GEMINI® VII

SURFACE AREA ANALYZER

OPERATOR MANUAL

239-42828-01
May 2021
(Rev H)

micromeritics®

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WARRANTY

MICROMERITICS INSTRUMENT CORPORATION warrants for one year from the date of shipment each instrument it manufactures to be free from defects in material and workmanship impairing its usefulness under normal use and service conditions except as noted herein.

Our liability under this warranty is limited to repair, servicing and adjustment, free of charge at our plant, of any instrument or defective parts when returned prepaid to us and which our examination discloses to have been defective. The purchaser is responsible for all transportation charges involving the shipment of materials for warranty repairs. Failure of any instrument or product due to operator error, improper installation, unauthorized repair or alteration, failure of utilities, or environmental contamination will not constitute a warranty claim. The materials of construction used in MICROMERITICS instruments and other products were chosen after extensive testing and experience for their reliability and durability. However, these materials cannot be totally guaranteed against wear and/or decomposition by chemical action (corrosion) as a result of normal use.

Repair parts are warranted to be free from defects in material and workmanship for 90 days from the date of shipment.

No instrument or product shall be returned to MICROMERITICS prior to notification of alleged defect and authorization to return the instrument or product. All repairs or replacements are made subject to factory inspection of returned parts.

MICROMERITICS shall be released from all obligations under its warranty in the event repairs or modifications are made by persons other than its own authorized service personnel unless such work is authorized in writing by MICROMERITICS.

The obligations of this warranty will be limited under the following conditions:

1. Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their respective brand names or trade names. We, therefore, make no express or implied warranty as to such products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree to in writing.
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.

Purchaser agrees to hold MICROMERITICS harmless from any patent infringement action brought against MICROMERITICS if, at the request of the purchaser, MICROMERITICS modifies a standard product or manufactures a special product to the purchaser's specifications.

MICROMERITICS shall not be liable for consequential or other type damages resulting from the use of any of its products other than the liability stated above. This warranty is in lieu of all other warranties, express or implied, including but not limited to, the implied warranties of merchantability or fitness for use.

CORPORATE PROFILE

Micromeritics Instrument Corporation is the world's leading supplier of high-performance systems to characterize particles, powders and porous materials with a focus on physical properties, chemical activity, and flow properties. Our technology portfolio includes: pycnometry, adsorption, dynamic chemisorption, particle size and shape, intrusion porosimetry, powder rheology, and activity testing of catalysts. The company has R&D and manufacturing sites in the USA, UK, and Spain, and direct sales and service operations throughout the Americas, Europe, and Asia. Micromeritics systems are the instruments-of-choice in more than 10,000 laboratories of the world's most innovative companies, prestigious government, and academic institutions. Our world-class scientists and responsive support teams enable customer success by applying Micromeritics technology to the most demanding applications. For more information, please visit www.Micromeritics.com.

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ABOUT THIS MANUAL

The following can be found on the Micromeritics web page (www.Micromeritics.com).

- Calculations document (PDF)
- Error Messages document (PDF)
- Parts and Accessories
- Smart VacPrep Operator Manual (PDF)
- Vacuum Pump Guide (PDF)

All references to Gemini VII in this document encompass the Gemini VII models 2390a, 2390p, and 2390t unless otherwise noted. It also encompasses all references to the Gemini VII and Gemini VII Confirm environments unless otherwise noted.

This manual contains instructions for installations in standard and 21CFR11 environments. For instructions on installing the software in a 21CFR11 environment, see the Confirm Administrator Guide [*part number 004-42821-01*].

The following symbols or icons indicate safety precautions and/or supplemental information and may appear in this manual:

NOTE — Notes contain important information applicable to the topic.

CAUTION — Cautions contain information to help prevent actions that may damage the analyzer or components.

WARNING — Warnings contain information to help prevent actions that may cause personal injury.

CFR Note

NOTE — Notes that apply to 21CFR11 environments only (Confirm applications).

GENERAL SAFETY

Do not modify this instrument without the authorization of a Micromeritics service personnel.

Toxic or flammable gases require proper venting of exhaust.

Any piece of laboratory equipment can become dangerous to personnel when improperly operated or poorly maintained. All employees operating and maintaining Micromeritics instruments should be familiar with its operation and should be thoroughly trained and instructed on safety.

- Read the operator manual for any special operational instructions for the instrument.
- Know how the instrument functions and understand the operating processes.

- Wear the appropriate personal protective equipment when operating this instrument — such as eye protection, lab coat, protective gloves, etc.
- When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument. The Gemini 2390 weighs approximately 32 kg (75 lb).
- Always pay attention to the safety instructions provided on each label affixed to the instrument and do not alter or remove the labels. When inspecting the instrument, ensure that the safety labels have not become worn or damaged.
- The Gemini VII sound level is below 80 dBA. Hearing protection is optional.
- The Gemini VII has a safety shield. Ensure it is in place when operating the instrument.
- Proper maintenance is critical to personnel safety and smooth instrument operation and performance. Instruments require regular maintenance to help promote safety, provide an optimum end test result, and to prevent costly down time. Failure to practice proper maintenance procedures can lead to unsafe conditions and shorten the life of the instrument.
- Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

INTENDED USE

The Gemini VII series analyzers provide single and multipoint surface area and pore size measurements. Three models are available — the Gemini 2390a, 2390p, and 2390t.

The Gemini 2390a and 2390p are enclosed in the same size cabinet. The only physical difference is the P_0 (saturation pressure) tube which is installed on the Gemini 2390p, allowing continuous measurement of the saturation pressure.

The Gemini 2390t is in a slightly larger cabinet allowing the use of a larger dewar and longer sample tubes for extended analyses. This model also is equipped with a P_0 tube.

Most application features are available on all three models, with the exception of a continuous P_0 measurement and a few reports.

The instrument is intended to be operated by trained personnel familiar with the proper operation of the equipment recommended by the manufacturer as well as relevant hazards involved and prevention methods. All use, other than that described in this manual, is seen as unintended use and can cause a safety hazard.

The instrument is intended to be used as per applicable local and national regulations.

TRAINING

It is the responsibility of the customer to ensure that all personnel operating or maintaining the equipment participate in training and instruction sessions. All personnel operating, inspecting, servicing, or cleaning this instrument must be properly trained in operation and machine safety before operating this instrument.

ENVIRONMENTALLY FRIENDLY USE PERIOD

Hazardous Substances Table

Part Name	Hazardous Substances					
	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Cover	x	o	o	o	o	o
Power Supplies	x	o	o	o	o	o
Printed Circuit Boards	x	o	o	o	o	o
Cables, Connectors & Transducers	x	o	o	o	o	o

o Hazardous substance is below the specified limits as described in SJ/T11363-2006.

x Hazardous substance is above the specified limits as described in SJ/T11363-2006.

The Environmentally Friendly Use Period (EFUP) for all enclosed products and their parts are per the symbol shown here, unless otherwise marked. Certain parts may have a different EFUP (for example, battery modules) and so are marked to reflect such. The Environmentally Friendly Use Period is valid only when the product is operated under the conditions defined in the product manual.

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Table of Contents

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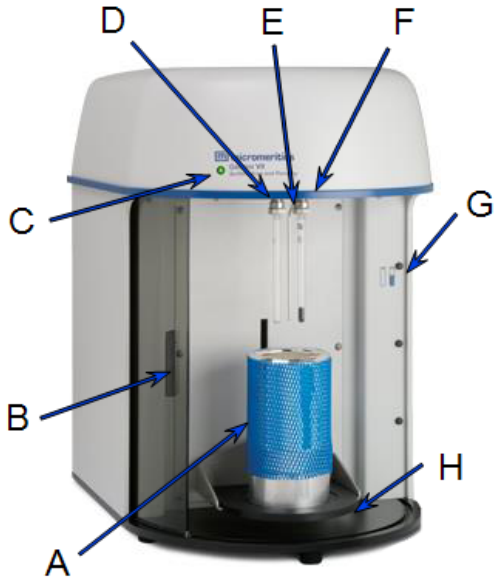
1 Analyzer Components

Parts and accessories can be found online at www.Micromeritics.com.

Front Components



A. Dewar
B. Sample safety shield
C. Power indicator light
D. Balance port and balance tube
E. P_0 port and tube
F. Sample port and sample tube
G. Tube position diagram
H. Elevator

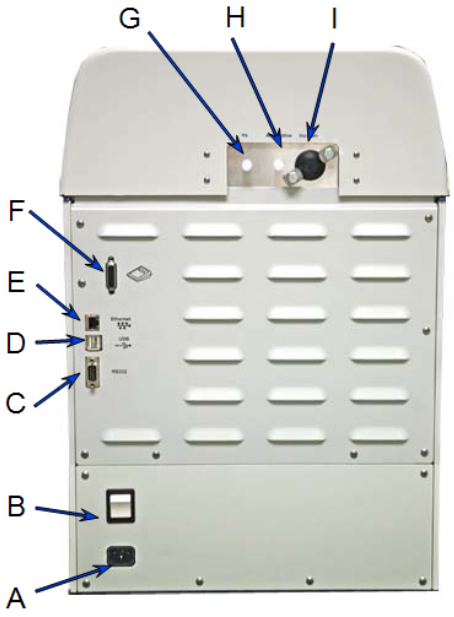
Front Components

Components	Description
Dewar	For housing cryogens used for analysis. The Dewar rests on an elevator.
Sample safety shield	Closes to cover the Dewar.
Power indicator light	Blinks when power is applied to the analyzer; illuminates when the analysis program is initiated and ready for operation.
Balance port and balance tube	The balance tube is identical to the sample tube. This design negates free-space errors introduced by thermal gradient variations or by initial mis-measurement of free space.
P_0 port and tube	Models 2390p and 2390t only. For measuring the saturation pressure.

Front Components (continued)

Components	Description
Sample port and sample tube	Holds the material to be analyzed.
Tube position diagram	Diagram of the positions of the balance and sample tubes. The empty tube represents the balance tube installed on the left port. The filled tube represents the sample tube installed on the right port.
Elevator	Allows placement of the Dewar around the sample and P_0 tubes. The elevator is raised automatically when the analysis is started and lowers automatically upon completion.

BACK PANEL COMPONENTS



A. Power connector
B. Power switch
C. RS232 connector - for service personnel use
D. USB ports - not used
E. Ethernet port
F. Keypad connection - used only wit keypad configurations
G. Helium inlet port for helium gas supply
H. Adsorptive gas inlet
I. Vacuum pump connector

EQUIPMENT OPTIONS AND UPGRADES

Parts and accessories can be found online at www.Micromeritics.com.

Option	Description
Chiller Dewar	For Gemini model 2390t. A closed loop recirculating system that utilizes a high surface area copper coil to provide excellent heat transfer between the Dewar and the recirculating liquids. The Chiller Dewar Quick Start Guide [*part number 025-42801-00*] can be found on the Micromeritics web page (www.Micromeritics.com).
Vacuum Pump	The analyzer requires a vacuum pump for sample analysis. Vacuum pumps used must meet the following criteria: ■ Achieve vacuum levels of 20 µmHg at the analyzer inlet ■ Contain an anti-suckback valve to prevent vacuum pump oil from back streaming into the analyzer in the event of a vacuum pump failure ■ Contain an NW16 inlet port for connection to the analyzer  A device to reduce oil vapor backstreaming is recommended.  The vacuum pump must have an anti-suckback valve to prevent oil from being admitted into the analyzer should the power fail while the system is under vacuum. Pumps available from Micromeritics are equipped with an anti-suckback valve. An oil-based or oil-free vacuum pump can be used with the analyzer. Appropriate vacuum pumps are available from Micromeritics.

Degasser Options	Description
FlowPrep	The FlowPrep applies both heat and a stream of inert gas to the sample to remove adsorbed contaminants from the surface and pores in preparation for analysis for up to six samples. Choose the temperature, gas, and flow rate best suited for your sample material. The FlowPrep is an independent unit and not controlled by the analyzer.
Smart VacPrep	The Smart VacPrep prepares samples by heating and evacuation. It contains six sample ports in which up to five temperatures, ramp rates, and soak times per sample are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. Samples can also be prepared, started, and completed independently. There is no need to wait for samples on other ports to finish. Front panel buttons allow a QuickStart operation with preprogrammed conditions. Up to three additional Smart VacPrep degassers can be connected to one computer permitting 24 preparation ports to be used. The Smart VacPrep is the recommended degassing unit.
VacPrep	The VacPrep offers two methods for removing contaminants. In addition to flowing gas, it provides vacuum to prepare samples by heating and evacuation of up to six samples. This combination provides preparation method options best suited to your material or application. Needle valves are also provided for introducing the vacuum slowly to prevent fluidization of samples. The VacPrep is an independent unit and not controlled by the analyzer.

GAS REQUIREMENTS AND PURITY

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

Compressed gases are required for analyses. Gas cylinders 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. Pressure relief valves should be set to no more than 30 psig (200 kPag). All gases should be of a purity listed below. Gas regulators can be ordered from Micromeritics. Parts and accessories can be found online at www.Micromeritics.com.

When helium is used for differential free-space measurement and nitrogen is used as the adsorbate gas, they should be of the following purity or better:

- Helium — purity of 99.9%. For analysis of materials with very low surface areas, Micromeritics recommends use of helium with purity of 99.995%. (CGA 580)
- Nitrogen — purity of 99.9%. For analysis of materials with very low surface areas, Micromeritics recommends use of nitrogen with purity of 99.995%. (CGA 580)

CRYOGEN REQUIREMENTS

Liquid nitrogen is commonly used as the cryogen to cool the sample during analysis. A liquid nitrogen transfer system eliminates the need to pressurize storage Dewars. The Model 021 liquid nitrogen transfer system is available from Micromeritics (www.Micromeritics.com).

