-   Value is out of the valid range.
2495-   Value is out of the valid range. Enter a value between (value) and (value).

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter or select an appropriate value.

2496-   Invalid number.

Cause: The number you entered in the highlighted field is invalid.

Action: Check the entry and enter or select a valid number.

2497-   This field contains an invalid character.

Cause: You entered an invalid character in the highlighted field.

Action: Check the entry and enter valid characters.

2498-   The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status (for example, from automatically collected to manually entered) could not be done.

Action: Contact your service representative if you continue to receive this error message. Record the name of the sample file in which the problem occurred.

2499-   Sequence number must contain at least 3 digits.

Cause: You tried to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500 Series

2500-   All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.

Action: Select **Options** > **Sample Defaults** from the Main Menu and enter a new sequence number.

2501-   System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows on the screen. Contact your service rep-
                  resentative if you continue to receive this error message.

2502-   Error writing to file (name) during print. Error code: (number).

Cause: An error occurred in the file being written to during a print operation.

Action: Ensure that there is sufficient space on the drive containing the file.

2503-   Error converting file (file name). Could not create DIO intermediate file.

Cause A: Insufficient space is available on the hard disk. The DIO file is placed
                  in the directory specified by the TEMP environment variable.

Action A: Determine if there is sufficient space on the drive where the TEMP
                  directory is located.

Cause B: An internal processing and/or hardware error has occurred.

Action B: Contact your service representative if you continue to receive this error
                  message.

2504-   Cannot create output file for sample (sample name).

Cause: Insufficient space may be available on the hard disk.

Action: Ensure that sufficient space is available. Contact your service repre-
                  sentative if you continue to receive this error message.

2505-   Error Logger cannot be initialized! Error code (number). Program will exit.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative.

2506-   (sample file) Output device (name) is not installed. Printing cannot be accomplished.

Cause:	The selected output device is not installed in Windows.
Action:	Select a different output device in the System Configuration dialog box. Install the device using the Control Panel, Printers operation.

2508-   (sample file) Overlay file (name) was not found. It will not be included in the reports.

Cause:	The specified overlay file could not be found.
Action:	Ensure that the file specified as an overlay does exist.

2509-   (sample file) Error opening file (name): (error). Reports cannot be produced.

Cause:	An error occurred while the program was opening a file necessary to the report operation.
Action:	Use the name given in the error message to investigate. Contact your service representative if you continue to receive this error message.

2510-   (sample file) Error parsing reports from file (name). Reports cannot be produced.

Cause A:	One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).
Action A:	Review the data entry fields (for example, the Sample field) and remove the invalid character.
Cause B:	The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.
Action B:	Check the space available on the hard disk.

Cause C: An internal processing error occurred.

Action C: Contact your service representative.

2511-   Print job (name) has been cancelled due to insufficient disk space. Delete unnecessary files and restart the report.

Cause: The disk drive does not have enough space for the temporary file required by the Windows Print Manager. Therefore, printing of the requested report has been canceled.

Action: Delete unnecessary files from the disk. You will require at least five megabytes of free space for normal operation.

2512-   Print job (name) has been cancelled.

Cause: The requested print job was canceled at your request.

Action: None required.

2513-   Unable to read the calibration file (file name).

Cause: You selected an invalid calibration file or one that cannot be read.

Action: Be sure the media containing the calibration file has no problems.

2514-   Unable to write the calibration file (file name).

Cause: An attempt to Save calibration data has failed due to possible media problems.

Action A: Be sure the media you want to Save the file to has no problems.

Action B: Choose an alternate media to Save the calibration data.

2515-   Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

   Cause: You have started the process of performing a calibration operation.

   Action: Calibration operations should only be done by or under the direction of qualified service personnel.

2516-   Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

   Cause: You have performed a calibration operation; a backup copy is recommended.

   Action: Perform a calibration **Save** operation.

2517-   Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

   Cause: You have not accepted the calibration you performed.

   Action: If the calibration operation was successful, press **Accept**.

2521-   Unable to program controller.

   Cause: A hardware malfunction has occurred.

   Action: Contact your local Micromeritics service representative.

2522-   Invalid controller application file.

   Cause: The application's control file has been corrupted or deleted.

   Action: Reinstall the ASAP 2050 analysis program.

2523-   Programming the controller failed.
2524-   CRC check failed on programming controller.
2525-   Unknown error programming controller.
2526-   Controller download was not successful.
2527-   Controller CRC error on boot block.

2528-   Controller DRAM error.
2529-   Controller Com1: error.
2530-   Controller Com2: error.
2531-   Controller debug port error.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error
                message.

2532-   The instrument contains a different software version. Do you want to reset it?

Cause: The application has discovered a different version of software operat-
                ing in the analyzer.

Action: If there are no analyzers other than the ASAP 2050 connected to the
                computer, select **Yes** and allow the updated software to load.

2533-   Analyzer initialization failed.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error
                message.

2534-   Error opening file (name) for printing. Error code: (number).

Cause: An error occurred in the selected file for print output.

Action: Ensure that sufficient space is available on the drive containing the file.

4000 Series

4000-   Memory Allocation Error.

Cause: An internal processing and/or hardware error occurred during report generation.

Action: Contact your service representative if you continue to receive this error message.

4002-   Thermal Transpiration correction had no effect.

Cause: The Thermal transpiration correction option was selected on the Report Options dialog box. However, the correction did not change any pressure by more than one percent.

Action: De-select this option to disable this message. This correction is only meaningful for very low pressures.

4003-   Error Converting Pressures.
4004-   Error Computing Volume Adsorbed.

Cause: An internal processing and/or hardware error occurred during report generation.

Action: Contact your service representative if you continue to receive this error message.

4005-   Pressures were not smoothed. Not enough pressures below 0.10 P/Po.

Cause: The **Smooth pressures below 0.10 P/Po** option was selected on the Report Options dialog. There must be at least 10 pressures within this range for smoothing to occur.

Action: Deselect this option to disable this message.

4006-   Report Type Not Found.
4007-   Error Processing Report.

> Cause: An internal processing and/or hardware error occurred during report
>   generation.

> Action: Contact your service representative if you continue to receive this error
>   message.

4008-   Report requested on sample file with no data points.

> Cause: You selected a file for reporting which contains no collected data.

> Action: Select another file which contains collected data and restart the report.

4009-   Error, No Reports Selected.

> Cause: You attempted to generate reports for a sample that has no reports
>   selected in its report options.

> Action: Select the desired reports in the sample's report options and save the
>   sample file.

4010-   Summary Report was not included in the Selected Reports.

> Cause: You did not select a plot or a table for any of the Selected Reports in
>   the Report Options dialog; you selected the reports only to generate an
>   entry in the summary. However, the Summary was not included in the
>   Selected Reports.

> Action: Select a plot or table in one or more of the Selected Reports, and/or add
>   the Summary to the Selected Reports.

4011-   Analysis gas in sample file does not match analysis gas in unit.

> Cause: You attempted to start an analysis using a sample information file in
>   which the analysis gas specified does not match the analysis gas
>   entered in the unit configuration.

Action: If necessary, attach the appropriate gas bottle, then enter the gas in the
                 Unit Configuration dialog box.

4012- Psat gas in sample file does not match Psat gas in unit.

Cause: You attempted to start an analysis using a sample information file in
                 which the Psat gas does not match the Psat gas entered in the unit con-
                 figuration.

Action: If necessary, attach the appropriate gas bottle, then enter the gas in the
                 Unit Configuration dialog box.

4015- Error creating export file for sample <sample file name>.

Cause: A file error occurred during creation of an export output file.

Action: The output file name may be invalid. Ensure that the target directory
                 exists. Ensure that the target diskette is not full or write protected. The
                 target disk drive may be damaged or inoperative. Verify that other files
                 may be created on the same drive. Contact your service representative
                 if you continue to receive this error message.

4016- Sample (file name) has no data for export.

Cause: The file selected for export has a status of No Analysis. No export file
                 will be created.

Action: Select a file which contains analysis data.

4017- Damage to the instrument will result if the sample has not been manually evac-
uated. Have you evacuated the sample?

Cause: You did not select **Backfill sample at start of analysis** on the Sample
                 Backfill Options dialog box. The sample tube is normally at atmo-
                 spheric pressure when an analysis is started, and it must be backfilled
                 before the analysis begins to prevent sample material from being
                 drawn into the manifold.

Action: If you have manually evacuated the sample tube, select **Yes**. If you
                 have not, select **No** and then either perform a manual evacuation or go
                 to the Sample Backfill Options dialog box and select **Backfill sample
                 at start of analysis**.

**4020-   Disabling this option may damage the instrument. Are you sure that the sample
should not be backfilled?**

Cause: You did not select **Backfill sample at start of analysis** on the Sample
                 Backfill Options dialog. The sample tube is normally at atmospheric
                 pressure when an analysis is started; it must be backfilled before the
                 analysis begins to prevent sample material from being drawn into the
                 manifold.

Action: If you want to manually evacuate the sample prior to the start of the
                 analysis, select **Yes**. Otherwise, select **No**, go to the Sample Backfill
                 Options dialog, and select **Backfill sample** at start of analysis.

**4021-   The entered Po value (Po and Temperature Options of the Analysis Conditions)
is outside the range of the pressures listed in the Psat vs. Temperature Table (Adsorp-
tive Properties).**

Cause: The temperature that corresponds to the entered Po cannot be found
                 because the entered Po is outside of the range of values in the Psat vs. T
                 table.

Action A: Enter a Po that is within the range of the table.

Action B: Use a different Psat vs. T table.

**4022-   The entered bath temperature value (Po and Temperature Options of the Anal-
ysis Conditions) is outside the range of the temperatures listed in the Psat vs.
Temperature Table (Adsorptive Properties).**

Cause: The Po that corresponds to the entered temperature cannot be found
                 because the entered temperature is outside of the range of values in the
                 Psat vs. T table.

Action A: Enter a temperature that is within the range of the table.

Action B: Use a different Psat vs. T table.

4023-   The file (sample file) cannot be prepared for analysis. It is open for editing and contains errors.

Cause: You attempted an analysis using a sample file that is being edited and has invalid values in one or more of its fields.

Action: Enter valid values into the fields that have errors, save the changes, and close the dialog box.

4024-   Backfill gas in sample file does not match any gas in unit.

Cause: You selected a backfill gas that is not included in the gas configuration.

Action: Make sure that gas selections match the gases that are attached to the instrument. Choose one of the gases as the backfill gas, or edit the sample file and choose one of the existing gases as the backfill gas.

4025-   There is no Helium attached to the unit.

Cause: A measured free space has been requested but Helium does not appear to be available.

Action: Make sure the helium cylinder is attached to the unit at its designated port.

4026- Cannot calculate Dubinin-Astkahov: bad least squares data.

Cause: Less than two selected data points are within the fitted pressure range.

Action: Edit the calculation assignments on the Collected Data dialog, or change the fitted pressure range on the Dubinin report options dialog.

4027-   Fewer than two sample files have data suitable for heat of adsorption reports.

Cause: Less than two of the sample files you selected for heat of adsorption reports contain appropriate data.

Action: Edit the Quantity Adsorbed table, or select other sample files.

4028-   Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

Cause: Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.

Action: Access the Dubinin Report Adsorptive options in the sample file and enter an appropriate value for the analysis gas.

4029-   At least two fitted data points are needed for Alpha-S calculations.

Cause: Fewer than two data points fall within the selected Alpha-s range.

Action: Edit the calculation assignments or the fitted Alpha-s range, or use a different reference curve.

4030-   Preparations failed in primary data.

Cause: Appropriate data were not available to generate the report.

Action: This message was preceded by a different error message; refer to the cause/action of the preceding message.

4031-   Not enough points with a relative pressure in the range (n,n)

Cause: Fewer than two data points selected for the Dubinin report falls within the selected relative pressure range.

Action: Edit the calculation assignments or the fitted relative pressure range.

4033-   Not enough points to generate Dubinin Tabular Report.

Cause: There are fewer than two valid data points available for the Dubinin tabular reports.

Action: Examine the calculation assignments in the Collected Data dialog of the sample file. You must have at least two micropore pressures selected for inclusion in the Dubinin report.

4034- Fewer than 2 points available for Dubinin calculations.

    Cause: There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.

    Action: Examine the calculation assignments in the Collected Data dialog of the sample file. You must have at least two micropore pressures selected for inclusion in the Dubinin report.

4035- Cannot calculate optimized Astakhov exponent: Not enough points with a relative pressure in the range [(pressure), (pressure)].

    Cause: There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.

    Action: Examine the calculation assignments in the Collected Data dialog for the sample file. You must have at least two pressures in the given range selected for inclusion in the Dubinin report.

4036- Fewer than 2 points available for Horvath-Kawazoe calculations.

    Cause: You must select at least two data points for inclusion in the Horvath-Kawazoe analysis on the Collected Data dialog. No report will be produced.

    Action: From the Collected Data dialog, select the data points to be analyzed.

4037- Computations failed while processing the primary data set. No reports will be produced.

    Cause: The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded with another one containing additional information.

    Action: Refer to the number of the error message which preceded this one for an explanation.

4038-   Fewer than 2 points available for the Langmuir Qm computation. Cheng/Yang correction will not be applied.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.

Action: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to prevent this message from appearing on future reports.

4039-   The isotherm does not meet the constraints of the Cheng/Yang assumption. Cheng/Yang correction will not be applied.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.

Action A: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to prevent this message from appearing on future reports.

Action B: Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them and reproduce the Horvath-Kawazoe reports.

4040-   The value of Qm computed from the Langmuir equation is too low. The pore size will not be computed for all data points.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.

Action: The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of Vm. The pore size will not be calculated for data points with an invalid volume adsorbed. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to clear this message.

4041-   Cheng/Yang correction is inappropriate for some P/Po.

Cause: The Cheng/Yang correction is usually inappropriate for any P/Po above the isotherm knee. In some instances, the computed pore sizes may decrease above the knee. While it is possible to include these relative pressures (usually above 0.1 P/Po) in the analysis, the computed pore sizes for these pressures are usually meaningless.

Action: Change the data points selected for the Horvath-Kawazoe report to include only relative pressures at or below the knee of the isotherm, or change the Horvath-Kawazoe report options so that the Cheng/Yang correction is not applied.

4042-   0.0 cannot be a starting or ending pressure for a geometric progression from low pressure.

Cause: You selected to generate a pressure table from a geometrically progressing range.

Action: Change the 0.0 entered value.

4043-   1.0 cannot be a starting or ending pressure for a geometric progression toward saturation.

Cause: You selected to generate a pressure table from a geometrically progressing range.

Action: Change the 1.0 entered value.

4044-   Points in the Langmuir report pressure table lie outside the collected data.

Cause: Calculation assignments are not being used and more than one of the report pressure table points is above the range of the collected data, and more than one is below.

Action: Change the report pressure table to be more consistent with the col-
                 lected data.

4045-   Points in the report pressure table lie outside the collected data.

Cause: Calculation assignments are not being used and more than one of the
                 report pressure table points is above the range of the collected data, and
                 more than one is below.

Action: Change the report pressure table to be more consistent with the col-
                 lected data.

4046- (file name) could not be opened for reading.

Cause: A thickness curve file could not be opened.

Action: If the problem persists, restart your computer and optionally perform a
                 media integrity check (using ScanDisk).

4047-   Warning: An error occurred while reading (file name).

Cause: An error happened during a read operation of a thickness curve file.

Action: If the problem persists, restart your computer and optionally perform a
                 media integrity check (using ScanDisk).