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

SPECIFICATIONS FOR THE GEMINI VII

Analysis

Surface Area	From 0.1 m^2/g, total From 0.01 m^2/g, specific
Pore Volume	From 4×10^{-6} cm^3/g

Electrical

Frequency	50/60Hz
Power	50VA
Voltage	100-240VAC
Overvoltage Category	II

Environment

Humidity	20% relative, non-condensing
Temperature	10 °C to 35 °C (50 °F to 95 °F), operating 0 °C to 50 °C (32 °F to 122 °F), non-operating
Indoor/Outdoor Use	Indoor only (not suitable for wet locations) Altitude: 2000 m max Pollution degree of the intended environment: 2
Degree of Ingress Protection	IPX0

Gases

Adsorbate	Optimized for nitrogen in a liquid nitrogen sample bath. Gemini may be used with non-corrosive adsorbate gases having vapor pressures at both room and bath temperatures that are acceptably high relative to the resolution of the 1,000 mmHg pressure transducer. Typically, oxygen, argon, carbon dioxide, butane, methane, and other light hydrocarbons will produce useful data above absolute pressures of a few mmHg.

Physical

Height	59 cm (23 in.) - Gemini VII 2390a and 2390p 74 cm (29 in.) - Gemini VII 2390t
Width	40 cm (16 in.)
Depth	51 cm (20 in.)
Weight	32 kg (75 lbs.) - Gemini VII 2390a and 2390p 35 kg (78 lbs.) - Gemini 2390t

Pressure Measurement

Range	0 to 950 mmHg
P/P_0 Resolution	$<10^{-4}$
Relative Pressure Range	0 to 1.0 P/P_0
Pressure Resolution	<0.1 mmHg
Accuracy and Linearity (transducer manufacturer specification)	Better than ± 0.5% full scale

Sample Tube and Dewar

Standard Tube	■ **Gemini VII 2390a and 2390p.** 0.95 cm (3/8 in.) OD, 15.5 cm (6.1 in.) long with 6.5 cm^3 of volume. Sample capacity is approximately 2.0 cm^3. ■ **Gemini VII 2390t.** 0.95 cm (3/8 in.) OD × 20.5 cm (8.1 in.) long with 8.9 cm^3 of volume. Sample capacity is approximately 2.0 cm^3.
Dewar	■ **Gemini VII 2390a and 2390p.** 8 hours ■ **Gemini VII 2390t.** >24 hours

Vacuum System

An external vacuum source achieving 20 × 10^{-3} mmHg (or better) at the instrument inlet for oil-based or oil-free pumps.

For oil-based pumps, an anti-suckback valve is required to prevent oil from being admitted into the analyzer should there be a power failure. A device to reduce oil vapor backstreaming is also recommended.

Computer Requirements

Operating System	32- bit Windows 7 Professional or higher operating system is recommended for the best user experience. For 21 CFR Part 11 environments, Windows 10 Professional or Windows 10 Enterprise or higher is required.
Desktop Installation Required	The application should not be installed on a network drive with shared access. Multiple users cannot operate the application at the same time.
10 Base T or 100 Base T Ethernet Port	If the computer is to be connected to a network, two Ethernet ports are required. If more than one Ethernet based unit is connected to the same computer, an Ethernet switch will also be required. If a Smart VacPrep is to be used, an Ethernet switch is required.
Read/Write Permissions	All users of the application will need Read/Write permission to all directories and subdirectories where the application is installed. For 21 CFR Part 11 environments, permission may be limited to the installation directory.
Drives	USB port

Due to continuous improvements, specifications are subject to change without notice.

2 ABOUT THE SOFTWARE

Software in 21CFR11 Environments on page 2 - 24

The analyzer allows other computer programs to run while an automatic operation is in progress. The *Help* menu provides access to the online operator manual.

Report options can be specified when creating the sample file. When running an analysis, data gathered during the analysis process are compiled into the predefined reports. Reports can also be defined and generated after an analysis has been run. Each selected report is displayed on its own tab and reflects data collected during the analysis.

The MicroActive feature offers a Windows interface with an easy way to collect, organize, archive, reduce raw data, and store sample files for later use. Scalable and editable graphs, and copy and paste graphics, are easily generated. Customized reports can be viewed on a computer monitor, printed, or exported for use in other programs.

MENU STRUCTURE

All program functions use standard Windows menu functionality. The title bar contains a *Unit [n]*. If multiple analyzers are installed, ensure the appropriate unit is selected before continuing.

Main Menu Bar Options

Option	Description
File	Use to manage files used by the application — such as sample files, analysis conditions files, report options files, etc.
Unit [*n*]	Use to perform analyses, calibrations, and other analyzer operations. *Unit [n]* displays on the menu bar for each analyzer attached to the computer.
Smart VacPrep	(If installed.) Use to access the menu for each installed Smart VacPrep.
Reports	Use to start or initiate reports and view the results.
Options	Use to edit the default method, specify system configuration, specify units, and change presentation options.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Provides access to the embedded operator manual, the Micromeritics web page (www.Micromeritics.com), and information about the application.

COMMON FIELDS AND BUTTONS

The fields and buttons in the following table are located in multiple windows throughout the analyzer application and have the same description or function. Fields and button descriptions not listed in this table are found in tables in their respective sections. All entry fields will accept information when using a bar code reader.

Common Fields and Buttons

Field or Button	Description
Add	Adds an item to the list.
Add Log Entry	Use to enter information that will display in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Append	Use to insert one row at the end of a table.
Autoscale	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data are shown. If *Autoscale* is not selected, the entered range is used.
Axis Range	On report parameters windows, the *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes.
Bar Code *(default field label name)*	Use to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file.
Cancel	Discards any changes or cancels the current process.
Clear	Use to clear the table entries and display only one default value.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Copies	Selects the number of copies to print. This field is only enabled when *Print* is selected.
Delete	When working with tables deletes the selected information.
Destination	Selects the report destination.
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.

Common Fields and Buttons (continued)

Field or Button	Description
Exit	Exits the application. If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes. If an analyzer is currently operating, an additional prompt displays to confirm exiting from the software.
Export	Exports data in a sample file as a .TXT, .XML or .XLS file. When saved to a file, the data can be imported into other applications.
File	Selects the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
File name	Selects a file name from the list shown or enter a file name. If the required file type is not shown, select the type of file from the list.
From / To	When working with report parameters windows, indicates the *From* and *To* range for x- and/or y-axes.
Insert	Inserts one row above the selected row in the table.
List	Creates a list of sample or other type files. The list will contain file name, date / time the file was created or last edited, file identification, and file status.
Name	Contains a list of files in the selected directory or library.
Next	Moves to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double-click the file name in the Name column to open the file.
Prev	Moves to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer, or file).
Remove	Removes the selected file or files from the list.
Replace	Selects another file where the values will replace the current file's values.
Replace All	Selects another .SMP file where the values will replace all values for the active sample file. The original file will remain unchanged. No analysis data is added to the file. The only information added is sample information, material properties, liquid properties, analysis and reporting parameters.

Common Fields and Buttons (continued)

Field or Button	Description
Report	Displays a window to specify report output options.
Save	Saves changes.
Save As	Saves a file in the active window under a different file name. When saving sample information, a portion can be saved as a separate, stand-alone file, such as Analysis Conditions or Report Options.
Start	Starts the report, test, analysis, or operation.
Start Date	Displays a calendar to select the start date for the report.
View	**Operation**. Displays the data from the current analysis. **Instrument Log**. Displays recent analyses, calibrations, errors, or messages. **Instrument Schematic**. Displays a schematic of the analyzer system.

FILE STATUS

In the *File Selector* window, the *Mic Description* column and the *Mic Status* column display file description and file status. The *File Selector* incorporates standard Windows features for resizing windows, reordering and repositioning columns, and right-clicking an entry to display a menu of standard Windows functions.

File Status

File Status	Description
Analyzing	Sample files that are currently being used for analysis.
Complete	Sample files used in an analysis that has been completed.
Entered	Sample files containing manually entered data.
No Analysis	Sample files which have not been used to perform an analysis.
Prepared	Sample files that have been used in an automatic degas operation but have not been analyzed. This status is applicable only if using the Smart VacPrep degasser.
Preparing	Sample files that are currently being used in an automatic degas operation. This status is applicable only if using the Smart VacPrep degasser.

File Type and File Name Extension

File Type	File Name Extension
Alpha-s Curve[1])	.ALS
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Degas Conditions	.DEG
Heat of Adsorption Report	.HOA
Methods	.MTH
Report Options	.RPO
Sample Information	.SMP
SPC Report	.SPC

1) Saves the relative pressures and resulting quantities adsorbed as an ASCII text file. These data are derived by dividing the isotherm by the quantity adsorbed at 0.4 relative pressure.

File Type and File Name Extension (continued)

File Type	File Name Extension
Thickness Curve[1])	.THK

File Types for Printing or Exporting

File Type	File Name Extension
Portable document format	.PDF
Report	.REP
Spreadsheet	.XLS
Unicode	.TXT
Extensible markup language	.XML

1) Saves the relative pressures and corresponding thicknesses as an ASCII text file. 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.

KEYBOARD SHORTCUTS

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (when applicable) are listed to the right of the menu item.

Certain menus or functions can also be accessed using the **Alt** key plus the underlined letter in the menu command. For example, to access the *File* menu, press **Alt + F**, then press the underlined letter on the submenu (such as pressing **Alt + F** then pressing **O** opens the *File Selector*).

If the underscore does not display beneath the letter on the menu or window, press the **Alt** key on the keyboard.

Keyboard Shortcuts

Field or Button	Description
Alt +*[Unit n]*	Opens the *Unit [n]* menu.
Alt + F	Opens the *File* menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the *Help* menu.
Alt + I	Opens the *Options* menu.
Alt + R	Opens the *Reports* menu.
Alt + V	Opens the *Smart VacPrep* menu.
Alt + W	Opens the *Window* menu.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector* window.
Ctrl + P	Opens the *File Selector* to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.
F2	Opens the *File Selector* window.
F3	When in the *File Selector* window, opens the file search box.
F4	When in the *File Selector* window, opens the address bar.
F5	Opens the *File Selector* window listing report options files.
F6	Cascades open windows.

Keyboard Shortcuts (continued)

Field or Button	Description
F7	Tiles all open application windows.
F8	Opens the *File Selector* to start a report from a selected .SMP file.
F9	Closes all open reports.
F10	Opens the *Heat of Adsorption* window.
Shift + F2	Opens the *File Selector* window listing sample information files.
Shift + F9	Opens the shortcut menu of either the selected component on the analyzer schematic when manual control is enabled or onscreen reports.

OPTION PRESENTATION

Options > Option Presentation

CFR Note

For 21CFR11 environments, see *Software in 21CFR11 Environments on page 2 - 24*.

Use to change the way sample files and parameter files display: *Advanced*, *Basic*, or *Restricted*. Each display option shows sample information and options differently.

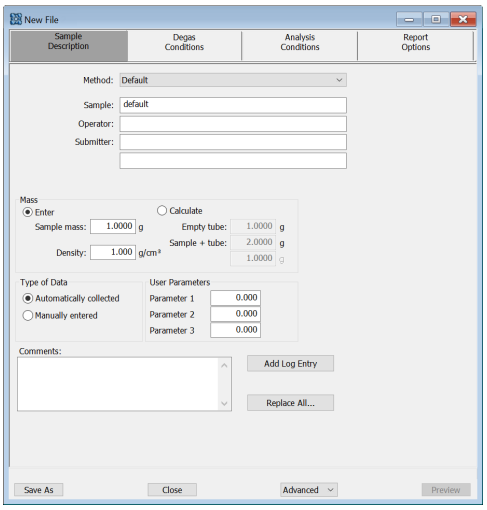
Option Presentation Display

Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample file in a single window similar to the *Basic* display option with certain functions disabled. A password is set when the *Restricted* option is selected. That same password must be entered to change to the *Basic* or *Advanced* display option. This display type is typically used in laboratories — such as the pharmaceutical industry — where analysis conditions must remain constant. The *Advanced* option is not available in the view selector at the bottom of the window when using the *Restricted* display option.
Always Open Edit View	Opens files with a *Complete* status in the tabbed file editor rather than in the editor view.
Show Degas Conditions	When enabled, displays the *Degas Conditions* tab when using *Advanced* option presentation and the Degas Conditions drop-down list when using *Basic* or *Restricted* option presentation. This option may be deselected to hide the *Degas Conditions* tab if not using a Smart VacPrep.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.

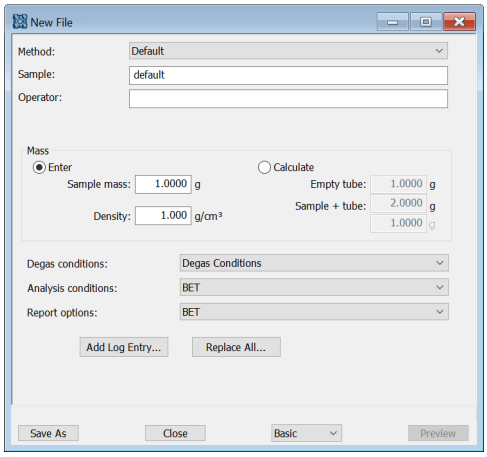
To change the view for the selected window, use the drop-down list at the bottom of the sample file editor.

The following examples show the same sample file in *Advanced* and *Basic* display. *Basic* and *Restricted* displays will look the same. A password is required if using *Restricted* format.

Option Presentation Examples



Advanced view



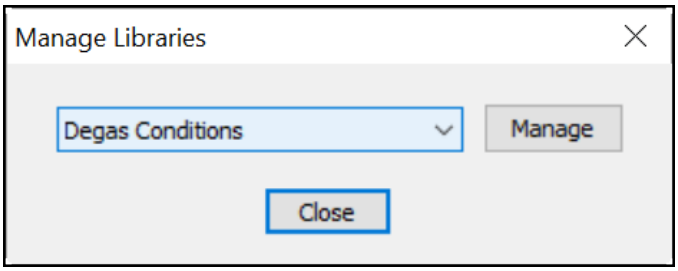
Basic or Restricted view

A sample file must be created for each analysis. The file can be created prior to or at the time of analysis. The sample file identifies the sample, guides the analysis, and specifies report options.

LIBRARIES

Options > Manage Libraries

This feature is not available when using *Restricted* option presentation.



The library provides an easy way to locate and open specific analyzer files. Libraries are located within the *File Selector* window and can be viewed only within the application.

The library gathers sample and parameter files that are stored in multiple locations — such as folders on a C: drive, a network location, a connected external hard drive, or a connected USB flash drive — providing access to all files. Even though libraries do not store actual sample and parameter files, folders can be added or removed within each library.

One library can include up to 50 folders. Other items — such as saved searches and search connectors — cannot be included.

When *removing* a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when *deleting* files or folders from within a library, they are deleted from their original file storage location. Deleted files and folders can be recovered from the Recycle Bin located on the Windows desktop.

METHODS

File > New Method

Options > Default Method

File > Open > [.MTH File]

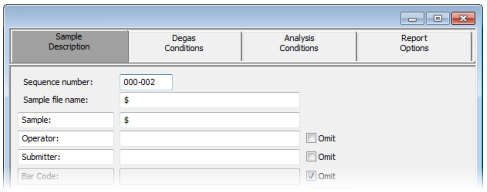
CFR Note

For 21CFR11 environments, this section is applicable only to members of the Developer group; however, members of the Analyst group may find information in this section helpful. Sample file information that is available to Analysts is created by a member in the Developer group using information in this section.

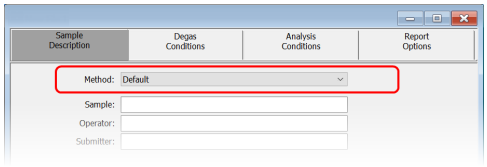
A *Method* determines the default sample identification format and sequence number. A *Method* is a template of specifications that go into a newly created sample file. It allows for the definition of complete sets of parameters for each type of sample commonly analyzed, so that only a single selection is required for each new sample file created.

The *Method* drop-down list displays only those methods applicable to the open sample file type.

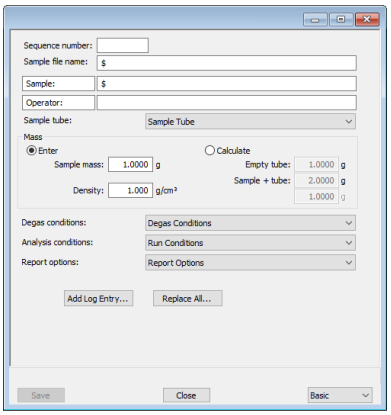
Developer Group view in a CFR21 environment



Default Method Examples



Default Method Examples



Analyst Group view in a 21CFR environment

Default Methods

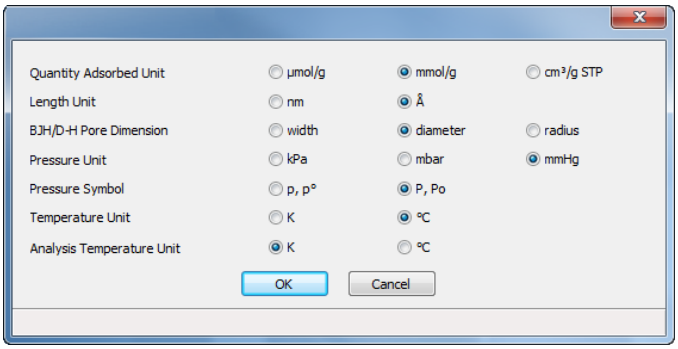
Field or Button	Description
Sample file name [*text box*]	Enter a format for the sample identification. The entry in this field becomes a part of the saved sample file name. Include the $ symbol to have the sample file number included as part of the identification.
Sample **Operator** **Submitter** **Bar Code** [*text box*]	These field labels may be renamed and the new label becomes a part of all new sample files.
Sequence Number [*text box*]	Specify a default numeric string to be used as a prefix in the *Sample* field when a new sample file is created. This number increments with each sample file created.

CONFIGURE THE ANALYZER

UNIT SELECTION

Options > Units

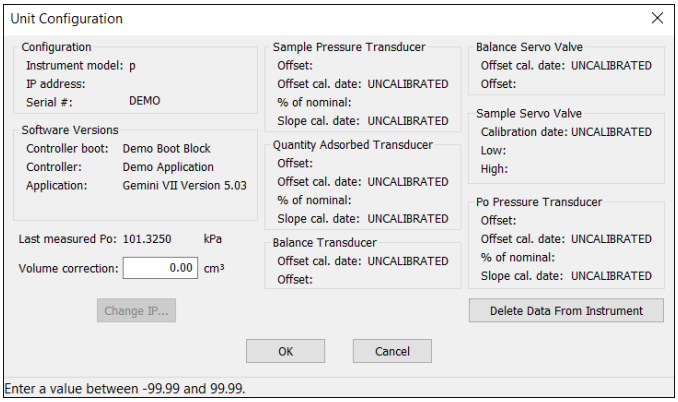
Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation in a standard installation environment.



UNIT CONFIGURATION

Unit [n] > Unit Configuration

Use to display and confirm hardware / software configurations and calibrations of the analyzer.



Unit Configuration

Field or Button	Description
Volume Correction [*text box*]	Displays the volume correction of the analyzer. If a different value is entered in this field, it is stored with sample data at the beginning of the analysis. The value in this field is used in calculating free space.
Change IP [*button*]	Click to display the *Unit IP Setup* window. Use to change the IP address and subnet mask assigned during installation. Do not edit these fields unless instructed by a Micromeritics Service Representative.
Delete Data From Instrument [*button*]	Restores the controller to factory defaults.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

INSTRUMENT STATUS

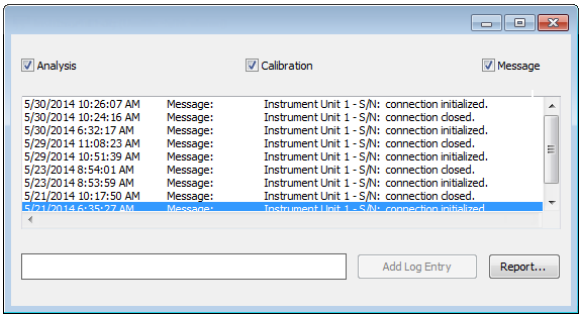
SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

CFR Note

In 21CFR11 environments, see *System Audit Trail on page 2 - 27*.

Use to display a log of recent analyses, calibrations, errors, or messages.



Instrument Log

Field or Button	Description
Add Log Entry [*button*]	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Analysis/ Calibration/ Message [*check box*]	Select the logs to display.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

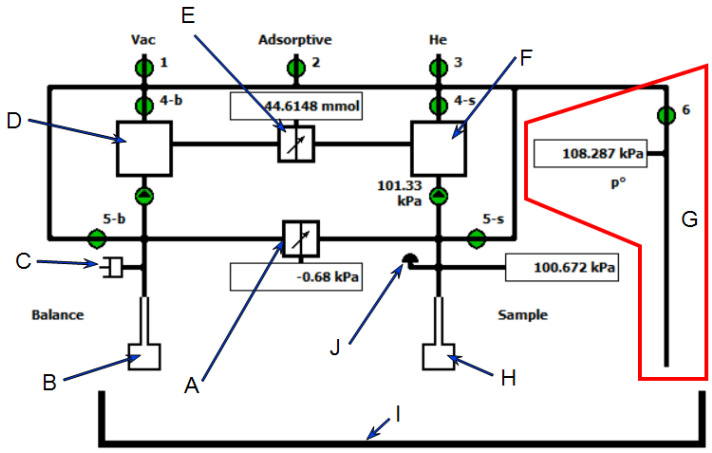
SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic

Use to enable the manual control of certain system valves and pump components on the analyzer schematic.

When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***.

If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.

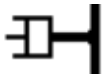


A. Differential pressure transducer
B. Balance tube
C. Balance volume adjustment
D. Balance tube reservoir
E. Quantity adsorbed transducer
F. Sample tube reservoir
G. P_o tube, valve, and transducer reading are not displayed on Model 2390a
H. Sample tube
I. Elevator
J. Pressure relief valve

Analyzer Schematic Icons

Icon or Symbol	Description
	**Open Valve**. Green indicates an open valve.
	**Closed Valve**. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
	**Servo Valve**. Closed.
	**Servo Valve**. Open.

Analyzer Schematic Icons (continued)

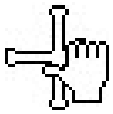
Icon or Symbol	Description
	**Elevator.**
	**Reservoir.** For the balance tube and sample tube.
	**Volume adsorbed transducer** (upper). **Differential pressure transducer** (lower). The reading for the volume adsorbed transducer is displayed just above the symbol. The reading for the sample pressure transducer is displayed just below the analysis servo valve and valve 5-s.
	**Balance volume adjustment.** Can be adjusted by a Micromeritics Service Representative.
	**Pressure relief valve.** Prevents excessive pressure build-up in the event of abnormal operation.
	**Balance tube** and **sample tube**.

Analyzer Schematic Components

Schematic Components	Description
1	Vacuum valve
2	Adsorptive valve
3	Helium valve
4-b	Balance reservoir valve
4-s	Sample reservoir valve
5-b	Balance port valve
5-s	Analysis port valve
6	P_0 port valve (models 2390 and 2390t only)

Instrument Schematic Shortcut Menus

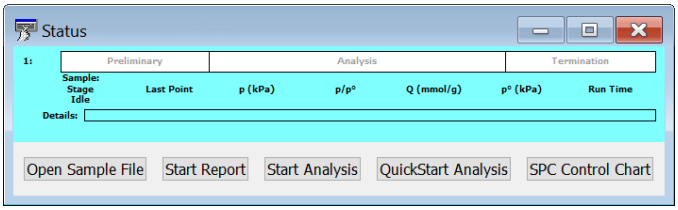
Schematic Shortcuts

Icon or Symbol	Description
Valve options 	For valves: ■ **Close.** Closes the selected valve. ■ **Close both.** Closes valves 4b and 4s or valves 5b and 5s. ■ **Open.** Opens the selected valve. ■ **Open both.** Opens valves 4b and 4s or valves 5b and 5s. ■ **Pulse.** Use to quickly turn the valve on and off allowing the operation to proceed in small increments. For servo valve: ■ **On/Off.** Turns the servo valve on or off. ■ **Increase / Decrease.** Increases pressure by dosing the sample. Decreases pressure by evacuating the sample. ■ **Enter target.** Displays the servo valve settings window. The entered target pressure display to the left of the sample servo valve icon. For balance servo valve: ■ **Disable**. Select to disable the balance servo valve. Operations for the sample servo will also be performed by the balance servo. ■ **Enable**. Select to enable the balance servo valve. Operations for the sample servo will be ignored by the balance servo.
Elevator options 	**Raise.** Select *Raise* to raise the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select *Stop* from the menu). **Lower.** Select *Lower* and press the keyboard space bar to lower the elevator. **Stop.** Stops the elevator from moving.

SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



If multiple units are attached to the computer, select *Show Status* on each *Unit [n]* menu. The status for all units displays.

EXPORT FILES

File > Export

Exported Data Example on page C - 1

Provides the option to print the contents of one or more sample or parameter files to either the screen, a printer, or a file. Data can be exported as a .PDF, .TXT, .XML, or .XLS file format. The type of data to include or exclude can be selected during the export process. When exported to a file, the data can be imported into other applications that read these file formats.

LIST FILES

File > List

Provides the option to create a list of sample file information —such as file name, date, time the file was created or last edited, file identification, and file status.

Select one or more files from the file selector, click **List**, then provide the file destination.

File Listing

No.	File Name	Date	Time	Description	Status
1	13x with CO2 at 0C Port 1B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 1	Complete
2	13x with CO2 at 0C Port 2B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 2	Complete
3	13x with CO2 at 0C Port 3B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 3	Complete
4	13x with N2 and TranSeal Port 2.SMP	8/10/2020	3:53:54 PM	13X Zeol Tube 2 w/ FS @ end of analysis, Port 2	Complete
5	13x with N2 and TranSeal Port 3.SMP	8/10/2020	3:53:54 PM	13X Zeol Tube 1A w/ FS @ end of analysis, Port 3	Complete
6	Activated Carbon with Butane C3 Port 1.SMP	8/10/2020	3:53:55 PM	Activated Carbon Tube C3 Butane Port 1	Complete
7	Activated Carbon with Butane C4 Port 3.SMP	8/10/2020	3:53:55 PM	Activated Carbon Tube C4 Butane Port 3	Complete

Example of File List

SOFTWARE SETUP

If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose.

The *Setup* program is located on the installation media and is used to reinstall the software and make analyzer changes — such as adding, moving, or removing a unit, etc.

If the IP address needs to be changed on the computer connected to the analyzer, refer to the computer's operating system manual or the internet for instructions. The IP address for the computer and the IP address specified in the setup program must match. The IP address must be 192.168.77.100.