4048-   Warning: An error occurred while restoring the heat of adsorption report editor.

Cause: The state of the heat of adsorption report editor could not be restored.
                 Default settings will be used.

Action: No action.

**4049- The sample (file name) does not have enough data. A minimum of two adsorp-
tion points is required.**

    Cause: A sample file has been included in the Heat of Adsorption report that
                    does not have enough data.

    Action: Remove the file from the selected file list.

**4050- None of the requested quantities adsorbed is within the range of the collected
data of more than one sample file.**

    Cause: The Heat of Adsorption report failed because the specified quantities
                    are not within the range of the collected data.

    Action A: Edit the quantities adsorbed so that they are within the range of the col-
                    lected data, or select other sample files.

**4051- The sample (file name) does not have any data in the range of the requested
quantities adsorbed.**

    Cause: The sample's data cannot be interpolated to any of the quantities
                    adsorbed.

    Action: Edit the quantities adsorbed so that they are within the range of the col-
                    lected data.

4052- Fewer than two points are selected for this report.

    Cause: At least two points are required for the BET calculations.

    Action: Edit the calculation assignments for the BET report.

4053- At least two data points must be selected for t-Plot calculations.

    Cause: At least two points are required for the t-Plot calculations.

    Action: Edit the calculation assignments for the t-Plot report.

4054- Fewer than two data points are inside the fitted thickness range.

Cause: At least two points must be within the fitted thickness range for the t-Plot calculations.

Action A: Edit the calculation assignments for the t-Plot report.

Action B: Edit the fitted thickness range in the t-Plot report editor.

4055- The BET surface area could not be calculated.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the t-Plot report editor.

4056- The Langmuir surface area could not be calculated.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the t-Plot report editor.

4057- At least two data points are needed for Freundlich calculations.

Cause: Less than two data points have been selected for the Freundlich report; at least two are required.

Action: Select Freundlich points on the Collected Data dialog. If calculation assignments are not being used, edit the Freundlich Report options, **Absolute pressure range** in the sample file.

4058-   At least two data points are needed for Temkin calculations.

Cause: Less than two data points have been selected for the Temkin report; at least two are required.

Action: Select Temkin points on the Collected Data dialog. If calculation assignments are not being used, edit the Temkin Report options, **Absolute pressure range** in the sample file.

Cause: You have selected at least one point with a negative pressure value to include in the Temkin report.

Action: Negative pressure points void Temkin calculations. Open the sample file and click the **Collected Data** tab. Locate the negative value(s) in the Temkin column and deselect it (them).

4059-   Fewer than 2 points available for MP-Method calculations.

Cause: At least two points are required for the MP-Method calculations.

Action: Edit the calculation assignments for the MP-Method report.

4060-   Sample (file name) contains no data points.

Action: An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.

Cause: Repeat the save as t-curve or save as alpha-S operation after opening a sample that has collected data.

4061-   The t-curve must contain at least 2 points.

Cause: At least two points are required in a thickness curve definition.

Action: Edit the thickness curve.

4062-   Error during report generation.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

4063-   The data requested on this report are not available.

    Cause: There is no information in the sample log to report.

    Action: You selected a sample file for which no instrument operations have been used. Select a sample file with a status of **Prepared**, **Preparing**, **Analyzing**, or **Complete** to obtain a valid sample log report.

4067-   No data points are within the range of pressures in the reference isotherm.

    Cause: There are no collected data points within the range of pressures in the reference isotherm.

    Action: Select data points that are in the range of the reference isotherm, or select a more appropriate reference isotherm.

4068-   No points were selected for the f-Ratio report.

    Cause: The collected data column for the f-Ratio report does not have any points selected.

    Action: Edit the collected data dialog and select points for the f-Ratio report.

4069-   Dosing method choice is invalid. The Analysis Conditions choice of Absolute pressure dosing requires that the Adsorptive Properties (Dosing Method) is set to Normal.

    Cause: There is an incompatibility between the analysis conditions choice of **Absolute pressure dosing** and the adsorptive properties **dosing** method.

    Action: Edit one of the choices.

4070-   Unable to load deconvolution model (name).

    Cause: For some reason, the list of available models was corrupted, therefore, the model selected could not be loaded for the deconvolution.

    Action: Exit the program and reinstall the software, then try again.

4071- The selected pressures points do not form a valid set for deconvolution.

Cause: The data points selected for analysis do not contain enough informa-
                    tion to allow a DFT data reduction.

Action A: Edit data points in the table of the Collected Data dialog, or select
                    another sample file. At least two points with strictly increasing pres-
                    sures and volumes adsorbed are required for a DFT Plus data reduc-
                    tion.

Action B: Edit the pressure range on the DFT report options dialog.

4072- The range of pressures selected is too small to deconvolute using this model.

Cause: A null result was found using the selected model.

Action: Edit data points in the table of the Collected Data dialog, or select
                    another sample file. At least two points with strictly increasing pres-
                    sures and volumes adsorbed are required for a DFT data reduction.

4073- The analysis gas (name) does not match the model gas (name).

Cause: The model assumes a specific gas and the sample file uses a different
                    one.

Action: Select a model that assumes the same gas.

**4076-   Invalid sample file - Adsorptive Properties "Non-condensing adsorptive" can-
not be used unless Analysis Conditions "Absolute pressure dosing" is selected.**

Cause: You selected a sample file that has **Non-condensing adsorptive**
                    selected in the Adsorptive Properties but does not have **Absolute pres-
                    sure dosing** selected in Analysis Conditions.

Action A: If you plan to use a non-condensing adsorptive, select **Absolute pres-
                    sure dosing** on the Analysis Conditions dialog.

Action B: If you are not using a non-condensing adsorptive, deselect the **Non-
                    condensing adsorptive** option on the Adsorptive Properties dialog, or
                    choose an adsorptive that you are sure in not non-condensing.

4077-   Cannot get surface area for: (file name)

Cause: The Isotherm report for the named overlay file has **Per gram** selected
                for the **Volume Adsorbed**, and the Isotherm report for the primary file
                has a surface area option selected for the **Volume Adsorbed**.

Action A: Edit the Isotherm report for the named overlay file and select a surface
                area option for **Volume Adsorbed**.

Action B: Click **Overlays** on the Report options dialog of the primary file and
                remove the named overlay file from the list.

4078-   Slope and Y-Intercept cannot be determined from the selected points.

Cause: The Langmuir report cannot be generated from the selected points.

Action: Edit the calculation assignments in the Langmuir column on the
                Collected Data dialog.

4079-   Points found with negative pressure values will not appear on the graph.

Cause: Collected data contains negative pressure values.

Action: Negative pressure points are automatically removed from isotherm
                graphs. If you wish to have negative pressure values appear on the
                graph: Open the sample file and click the **Collected Data** tab.
                Deselect **Use calculation assignments** and then select the points with
                negative pressures as Outliers.

**4080- The compressibility factor table has (number) pressure entries. Only (number)
are allowed.**

Cause: The compressibility factor table in the adsorptive properties has too
                many pressure values for analysis.

Action: Edit the table to remove unneeded pressure entries.

4081- In the compressibility factor table, the 4 temperatures below (temperature) and 7 temperatures above must span at least 10 K.

Cause: The compressibility factor table in the adsorptive properties has too many entries for temperatures near ambient.

Action: Edit the table to remove unneeded temperature entries.

4082- Compressibility factors must be greater than zero for temperatures near ambient and pressures up to the maximum manifold pressure.

Cause: The compressibility factor table in the adsorptive properties contains zeros or negative values for the temperatures and pressures expected during analysis.

Action: Edit the table to add missing compressibility factors.

4083- All rows of (file name) do not have the same number of entries. Some rows will be filled or truncated.

Cause: This is a warning that all rows in the compressibility factor table do not have the same number of enties.

Action: Edit the table to make all rows the same length.

4084- The temperatures of (file name) are not strictly ascending.

Cause: The temperature values in the compressibility factor table are not in ascending order.

Action: Edit the table to put the temperature columns in ascending order.

4085- The pressures of (file name) are not strictly ascending.

Cause: The pressure values in the compressibility factor table are not in ascending order.

Action: Edit the table to put the pressure rows in ascending order.

4086- Error reading (file name)

Cause A: An error ocurred while reading the compressibility factor table.

Action A: Check the performance of your computer devices or network.

Cause B: A software error occurred when the file was accessed.

Action B: Contact your service representative.

4087- Error writing (file name)

Cause: An error ocurred while writing the compressibility factor table.

Action: The hard disk does not have enough space left to perform the opera-
                tion. Copy files not used regularly from the hard disk to a diskette, CD,
                or network directory, delete them from the hard disk, and then try the
                operation again.

4088- Can not open (file name) for reading

Cause A: The specified compressibility factor table could not be read.

Action A: Check the performance of your computer devices or network.

Cause B: A software error occurred when the file was accessed.

Action B: Contact your service representative.

4089- Can not open (file name) for writing

Cause: The specified compressibility factor table could not be written.

Action: The hard disk does not have enough space left to perform the opera-
                tion. Copy files not used regularly from the hard disk to a diskette, CD,
                or network directory, delete them from the hard disk, and then try the
                operation again.

4090- The compressibility factor table is empty.

   Cause: The compressibility factor table in the adsorptive properties does not
                   have any data.

   Action: Load an appropriate compressibility factor table.

6200 Series

6201- 'GetOpenFile()' reports error code (number). File selection cannot be made.

   Cause: You specified a file name which does not exist.

   Action: Confirm that the file name used is valid and that the file exists.

**6202-   Invalid communications port (port name) specified in the application's config-
uration settings. Unit cannot initialize.**

   Cause A: The communications port assigned to the ASAP 2050 cannot be made
                   available for proper communications.

   Action A: Run the ASAP 2050 Setup program, select Change analyzer setup, and
                   enter the correct port information. If the message continues, contact
                   your service representative.

   Cause B: The ASAP 2050 control file was manually modified and an invalid
                   communications port was specified.

   Action B: Use the ASAP 2050 Setup program to change the communications port
                   assigned to the ASAP 2050.

6203-   No serial number available. Unit cannot initialize.

   Cause: The ASAP 2050 control file does not contain a serial number for the
                   unit.

   Action: Run the ASAP 2050 Setup program, select Change analyzer setup, and
                   define the serial number for the instrument.

6204- The disk does not have enough room for the ROA data. The space required is (number) megabytes.

Cause: The computer's disk is almost out of disk space.

Action: Delete unwanted files.

6209- Analysis startup failed. (Secondary message number and text.)

Cause: The ASAP 2050 Program was unable to start an analysis with the indicated unit.

Action: Refer to the specified error number and text for instructions.

6212- Calibrating. This action must be completed before the application exits.

Cause: The unit is calibrating.

Action: Do not exit the application until the calibration is complete.

6216- Instrument could not initialize. Reason: (reason).

Cause: An internal processing and/or hardware error has occurred.

Action: The specific reason given may identify a possible corrective action. Contact your service representative if you continue to receive this error message.

6235- (Unit [number]) Analysis canceled: Time limit exceeded while dosing Psat tube to 925 mmHg.

Cause A: A pressure of 925 mmHg was not attained within the allowed time. The Psat gas regulator may be set too low or turned off.

Action A: Set the Psat gas regulator to 10 psig (0.7 bar). Then restart the analysis.

Cause B: The Psat gas bottle is empty.

Action B: Connect a new Psat gas bottle. Then restart the analysis.

Cause C: The Psat tube fitting is loose.

Action C: Check the Psat tube fittings and ensure that the tube is attached
                 securely to the port. Then restart the analysis.

**6237-   Unit (number) Analysis canceled: Time limit exceeded while evacuating Psat
tube.**

Cause: The maximum time for evacuating the Psat tube was exceeded before
                 the vacuum set point was achieved.

Action: Check the Psat tube fittings. Tighten the connector nut if necessary;
                 then restart the analysis.

6240-   Leak test failed.

Cause: With the sample port valve closed, the sample pressure increased by
                 0.15 mmHg before the leak test duration was completed.

Action: Check sample tube fitting and ensure that it is securely attached to the
                 port. Then restart the analysis.

**6241-   Unit (number) Analysis canceled: Maximum time exceeded before the elevator
reached the UP (or DOWN) position.**

Cause A: The maximum time for the analysis was exceeded before the elevator
                 reached the UP (or DOWN) position. Ice may be present in the bottom
                 or the neck of the Dewar.

Action A: Check the Dewar and remove ice if necessary. Refer to Checking the
                 Analysis Port Dewar in Chapter 8. Then restart the analysis.

Cause B: The Psat tube is interfering with elevator movement.

Action B: Make sure the Psat tube is close to the sample tube and the Dewar
                 cover is over both the sample and Psat tubes. Then restart the analysis.

6500 Series

6500- Failed to evacuate manifold to VAC SET in (number of) seconds. Calibration canceled.

Cause A: The vacuum set point is set too low.

Action A: Ensure that the vacuum set point is at 5.0 mmHg. If the set point is already at 5.0 mmHg or above, the vacuum gauge may need servicing.

Cause B: Leak in manifold.

Action B: Locate the leak and repair it. Refer to Chapter 9, Troubleshooting and Maintenance. Restart calibration.

Cause C: Valve failure.

Action C: Identify the leaking valve. Contact your service representative.

6501- The Master transducer offset exceeds recommended limits: (number)

Cause: The Pressure Gauge Calibration operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the Pressure Gauge Calibration operation, if this message occurs again contact a Micromeritics service representative.

6504- Unable to write the calibration file (name).

Cause: A Save to File operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6505- Unable to read the calibration file (name).

Cause: A Load from File operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6506- Calibration file for (file name) is invalid.

Cause: A Load From File operation failed due to invalid information in the file.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6509- The sample has an invalid status and cannot be used for degassing.

Cause: A file selected for degassing has a status other then No Analysis or Prepared.

Action: Select a different file.

6510- Error evacuating.

Cause: An evacuation error occurred during a degas operation.

Action: If the problem persists, contact a Micromeritics service representative.

6511- Error dosing.

Cause: A dosing error occurred during a degas operation.

Action: If the problem persists, contact a Micromeritics service representative.

6512-   Error calibrating the servo.

    Cause: An error occurred while calibrating the servo valve.

    Action: If the problem persists, contact a Micromeritics service representative.

6513-   Error waiting for pressure to drop.

    Cause: A pressure error occurred during a degas operation.

    Action: If the problem persists, contact a Micromeritics service representative.

6514-   Problem encountered dosing to target [PR2] [PR-U], last pressure = [PR4] [PR-U], elapsed time = [0]:[0].

    Cause: Dosing during an analysis did not come within the allowed range of the target.

    Action: Check that the outlet stage of the gas regulator is within specification. If the problem occurs frequently contact a Micromeritics service representative.

6516-   Unit (number) Analysis canceled: Sample pressure greater than (pressure) mmHg is not allowed.

    Cause: An absolute pressure greater than (pressure) mmHg was attained during Low pressure dosing (either fixed dose mode or incremental dose mode).

    Action: The analysis was canceled. All previously collected data were stored.

6517-   Unit (number) Analysis canceled: Total volume dosed greater than (volume) cm3 is not allowed.

    Cause A: More than (volume) cm^3 has been dosed onto the sample, possibly due to leaks in the system.

    Action A: Perform leak checks on the system. Refer to Appendix E, Testing for Leaks.

Cause B: The value for the Po may be too low, causing the instrument to mistake condensation for actual adsorption.

Action B: Change the analysis variables to directly measure the value of Po during the experiment.

Cause C: The amount of sample you used may be too large. Multiply the maximum value from the Volume Adsorbed column of the Isotherm Report by the sample weight. If the result exceeds (volume) cm^3, the amount of sample is too large.

Action C: Reduce the amount of sample.

6518-   Unit (number) Analysis canceled: Pressure of (pressure point) mmHg exceeds the maximum manifold pressure of (pressure point) mmHg.

Cause: An absolute pressure greater than (pressure) mmHg was attained that exceeded the specified maximum manifold pressure.

Action: The analysis was canceled. All previously collected data were stored. Change the maximum manifold pressure value in the Adsorptive Properties file.

6519-   Unit (number) Analysis canceled: Psat gas is not condensing.

Cause A: The working Dewar does not contain enough bath liquid.

Action A: Retry the operation after filling the Dewar.

Cause B: The Psat gas is contaminated.

Action B: Replace the Psat gas supply.

Cause C: The Psat tubing from the regulator to the instrument is contaminated.

Action C: Pump out the tubing.

6520-   Power failure detected.

Cause A:	Main power failed but UPS (if attached) is working.
Action A:	Restore power to the analyzer.
Cause B:	The value for the Po may be too low, causing the instrument to mistake condensation for actual adsorption.
Action B:	Change the analysis variables to directly measure the value of Po during the experiment.
Cause C:	The amount of sample you used may be too large. Multiply the maximum value from the Volume Adsorbed column of the Isotherm Report by the sample weight. If the result exceeds (volume) cm3, the amount of sample is too large.
Action C:	Reduce the amount of sample.

6522-   Unit (number) Analysis canceled: Time limit exceeded while evacuating manifold.

Cause A:	Maximum manifold evacuation time was exceeded before the vacuum set point was achieved. Vacuum pump may be turned off.
Action A:	Turn on vacuum pump switch. Then restart the analysis.
Cause B:	The vacuum pump oil level is low.
Action B:	Check the vacuum pump oil level and add more oil if necessary. Then restart the analysis.
Cause C:	The manifold is contaminated or leaking.
Action C:	Correct the problem. Refer to **Appendix D, Testing for Leaks**. Then restart the analysis.

6523- Unit (number) Analysis canceled: Time limit exceeded while evacuating sample (unrestricted).

Cause: The maximum time for evacuating the sample through the unrestricted valve was exceeded. Possible causes are a leak in the sample tube fitting or a crack in the sample tube.

Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Then restart the analysis.

6524- Unit (number) Analysis canceled: Time limit exceeded while evacuating sample (restricted).

Cause: The maximum time for evacuating the sample through the restricted valve was exceeded. Possible causes are a leak in the sample tube fitting, a crack in the sample tube, or a poorly degassed sample.

Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Verify that the sample is properly degassed; then restart the analysis

6525- Power failure lasted too long.

Cause: A power failure on an analyzer which has an Uninteruptible Power Supply attached has lasted for an hour or more; therefore, the analysis has been canceled.

Action: Determine the cause of the power failure and correct it.

6526- Time limit exceeded while backfilling manifold to [PR4] [PR-U] with [G].

Cause A: The maximum time was exceeded before the target pressure point was reached. The gas regulator may be set too low or turned off.

Action A: Set the gas regulator to 150 psig. Then resume the analysis.

Cause B: The gas bottle is empty.

Action B: Connect a new gas bottle. Then resume the analysis.

6527 - Unit (number) Analysis suspended: Time limit exceeded while dosing manifold to (target pressure point) mmHg with (gas name).

Cause A: The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.

Action A: Set the analysis gas regulator to 150 psig. Then resume the analysis.

Cause B: The analysis gas bottle is empty.

Action B: Connect a new analysis gas bottle. Then resume the analysis.

6529-   Master pressure gauge offset is too high - [PR1] [PR-U].

Cause: A check of the Master gauge's offset during an automatic operation indicated it was too high.

Action: If this message occurs repeatedly, contact a Micromeritics service representative

6530-   Volume calibration canceled due to failure (code [0]).

Cause: A problem occurred during volume calibration.

Action: Contact a Micromeritics service representative.

6531-   The gas configuration file for (unit number) is invalid.

Cause: The contents of the gas configuration file are not valid.

Action A: Exit the application, navigate to the application hardware directory, and delete the <S/N>.SST file. Then restart the application and reassign gasses.

Action B: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists, contact a Micromeritics service representative.

6532-   The instrument is currently running the Physi software. Do you want to reset it?

Cause: The analyzer is under the control of the 2050 Physisorption software.

Action: Selecting Yes will reset the analyzer and download the 2050 Chemisorption control software.

6534-   Instrument (unit number) is not calibrated.

Cause: Calibration information for various analyzer components are missing.

Action: Run the application setup program and reinstall calibration information for the specified unit.

6535-   Problem encountered evacuating.

Cause: Evacuation during the manifold dosing operation did not come within the allowed range of the target.

Action: Check that the outlet stage of the gas regulator is within specification. If the problem occurs frequently, contact a Micromeritics service representative.

6536-   There is no Nitrogen attached to the unit.

    Cause: A volume calibration has been requested but N2 is not one of the gases selected in the unit/gas configuration.

    Action: Make sure the nitrogen cylinder is attached to the unit and edit unit configuration to match the gases that are present.

6537-   Error reading SmartVac ADC.

    Cause: There was a problem reading one of the signals on the SmartVac board.

    Action: Exit the application, turn off the power switch on the analyzer, then turn the power back on. Restart the application and the degas operation. If the problem persists, contact your service representative.

6538-   Power failure detected. The sample is in an unknown condition. A run termination will be performed for safety.

    Cause: A total power failure occurred (a UPS was not connected) while an analysis was in progress.

    Action: Allow the analysis to terminate.

6539-   Error overheating Current = (no.), Target = (no.), Limit = (no.)

    Cause A: The heating mantle is reporting a higher temperature than expected.

    Action A: Ensure that the heating mantle and thermocouple cables are plugged in fully on the port(s).

    Cause B: An internal failure of the degas system may have occurred.

    Action B: Unplug the heating mantle power connector; do not unplug the thermocouple. Allow the heating mantle to cool down (approximately 15-30 minutes). Then exit the analysis program and turn off the analyzer. Wait a couple of minutes, then turn the instrument on and restart the program. If the problem recurs, contact your Micromeritics service representative.

C. CALCULATIONS

Saturation Pressure (Po)

Po method is selected by the user on the Po and Temperature Options dialog box. It may be entered, measured, or calculated from temperature.

If entered, Po = user entered value

If measured, Po = equilibrated pressure reading after saturating Po tube

If calculated from temperature, interpolate Po from the Psat vs Temperature table on the Po and Temperature Options dialog box, given the analysis bath temperature

Relative Pressure

If Po was measured:

For points taken before the previous Po measurement,

$$Po_I = Po_1 + \frac{(Po_2 - Po_1) \times (T_I - To_1)}{(To_2 - To_1)}$$

For points taken after the previous Po, use the previous Po:

$$Po_I = Po_1$$

If Po was entered, use the entered Po:

$$Po_I = Po_E$$

If Po was calculated, use the calculated Po:

$$Po_I = Po_C$$

Calculate relative pressure for the Ith data point:

$$P_{rel_1} = \frac{P_1}{Po_I}$$

where

Po_I =   saturation pressure for the Ith data point (mmHg)

Po_1 =   most previous measured saturation pressure before I^{th} data point (mmHg)

Po_2 =   first measured saturation pressure after the Ith data point (mmHg)

Po_C =   calculated Po

Po_E =   entered saturation pressure (mmHg)

T_I =   time when the Ith data point was taken (minutes)

To_1 =   time when Po_1 was measured (minutes)

To_2 =   time when Po_2 was measured (minutes)

P_{rel_I} =   relative pressure for the Ith data point (mmHg)

P_I =   absolute pressure for the Ith data point, taken at equilibrium (mmHg)

Free Space

Free-space volumes are calculated using the following equations:

$$V_{FW} = \frac{V_{LOW}}{T_1} \times \left[\frac{P_1}{P_2} - 1 \right] \times T_{STD}$$

$$V_{FC} = \frac{V_{LOW}}{T_2} \times \left[\frac{P_1}{P_3} - 1 \right] \times T_{STD}$$

$$V_{ABT} = \frac{V_{FC} - V_{FW}}{1 - \dfrac{T_{ABT}}{T_{RM}}}$$

where:\

P_1 =   system manifold pressure before dosing helium onto sample (mmHg)

P_2 =   system manifold pressure after dosing helium onto sample (mmHg)

P_3 =   sample pressure after raising Dewar and equilibrating with helium (mmHg)

T_{STD} =   standard temperature (273.13 K)

T_{RM} =   approximate room temperature (298 K)

T_1 =   system manifold temperature before dosing helium onto sample (K)

T_2 =   system manifold temperature after raising Dewar and equilibrating with helium (K)

V_{LOW} =   lower manifold volume (cm^3)

V_{FC} =   volume of free space, cold (cm^3 at standard temperature)

V_{FW} = volume of free space, warm (cm^3 at standard temperature)

V_{ABT} = portion of cold free space at analysis bath temperature: (cm^3 at standard temperature)

T_{ABT} = analysis bath temperature (K), from the Po and Temperature Options dialog box

Volume Adsorbed

For the Ith dose:

$$Ngas_I = Ngas_{I-1} + V_{LOW} \times \left[\frac{Psys1_I}{Tsys1_I} - \frac{Psys2_I}{Tsys2_I}\right] \times \frac{T_{STD}}{P_{STD}}$$

$$Nads_I = Ngas_I - \frac{Psam_I}{P_{STD}}\left(\frac{V_{ABT}}{Z_{FC}} + \frac{V_{FA}}{Z_{FA}}\right)$$

$$V_I = \frac{Nads_I}{Wsam}$$

where:

$Ngas_I$ = total amount of gas dosed into sample tube after Ith dose (cm^3 STP)

$Nads_I$ = amount of gas adsorbed after equilibrating Ith dose (cm^3 STP)

P_{STD} = standard pressure (760 mmHg)

$Psys1_I$ = system manifold pressure before Ith dose of gas onto sample (mmHg)

$Psys2_I$ = system manifold pressure after Ith dose of gas onto sample (mmHg)

$Psam_I$ = sample pressure after equilibrating Ith dose of gas onto sample (mmHg)

$Tsys1_I$ = system manifold temperature before Ith dose of gas onto sample (K)

$Tsys2_I$ = system manifold temperature after Ith dose of gas onto sample (K)

$Wsam$ = weight of sample (g)

V_I = amount of gas adsorbed per gram of sample (cm^3/g STP)

Z_{FC} = compressibility factor for the analysis gas at $Psam_I$ and the analysis temperature

Z_{FA} = compressibility factor for the analysis gas at $Psam_I$ and the ambient temperature

V_{FA} = volume of the sample tube at ambient temperature $V_{FC} - V_{ABT}$

Equilibration

Equilibration is reached when the pressure change per equilibration time interval (first derivative) is less than 0.01% of the average pressure during the interval. Both the first derivative and average pressure are calculated using the Savitzky-Golay[1] convolution method for polynomial functions. The equations below are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

$$P_{AVG} = \frac{-36(P_{11} + P_1) + 9(P_{10} + P_2) + 44(P_9 + P_3) + 69(P_8 + P_4) + 84(P_7 + P_5) + 89(P_6)}{429}$$

$$P_{CHG} = \frac{5(P_{11} - P_1) + 4(P_{10} - P_2) + 3(P_9 - P_3) + 2(P_8 - P_4) + (P_7 - P_5)}{110}$$

where the numerical constants are from the Savitzky-Golay convolution arrays, and

P_{AVG} =   average pressure (mmHg)
P_{CHG} =   change in pressure (mmHg)
P_I = I^{th} pressure reading taken at equilibrium intervals (mmHg)

After equilibration has been reached, if the user-entered minimum equilibration time from the Low Pressure Options dialog box has not elapsed, the equilibration continues until the entered time has elapsed.

If the user entered a non-zero maximum equilibration time from the Low Pressure Options dialog box, and this time period has elapsed before equilibration has been reached, the equilibration ends as if equilibration had been reached, and the point is collected.

> **If a non-zero value that is too small is entered for the maximum equilibration time, the points are collected before equilibration is reached.**

> **If P_{AVG} is greater than 0.995 times the current Po, equilibration will not take place until the *Minimum equilibration delay for P/Po 0.995* has expired, in addition to the standard equilibration criteria.**

Thermal Transpiration Correction

During data reduction, thermal transpiration correction is applied to the data if the user selected **Thermal transpiration correction** from the Report Options dialog box. Starting with the first collected pressure, the following calculations are performed until the pressure ratio (PC/P) is greater than or equal to 0.99.

$$Y = \frac{P \times SD \times MD^2}{2.33 \times T} \times 10^3$$

$$\mu = \frac{1 + G \times Y}{1 + H \times Y}$$

$$F = \frac{1}{(\alpha \times Y^2) + (\beta \times Y) + \mu}$$

$$PC = \left\{ 1 - \left[F - \left(F \left(\frac{T_{ABT}}{T_{RM}} \right)^{0.5} \right) \right] \right\} \times P$$

where:

P	=	equilibrated collected pressure measured by gauge at temp T_{RM} (mmHg)
SD	=	inside diameter of sample tube (mm), from the Report Options dialog box
MD	=	thermal transpiration hard sphere diameter of gas (Å), from the Adsorptive Properties dialog box
T	=	average temperature $[T_{ABT} + T_{RM}]$ / 2 (K)
α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
G	=	Weber's coefficient, 2.5
H	=	Weber's coefficient, 2
T_{ABT}	=	analysis bath temperature (K), from the Po and Temperature Options dialog box
T_{RM}	=	room temperature (298 K)
PC	=	corrected equilibrated pressure at temperature T_{ABT} (mmHg)
F,Y,μ	=	intermediate values for subsequent calculations

BET Surface Area

For each point designated for surface area calculations, the BET[2] transformation is calculated as follows:

$$B_1 = \frac{P_{rel_1}}{(1.0 - P_{rel_1}) \times N_{ads_1}}$$

where B_I is in units of g/cm^3 STP.