SOFTWARE UPDATES

User Account Control in the Windows operating system must be enabled to ensure all components of the Micromeritics application are installed properly. If UAC is not enabled, right-click the *setup.exe* installer file and select *Run as administrator*.

The most current version of the instrument software can be found on the Micromeritics web page (www.Micromeritics.com).

When performing a software update, existing data files are not overwritten. There are three types of subsequent installation:

- Later version than the current installation.
- Same version as the current installation.
- Earlier version than the current installation.

Insert the setup media into the media drive. The setup program starts automatically. If the program does not start automatically, navigate to the installation media drive, locate and double-click the *setup.exe* file.

CFR Note

Existing Confirm application users and groups are not affected by software updates. Any changes to Confirm users and Confirm groups must be made using Windows Users and Groups.

SOFTWARE UNINSTALL

Uninstall Software in 21CFR11 Environments on page 2 - 28

The software can be uninstalled two ways. Either method removes only the files required to run the software - not he analysis files.

- Click the Windows *Start* icon. Scroll to the Micromeritics entry. Select the *Uninstall [analyzer]* option, then follow the prompts.
- Locate the *uninstall.exe* file in *C:\Program Files (x86)\Micromeritics\[analyzer name]* (or wherever the application was installed). Double-click the *uninstall.exe* file then follow the screen prompts.

SOFTWARE IN 21CFR11 ENVIRONMENTS

The Micromeritics Confirm applications for 21CFR11 environments require an operating system of Windows 10 Professional or Windows 10 Enterprise or higher. Management of users and groups is performed in Windows Users and Groups.

The Micromeritics Confirm application enables laboratory managers to develop analysis methods, enforce industry standards, and produce audit trails. It also enables laboratory analysts to perform analyses and produce reports.

USER PERMISSIONS

Confirm User Name	Description
mic_[*analyzer model number*]_controller	mic_[*analyzer model number*]_controller is the user name used by all installations. ■ This user should have complete control over the installation directory. ■ The application is launched under this user name and has this user's privileges to the windows file system. ■ This user should not be used by anyone or any other software that is not a Micromeritics application. ■ The system administrator has the option of modifying this account so that the password never expires. Alternatively, if the password does expire while the application is running, the application automatically changes the password for this account.

Confirm Group Name	Description
Developer Group	The default Developer group name is *mic_[analyzer model number]_developer*. Members of the Developer group: ■ have rights to all functions of the Micromeritics application - including Advanced option presentation which allows the creation and modification of methods, sample files, and parameter files. ■ can run an analysis. ■ can also be assigned Administrator rights which control the user profiles.
Analyst Group	The default Analyst group name is *mic_[analyzer model number]_analyst*. Members of the Analyst group: ■ have access to the *Basic* presentation option only. ■ may create sample files from pre-defined methods and can modify only a limited number of input fields.

OPTION PRESENTATION FOR 21CFR11 ENVIRONMENTS

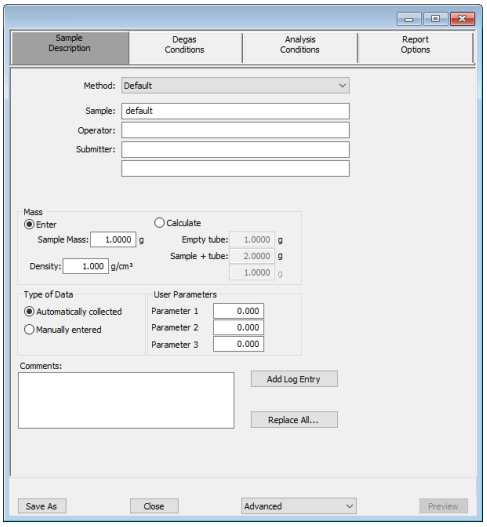
Options > Option Presentation

Option Presentation Display

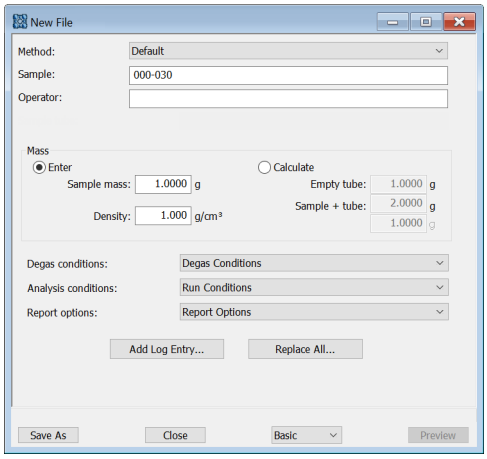
Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.

CFR Note

For members of the Developer group only. To change the view from *Advanced* (for Developers) to *Basic* (for Analysts), click the view selector drop-down list at the bottom of the window. Select either *Advanced* (when in *Basic* view) or *Basic* (when in *Advanced* view).



Advanced view / Developer group



Basic view / Analyst group

CFR Note

A sample file must be created for each analysis. The file can be created prior to or at the time of analysis. The sample file identifies the sample, guides the analysis, and specifies report options.

- The **Save** button is disabled on sample files with a *Complete* status.
- When the **Preview** button is used to view reports for sample files with an unsaved status, the report will have a *Preview* watermark.
- The **Save As** and **Print** buttons on the report window are also disabled.

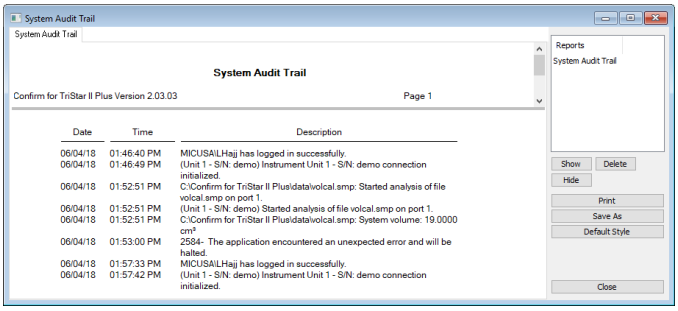
CREATE A NEW FOLDER

File > Create New Folder

Provides the option to create and name a new folder in the Confirm application folder. This option may not be available depending on how the IT Administrator configures Windows permissions.

SYSTEM AUDIT TRAIL

File > System Audit Trail



Lists the current user, successful and failed application user login attempts, and contains a description of all the changes made to sample files. Contains an audit trail of all system initializations, user login attempts, and sample analyses.

UNINSTALL SOFTWARE IN 21CFR11 ENVIRONMENTS

When the software is uninstalled using *uninstall.exe*, only the files required to run the application are removed. Parameter files, sample files, reports, calibration files, and data files are not removed.

To uninstall the software, double-click the *uninstall.exe* file located in the software installation directory, then follow the prompts.

CFR Note	To uninstall the Micromeritics Confirm application, the owner of the application directory and its contents must be set to the account of the administrator that is removing the application installation. This account must also have permission to modify the application directory and its contents. This may require modification to the owner and to the access permissions of the application directory and its contents. Upon uninstalling the Confirm application, the system administrator should go into Windows Users and Groups to remove the Confirm users and groups. See the Confirm Administrator Guide [*part number 004-42821-01*].

Depending on the network, Windows may not allow the uninstall.exe program to run. If this happens. follow these steps:

1. In Windows Users and Groups, verify that the current user is not a member of the analyst group or developer group. If so, remove the user from the group(s). Log OFF, then log back ON to the computer.
2. In Windows Explorer, in the Confirm installation directory, double-click the *uninstall.exe* file to run the uninstall program.

3 SAMPLE FILES

Software in 21CFR11 Environments on page 2 - 24

Sample files include the information required by the analyzer to perform analyses and collect data. A sample file identifies the sample, guides the analysis, and specifies report options and may be displayed in *Advanced*, *Basic*, or *Restricted* presentation display mode.

A sample file consists of parameter sets; however, parameter sets can also stand alone. A sample file may be created either prior to or at the time of analysis.

Parameter files allow for repeated use of parameter sets. For example, if the same analysis conditions exist for multiple analyses, an *Analysis Conditions* file containing the recurring conditions can be created. When the sample file is created, the *Analysis Conditions* file can be selected for the analysis conditions. Once it becomes part of the new sample file, the new file can be edited as needed without affecting the original *Analysis Conditions* file.

The analysis application contains a default method. A method is a template for sample files that contains the parameters to be used for an analysis. When a new sample file is created, all the parameters are filled with the values in the default method.

To change the view for the selected window, use the drop-down list at the bottom of the sample file editor.

CREATE SAMPLE FILES

Options > Option Presentation > Show Degas Conditions

File > New Sample > [.SMP File]

File > Open > [.SMP File]

CFR Note

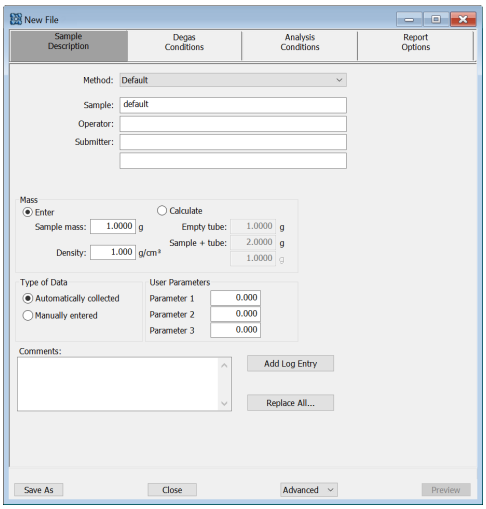
For 21CFR11 environments, this section is applicable only to members of the Developer group; however, members of the Analyst group may find information in this section helpful. Sample file information that is available to Analysts is created by a member in the Developer group using information in this section.

Each analysis must be linked with a sample file before the analysis can proceed. A sample file can consist of parameter files; however, parameter files can also stand alone.

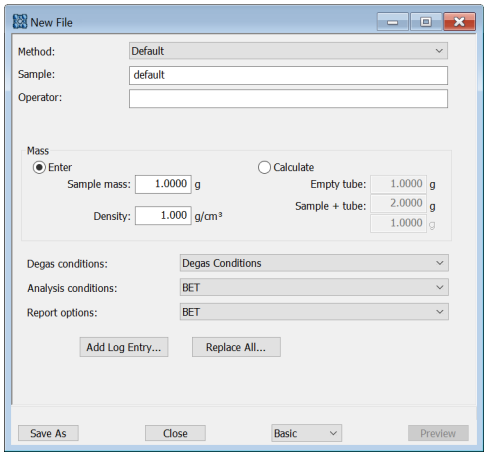
Specify or change the option presentation by selecting ***Options > Option Presentation,*** or use the view selector drop-down list at the bottom of the window.

Sample files created in the *Basic* option presentation must be selected from parameter files created in the *Advanced* option presentation. The values specified in the parameter portions of the default method are the defaults for new sample files. To navigate from one set of parameters to another, select the parameter tab across the top of the window.

The *Degas Conditions* tab displays only if enabled in ***Options > Option Presentation > Show Degas Conditions***



Advanced or Developer view



Basic or Analyst view

A bar code reader may be used to enter text into many of the fields on the *Sample Description* window. Use a mouse to click in the field first where information is to be entered then scan the bar code with the bar code reader.

Sample Files

Field or Button	Description
Add Log Entry [*button*]	Use to enter information that will display in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Bar Code [*text box*] *	Use to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Comments [*text box*]	Enter comments about the sample or analysis. Comments display in the report header.
Mass [*group box*]	Enter a value for sample mass. Mass can be changed any time before, during, or after analysis. **Enter.** Enables the *Sample mass* field. Enter a value for the sample mass. **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$
Method [*drop-down box*]	Select a method from the drop-down list.
Operator [*text box*] *	Enter operator identification information.
Sample [*text box*] *	Enter a sample description.
Submitter [*text box*] *	Enter submitter identification information.
Type of Data [*group box*]	**Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running. **Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Isotherm Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file. See ***Manually Enter Data on page 3 - 7***.

Sample Files (continued)

Field or Button	Description
User Parameters [*group box*] *	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *SPC Report*. See ***SPC Report on page 6 - 2***.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

* This field label may have been renamed or may not display if modified in ***Options > Default Methods***.

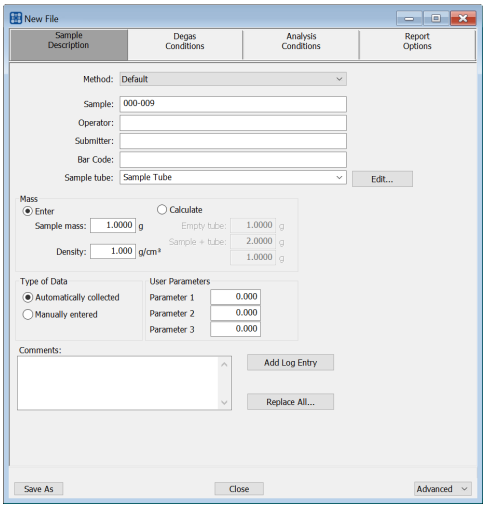
OPEN A SAMPLE FILE

File > Open > [.SMP File]

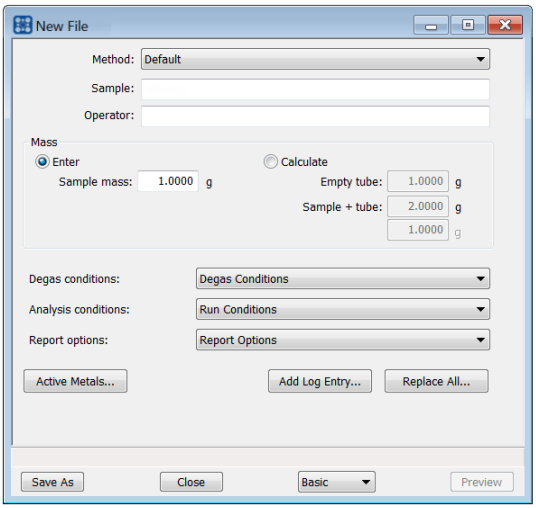
When working with an existing sample file, consider making a copy of the sample file to maintain the original configuration options.