A least-squares fit is performed on the (P_{relI}, B_I) designated pairs where P_{relI} is the independent variable and B_I is the dependent variable. The following are calculated:

    a.   Slope (S g/cm^3 STP)

    b.   Y-intercept (Y_{INT} g/cm^3 STP)

    c.   Error of the slope (S_{ERR} g/cm^3 STP)

    d.   Error of the y-intercept (YI_{ERR} g/cm^3 STP)

    e.   Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

BET Surface Area (m^2/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414 \ cm^3 \ STP) \times (10^{18} \ nm^2/m^2) \times (S + Y_{INT})}$$

where

    CSA   =   analysis gas molecular cross-sectional area (nm^2), user-entered on the Adsorptive Properties dialog box

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

Error of the BET Surface Area (m²/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}{}^2 + YI_{ERR}{}^2)^{0.5}}{Y_{INT} + S}$$

Langmuir Surface Area

For each point designated for surface area calculations, the Langmuir[3] transformation is calculated as follows:

$$L_I = \frac{P_{rel_I}}{N_{ads_I}}$$

where L_I is in units of g/cm³ STP

A least-squares fit is performed on the (P_{rel_I}, L_I) designated pairs where P_{rel_I} is the independent variable and L_I is the dependent variable. The following are calculated:

   a.   Slope (S g/cm³ STP)

   b.   Y-intercept (Y_{INT} g/cm³ STP)

   c.   Error of the slope (S_{ERR} g/cm³ STP)

   d.   Error of the y-intercept (YI_{ERR} g/cm³ STP)

   e.   Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Langmuir Surface Area (m²/g):

$$SA_{LAN} = \frac{CSA \times (6.023 \times 10^{23})}{(22414 \ cm^3 \ STP) \times (10^{18} \ nm^2/m^2) \times S}$$

where

   CSA   =   analysis gas molecular cross-sectional area (nm²), user-entered on the
               Adsorptive Properties dialog box

Volume of the Monolayer (cm³/g STP):

$$V_M = \frac{1}{S}$$

Langmuir b Value:

$$b = (Y_{INT})(V_M)$$

Error of the Langmuir Surface Area (m²/g):

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

t-Plot

For each point designated for t-Plot[4] calculations, the following calculations are done:

Thickness for the Ith point (Å):

$$t_I = HP1 \times \left[\frac{HP2}{\ln(P_{rel_I})} \right]^{HP3} \qquad \text{(Halsey[5])}$$

or

$$t_I = \left[\frac{HJP1}{HJP2 - \log(P_{rel_I})} \right]^{HJP3} \qquad \text{(Harkins and Jura[6])}$$

where:

t_I	=	thickness for I[th] point
HP1	=	Halsey parameter #1
HP2	=	Halsey parameter #2
HP3	=	Halsey parameter #3
HJP1	=	Harkins and Jura parameter #1
HJP2	=	Harkins and Jura parameter #2
HJP3	=	Harkins and Jura parameter #3
P_{relI}	=	relative pressure for the Ith point (mmHg)

A least-squares analysis fit is performed on the (t_I, N_{adsI}) data pairs where t_I is the independent variable and $Nads_I$ is the dependent variable. Only the values of t_I between t_{MIN} and t_{MAX}, the minimum and maximum thickness, are used. The following are calculated:

a. Slope (S cm³/g-Å STP)

b. Y-intercept (Y_{INT} cm³/g STP)

c. Error of the slope (S_{ERR} cm³/g-Å STP)

d. Error of the Y-intercept (YI_{ERR} cm³/g STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

External Surface Area (m²/g):

$$SA_{EXT} = \frac{(S\ cm^3/g - A\ STP) \times (10^{10}\ A/m) \times (D\ cm^3\ liquid/cm^3\ STP)}{F \times (10^6\ cm^3/m^3)}$$

where

> F   =   surface area correction factor, user-entered on the t-Plot Report Options dialog box
>
> D   =   density conversion factor, user-entered on the Adsorptive Properties dialog box

Micropore Surface Area (m^2/g):

$$SA_{\mu P} \; = \; SA_{TOT} + SA_{EXT}$$

where SA_{TOT} is the BET surface area if the user enabled the BET report exclusively, or Langmuir surface area if the user enabled the Langmuir report exclusively. If neither report has been selected, SA_{TOT} is the BET surface area value calculated using a set of default parameters.

Micropore Volume (cm^3 liquid/g):

$$V_{\mu P} \; = \; (Y_{INT} \; cm^3/g \; STP) \times (D \; cm^3 \; liquid/cm^3 \; STP)$$

BJH Pore Volume and Area Distribution

For adsorption data, the relative pressure and volume adsorbed data point pairs collected during an analysis must be arranged in reverse order from which the points were collected during analysis. All calculations are performed based on a desorption model, regardless of whether adsorption or desorption data are being used.

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (Pr), volume adsorbed (Va) pairs from (Pr_1, Va_1) to (Pr_N, Va_N) where $(Pr_N = 0, Va_N = 0)$ is assumed as a final point. Each data pair represents an interval boundary (or desorption step boundary) for intervals i=1 to i=N-1 where N = total number of (Pr, Va) pairs.

Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin[7] equation. After the core has evaporated, a layer of adsorbate remains on the wall of the pore. The thickness of this layer is calculated for a particular relative pressure from the thickness equation. This layer becomes thinner with successive decreases in pressure, so that the measured quantity of gas desorbed in a step is composed of a quantity equivalent to the liquid cores evaporated in that step plus the quantity desorbed from the pore walls of pores whose cores have been evaporated in that and previous steps. Barrett, Joyner, and Halenda[8] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used on the ASAP 2020 is an implementation of the BJH method.

Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

a. The *core* - evaporates all at once when the critical pressure for that radius is reached; the relationship between the core radius and the critical pressure is defined by the Kelvin equation.

b. The *adsorbed layer* - composed of adsorbed gas that is stripped off a bit at a time with each pressure step; the relationship between the thickness of the layer and the relative pressure is defined by the thickness equation.

c. The ***walls of the cylindrical pore*** - the diameter of the empty pore is required to determine the pore volume and pore area. End area is neglected.
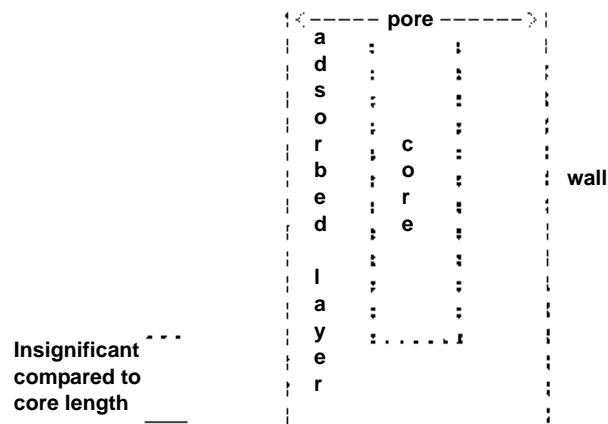
Calculations

The volumes adsorbed (Va) are converted to the liquid equivalent volumes (V1, cm^3/g):

$$VI_I = (Va_I)(D)$$

where D is the Density Conversion Factor from the Adsorptive Properties dialog box.

The relative pressure (Pr$_1$) is assumed to be close to unity so that substantially all the pores in the sample are filled.

The corresponding Kelvin core radius is calculated. Only pores smaller than this size will be included:

$$Rc_I = \frac{-A}{(1 + F)[\ln(Pr_I)]}$$

where

A   = adsorbate property factor (the BJH Adsorptive Options dialog box)
F   = fraction of pores open at both ends (from the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box); assumed to be zero for desorption

This radius will be adjusted for the thickness of the adsorbed layer during subsequent calculation steps.

The thickness of the remaining adsorbed layer at this relative pressure is calculated:

$$Tw_1 = HP1\left[\frac{HP2}{\ln(Pr_1)}\right]^{HP3}$$

where

HP1, HP2, and HP3 are Halsey Parameters 1, 2, and 3 (respectively) from the Halsey Thickness Equation dialog box.

These calculations illustrate the use of the Halsey thickness equation. If the Harkins/Jura equation was selected, substitute the following wherever the thickness equation appears:

$$Tw_1 = \left[\frac{HJ1}{HJ2 - \log(Pr_1)}\right]^{HJ3}$$

where

HJ1, HJ2, and HJ3 are Harkins-Jura Parameters 1, 2, and 3 (respectively) from the Harkins-Jura Thickness Equation dialog box.

The following calculations (a-c) are made for each relative pressure interval based on the increment of volume desorbed during that interval. The variable I refers to the interval number, that is I=1 for the first interval from Pr_1 to Pr_2, and so on. J refers to each previous interval during which new pores were found. K refers to the total number of intervals in which new pores have been found. K is also the number of lines reported on the BJH table for collected data.

    a.   The thickness of the adsorbed layer at the end of the interval is calculated as follows:

$$Tw_{I+1} = HP1\left[\frac{HP2}{\ln(Pr_{I+1})}\right]^{HP3}$$

(For the last pressure interval from the lowest Pr_I to zero relative pressure, $Tw_{I+1} = 0$.)

For the first pressure interval, there are no previously opened pores so the volume desorbed from walls of previously opened pores is zero ($Vd_1 = 0$), and the remainder of Step a is skipped.

The change in thickness of the wall layer due to desorption from preivously opened pores is calculated:

$$\Delta Tw = Tw_1 - Tw_{I+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all pre-
viously opened pores:

$$CSA_J = \pi[(Rc_j + \Delta Tw)^2 - Rc_j{}^2](10^{-16}\ cm^2/A^2)$$

The total volume of gas desorbed from walls of previously opened pores is
calculated:

$$Vd_I = \sum(Lp_J)(CSAa_J)\ \text{for all previously opened pores}$$

where LP_J = length of previously opened pores as calculated in Step b(2).

b. The physical processes occurring for this pressure interval are determined as follows:

(1) If Vd_I is greater than the current increment of volume desorbed (Vl_I - Vl_{I+1}),
    desorption from walls only is occurring. Total surface of walls exposed thus far
    (cm^2/g) is calculated as follows:

$$SA_W = \sum\pi(LP_J)(D_{avg_J})(10^{-8}\ cm/A)\quad \text{for all previously opened pores}$$

where

D_{avgJ} = weighted average pore diameter calculated below in Step b(2).

A new layer thickness (ΔTw) that will not overcompensate for the actual volume
desorbed in this interval is calculated:

$$\Delta Tw = \frac{(V1_I - V1_{I+1})(10^8\ A/cm)}{SAw_I}$$

Since no cores are evaporated in this pressure interval, no new pores are revealed.
Thus no ending Kelvin radius and average pore diameter are calculated for this
interval. Note that this means the report may have fewer tabulated intervals on
the collected data report than experimental pressure intervals.

(2) If Vd_I is less than the volume increment desorbed during this interval

$(Vl_1 - Vl_{l+1})$, the remaining volume is due to new pores with core evaporation
taking place in this interval. K, the number of intervals with new pores exposed,
is increased by 1. (For the interval from the lowest Pr_1 to zero relative pressure,
no new pore volume is calculated and the rest of Step b is skipped.)

The volume desorbed from newly opened pores in this interval is calculated as
follows:

$$Vc_I = (VI_I - VI_{I+1}) - Vd_I$$

The Kelvin radius for the end of the interval is calculated as follows:

$$Rc_{K+1} = \frac{-A}{(1+F)[\ln(Pr_{I+1})]}$$

All new pores opened in this interval are represented by one pore having a length-weighted average pore diameter and a corresponding length sufficient to account for the required volume of adsorbate. The weighted average pore diameter is calculated as follows:

$$D_{avg_K} = \frac{(2)(Rc_K + Rc_{K+1})(Rc_K)(Rc_{K+1})}{Rc_K{}^2 + Rc_{K+1}{}^2}$$

D_{avgK} is the diameter of a pore which would have a surface area that is the average of the areas for pores radius Rc_K and Rc_{K+1}, if its length was the mean of the lengths at those radii.

The relative pressure corresponding to is calculated as follows:

$$P_{avg_K} = \ln^{-1}\left[\frac{-2A}{(1+F)(D_{avg_K})}\right]$$

The thickness of the adsorbed layer at this pressure is calculated as follows:

$$Tw_{avg_K} = HP1\left[\frac{HP2}{\ln(P_{avg_K})}\right]^{HP3}$$

The decrease in thickness of the wall layer by desorption from the walls of new pores during the lower portion of the pressure interval is calculated as follows:

$$\Delta Td = Tw_{avg_K} - TW_{I+1}$$

The cross-sectional area of the newly opened pores is calculated as follows:

$$CSAc_K = \left[\frac{Davg_K}{2} + \Delta Td\right]^2 (10^{-16}\ cm^2/A^2)$$

The length of the newly opened pores is calculated as follows:

$$LP_K = \frac{Vc_I}{CSAc_K}$$

c. Pore diameters and radii are adjusted for the change in thickness of the adsorbed wall layer during this interval. If new pores were opened during this interval, the average diameter is adjusted by the change in layer thickness during the second portion of the desorption interval as follows:

$$D_{avg_{Knew}} = D_{avg_{Kold}} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as follows:

$$D_{avg_{Jnew}} = D_{avg_{Jold}} + 2(\Delta Tw)$$

(not including D_{avgK})

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as follows:

$$Rc_{Jnew} = Rc_{Jold} + \Delta Tw$$

for all except Rc_{K+1}.

Steps a to c are repeated for each pressure interval.

After the above calculations have been performed, the diameters corresponding to the ends of the intervals are calculated as follows:

$$Dp_J = 2(rc_J)$$

for all Rc_J including Rc_{K+1}.

The remaining calculations are based on $DP_{(I)}$, $D_{avg(I)}$, and $LP_{(I)}$. These calculations are only done for $D_{avg(I)}$ values that fall between the Minimum BJH diameter and the Maximum BJH diameter specified by the operator on the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box.

1) Incremental Pore Volume (Vp_I, cm^3/g):

$$Vp_I = \pi(Lp_I)\left[\frac{D_{avg_I}}{2}\right]^2 10^{16} \ cm^2/A^2$$

2) Cumulative Pore Volume ($Vp_{CUM(1)}$, cm^3/g):

$$VP_{CUM_{(1)}} = \sum Vp_J \, for \, (J \le 1)$$

3) Incremental Surface Area (SA_I, m^2/g):

$$SA_I = \pi(LP_I)(10^{-2} \, m/cm)(D_{avg_I})(10^{-10} \, m/A)$$

4) Cumulative Surface Area ($SA_{CUM(I)}$, m^2/g):

$$SA_{CUM_{10}} = \sum SA_J \, for \, J \le 1$$

5) dV/dD pore volume (dV/dD_I, cm^3/g-A):

$$dV/dD_I = \frac{VP_I}{Dp_I - Dp_{I+1}}$$

6) dV/dlog(D) pore volume ($dV/dlog(D)_I$, $cm^3 g$):

$$lDv/d\log(D)_I = VP_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right)$$

7) dA/dD pore area (dA/dD_I, m^2/g-A):

$$dA/dD_I = \frac{SA_I}{Dp_I - Dp_{I+1}}$$

8) dA/dlog(D) pore area [$dA/dlog(D)_I$, m^2/g]:

$$dA/d\log(D)_I = SA_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right)$$

For fixed pore size tables (if selected), the following calculations are performed:

1) Average Fixed Pore Size (DF_{avgJ}, A):

$$DF_{avg_J} = \frac{Dp_{F_J} + Dp_{F_{J+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with between the Minimum BJH diameter and the Maximum BJH diameter.

2) Cumulative Pore volume (VpF_{CUMI}, cm^3/g):

$$VpF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = Dp_{J+1}$ and $Y = VP_{CUMJ}$, using an AKIMA semi-spline interpolation.

3) Incremental Pore Volume (VpF_I, cm^3/g):

$$VpF_I = VpF_{CUM_I} - VpF_{CUM_{I-1}}$$

where $VpF_{CUM0} = 0$.

4) Cumulative Surface Area (SAF_{CUMI}, m^2/g):

$$SAF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = Dp_{J+1}$ and $Y = SA_{CUMJ}$.

5) Incremental Surface Area (SAF_I, m^2/g):

$$SAF_I = SAF_{CUM_I} - SAF_{CUM_{I-1}}$$

where $SAF_{CUM0} = 0$.

6) dV/dD pore volume ($dV/dDpF_I$, $cm^3/g\text{-}A$)):

$$dV/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = D_{avgJ}$ and $Y = dV/dD_J$.

7) dV/dlog(D) pore volume [$dV/dlog(DpF)_I$, cm^3/g]:

$$dV/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = D_{avgJ}$ and $Y = dV/dlog(D)_J$.