File Status	Displays
Preparing **Prepared** **No Analysis**	Tabbed file editor
Complete **Analyzing** **Entered**	MicroActive report window

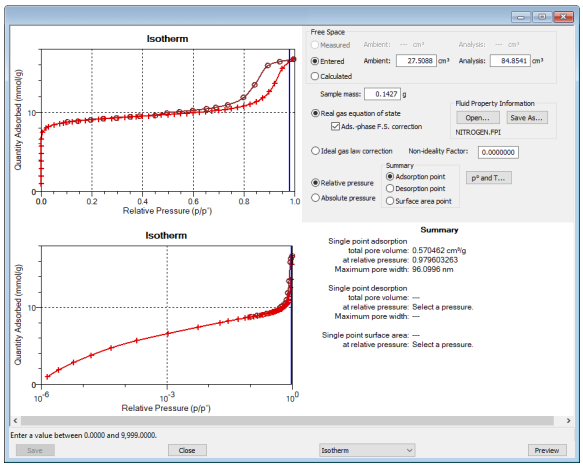
File Editor Example for CFR21



Advanced or Developer view



Basic or Analyst view



Example of a Report window

If a sample file with a *Complete* status is opened, to return to the tabbed file editor, select *Advanced* or *Basic* from the view selector drop-down list at the bottom of the window.

CFR Note	In 21CFR11 environments, this feature is applicable to members of the Developer group only.

MANUALLY ENTER DATA

CFR Note

In 21CFR11 environments, this feature is applicable to members of the Developer group only.

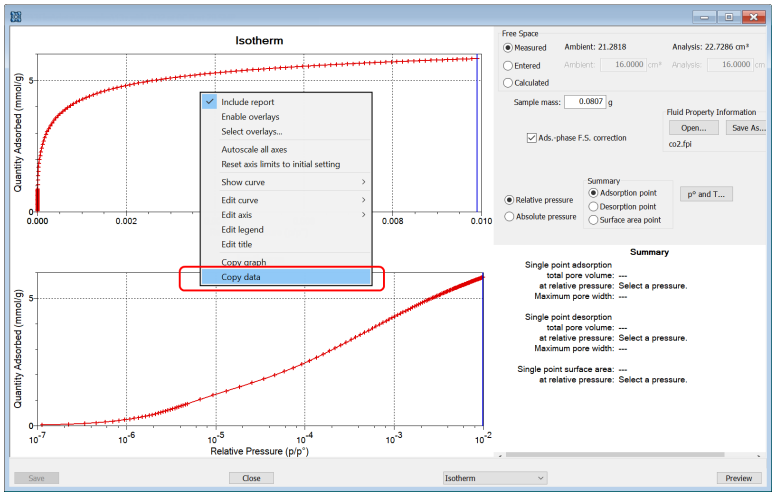
This process allows the manual entry of pressure data from a sample file with a *Complete* status. There are two methods for manually entering data into a sample file:

- Copy and paste onto the graph area of the interactive window.
- Import data into the interactive window.

COPY AND PASTE MANUALLY ENTERED DATA

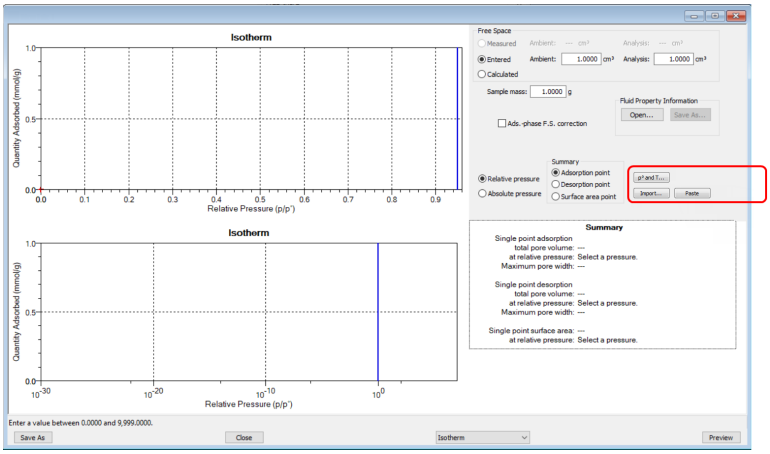
To display the file status in a search window, go to ***File > Open***. Right-click the column header then click *More...* Scroll to the *MIC* entries and enable *MIC Status*.This is a snippet

1. Open a sample file with a *Complete* status. The file will open in the interactive reports window.
2. Right-click in the graph area of the interactive reports window, then select *Copy data*.



Example of Report window

3. Open another sample file using the *Advanced* option presentation.
4. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
5. In the view selector drop-down list at the bottom of the window, click *Advanced*, then select *Isotherm*.



6. Ensure that all parameter fields are set appropriately, then click **Paste**.

IMPORT MANUALLY ENTERED DATA

When importing isotherm data from an external ASCII text file using the **Import** button on the interactive window, the ASCII text file must use the following rules:

ASCII text file format rules

Data must be in two columns and separated by a comma or white-space. Acceptable column headings are:

- Relative Pressure
- Absolute Pressure (mmHg)
- Absolute Pressure (kPa)
- Absolute Pressure (mBar)
- Quantity Adsorbed (mmol/g)
- Quantity Adsorbed (cm³/g STP)
- Quantity Adsorbed (cm3/g STP)

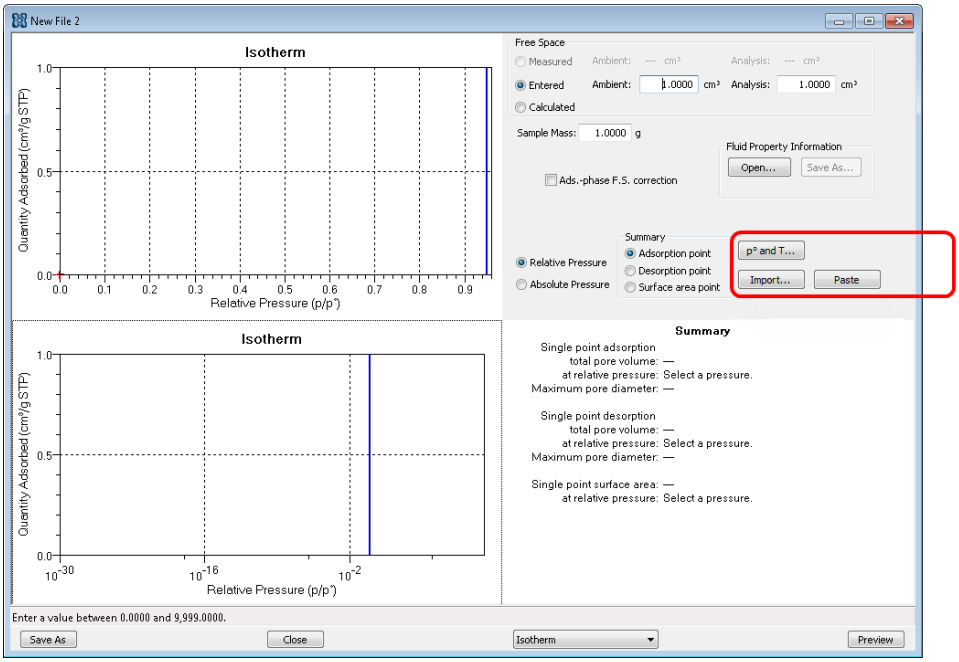
Sample Physisorption ASCII Text File

```
Silica Alumina : Adsorption
Relative Pressure        Quantity Adsorbed (cm3/g STP)
0.108629                 50.6657
0.22288                  60.7813
0.339909                 71.3095
0.459512                 84.4172
0.577447                 102.672
0.654583                 121.707
0.760074                 179.096
0.855713                 334.565
0.958511                 394.675
0.996251                 403.793

Silica Alumina : Desorption
Relative Pressure        Quantity Adsorbed (cm3/g STP)
0.996251                 403.793
0.86016                  389.626
0.753567                 256.264
0.664418                 133.099
0.542416                 96.7366
0.422295                 79.7351
0.346371                 71.5994
0.2519                   62.8256
0.152718                 54.2336
0.103389                 49.5803
```

To import the ASCII text file

1. Open a new sample file in *Advanced* option presentation.
2. On the *Sample Description* tab, select *Manually entered*.
3. In the view selector drop-down list at the bottom of the window, click *Advanced*, then select *Isotherm*.



4. Ensure that all parameter fields are set appropriately, then click **Import**.
5. Open the .TXT file. The data from the original sample file is imported and displayed. If an error message displays instead, verify that the .TXT file format is correct.

4 PARAMETER FILES

CFR Note

In 21CFR11 environments, this section is applicable only to members of the Developer group; however, members of the Analyst group may find information in this section helpful. Parameter file information that is available to Analysts is created by a member in the Developer group using information in this section.

Parameter files allow for repeated use of parameter sets. For example, if the same analysis conditions exist for multiple analyses, an *Analysis Conditions* file containing the recurring conditions can be created. When the sample file is created, the *Analysis Conditions* file can be selected for the analysis conditions. Once it becomes part of the new sample file, the new file can be edited as needed without affecting the original *Analysis Conditions* file.

Methods include both analysis conditions and report options, offering the most convenient way to repeat most analyses.

Predefined parameter files are included with the program and can be edited, as needed, or new parameter files can be created.

The following file types can exist as part of the sample file as well as individual parameter files.

Parameter File Types

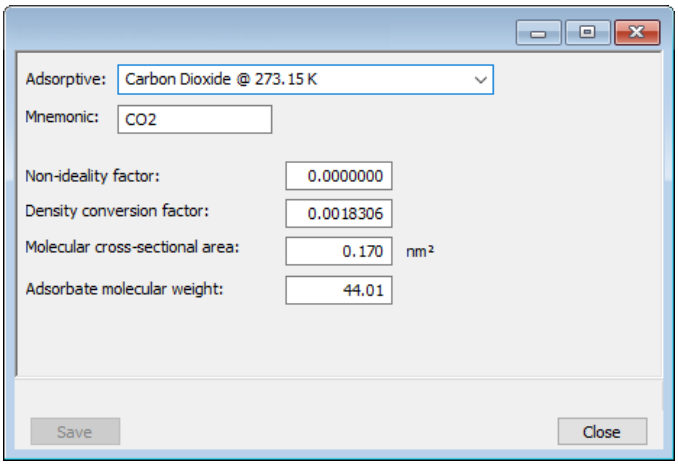
File Type	File Extension
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Degas Conditions	.DEG
Method	.MTH
Report Options	.RPO
Sample Tube	.STB

ADSORPTIVE PROPERTIES

File > Open > [.ADP File]

Or, click **Edit** next to the *Adsorptive* selection on the *Analysis Conditions* tab when in *Advanced* option presentation.

Adsorptive properties provide the adsorptive (analysis gas) characteristics for the analysis.



Adsorptive Properties

Field or Button	Description
Adsorbate molecular weight [*text box*]	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
Adsorptive [*text box*]	Name of the adsorptive gas whose properties are being defined.
Density Conversion Factor [*text box*]	Factor determined by obtaining the ratio of the gas density (STP) to the liquid density.
Mnemonic [*text box*]	Enter the mnemonic name for the adsorptive.
Molecular cross-sectional area [*text box*]	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
Non-ideality factor [*text box*]	Compensates for the forces of attraction between molecules in a real gas. This value is used for a calculated free space.

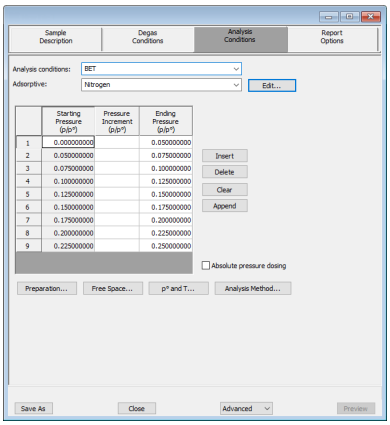
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

ANALYSIS CONDITIONS

File > Open > [.ANC File]

Or, click the *Analysis Conditions* tab when in *Advanced* option presentation.

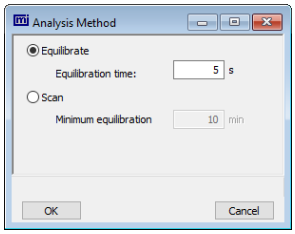
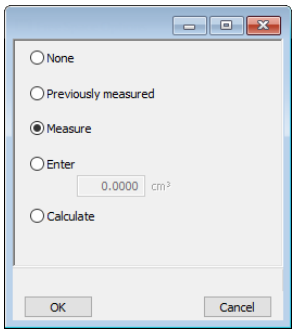
Analysis conditions specify the parameters used to guide an analysis.



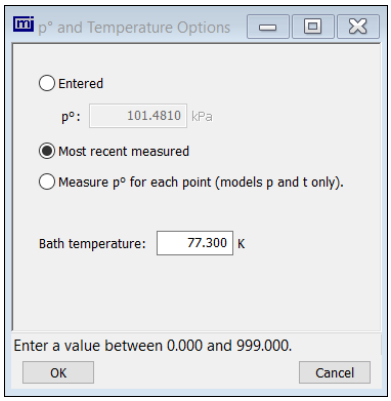
Analysis Conditions

Field or Button	Description
Absolute pressure dosing [*check box***]**	Specifies pressure targets in mmHg, mbar, or kPa instead of relative pressure. If this option is selected, the *Relative Pressure* labels and entries change to *Absolute Pressure* in the selected pressure units. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H2 adsorption on carbon at liquid nitrogen temperature.
Adsorptive [*text box***]**	Select an *Adsorptive Properties* file from the list of defined gases to be used for analysis. After selection, click **Edit** to modify adsorptive properties.
Analysis conditions [*drop-down box***]**	Use to browse for an *Analysis Conditions* file that contains analysis condition parameters to be used in the analysis.

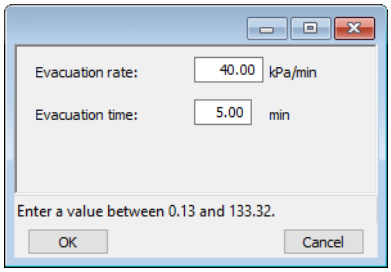
Analysis Conditions (continued)

Field or Button	Description
Analysis Method [*button*]	Use to enter equilibration time in seconds or enter the minimum equilibration time for scanning in minutes 
Free Space [*button*]	Use to specify how free space is determined.  **None.** Select if free space will not be used. **Previously measured.** Select if free space has been previously measured. Recommended if measuring a series of samples with similar volumes. **Measure.** Select if the free space is to be measured before the analysis begins. **Enter.** Allows manual entry of free space. **Calculate.** Select to have the free-space calculated using the value entered for *Volume Correction* (entered on the ***Unit [n] > Unit Configuration*** window) and the non-ideality factor (entered on the *Adsorptive Properties* window).

Analysis Conditions (continued)

Field or Button	Description
p° and T [*button*]	 Use to select options for obtaining the saturation pressure (p°) and analysis bath temperature. **Entered.** Select to manually enter the p°. **Most recent measured.** Select to use the most recently measured saturation pressure. **Measure p° for each point (models p and t only).** Select if p° is to be measured for each point. **Bath temperature.** Calculates p° using the entered bath temperature.

Analysis Conditions (continued)

Field or Button	Description
Preparation [*button*]	Use to enter analysis preparation details.  **Evacuation rate.** The maximum rate of change of pressure when evacuating the sample tube. **Evacuation time.** The length of time for preliminary evacuation which takes place prior to the free space measurement or sample analysis if free space is to be entered or calculated.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DEGAS CONDITIONS

File > Open > [.DEG File]

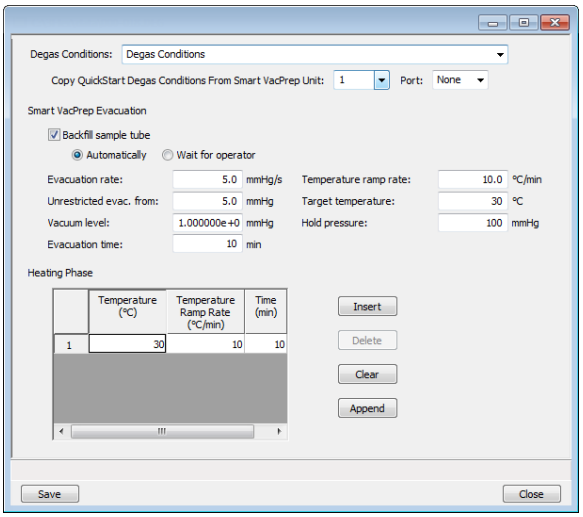
Or, click the *Degas Conditions* tab when using *Advanced* option presentation.

Use this option only when the Smart VacPrep is installed. Degassing is a required step in preparation for an analysis. The *Degas Conditions* tab provides settings that will be automatically applied during the degassing procedure when using the Smart VacPrep.

If using a FlowPrep or a VacPrep use the heating phase section for reference only.

The *Degas Conditions* tab displays only if enabled in ***Options > Option Presentation > Show Degas Conditions***.

The *Smart VacPrep Operator Manual* can be found on the Micromeritics web page (www.Micromeritics.com).



Degas Conditions

Field or Button	Description
Copy QuickStart degas conditions from Smart VacPrep Unit [*drop-down box***]**	Use to copy the degas conditions settings from the selected Smart VacPrep unit and port.
Degas conditions [*drop-down box***]**	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.

Degas Conditions (continued)

Field or Button	Description
Heating Phase [*table*]	This option is applicable when degassing with a Smart VacPrep . Enter up to five stages of degas conditions. **Temperature.** Temperature at which the sample is to be held while degassing. **Time.** How long the sample is to be held at the specified temperature before beginning to cool down. **Temperature Ramp Rate.** The rate at which the temperature will change while advancing to the hold temperature.
Smart VacPrep Evacuation [*group box*]	**Backfill sample tube.** Indicate if the sample tube should be backfilled automatically or wait for operator response. **Evacuation rate.** Rate used for evacuation. **Evacuation time.** Length of time for preliminary evacuation before proceeding with the *Heating Phase* temperature schedule. The timer starts when the vacuum level is reached. **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold* pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity. **Target temperature.** Targeted temperature for evacuation. **Temperature ramp rate.** Rate at which the temperature is to change when advancing to the target pressure. **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. **Vacuum level.** Evacuation time starts when the vacuum level is reached.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

REPORT OPTIONS

File > Open > [.RPO File]

Or, click the *Report Options* tab when in *Advanced* option presentation.

About Reports on page 6 - 1
Selected Report Options on page 7 - 1

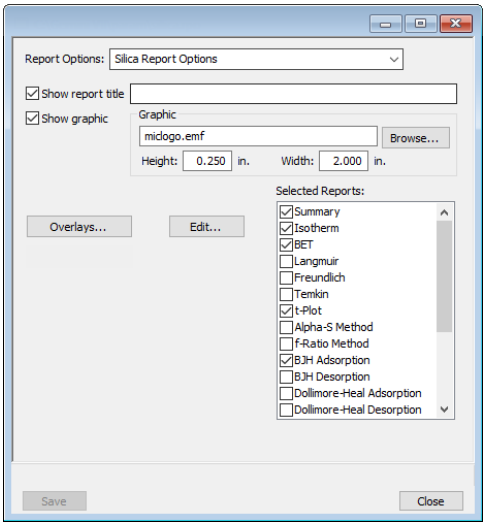
The *Calculations* document can be found on the Micromeritics web page (www.Micromeritics.com).

Additional reports are available using the *Reports* menu.

Use to specify report options for data collected from an analysis or manually entered data. *Report Options* files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations. These files may contain tabular reports, plots, or both, as well as advanced report tables.

Customized report options files can be created then loaded into a sample file, allowing quick generation of reports.

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis.



Report Options

Field or Button	Description
Edit [*button*]	Opens a dialog box for settings options for the selected report. Select the reports to include in the report preview, printed report, or report file.
Overlays [*button*]	See *Graph and Sample Overlays on page 6 - 27*.
Report Options [*drop-down box*]	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports [*group box*]	Select the report names to include in the report.
Show graphic [*text box*]	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [*check box*]	Select then enter a report title to appear on the report header.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

5 PERFORM AN ANALYSIS

CFR Note	In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

DEWAR PRECAUTIONS

	Always handle glass Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. If in doubt, contact your safety officer.
	Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.
	Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.

When handling Dewars containing liquefied gases or cryogenic liquids:

- Wear protective equipment:
  - goggles or face shield
  - an insulated or rubber apron
  - insulated gloves
- When pouring liquefied gases from one container to another:
  - cool the receiving container gradually to minimize thermal shock
  - pour the liquified gas slowly to prevent splashing
  - vent the receiving container to the atmosphere

For Glass Dewars

- 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 has a protective coating.
- Do not handle heavy objects above the Dewar. If unavoidable, place a protective cover over the Dewar opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.
- If the Dewar has a protective mesh covering, do not remove it. This cover minimizes the risk of flying particles should the Dewar be knocked over, dropped, or broken.

PREPARE FOR ANALYSIS

The steps in this topic properly prepare the equipment for an analysis. It is recommended to perform the tasks in the provided order.

CLEAN AND LABEL SAMPLE TUBES

The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

Sample tubes and filler rods must be clean and dry before samples are added and weighed. The following table indicates which materials are needed for cleaning. The procedures following the materials list are recommended.

Supplied by Micromeritics	Supplied by User
■ Filler rod ■ Funnel ■ Sample data worksheet ■ Sample tube ■ Sample tube brush ■ Sample tube rack ■ Sample weighing support ■ Stopper for sample tube	■ Acetone or isopropyl alcohol ■ Analytical balance ■ Detergent (such as Alconox) ■ Drying oven ■ Forceps ■ Insulated gloves ■ Pipe cleaners ■ Rubber gloves or clean, lint-free cloth ■ Safety glasses ■ Ultrasonic cleaning unit ■ Waste container

1. Preheat drying oven to 110 ºC.
2. Verify that the ultrasonic cleaning unit is clean.
3. Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water and fill the ultrasonic unit with enough water to cover the sample tubes and filler rods (if used). If too much detergent is used, it may be difficult to rinse from the sample tubes. Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water.
4. Fill the sample tubes with warm water and place them in the ultrasonic cleaning unit, then place the filler rods in the unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.

5. Use rubber gloves to ensure no oils or residue are transferred to the clean tubes and filler rods, then remove the sample tubes and filler rods from the unit.
6. Clean the interior of the sample tubes with the brush supplied with the analyzer.
7. Rinse the sample tubes and filler rods thoroughly with hot water. Rinse again with isopropyl alcohol or acetone. If isopropyl alcohol or acetone is not available, deionized water may be used.

8. Stand the sample tubes on the sample tube rack and place the filler rods in a basket or in the rack. Bake in a vacuum oven for two hours at 110 °C.

Samples tubes can also be cleaned with high purity acetone or isopropyl alcohol and dried for about 10 minutes under heat. If using this method, continue with step 10.

9. Remove the sample tubes and filler rods from the oven and allow to cool.

Do not insert the filler rods at this time. Filler rods are inserted before the sample tube is installed on the analysis port.

10. Blow out the sample tubes with oil-free compressed air.
11. Rinse the sample tube closure with isopropyl alcohol, then wipe the sample tube closure dry with a clean, lint-free cloth.
12. Label the sample tube and stopper for identification.
13. Replace the rubber stopper.

DETERMINE THE SAMPLE MASS

Sample Data Worksheet for Gas Adsorption on page E - 2

The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

Clean, dry sample tubes are essential for accurate results. How much sample to use can be determined best by experiment. In general, a sample providing 40 to 120 square meters of total surface area is recommended for nitrogen analysis. Less than 40 square meters may cause unreliable results. More than 120 square meters will extend analysis time.

Smaller quantities are required for samples having high surface areas. These samples require careful weighing after degassing because a small error may represent a considerable percent of total weight. Proper weighing techniques are most important in this case. Use no less than 100 mg to reduce the effect of weighing errors.

Care should be taken when loading powders: the accessory funnel is useful for this purpose. Large granules or chunks may be loaded with forceps.

Analysis results are expressed in units of surface area per gram of sample; therefore, it is important to know the true sample mass.

Follow the instructions on the *Sample Data Worksheet* and complete all fields to find the true sample mass.

1. Record the sample tube identification on the *Sample Data Worksheet.*
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).
3. Place the empty sample tube set (empty sample tube and stopper) on the sample weighing support and place it on the balance.
4. Record the stabilized mass on the *Sample Data Worksheet* . Remove the sample tube set from the balance.

Do not touch the sample with bare hands while performing the following steps. Doing so could affect the accuracy of results.

5. Place a sample container on the balance. Tare the balance and allow it to stabilize to zero.
6. Slowly pour the specified amount of sample into the sample container.
7. Remove either the rubber stopper from the sample tube.
8. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.

9. Replace the rubber stopper.
10. On the *Sample Data Worksheet,* record the following:
    - Mass of the sample tube set with the sample.
    - Subtract the *Mass of empty sample tube set* from the *Mass of sample tube set plus sample.*

DEGAS THE SAMPLE

If using the Smart VacPrep degasser, go to ***Smart VacPrep > Unit [n] > Start Degas***, then degas the sample using menu commands and information entered on the *Degas Conditions* tab. The Smart VacPrep Operator Manual can be found on the Micromeritics web page (www.Micromeritics.com).

After the sample has been weighed, use a degassing unit to remove any contaminants which may have adsorbed to the surface or pores. Appropriate degassing units are available from Micromeritics.

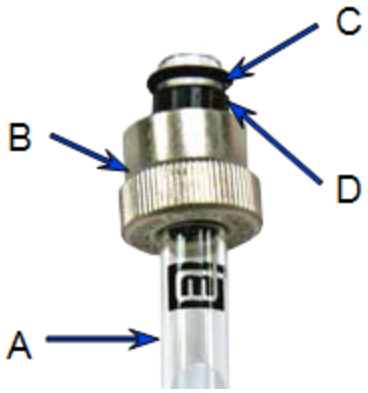
After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample. Record the mass on the ***Sample Data Worksheet for Gas Adsorption on page E - 2*** as *Mass of Sample tube set plus sample (After Degas)*.
2. Subtract the *Mass of empty sample tube set (Before Degas)* from the *Mass of Sample tube set plus sample (After Degas)* to obtain the sample's mass. Record this value as *Mass of sample (After Degas)*.

SAMPLE TUBE INSTALLATION

The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

1. Remove the sample tube stopper, if used.
2. Place the connector nut, ferrule, and O-ring onto the sample tube stem. The sample tube ferrule is tapered slightly on one end, however it may be installed with the tapered end in the up or down position.