8) dA/dD pore area (dA/dDpF$_I$, m^2/g-A):

$$dA/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D$_{avgJ}$ and Y = dA/dD$_J$.

9) dA/dlog(D) pore area [dA/dlog(DpF$_I$), m^2/g]:

$$lA/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D$_{avgJ}$ and Y = dA/dlog(D)$_J$.

Compendium of Variables

Va	=	quantity adsorbed expressed as a volume (cm^3/g STP)
Vl	=	liquid equivalent volume of volume adsorbed (cm^3/g)
D	=	density conversion factor from the Adsorptive Properties dialog box (cm^3/cm^3 STP)
Pr	=	relative pressure
Dp	=	pore (or core) diameter (A)
Rc	=	Kelvin radius (A) of core
A	=	adsorbate property factor; from the BJH Adsorptive Options dialog box
F	=	fraction of pores open at both ends; from the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box
ΔTw	=	thickness of adsorbed layer desorbed during interval (A)
Tw	=	thickness of remaining adsorbed wall (A)

HP1, HP2, and HP3 are Halsey Parameters from the Halsey Thickness Equation dialog box.

HJ1, HJ2, and HJ3 are Harkins and Jura Parameters from the Harkins/Jura Thickness Equation dialog box.

Vd	=	volume of gas desorbed from walls of previously opened pores (cm^3/g)
Davg	=	average pore diameter (A)
CSAa	=	annular cross-sectional area of the desorbed layer (cm^2)
CSAc	=	cross-sectional area of opening of newly opened pores (cm^2)
SAw	=	total surface area of walls exposed (cm^2/g)
ΔTd	=	thickness of layer desorbed from walls of newly opened pores (A)
Vc	=	volume desorbed from cores of newly opened pores (cm^3/g)
Lp	=	length of pore (cm/g)

Horvath-Kawazoe

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/Po) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito/Foley)
- Sphere Pore Geometry (Cheng/Yang)

Slit Pore Geometry (original HK)

When you use the original Horvath-Kawazoe[9] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\frac{P}{Po} = \frac{K}{RT} \times \frac{IP \times 10^{32} \ JA^4/J \ cm^4}{\sigma^4 \times (L - 2 \times d_0)} \times \left[\frac{\sigma^4}{3 \times (L - d_0)^3} - \frac{\sigma^{10}}{9 \times (L - d_0)^9} - \frac{\sigma^4}{3 \times d_0^3} + \frac{\sigma^{10}}{9 \times d_0^9} \right]$$

where

K	=	Avogadro's number (6.023 x 10^{23})
R	=	gas constant (8.31441 x 10^7 ergs/mole K)
T	=	analysis bath temperature (K), from an entered or calculated value on the Po and Temperature Options dialog box
σ	=	gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

where:

Z_S = sample equilibrium diameter at zero interaction energy (Å) from the Horvath-Kawazoe Physical Properties dialog box

Z_A = zero interaction energy diameter from the Horvath-Kawazoe Physical Properties dialog box

$d_0 \quad = \quad \dfrac{D_A + D_S(A)}{2}$

where:

D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical Properties dialog box

D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical dialog box

L   =   pore width (nucleus to nucleus) (Å)
P   =   equilibrium pressure (mmHg)
Po   =   saturation pressure (mmHg)
IP   =   interaction parameter (10^{-43} ergs-cm^4) from the Horvath-Kawazoe
               Report Options dialog box

Cylinder Pore Geometry (Saito/Foley)

When you use the Saito-Foley[10] method, the following equation is solved for each value of P.
The value of L is determined when the solved-for relative pressure is within 0.1% of the
collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{3}{4}\frac{\pi K}{RT} \times \frac{IP \times 10^{32}\ JA^4/J\ cm^4}{d_0^{\ 4}} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1 - \frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} - \beta_k\left(\frac{d_0}{r_p}\right)^{4}\right\}\right]$$

where

K   =   Avogadro's number (6.023×10^{23})
R   =   gas constant (8.31441×10^7 ergs/mole K)
T   =   analysis bath temperature (K), from an entered or calculated
               value on the Po and Temperature Options dialog box
L   =   pore width (nucleus to nucleus) (Å)
P   =   equilibrium pressure (mmHg)
Po   =   saturation pressure (mmHg)
IP   =   interaction parameter (10^{-43} ergs-cm^4) from the Horvath-Kawazoe Report
               Options dialog box
d_0 = $\dfrac{D_A + D_S(A)}{2}$

          where:
          D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical
                Properties dialog box
          D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical
                dialog box

α_k = $\left(\dfrac{-4.5-k}{k}\right)^2 \alpha_{k-1}, \alpha_0 = 1.0$

β_k = $\left(\dfrac{-1.5-k}{k}\right)^2 \beta_{k-1}, \beta_0 = 1.0$

r_p =   radius of the cylindrical pore, $\dfrac{L}{2}$

Sphere Pore Geometry (Cheng/Yang)

When you use the Cheng/Yang[11] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{6(N_1\varepsilon^*{}_{12} + N_2\varepsilon^*{}_{22})L^3 \times 10^{32} \ J \ A^4/J \ cm^4}{RT(L - d_0)^3}$$

$$\left[-\left(\frac{d_0}{L}\right)^6\left(\frac{1}{12}T_1 + \frac{1}{8}T_2\right) + \left(\frac{d_0}{L}\right)^{12}\left(\frac{1}{90}T_3 + \frac{1}{80}T_4\right)\right]$$

where

R   = gas constant (8.31441×10^7 ergs/mole K)

T   = analysis bath temperature (K), from an entered or calculated value on the Po and Temperature Options dialog

d_0 $= \dfrac{D_A + D_S(A)}{2}$

where:

D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical Properties dialog

D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical dialog

L   = pore width (nucleus to nucleus) (Å)

P   = equilibrium pressure (mmHg)

Po   = saturation pressure (mmHg)

N_1 $= 4\pi \, L^2 \, N_S$, where N_S = number of sample atoms/cm^2 at monolayer

N_2 $= 4\pi \, (L - d_0)^2 \, N_A$, where N_s = number of gas molecules/cm^2

$e^*{}_{12}$ $= \dfrac{A_S}{4d_S{}^6}$, where $A_S = \dfrac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\dfrac{\alpha_S}{\chi_S} + \dfrac{\alpha_A}{\chi_A}}$

$\varepsilon^*{}_{22}$ $= \dfrac{A_A}{4D_A{}^6}$, where $A_A = \dfrac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$

T_1 $= \dfrac{1}{(1 - S)^3} - \dfrac{1}{(1 + S)^3}$

T_2 $= \dfrac{1}{(1 + S)^2} - \dfrac{1}{(1 - S)^2}$

T_3 $= \dfrac{1}{(1 - S)^9} - \dfrac{1}{(1 + S)^9}$

$$T_4 = \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8}$$

$$\text{where} \quad S = \frac{L - d_0}{L}$$

Cheng/Yang Correction

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometrics:

$$\ln\left(\frac{P}{Po}\right) = G(L) - \left[1 - \frac{1}{\theta}\ln\left(\frac{1}{1-\theta}\right)\right]$$

where

G(L)	=	one of the Horvath-Kawazoe equations given above
θ	=	degree of void filling; θ is estimated by first computing the monolayer capacity (V_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the volume adsorbed over V_m.

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients -

$$A_S = \frac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\dfrac{\alpha_S}{\chi_S} + \dfrac{\alpha_A}{\chi_A}}$$

$$A_A = \frac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$$

where

MC^2	=	kinetic energy of electron (0.8183×10^{-6} erg)
α_S	=	polarizability of sample atoms (cm^3)
α_A	=	polarizability of gas molecule (cm^3)
χ_S	=	diamagnetic susceptability of sample atom (cm^3)

χ_A = diamagnetic susceptability of gas molecule (cm³)

$$IP \ = \ (N_A \times A_A) + (N_S \times A_S)$$

where:

N_A = number of gas molecules/cm² at monolayer from the Horvath-Kawazoe
               Physical Properties dialog

N_S = number of sample atoms/cm² from the Horvath-Kawazoe Physical Properties
               dialog

Refer to **Interaction Parameter Components** later in this Appendix for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):
   (Shell to Shell)

$$AL_I \ = \ L_I - DS$$

Cumulative Pore Volume (cm³/g):

$$V_{CUM_I} \ = \ V_I \times D$$

where

D   = density conversion factor (cm³ liquid/cm³ STP) on the Adsorptive Properties
           dialog box

dV/dD Pore Volume (cm³/g-Å):

$$\frac{dV}{dD_I} \ = \ \frac{V_{CUM_I} - V_{CUM_{I-1}}}{AL_I - AL_{I-1}}$$

Median Pore Width (Å):

$$V_{HALF} \;=\; \frac{V_{CUM_N}}{2}$$

$$D_{MED} \;=\; 10\!\left[\log(D_L) + [\log(V_{HALF}) - \log(V_L)] \times \frac{\log(D_G) - \log(D_L)}{\log(V_G) - \log(V_L)}\right]$$

where

V_{CUMN} = total cumulative pore volume (V_{CUMI}) for points designated for Horvath-Kawazoe calculations

V_{HALF} = 50% of total cumulative pore volume

V_L = cumulative pore volume (V_{CUMI}) for first point less than V_{HALF}

V_G = cumulative pore volume (V_{CUMI}) for first point greater than V_{HALF}

D_L = pore width (L_I) that corresponds to V_L

D_G = pore width (L_I) that corresponds to V_G

Interaction Parameter Components

Table C-1. Interaction Parameters

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value*
Argon	87.3	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.61 5.89 3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.20 9.20 5.08
	273.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.34 9.35 5.22
	194.65	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.72 9.72 5.60
Nitrogen	77.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.84 6.53 3.49

*The interaction parameter is entered in the Horvath-Kawazoe Report Options dialog in the following field:

Interaction parameter: (calculated value) x 10^{-43} ergs-cm^4

The following values were used to calculate the values in Table C-1.

Carbon-Graphite
D_S = 3.40
N_S = 3.845 x 10^{15}
χ_S = 1.05 x 10^{-29} (Ross/Olivier)
        13.5 x 10^{-29} (Horvath/Kawazoe, implicit)
α_S = 1.02 x 10^{-24}

Zeolite
D_S = 3.04
N_S = 3.75 x 10^{15}
χ_S = 1.94 x 10^{-29}
α_S = 0.85 x 10^{-24}

Nitrogen
D_A = 3.00
N_A = 6.71 x 10^{14}
χ_A = 3.6 x 10^{-29}
α_A = 1.76 x 10^{-24}

Argon
D_A = 2.95
N_A = 7.608 x 10^{14}
χ_A = 3.22 x 10^{-29}
α_A = 1.63 x 10^{-24}

Carbon Dioxide
$D_A = 3.23$
$N_A = 4.567 \times 10^{14}$ (25 ºC)
$\qquad 5.45 \times 10^{14}$ (0 ºC)
$\qquad 7.697 \times 10^{14}$ (-78 ºC)
$\chi_A = 5.0 \times 10^{-29}$
$\alpha_A = 2.7 \times 10^{-24}$

D_A values are from van der Waal's constant.
N_A values are from liquid densities.
χ and α values are derived from data found in Ross and Olivier[12].

The physical parameters referenced in Saito/Foley are as follows:

Aluminophosphate
$D_S = 2.60$
$N_S = 1.48 \times 10^{15}$
$\chi_S = 1.3 \times 10^{-29}$
$\alpha_S = 2.5 \times 10^{-24}$

Aluminosilicate
$D_S = 2.76$
$N_s = 1.31 \times 10^{15}$
$\chi_S = 1.3 \times 10^{-29}$
$\alpha_S = 2.5 \times 10^{-24}$

Dubinin-Radushkevich

The Dubinin-Radushkevich[13] equation is as follows:

$$\log(V) \;=\; \log(Vo) - \frac{B \times T^2}{\beta} \times \left[\log\frac{Po}{P}\right]^2$$

where:

V	=	volume adsorbed at equilibrium pressure (cm^3/g STP)
Vo	=	the micropore capacity (cm^3/g STP)
Po	=	saturation vapor pressure of gas at temperature T (mmHg)
P	=	equilibrium pressure (mmHg)
B	=	a constant
β	=	the affinity coefficient of analysis gas relative to Po gas (for this application β is taken to be 1)
T	=	analysis bath temperature (K), from the Po and Temperature Options dialog box

For each point designated for Dubinin-Radushkevich calculations, the following calculations are done:

$$LV = \log (V)$$

$$LP \;=\; \log\left(\frac{Po}{P}\right)^2$$

The intercept, log(Vo) can be found by performing a least-squares fit on the (LP,LV) designated pairs where LP is the independent variable and LV is the dependent variable. Assuming the adsorption of gas is restricted to a monolayer, Vo is the monolayer capacity. Based on this assumption, the following are calculated:

a. Slope (S cm^3/g STP)

b. Y-intercept (YI cm^3/g STP)

c. Error of the slope (S$_{ERR}$ cm^3/g STP)

d. Error of the y-intercept (YI$_{ERR}$ cm^3/g STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Monolayer Capacity (cm³/g STP):

$$Vo = 10^{YI}$$

Error of Monolayer Capacity (cm³/g STP):

$$Vo_{ERR} = Vo \times (10^{YI_{ERR}} - 1.0)$$

Micropore surface area (m²/g):

$$SDP = \frac{\sigma \times Vo \times (6.023 \times 10^{23})}{22414 \ cm^3 \times (10^{18} \ nm^2/m^2)}$$

where

σ   =   molecular cross sectional area of gas (nm²)from the Adsorptive Properties
              dialog box

Dubinin-Astakhov

The Dubinin-Astakhov equation is a follows:

$$\log(V) = \log(Vo) - \left[\frac{RT}{\beta E_0}\right]^N \times \left[\log\frac{Po}{P}\right]^N$$

where

V	=	volume adsorbed at equilibrium pressure (cm^3/g STP)
Vo	=	the micropore capacity (cm^3/g STP)
Po	=	saturation vapor pressure of gas at temperature T (mmHg)
P	=	equilibrium pressure (mmHg)
T	=	analysis bath temperature (K)
R	=	the gas constant (0.0083144 kJ/mol)
E_0	=	characteristic energy (kJ/mole)
N	=	Astakhov exponent, may be optimized or user entered from the Dubinin Report Options dialog box
β	=	the affinity coefficient of the analysis gas relative to the Po gas, from the Dubinin Adsorptive Options dialog box

For each point designated for Dubinin-Astakhov calculations, the following calculations are done:

$$LV = log(V)$$

$$LP = \left[\log\frac{Po}{P}\right]^N$$

A least-squares fit is performed on the (LP,LV) designated pairs where LP is the independent variable and LV is the dependent variable. If the user selected **yes** for the Optimize Astakhov Exponent prompt, a systematic search for the optimum value of N is conducted by recalculating the linear regression and selecting the value of N that gives the smallest standard error of the y-intercept. The exponent N is optimized to within 10^{-4}. If the optimum value for N is not found in this range, an exponent of 2 is used. The following are calculated:

a. Slope (S cm^3/g STP)

b. Y-intercept (YI cm^3/g STP)

c. Error of the slope (S_{ERR} cm^3/g STP)

d. Error of the y-intercept (YI_{ERR} cm^3/g STP)

e. Correlation coefficient (Cc)

f. Optimized Astakhov exponent (N)

Using the results of the above calculations, the following can be calculated:

Monolayer Capacity (cm³/g STP):

$$V_0 = 10^{YI}$$

Micropore Volume (cm³/g):

$$W_I = (V_I \times D)$$

where

D   = density conversion factor (cm³ liquid/cm³ STP) from the Adsorptive
        Properties dialog box

Limiting Micropore Volume (cm³/g):

$$W_0 = (V_0 \times D)$$

where

D   = density conversion factor (cm³ liquid/cm³ STP) from the Adsorptive
        Properties dialog box

Error of Limiting Micropore Volume (cm³/g):

$$W_{0ERR} = W_0 \times (10YI_{ERR} - 1.0)$$

Characteristic Energy (KJ/mole):

$$E = \frac{2.303 \times R \times T}{\beta(2.303 \times S)^{1/N}}$$

Modal Equivalent Pore Width (nm):

$$w_{mode} = 2 \times \left\{ \left[\frac{3N}{3N+1} \right]^{1/N} \times \left[\frac{k}{E_0} \right] \right\}^{1/3}$$

where

k   = the interaction constant for benzene, 8.969 kJ•nm³/mol

Maximum Differential Pore Volume (cm³/g-nm):

This value is also known as frequency of the mode

$$\frac{dV}{dw_{mode}} Max = 0.5 \times (3N+1) \times W_o \times \left[\frac{3N+1}{3N}\right]^{1/3N} \times \left[\frac{E_0}{k}\right]^{1/3} \times \exp\left(-\left[\frac{3N+1}{3N}\right]\right)$$

Mean Equivalent Pore Width (nm):

$$w_{mean} = 2 \times \frac{\left[\frac{k}{E_0}\right]^{1/3}}{\Gamma\left(\frac{3N+1}{3N}\right)}$$

Micropore surface area (m²/g):

$$SDA = 1000 \times 2.0 \times W_0 \times \left[\frac{E_0}{k}\right]^{1/3} \times \Gamma\left(\frac{3N+1}{3N}\right)$$

Γ is calculated by a polynomial approximation over the domain $0 \le x \le 1$ as follows:

$$\Gamma(x+1) = 1+b_1x + b_2x^2 + b_3x^3 + b_4x^4 + b_5x^5 + b_6x^6 + b_7x^7 + b_8x^8 + \varepsilon(x) \mid \varepsilon(x) \mid \le 3 \times 10^{-7}$$

where

$$b_1 = -0.57719\ 1652$$
$$b_2 = 0.98820\ 5891$$
$$b_3 = -0.89705\ 6937$$
$$b_4 = 0.91820\ 6857$$
$$b_5 = -0.75670\ 4078$$
$$b_6 = 0.48219\ 9394$$
$$b_7 = -0.19352\ 7818$$
$$b_8 = 0.03586\ 8343$$

and where

$$x + 1 = \left(\frac{3N+1}{3N}\right)$$

Equivalent Pore Width (nm):

$$w_i = 2 \times \left[\frac{-\left(\dfrac{k}{E_0}\right)^N}{\ln(W_i) - \ln(W_0)} \right]^{1/3N}$$

dV/dw Pore Volume (cm^3/g-nm):

$$\frac{dV}{dw_i} = 0.5 \times W_0 \times 3N \times \left[\frac{k}{E_0}\right]^N \times \left(\frac{w_i}{2}\right)^{-(3N+1)} \times \exp\left[-\left(\frac{k}{E_0}\right)^N \times \left(\frac{w_i}{2}\right)^{-3N}\right]$$

MP-Method

For each point designated for MP-method[14] calculations, the following calculations are done:

Thickness for the Ith point (Å):

$$t_I = HP1 \times \left[\frac{HP2}{\ln(P_{rel_I})}\right]^{HP3} \qquad\qquad (\text{Halsey}[6])$$

or

$$t_I = \left[\frac{HJP1}{HJP2 - \log(P_{rel_I})}\right]^{HJP3} \qquad\qquad (\text{Harkins and Jura}[7])$$

where:

t_I	=	thickness for Ith point
HP1	=	Halsey parameter #1
HP2	=	Halsey parameter #2
HP3	=	Halsey parameter #3
HJP1	=	Harkins and Jura parameter #1
HJP2	=	Harkins and Jura parameter #2
HJP3	=	Harkins and Jura parameter #3
P_{relI}	=	relative pressure for the Ith point (mmHg)

With the (t_I, V_I) data pairs, the Akima semi-spline interpolation method is used to interpolate volume adsorbed values based on thickness values that are evenly spaced 0.2 Angstrom apart starting at the first outlier point. Outliers are defined as those points have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_I = \frac{t_I + t_{I-1}}{2}$$

Remaining pore surface area for the Ith point (m²/g):

$$S_I = \frac{(V_I - V_{I-1}) \times (D \times 10^{-6} \ m^3/cm^3)}{(t_I - t_{I-1}) \times 10^{-10} m/A}$$

where

D   =   density conversion factor (cm³ liquid/cm³ STP) on the Adsorptive Properties dialog box

Incremental pore surface area occluded for the Ith point (m²/g):

$$S_{INC_I} = S_{I-1} - S_I$$

Cumulative pore surface area occluded for the Ith point (m²/g):

$$S_{CUM_I} = S_{INC_I} + S_{INC_{I-1}} + \ldots + S_{INC_1}$$

dA/dR pore surface area for the Ith point (m²/g-Å):

$$\frac{dA}{dR_I} = \frac{S_{INC_I}}{t_I - t_{I-1}}$$

Incremental pore volume occluded for the Ith point (cm³/g):

$$V_{INC_I} = (S_{INC_I} \times 10^4 \ cm^2/m^2) \times (R_I \times 10^{-8} \ cm/Å)$$

Cumulative pore volume occluded for the Ith point (cm³/g):

$$V_{CUM_I} = V_{INC_I} + V_{INC_{I-1}} + \ldots + V_{INC_1}$$

dV/dR pore volume for the Ith point (cm³/g-Å):

$$\frac{dV}{dR_I} = \frac{V_{INC_I}}{t_I - t_{I-1}}$$

Freundlich Isotherm

The Freundlich isotherm has the form:

$$\frac{Q}{Q_S} = CP^{1/m}$$

where

Q	=	quantity of gas adsorbed
Q_S	=	quantity of gas in a monolayer
C	=	temperature-dependent constant
m	=	temperature-dependent constant

The pressure is absolute; typically, m > 1. In terms of quantity adsorbed,

$$Q = Q_S CP^{1/m}$$

Taking the log of both sides yields:

$$\log Q = \log Q_S C + \frac{1}{m} \log P$$

Temkin Isotherm

The Temkin isotherm has the form,

$$\frac{Q}{Q_S} = \frac{RT}{q_0\alpha}\ln(A_0 P)$$

where

Q   =   quantity of gas adsorbed
Q_S =   quantity of gas in a monolayer
q_0 =   the differential heat of adsorption at zero surface coverage
A_0 = $a_0 \exp\{-q_0/RT\}$, where α_0 and a_0 are adjustable constants

In terms of quantity adsorbed,

$$Q = \frac{RTQ_S}{q_0\alpha}\left[\ln A_0 + \ln\left(\frac{P}{Po}\right)\right]$$

Thus, the plot of the natural log of absolute pressure vs. quantity adsorbed yields a straight line with slope RTQ_S/q_0 and intercept $(\ln A_0)\, RTQ_S/q_0\alpha$.

DFT (Density Functional Theory)

The adsorption isotherm is known to convey a great deal of information about the energetic heterogeneity and geometric topology of the sample under study. The data of physical adsorption have been used for many years as the basis for methods to characterize the surface area and porosity of adsorbents. Real solid surfaces rarely approach ideal uniformity of structure. It is accepted that in general, the surface of even a nonporous material presents areas of greater or lesser attraction for adsorbed molecules.

This energetic heterogeneity greatly affects the shape of the adsorption isotherm with the result that simple theories such as the Langmuir and BET formulas can, at best, give only approximate estimates of surface area. Porous solids virtually are never characterized by a single pore dimension, but instead exhibit a more or less wide distribution of sizes. The observed adsorption isotherm for a typical material is therefore the convolution of an adsorption process with the distribution of one or more properties which affect that process. This was first stated mathematically by Ross and Olivier[12] for the case of surface energy distribution and has become known as the integral equation of adsorption.

The Integral Equation of Adsorption

In a general form for a single component adsorptive, the integral equation of adsorption can be written as

$$Q(p) \; = \; \int da \; db \; dc \ldots q(p, a, b, c \ldots) \, f(a, b, c \ldots) \qquad (1)$$

where

$Q(p)$	=	the total quantity adsorbed per unit weight at pressure p,
a,b,c,...	=	a set of distributed properties,
f(a,b,c,...)	=	the distribution function of the properties, and
q(p,a,b,c,...)	=	the kernel function describing the adsorption isotherm on unit surface of material with fixed properties a,b,c,...

Equation (1), a Fredholm integral of the first kind, is a member of a class of problems known as ill-posed, in that there are an infinite number of functional combinations inside the integral that will provide solutions. Even when the kernel function is known, experimental error in the data can make solving for even a single distribution function a difficult task. Solving for multiple distribution functions requires more data than provided by a single adsorption isotherm.

Application to Surface Energy Distribution

Under certain conditions, an energetically heterogeneous surface may be characterized by a distribution of adsorptive energies. The conditions are that the sample is not microporous, i.e., that adsorption is taking place on essentially a free surface with no pore filling processes at least to about 0.2 relative pressure. Secondly, that each energetically distinct patch contributes independently to the total adsorption isotherm in proportion to the fraction of the total surface that it represents. This condition is satisfied if the patches are relatively large compared to an adsorptive molecule, or if the energy gradient along the surface is not steep. In mathematical terms, this concept is expressed by the integral equation of adsorption in the following form.

$$Q(p) = \int d\varepsilon \; q(p, \varepsilon) \, f(\varepsilon) \tag{2}$$

where

Q(p)	=	the experimental quantity adsorbed per gram at pressure p,
q(p,e)	=	the quantity adsorbed per unit area at the same pressure, p, on an ideal free surface of energy e, and
f(e)	=	the total area of surface of energy e in the sample.

The exact form of the energy-dependent term depends on the form of the model isotherms expressed in the kernel function and is provided in the model description (refer to Appendix G for model descriptions).

Application to Pore Size Distribution

Similarly, a sample of porous material may be characterized by its distribution of pore sizes. It is assumed in this case that each pore acts independently. Each pore size present then contributes to the total adsorption isotherm in proportion to the fraction of the total area of the sample that it represents. Mathematically, this relation is expressed by

$$Q(p) = \int dH \; q(p, H) \, f(H) \tag{3}$$

where

Q(p)	=	the experimental quantity adsorbed at pressure p,
q(p,H)	=	the quantity adsorbed per unit area at the same pressure, p, in an ideal pore of size H, and
f(H)	=	the total area of pores of size H in the sample.

Numerical values for the kernel functions in the form of model isotherms can be derived from modern statistical mechanics such as density functional theory or molecular simulations, or can be calculated from one of various classical theories based on the Kelvin equation. Several types are found in the models library.

Performing the Deconvolution

The integrations in equations (2) and (3) are carried out over all surface energies or pore sizes in the model. The functions q(p,e) and q(p,H), which we call the kernel functions, are contained in numeric form as model isotherms. Because, in general, there is no analytic solution for equation (1), the problem is best solved in a discrete form; the integral equation for any distributed property Z becomes a summation:

$$Q(p) \;=\; \sum_i q(p, Z_i)\, f(Z_i) \tag{4}$$

Given a set of model isotherms, q(p,Z), from a model chosen from the models library and an experimental isotherm, Q(p), contained in a sample information file, the software determines the set of positive values f(Z) that most nearly, in a least squares sense, solves equation (4). The distributed property, surface energy or pore size, is then displayed on the Report Options dialog box as a selection of tables or graphs.

Regularization

DFT allows a selectable regularization (also referred to as smoothing) constraint to be applied during the deconvolution process to avoid over-fitting in the case of noisy data or ill-fitting models. The method used is based on co-minimization of the second derivative of the distribution. The relative weight given to this term is determined by the value of the regularization parameter, which is set on the DFT Pore Size or Surface Energy dialog box and also is shown in the header of reports. The value of the regularization parameter varies from zero (for no second derivative constraint) to ten (indicating a weight equal to minimizing the residuals), or even larger. When the distribution and residuals obtained change little with the value of the regularization parameter, it indicates that the chosen model provides a good representation of the data. Conversely, a large sensitivity to the regularization parameter might indicate inadequate data or a poor choice of model to represent the data.

Summary Report

The following calculations and the results of previous calculations (as noted) are used to generate the summary report:

a. BET Surface Area

   See BET Surface Area Calculations

b. Langmuir Surface Area

   See Langmuir Surface Area Calculations

c. Single Point Surface Area (m^2/g)

$$S_{1PT} = \frac{[Va \times (1 - Pr)] \times CSA \times (6.023 \times 10^{23})}{22414 \ cm^3 \ STP \times 10^{18} \ nm^2/m^2}$$

   where

   Pr = pressure closest to 0.3 of the relative pressure points designated for surface area calculations.

   Va = volume corresponding to Pr

d. Micropore Area

   See t-Plot calculations.

e. Micropore Volume

   See t-Plot calculations.

f. Horvath-Kawazoe Maximum Pore Volume

   See Horvath-Kawazoe Calculations

g. Horvath-Kawazoe Median Pore Diameter

   See Horvath-Kawazoe Calculations

h. Dubinin-Radushkevich Micropore Surface Area

   See Dubinin-Radushkevich Calculations

i. Dubinin-Radushkevich Monolayer Capacity

   See Dubinin-Radushkevich Calculations

    j.   Dubinin-Astakhov Micropore Surface Area

       See Dubinin-Astakhov Calculations

    k. Dubinin-Astakhov Micropore Volume

       See Dubinin-Astakhov Calculations

    l. MP-Method Cumulative Surface Area of Pores

       $MPS_{TOT} = S_{CUMI}$, (see MP-method Calculations) for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative surface area was computed.

    m. MP-Method Cumulative Pore Volume of Pores

       $MPV_{TOT} = V_{CUMI}$, (see MP-method Calculations) for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative pore volume was computed.

    n. Average Pore Hydraulic Radius (A)

$$MP_{DAVER} = \frac{MPV_{TOT}}{MPS_{TOT}} \times 10^{-6} m^3/cm \times 10^{10} \text{ Å}/m$$

SPC Report Variables

Regressions Chart Variables

The line of best fit for the Regression Chart is calculated by the usual Least Squares methd. (Refer to *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.) If there is only a single point or all N points have the same x-value, there can be no line of best fit in the standard form.

$$XAve = \frac{\Sigma\, x_i}{N}$$

$$YAve = \frac{\Sigma\, y_i}{N}$$

$$Slope = \frac{\Sigma\, (x_i - XAve)(y_i - YAve)}{\Sigma\, (x_i - XAve)^2}$$

The coefficient of Correlation for this line is also calculated in the usual way. (Refer to *Mathematical Handbook for Scientists and Engineers*, by Granino A. Korn and Theresa M. Korn, Copyright 1961, 1968 McGraw Hill, Sec. 18.4.)

$$\sigma_x = \sqrt{\frac{\Sigma\, (x_i - XAve)^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\Sigma\, (y_i - YAve)^2}{N}}$$

$$Cov(x, y) = \frac{\Sigma\, (x_i - XAve)(y_i - YAve)}{N}$$

$$Correlation\ Coefficient = \frac{Cov(x, y)}{\sigma_x \sigma_y}$$

Control Chart Variables

$$Mean = \frac{\Sigma\ y_i}{N}$$

$$StdDev = \sqrt{\frac{\Sigma\ (y - Mean)^2}{N - 1}}$$

$$CoefVar = \frac{StdDev}{Mean}$$

$$PlusNSig = Mean + n \cdot StdDev$$

$$MinusNSig = Mean - n \cdot StdDev$$

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D. TESTING FOR LEAKS

This appendix contains general instructions for testing the ASAP 2050 for leaks. If the analyzer successfully performs a blank tube analysis using nitrogen, you do not need to test for leaks.