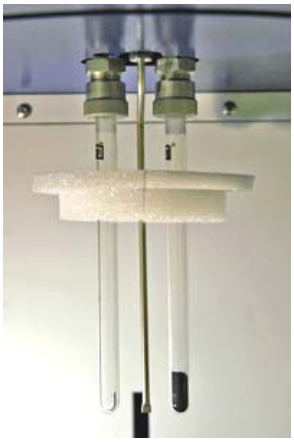
A. Sample tube or balance tube
B. Connector nut
C. O-ring
D. Ferrule

If analyzing samples with a surface area of 1.0 m^2 or less, use filler rods in the sample and balance tubes. See *Analyze Samples with a Total Surface Area of 1.0m2 or Less on page A - 1* .

3. Attach the sample tube to the analysis port. Ensure it is fully in the port. Secure it in place by screwing the connector nut into the analysis port. Hand tighten the connector nut.
4. If the balance tube is not installed, attach it to the balance port following the above procedures. If a filler rod was used in the sample tube, one must be used in the balance tube.

It is not necessary to remove and replace the balance tube between analyses unless it has been contaminated or if using a different size sample tube.

5. Place the Dewar cover under the sample and balance tubes and slide upward until it is 7.5 cm (3 in.) from connector nuts.



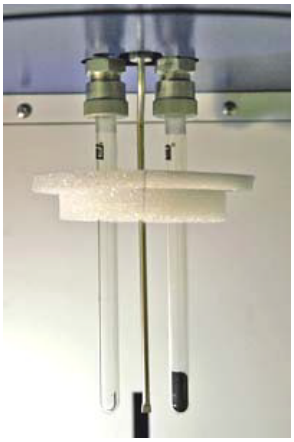
FILL AND INSTALL THE DEWAR

Dewar Precautions on page 5 - 1

1. Fill the Dewar with the analysis bath liquid (such as liquid nitrogen) to no higher than 1 in. (2.5 cm) from the top for a large Dewar or no higher than 3/4 in. (2 cm) from the top for a smaller Dewar.

Incorrect fluid levels can lead to measurement errors. Check the level of the bath liquid before each analysis.

2. Check the level of the analysis bath liquid. For best results, if the Dewar has not been used for a while, allow approximately 30 minutes for the temperature of the Dewar to stabilize with the bath liquid, then recheck the level of the bath liquid. Add additional liquid if necessary.
3. Slide the Dewar cover to approximately 7.5 cm (3 in.) from the sample port nuts to ensure a proper seal on the top of the Dewar.



4. Place the Dewar on the elevator. A Dewar support is not necessary for the larger Dewar used with the Gemini 2390t. Place the Dewar directly on the elevator.
5. Close the safety shield on the front of the analyzer.

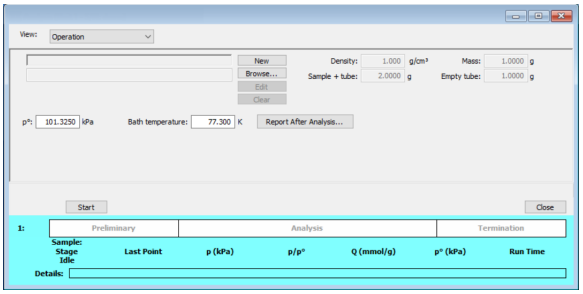
PERFORM A SAMPLE ANALYSIS

Unit [n] > Start Analysis

CFR Note

In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

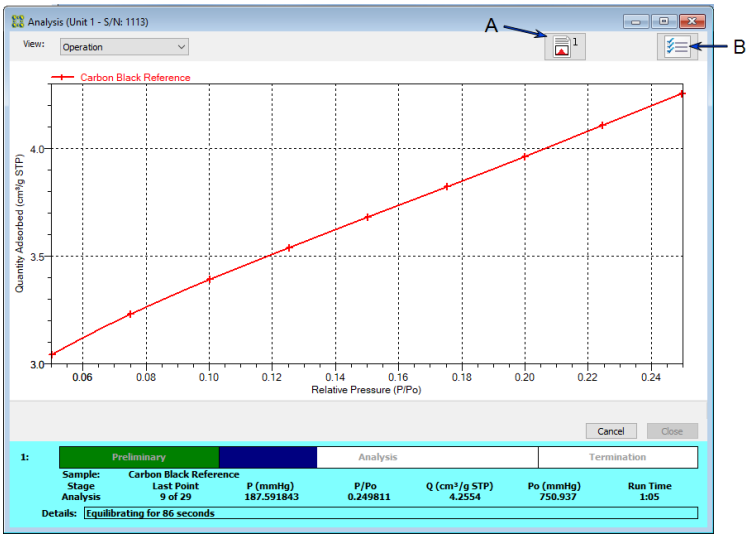
Ensure the analyzer safety shield is closed before beginning an analysis. If the analyzer is operated at an excessive pressure, the sample or balance tube could become dislodged from its port, possibly causing personal injury or damage to the equipment.



Sample Analysis

Field or Button	Description
Density / Mass / Sample + Tube / Empty Tube [*text box*]	Enter values for the sample's mass and density. These values may be edited after analysis.
New [*button*]	Creates a new sample file.
Sample tube [*drop-down box*]	Select the sample tube to be used for this analysis.
Start [*button*]	Click to start the analysis.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.



A. Port report buttons
B. Live graph settings

Sample Analysis Graph

Field or Button	Description
Live Graph Settings [*button*]	Select Thermal transpiration, x-axis Quantity (relative or absolute pressure), and the x-axis Scale (linear or logarithmic).
Report after analysis [*button*]	Generates reports to the selected destination when the analysis is complete.
Display Port [*button*]	Generates a report on data being collected . The reports are displayed on the computer monitor only.
Status window	Displays the last point pressure and relative pressure for each port.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

PERFORM AN EMPTY TUBE ANALYSIS

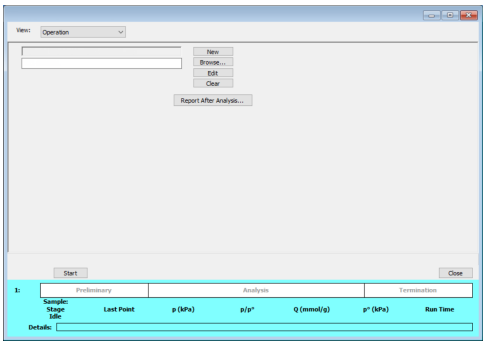
Unit [n] > Empty Tube Analysis

CFR Note

In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

The volume correction is only used when calculated free space is selected. It accounts for any difference in volume between the balance tube and the empty sample tube, and may be determined by the following procedure: Perform a liquid nitrogen analysis using two empty sample tubes of the same size. Select Measure for free-space option (see ***Perform an Empty Tube Analysis for Diagnostic Purposes on page 10 - 17***). After the analysis, record the reported free-space difference in the Unit Configuration dialog (cross-reference) and use it for these two tubes only for future analyses with calculated free space.

Ensure the analyzer safety shield is closed before beginning an analysis. If the analyzer is operated at an excessive pressure, the sample or balance tube could become dislodged from its port, possibly causing personal injury or damage to the equipment.



1. Click **Browse** to select a sample file.
2. Click **Report after analysis** to automatically generate reports when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK**.
3. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status changes from the Idle state at the bottom of the window. When analysis is complete, remove the sample tube and store (or dispose) of the sample material as applicable.

4. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status changes from the Idle state at the bottom of the window. When analysis is complete, remove the sample tube and store (or dispose) of the sample material as applicable.

Perform a Reference Material Analysis

CFR Note

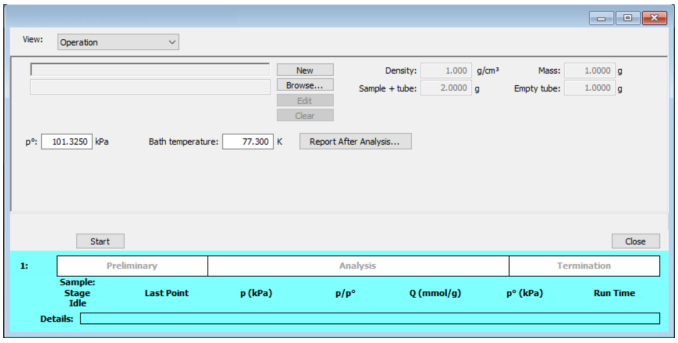
In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

A reference material analysis is used to verify the analyzer is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether quantities are in or out of specification.

When running a reference material analysis, use the appropriate reference material provided in the accessories kit to perform this analysis. The results should match those shown on the label of the reference material bottle, within the tolerance level.

Ensure the analyzer safety shield is closed before beginning an analysis. If the analyzer is operated at an excessive pressure, the sample or balance tube could become dislodged from its port, possibly causing personal injury or damage to the equipment.

1. Go to ***File > New Sample > [.SMP]*** to create a new sample file.
2. Click **Replace All**. Locate and select the file *Carbon.smp*. Click **Load**. This file contains the appropriate conditions for analyzing the carbon reference material. All values will be copied into the new sample file. Edit fields as needed.
3. Click **Save As**, enter a new file name if necessary, click **Save**, then click **Close**.
4. Clean and label a sample tube. If installing a new balance tube, clean and label it also.
5. Determine the sample mass.
6. Degas the sample using a degassing unit.
7. Install the sample tube with degassed sample on the sample port.
8. Install the balance tube (if not installed) on the balance port.
9. Fill the Dewar with liquid nitrogen to about 5 cm (2 in.) from the top. Allow the Dewar to equilibrate to ambient conditions (approximately 30 minutes).
10. Close the sample compartment door.
11. Go to ***Unit [n] > Start Analysis***.



12. Click **Browse**. Locate and select the sample file created previously for this analysis. Click **Open**.
    a. Click **Report After Analysis**.
    b. Select *Report after analysis* and *Preview* as the destination. Click **OK**.
13. Click **Start**.
14. Compare the BET Surface Area in the report with the BET Surface Area on the reference material bottle.

    - If results are within tolerance, the analyzer is operating properly.
    - If results are not within tolerance, refer to the following table for possible causes and actions. After performing the action, perform the reference material analysis again.

Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Clean and verify the gas lines.
The measured free space is too high. This indicates the helium may not be pure enough.	Helium must be 99.995% pure.

PERFORM A QUICKSTART ANALYSIS

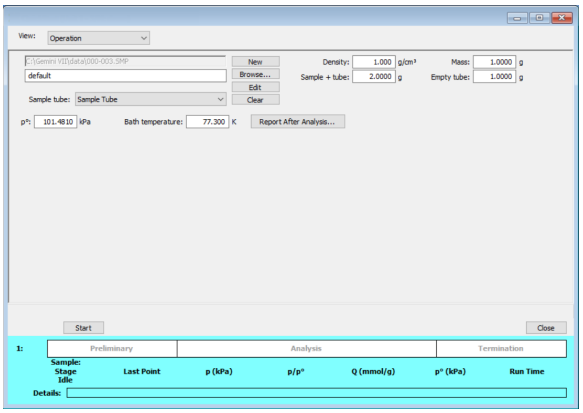
Unit [n] > QuickStart Analysis

CFR Note

In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

The *QuickStart Analysis* option automatically creates a sample file and assigns the default parameters. Click **Edit** to modify the parameters or **Browse** to select another file.

Ensure the analyzer safety shield is closed before beginning an analysis. If the analyzer is operated at an excessive pressure, the sample or balance tube could become dislodged from its port, possibly causing personal injury or damage to the equipment.



QuickStart Analysis Fields and Buttons

Field or Button	Description
Sample tube [*drop-down box*]	Click the drop-down arrow to select a sample tube file. This option is applicable only if the calculated method for measuring free space is selected for analysis conditions.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

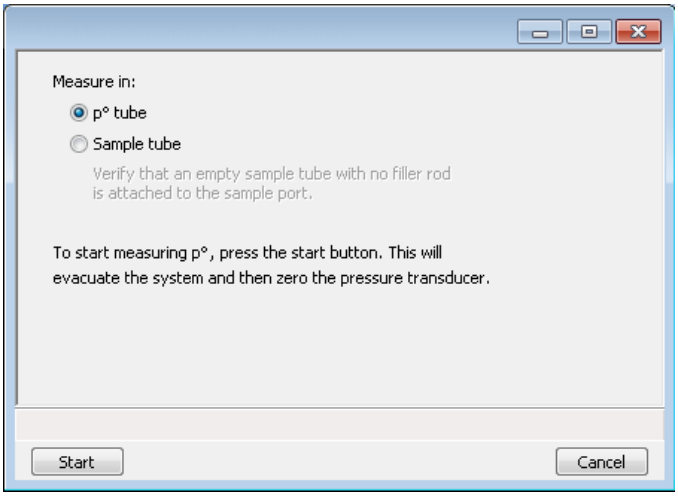
MEASURE SATURATION PRESSURE

Unit [n] > Start p° Measurement

CFR Note

In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

Starts a saturation pressure measurement.



Gemini Model	Description
2390a	p° can only be measured in the sample tube, therefore selections are not provided. Verify that a sample tube is attached to the sample port and that it does not contain a filler rod. Click **Start**.
2390p and 2390t	A p° tube is installed on these models. p° can be measured in the p° tube or in the sample tube. If using a sample tube, ensure that a sample tube is attached to the sample port and that it does not contain a filler rod. Click **Start**.

After analysis, go to ***Unit [n] > Unit Configuration***. The recorded value now displays in the *Last Measured Po* field.

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6 About Reports

Reports > Start Report

Generates a report on a sample analysis.

Reports can be generated for data collected on a sample that has completed analysis, collected on a sample currently being analyzed, or manually entered.

Reports > Close Reports

Closes all open reports. This option is unavailable if reports are being generated.