These procedures may be performed individually or sequentially. Record pressure readings on the Valve Test Data Sheet included at the end of this appendix. After you finish testing for leaks, replace any leaking valves.

You will need the following items to perform tests:

- Stopwatch
- Valve Test Data Sheet (included in this appendix)

Testing Individual Valves

Analysis Valves

This procedure removes differential pressure from all valves in the analysis system and establishes that there are no leaks in the analysis system. It should be performed before testing for individual valves.

1. Ensure that the analyzer is idle.

2. Close the regulator outlet valve for each gas supply line to the analyzer.

3. Attach the Po tube to the Po port.

4. Insert a plug (or attach a clean, empty sample tube) into the analysis port.

5. Insert a plug into the vapor inlet (if the vapor option is not installed).

6. Select **Show Instrument Schematic**, then **Enable Manual Control** from the Unit menu. Table D-1 lists descriptions of the analysis valves.
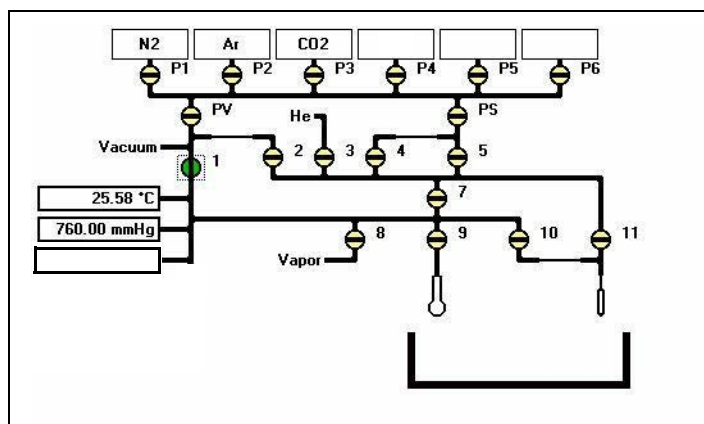
Table D-1. Analysis Valve Descriptions

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Helium inlet port valve
4	Highly restricted analysis gas
5	Partially restricted analysis gas
7	Lower manifold isolation
8	Vapor inlet port
9	Sample port
10	Restricted Psat tube port
11	Unrestricted Psat tube port
P1 through P6	Gas inlet port valves
PS	Supply valve for physisorption gases
PV	Vacuum valve for physisorption gases

7. Open valves 1, 2, 4, 5, and 7; close all other manifold valves.

8. Open valve PS.

9. Open valve 3 and allow the pressure to drop below 1000 μmHg (1 mmHg).

10. Repeat Step 9 for valves P1 through P6.

11. Open valve 9.

12. Open valves 10 and 11.

13. Open valve 8.

14. Open valve PV.

15. Allow the system to evacuate for a minimum of one hour; a four-hour or overnight evacuation is preferred.

16. Obtain the manifold outgassing rate using a vacuum gauge:

    a. Close valves 3, 4, 5, 8, 9, 10, 11, and P1 through P6.

    b. Evacuate manifold for another 30 minutes.

    c. Take a pressure reading; record it as P1 for the manifold.

    d. Close valves 1 and 2.

    e. Wait 10 minutes and take another pressure reading; record it as P2 for the manifold.

    f. Subtract P1 from P2 and divide the difference by 10 to obtain the outgassing rate per minute;

    $$\text{Outgassing rate} = \frac{P2 - P1}{10}$$

    Record this value as the outgassing rate for the manifold. This value should be less than 0.5 μmHg/min. for the lower manifold alone and less than 0.7 μmHg/min. for the upper and lower manifolds combined.

17. Obtain the gas inlet valve outgassing rate:

    a. Open valves PS and 5; valve 7 should still be open.

    b. Take a pressure reading; record it as P1 for the gas inlet valve.

    c. Wait 10 minutes and take another pressure reading; record it as P2 for the gas inlet valve.

    d. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for the gas inlet valve. This value should be less than 0.7 μmHg/min.

Valves 3 and P1 through P6

1. Open the regulator outlet valve for the gas connected to port 3.

2. Take a pressure reading; record it as P1 for valve 3.

3. Wait 10 minutes and take another pressure reading; record it as P2 for valve 3.

4. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for Valve 3. This value should be less than 0.7 µmHg/min.

5. Repeat Steps 1 through 4 for valves P1 through P6 to which a gas is attached. Record the values on the Valve Test Data Worksheet in the spaces provided.

Valves PS, 5, and 7

1. Close valve PS.

2. Open valve P1 for 60 seconds, then close it.

3. Take a pressure reading; store it as P1 for valve PS.

4. Wait 10 minutes and take another pressure reading; record it as P2 for valve PS.

5. Subtract P1 from P2 and divide the difference by 10; record the value as the outgassing rate for Valve PS. This value should be less than 0.7 µmHg/min.

6. Close valve 5 (close Valve 4 if open).

7. Open valve PS.

8. Open valve P1 for 30 seconds, then close it.

9. Take a pressure reading; record it as P1 for valve 5.

10. Wait 10 minutes and take another pressure reading; record it as P2 for valve 5.

11. Subtract P1 from P2 and divide the difference by 10.; record this value as the outgassing rate for Valve 5. This value should be less than 0.7 µmHg/min.

12. Close valve 7; open valve 5.

13. Open valve P1 for 30 seconds, then close it.

14. Take a pressure reading; record it as P1 for valve 7.

15. Wait 10 minutes and take another pressure reading; record it as P2 for valve 7.

16. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for Valve 7. This value should be less than 0.5 µmHg/min.

Valves 1, 2, 8, 9, 10, 11, and PV

Valves 10 and 11

1. Open valve PV; wait 5 minutes (Valves PS and 5 should be open.).

2. Open valve 7; allow the system to evacuate to below vacuum setpoint (should be 10 μmHg or less).

3. Close valves PV, PS, and 5.

4. Open valves 1 and 2; allow the system to evacuate for 30 minutes.

    Valves 1, 2, and 7 should be the only valves open at this time.

5. Remove the Po tube.

6. Take a pressure reading; record it as P1 for valves 10 and 11.

7. Wait 10 minutes and take another pressure reading; record it as P2 for valves 10 and 11.

8. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for Valves 10 and 11. This value should be less than 0.7 μmHg/min.

Valve 9

9. Remove the sample tube from the analysis port.

10. Take a pressure reading; record it as P1 for valve 9.

11. Wait 10 minutes and take another pressure reading; record it as P2 for valve 9.

12. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve 9. This should be less than 0.7 μmHg/min.

Valve 8

13. With valve 8 closed, remove the plug from the vapor inlet port on the side of the analyzer.

    If the vapor accessory is installed, disconnect it from the analyzer.

14. Take a pressure reading; record it as P1 for valve 8.

15. Wait 10 minutes and take another pressure reading; record it as P2 for valve 8.

16. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve 8. This value should be less than 0.7 μmHg/min.

17. Before proceeding, reinstall the sample tube, the Po tube, and the plug from the vapor inlet (or the vapor accessory).

Valves 1, 2, and PV

18. Open valves 1 and 2.

19. Open valves 4, 5, and PS.

20. Allow the system to evacuate for 15 minutes.

21. Close valves 1 and 2. Valve PV should be closed; if not, close it.

22. Take a pressure reading; record it as P1 for valves 1, 2, and PV.

23. Wait 10 minutes and take another pressure reading; record it as P2 for valves 1, 2, and PV.

24. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valves 1, 2, and PV. This value should be less than 0.7 μmHg/min.

25. Fill the manifold to 800 mmHg with N_2, then close the nitrogen port valve.

26. Close valve 4.

27. Take a pressure reading; record it as P1 for valve PV.

28. Wait 10 minutes and take another pressure reading; record it as P2 for valve PV.

29. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve PV. This value should be less than 50 μmHg/min.

     If the pressure drops more than 50 μmHg, close valve 5, the PV valve may be leaking.

30. Take another pressure reading; record it as P1 for valves 1 and 2.

31. Wait 10 minutes and take another pressure reading; record it as P2 for valves 1 and 2.

32. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valves 1 and 2. This value should be less than 50 μmHg/min.

     If the pressure drops more than 0.05 mmHg, close valve 7, valves 1 or 2 may be leaking.

33. Take another pressure reading; record it as P1 for valve 1.

34. Wait 10 minutes and take another pressure reading; record it as P2 for valve 1.

35. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve 1. This value should be less than 50 μmHg/min.

     If the pressure drops more than 50 μmHg, valve 1 may be leaking.

Degas Valves

Perform the following steps before testing the individual degassing valves.

1. Ensure that the analyzer is idle.

2. Close the regulator outlet valve for the backfill gas.

3. Insert a plug into (or attach a clean, empty sample tube) each degas port.

4. Select **Degas**, **Show Degas Schematic**, then **Degas**, **Enable Manual Control** from the Unit menu. Table D-2 lists descriptions of the degas valves.
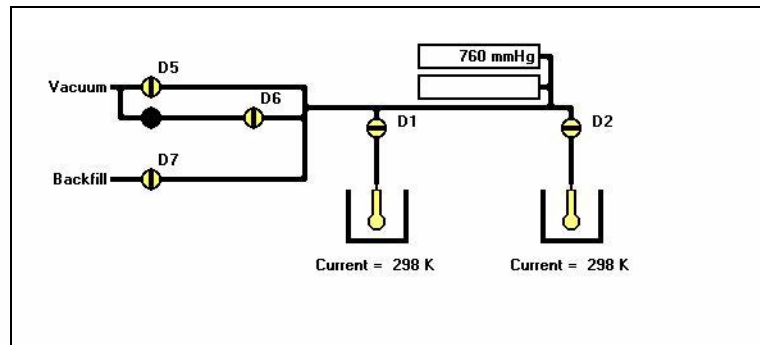
Table D-2.   Degas Valve Descriptions

Valve	Description
D1 and D2	Degas port valves
D5	Vacuum valve
D6	Servo isolation valve
D7	Gas inlet port valve

5. Open Valves D5, D1, D2, and then D7.

6. Open Valve D6.

7. Allow the degas system to evacuate to the vacuum setpoint, then evacuate for 60 minutes.

8. Close Valves D1, D2, D5, D6, and D7.

9. Take a pressure reading and record it as P1 for the preliminary test.

10. Wait 10 minutes and take another reading; record it as P2 for the preliminary test.

11. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for the preliminary test. This value should be less than 10 µmHg/min.

What To Do If You Detect a Leaking Valve

If you determine that a specific valve is leaking, remove differential pressure from the valve and continue testing the remaining valves for leaks.

> **As an alternative, you may repair or replace the leaking valve after removing the differential pressure on the valve. However, opening the analysis system to replace or repair a valve exposes the analyzer manifold to atmospheric gases and moisture. These gases and moisture greatly increase the time required to evacuate the analyzer and prepare it for further testing or operation.**

Removing Differential Pressure from a Leaking Valve

1. Select either of the following actions:

   • Close the regulator outlet valve for the gas supply line to the valve.
   • Reinstall the plug that was removed to test the valve.

2. Open the leaking valve.

3. Evacuate the analyzer until you obtain an outgas rate of less than 0.7 μmHg per minute. Refer to **Testing Individual Valves** on page **D-1** for detailed instructions on determining the outgas rate for specific valves.

Repairing or Replacing a Leaking Valve

Whether you repair or replace a valve depends on the type of valve that is leaking.

   • Valves on the Analysis manifold should be repaired
   • Valves on the Degas and inlet manifold should be replaced.

Repairing Valves on the Analysis Manifold

1. Backfill the manifold with nitrogen to approximately 800 mmHg.

   • Close off the helium gas supply if you are repairing the helium inlet valve.
   • Turn off the vacuum pump system if you are repairing a vacuum valve.

2. Remove and replace the plunger and gasket for the leaking valve.

3. Closely inspect the valve seat area for any debris which may cause another leak.

Gasket

Plunger
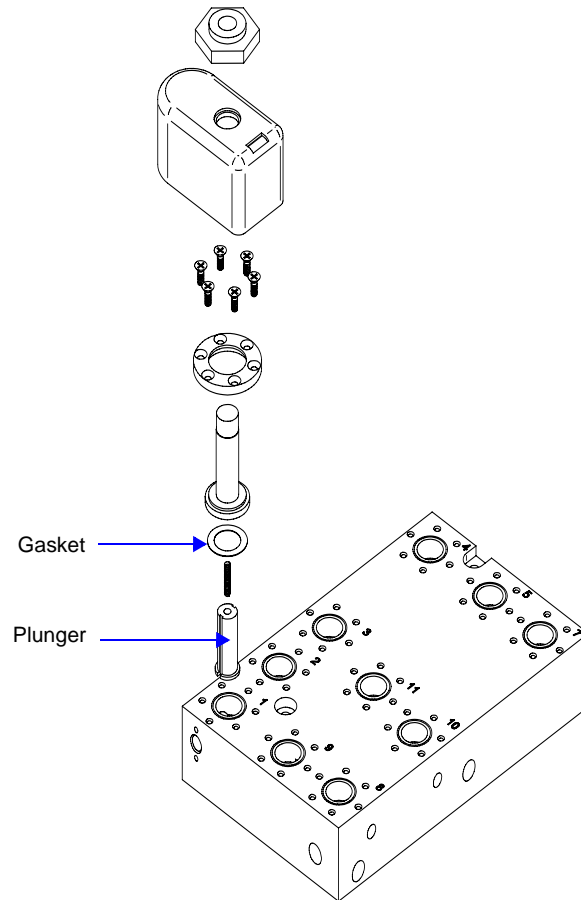
4. Reassemble the valve.

    • Reopen the helium supply if you are repairing the helium inlet valve.
    • Turn on the vacuum pump system if you are repairing a vacuum valve.

5. Open and close the repaired valve approximately 20 to 30 times; this allows the valve to seat properly.

6. Evacuate the analyzer until the outgas rate is less than 0.7 µmHg per minute. Refer to **Testing Individual Valves** on page **D-1** for detailed instructions.

Replacing Valves on the Degas and Gas Inlet Manifold

1. Backfill the manifold with nitrogen to approximately 800 mmHg.

2. Turn off the gas supply for the gas inlet valves.

3. Turn off the degas vacuum system for access to degas valves 5 or 6.

4. Disconnect the wiring from the valve.

5. Unscrew and remove the valve from the manifold.
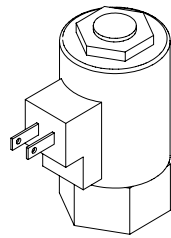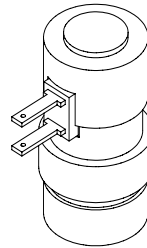
Inlet Valve   Degas Valve

6. Install the new valve, then reconnect the wiring.

7. Turn on the gas supply and vacuum pump system.

8. Evacuate the analyzer until the outgas rate is less than 0.7 µmHg/min. for inlet valves or 10 µmHg/min. for degas valves. Refer to **Testing Individual Valves** on page **D-1** for detailed instructions.

Valve Data Test Sheet

Make a copy of this form to record pressure readings and outgassing rates when leak-testing system valves.

VALVE(s)	P1 (1st pressure reading)	P2 (2nd pressure reading)	OUTGASSING RATE
Analysis Valves			
Manifold			
Gas inlet valve			
3			
P1			
P2			
P3			
P4			
P5			
P6			
PS			
5			
7			
10 and 11			
9			
8			
1, 2, and PV			
1, 2, and PV			
1 and 2			
1			
Degas Valves			
Manifold			

E. CALCULATING FREE-SPACE VALUES

Many microporous materials, such as zeolites and activated carbons, trap and hold helium in their complex pore structures for many hours after being exposed to helium. Helium trapped in micropores can interfere with the analysis at low pressures, causing an "S"-shaped curve at the lower end of the isotherm. For this reason, it is recommended that you enter the ambient and analysis free-space volumes when performing micropore analyses, therefore avoiding exposure of the sample to helium. Two techniques can be used for determining ambient and analysis free-space values.

The first method is to perform a short analysis on the sample after partial degassing (one pressure point with no incremental dosing), but prior to final sample preparation. Measure the free space during this analysis. The measured free-space values will be printed on the report and may then be entered into the sample file after more thorough sample preparation.

The second method requires prior tests using empty tubes that will be employed later for the sample analyses. The measured free-space data can be used thereafter on every analysis performed using these sample tubes. This small initial investment of time will save considerable time later. Perform an empty tube analysis on each sample tube you intend to use for micropore analysis. Measure the free space of each sample tube, taking only one pressure point. Four important things to remember are: 1) be consistent in using seal frits; for example, use the same seal frits for the analysis as you did for the empty tube test; 2) since the analysis free space is dependent on bath temperature, perform a test for each bath temperature to be used; 3) the isothermal jacket must be in the same position for the sample analysis as it was for the empty tube test; and 4) correct the free-space volumes obtained for the volume displaced by the sample when you use them.

To make the correction, subtract the amount of gas displaced by the sample. The calculations are simple and are given here with a brief explanation of their derivation. Remember to employ the appropriate free-space values for each bath temperature used, stating all temperatures in Kelvin.

To correct ambient free space:

$$V_{ws} = V_{wm} - \left(\frac{M_s}{\rho_s} \times \frac{T_{std}}{T_{amb}} \right)$$

where

V_{ws} = calculated ambient free space with sample present (in standard cm^3)

V_{wm} = ambient free space measured for the empty tube (in standard cm^3)

M_s = mass of sample to be analyzed (in grams)

r_s = approximate sample true density (in grams/cm^3)

T_{amb} = ambient temperature (in Kelvin)

T_{std} = standard temperature (273.15 Kelvin)

To correct analysis free space:

where

V_{cs}	=	calculated analysis free space with sample present (in standard cm^3)
V_{cm}	=	analysis free space measured for the empty tube (in standard cm^3)
M_s	=	mass of the sample to be analyzed (in grams)
r_s	=	approximate sample true density (in grams/cm^3)
T_{bath}	=	analysis bath temperature (in Kelvin)
T_{std}	=	standard temperature (273.15 Kelvin)

When you later analyze a sample, select Enter Free Space, and use the values calculated above.

Example:
The sample is 0.2345 grams of activated carbon with a density of 2.0000 g/cm^3. The room temperature is 22 °C or 295.15 K. The analysis tube has been measured previously in an argon bath. The measured ambient and analysis free spaces are 29.1234 and 89.4567 cm^3 STP atm-1, respectively. The analysis will be performed with argon at liquid argon temperature, 87.3 K.

With this information, enter the corrected ambient free space as follows:

$$V_{ws} = 29.1234 \ cm^3 \ STP - \left(\frac{0.2345 \ g}{2.0000 \ g/cm^3} \times \frac{273.2 \ K}{295.2 \ K} \right) = 29.0149 \ cm^3 \ STP \ atm^{-1}$$

and enter the corrected analysis free space as follows:

$$V_{cs} = 89.4567 \ cm^3 \ STP - \left(\frac{0.2345 \ g}{2.0000 \ g/cm^3} \times \frac{273.2 \ K}{87.3 \ K} \right) = 89.0898 \ cm^3 \ STP \ atm^{-1}$$

F. DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

Models Based on Statistical Thermodynamics

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

Theoretical Background

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (Vm), and the molar intensity of the gas-surface interaction, such as the Langmuir "K" constant or the BET "C" constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed.

A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.

Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal's forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.

If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:

$$U(s) = \left[\left(\frac{\sigma}{s}\right)^{12} - \left(\frac{\sigma}{s}\right)^{6}\right]$$

where

ε	=	a characteristic energy of the adsorptive,
σ	=	the diameter of the adsorptive molecule, and
s	=	the separation distance.

Molecular Simulation Methods

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

Molecular Dynamics Method

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

Monte Carlo Method

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is "equilibrated" through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerable less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

Density Functional Formulation

Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H. The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential[*] is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under **References** at the end of this appendix.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

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[*]Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

Figure F-1 shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.
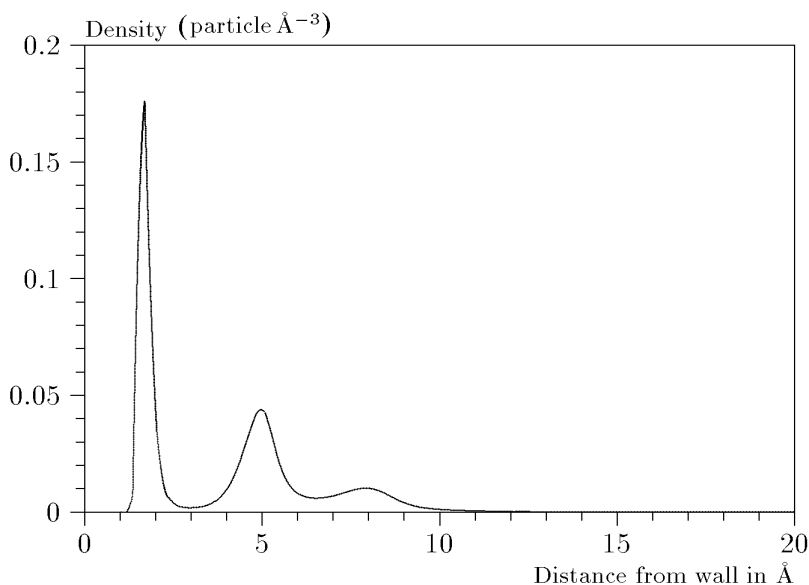
Figure F-1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

Figure F-2 shows how the profiles change with pressure for a model pore with H = 40 Angstroms. The insets show the density profiles for the corresponding points of the isotherm.
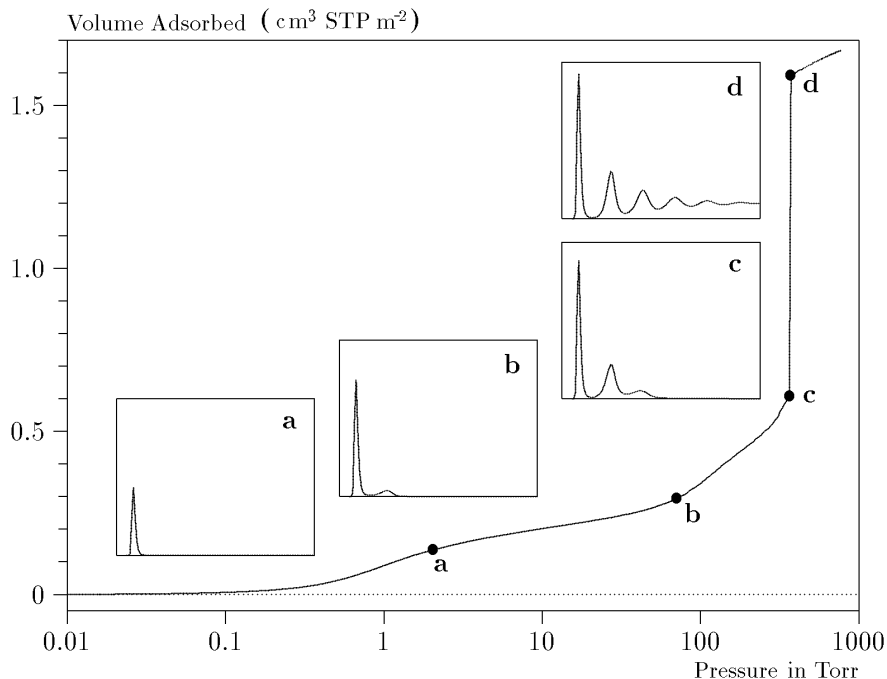
Figure F-2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

• Inset **a** shows the profile corresponding to point a on the isotherm where the surface is about half covered.

• At point **b**, the first layer is so full that it is more favorable for atoms to start a new layer.

• At point **c**, a third layer is forming. Point c, for this size slit, is the critical pore-filling pressure. In inset c, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.

• Inset **d** shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

Note that the adsorption isotherms for pores larger than the one shown in Figure 4-2 is identical up to point c. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in Figure F-3, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.
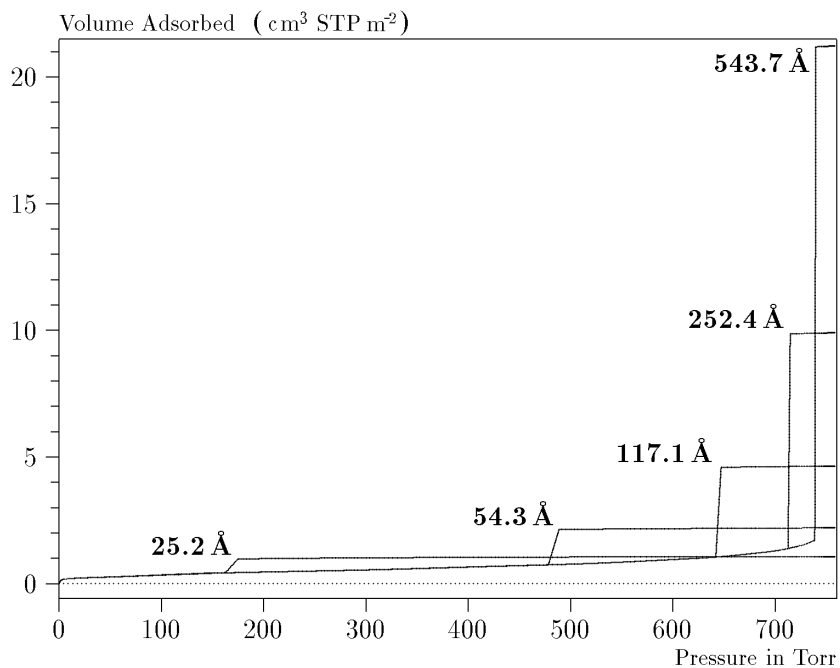
Figure F-3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure F-4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.
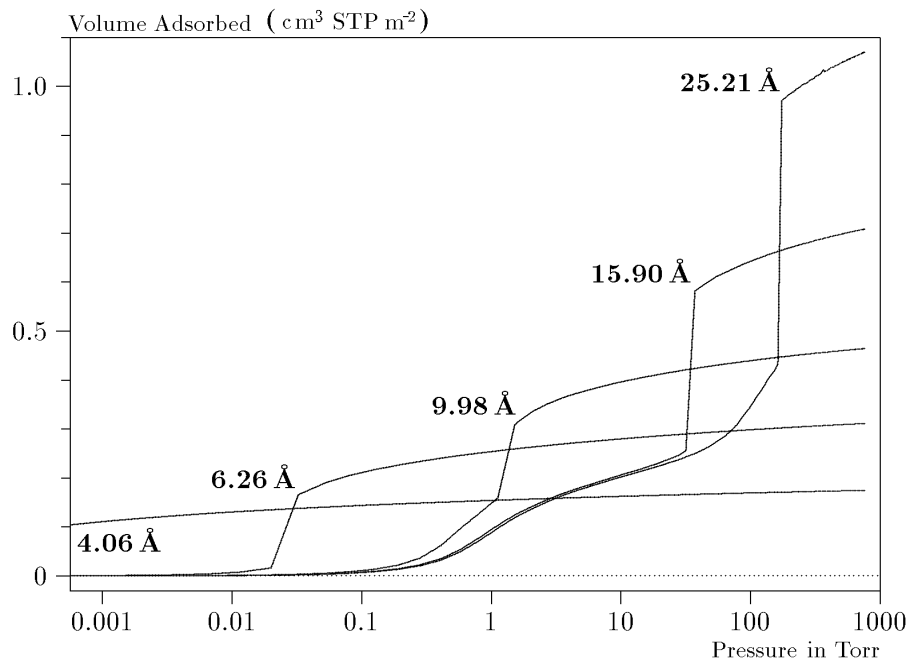
Figure F-4. Model Isotherms in the Micropore Size Range of Pore Width
Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density Independent Weights

N2 - DFT Model
AR - DFT Model

Geometry: Slit
Substrate: Carbon (graphite)
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near $1x10^{-6}$ to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Modified Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry: Free surface
Substrate: Surface energy
Method: Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (refer to references 3 and 4 on page), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the Select Data Points dialog box) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, e, for the adsorptive/adsorbent pair divided by Boltzmann's constant. The units are therefore degrees Kelvin.

N2 - Cylindrical Pores - Oxide Surface

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. This model reports pore sizes ranging from 3.8 to 387 Angstroms.

Reference: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999)

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry: Cylinder
Substrate: Crystalline Silicate
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillionite. The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al,1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts. This model reports pore widths from 3.8 to 387 Angstroms.

References: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occell, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.

References: M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization Of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface area and microporosity of pillared rectorite catalysts from a hybrid density functional theory method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Carbon dioxide at 273 K

Model isotherms were calculated, using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional

Geometry: Free Surface
Substrate: Any
Category: Surface energy
Method: Argon at 87 K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is /k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 Angstroms.

Reference: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
                  Idem, Phys. Rev. A 32: 3148 (1985).
                  P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

Models Based on Classical Theories

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions

Surface Energy

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity, or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-deBoer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

Pore Size

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, it finally will predict a larger increment of adsorptive for a given pressure increment than is actually observed; since a negative pore volume is non-physical, the algorithm must stop. Conversely, if the thickness curve used underestimates film thinning, accumulated error results in the calculation of an overly large volume of (possibly nonexistent) small pores.

The use of equation (1) represents an improvement over the traditional algorithm. Kernel functions corresponding to various classical Kelvin-based methods have been calculated for differing geometries and included in the list of models.

Models Included

Kelvin Equation with Halsey Thickness Curve

N2 - Halsey Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{-5.00}{\ln(P/\mathrm{Po})}\right)^{1/3}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension   =   8.88 dynes cm^{-1}
Molar density   =   0.02887 g cm^{-3}

N2 - Halsey Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948)

Kelvin Equation with Harkins and Jura Thickness Curve

N2 - Harkins and Jura Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left(\frac{13.99}{0.034 - \log(P/Po)}\right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density   = 0.02887 g cm^{-3}

N2 - Harkins and Jura Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

References: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944)
                    J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966)

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density   = 0.02887 g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

Reference: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, "The Surface Area in Intermediate Pores," Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969)

References

The papers listed below provide additional information on DFT models:

1. "Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids," J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).

2. "Classification of Adsorption Behavior: Siimple Fluids in Pores of Slit-shaped Geometry," Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).

3. "Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory," J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).

4. "The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory," J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

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