Reports > SPC Report Options

See *SPC Report on the next page*.

Reports > Regression Report

See *Regression Report on page 6 - 3*.

Reports > Control Chart

See *Control Chart Report on page 6 - 5*.

Reports > Heat of Adsorption

See *Heat of Adsorption Report on page 6 - 8* .

Start Reports

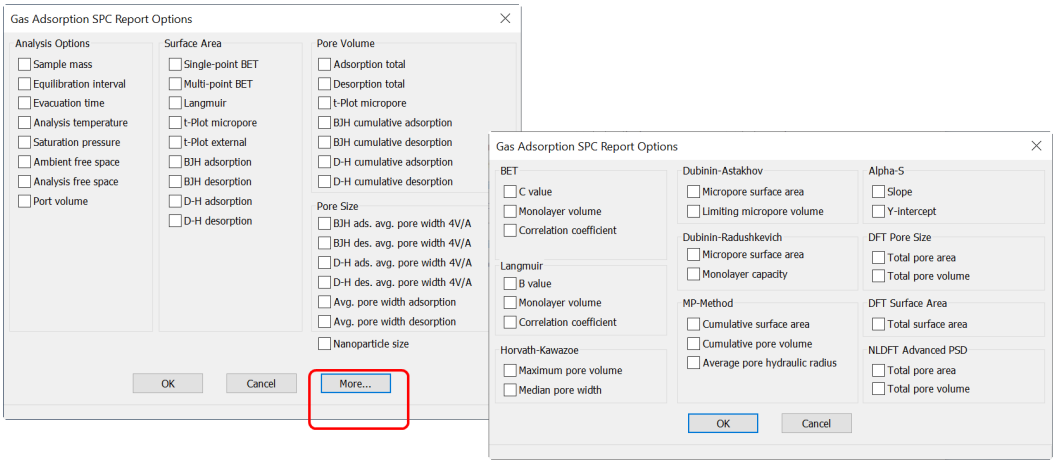
Reports > Start Report

Starts the selected report. Select a file from the *Files* list. Ensure the selected file has a status of either *Complete* or *Analyzing*.

SPC REPORT

Reports > SPC Report Options

Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.

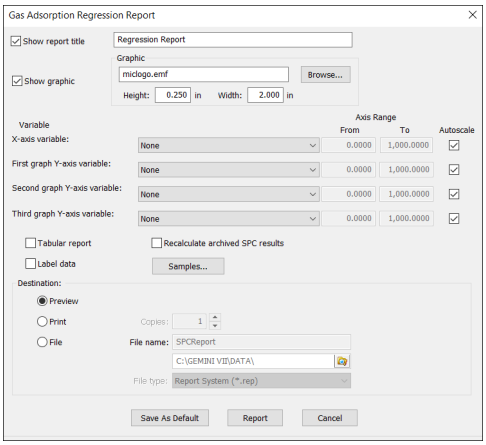


The selected items display as graph variable selections in ***Reports > Regression Report*** and graph selections in ***Reports > Control Chart***. If report options for NLDFT Advanced PSD are required, click **More**.

REGRESSION REPORT

Reports > Regression Report

Use to generate a Statistical Process Control (SPC) Regression report 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 is also reported.



Gas Adsorption Regression Report

Field or Button	Description
Autoscale [*check box*]	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range [*text box*]	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data [*check box*]	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results [*check box*]	The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report [*button*]	Generates the report.
Samples [*button*]	Select additional sample files to add to the report.
Save as Default [*button*]	Click to save selected report options as default report settings.

Gas Adsorption Regression Report (continued)

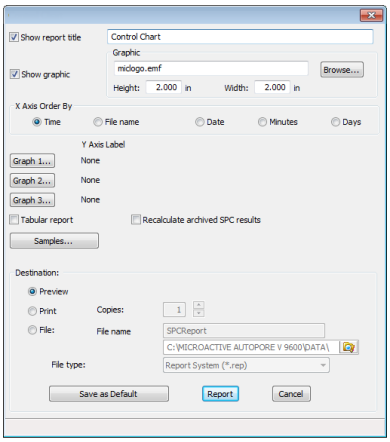
Field or Button	Description
Show graphic [*check box*]	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [*check box*]	Select then enter a report title to appear on the report header.
Tabular report [*check box*]	Generates a tabular report of the included samples that contains the numeric values contributed by each sample.
X- and Y-Axis variable [*drop-down box*]	Designates the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

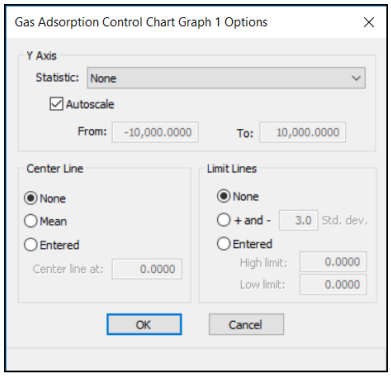
CONTROL CHART REPORT

Reports > Control Chart

Generates a Statistical Process Control *(SPC)* chart report which plots the changes in a statistic.



Gas Adsorption Control Chart Report

Field or Button	Description
Graph [*n*] [*button*]	Defines the y-axis of each graph.  **Statistic.** Displays the SPC variables selected on the ***Reports > SPC Report Options*** window. The selected variable will be plotted for each selected sample. This selection also becomes the y-axis label. **Autoscale.** Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the *From* and *To* fields.

Gas Adsorption Control Chart Report (continued)

Field or Button	Description
	Center Line. Displays placement options for the center line in the graph. Select *Entered* to specify placement of the line or *Mean* to place the center line at the calculated mean value for the selected samples. **Limit Lines.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.
Recalculate archived SPC results [*check box***]**	The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report [*button***]**	Generates the report.
Samples [*button***]**	Select additional sample files to add to the report.
Show graphic [*check box***]**	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [*check box***]**	Select then enter a report title to appear on the report header.
Tabular report [*check box***]**	Generates a tabular report of the included samples that contains the numeric values contributed by each sample.

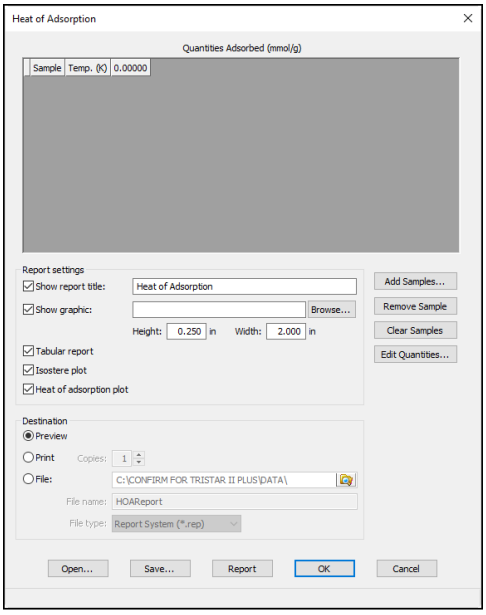
Gas Adsorption Control Chart Report (continued)

Field or Button	Description
X Axis Order By [*group box*]	Select the order in which x-axis statistics are placed. Sort by: **Time.** Time the files were analyzed. **File name.** Alphanumeric order. **Date.** Date the files were analyzed. **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption

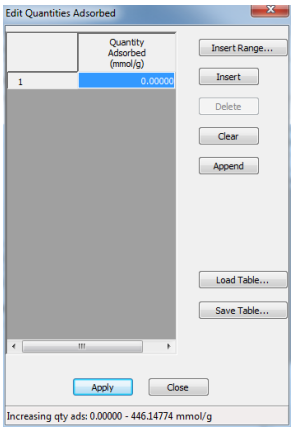


Use to select sample files, define quantities, and generate a *Heat of Adsorption* report. The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity and for providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms are obtained on the same sample using the same adsorptive but at different temperatures to obtain the heat of adsorption.

Heat of Adsorption Report

Field or Button	Description
Add Samples [*button*]	Adds a sample file to the table.
Clear Samples [*button*]	Removes all entries from the table.
Edit Quantities [*button*]	Specifies the range of surface coverage to include in the report.

Heat of Adsorption Report (continued)

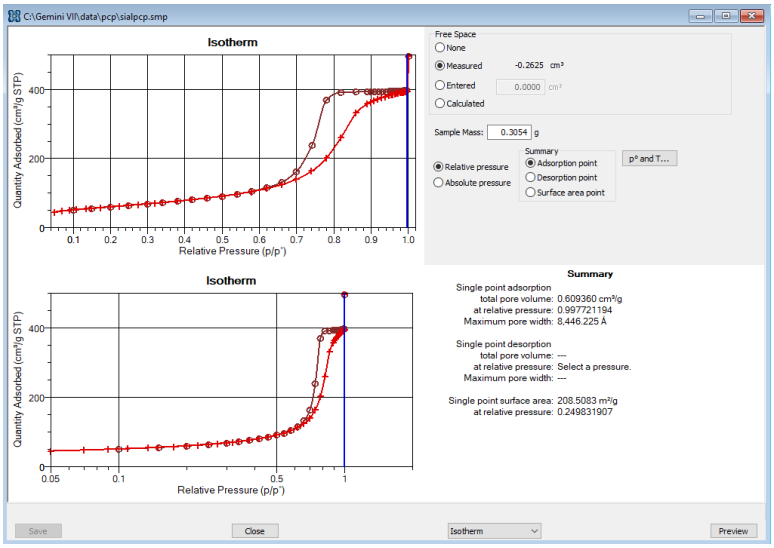
Field or Button	Description
	 **Insert Range.** Specifies the starting and ending quantities adsorbed and number of points to insert. **Load Table.** Imports values from another file. **Save Table.** Saves the current table as a .QNT file. **Apply.** Applies all table changes.
Heat of adsorption plot [*selection*]	Generates the *Heat of Adsorption* data in a graphical format.
Isostere plot [*selection*]	Generates a graph showing quantities of gas adsorbed versus the temperature.
Remove Sample [*button*]	Removes the selected sample from the list.
Show graphic [*check box*]	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [*check box*]	Select then enter a report title to appear on the report header.
Tabular report [*check box*]	Generates a tabular report of the included samples that contains the numeric values contributed by each sample.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

INTERACTIVE REPORTS

When opening a sample file that contains data from a complete or in-progress analysis, the interactive reporting feature is enabled.

1. When opening a sample file that contains analysis data, a window with the following information will display:
   - a linear plot and log plot of the data collected during analysis.
   - a summary of the analysis giving a single total pore volume and surface area.



2. To view the plots in either relative or absolute pressure, select either the *Relative Pressure* or *Absolute Pressure* option.
3. To view the reports selected for generation during the analysis, click **Preview**.
4. From the view selector drop-down list at the bottom of the window, do either of the following:
   - Change the option presentation of the sample description window to either *Basic* or *Advanced* to modify certain file parameters.
   - Select another plot from the list and edit the data contained in the plot.
5. When ranges are edited, the changes are reflected immediately in the plots and the summary data displayed in the window. Some editing options are:
   - Drag the blue bars to increase or decrease the range of data included in the plot.
   - Right-click to display a popup menu to include reports; enable or select overlays; edit curves, axes, legends, titles; and copy and paste the data in a graph or in tabular format.
6. Click **Save**.

MICROACTIVE REPORTS

MicroActive reports are generated automatically after an analysis is performed. This feature provides a quick and easy way to investigate and manipulate analysis data using a variety of reporting methods.

When a sample file with a status of *Complete*, *Analyzing* or *Entered* is opened, a linear plot and log plot of the data collected during analysis are displayed as well as a summary of the analysis giving the total pore volume. Numerous reports are accessible from a drop-down menu.

When a report is opened, plots and summary data are displayed, and in some reports certain parameters (for example, thickness curve type, pore geometry, and interaction parameters) are also displayed. Plots may be edited by selecting the data points or data point range to be included in the plots and modifying the parameters. When a report is edited, the results are immediately reflected in the plots and summary data.

Evaluate Report Results

Analysis reports provide a record of test conditions, experimental data, and information extracted from the experimental data by application of various reduction methods. This topic discusses the elements of various reports presented by Micromeritics' analyzers and suggests ways by which the merit of the reported information may be evaluated.

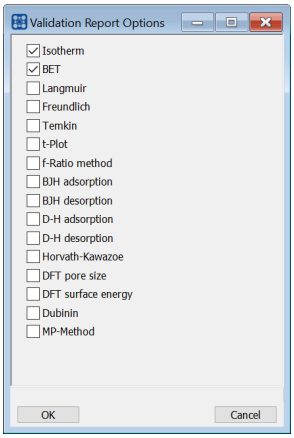
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the quality of the experiment is to repeat the analysis. Toward that end, Micromeritics' analyzers log and report the exact conditions of each analysis.

Analysis data can be evaluated by:

- Viewing the Validation Report
- Inspecting the Isotherm Plot
- Evaluating the Isotherm Tabular Data Set
- Reviewing Reduced Data

VIEW THE VALIDATION REPORT

The *Validation* report shows whether the data collected during an analysis are within typical ranges. Select the types of reports to include by selecting the report in the *Validation Report Options* window.



When a selected report is generated, if errors occur, a message is displayed across the top portion of the report and a unique symbol displays on the graph.

INSPECT THE ISOTHERM PLOT

Evaluation of data should begin with a visual inspection of the isotherm plot. The plot should be composed of data which have not been subjected to mathematical smoothing as far as possible. If the data describe a Type I isotherm, then the plot is best shown on a logarithmic pressure axis so that details of the low pressure region are revealed. Data in this region are important particularly for micropore studies. Examine the plot to determine if any points are outliers or if a region of the isotherm exhibits characteristics (spikes, steps, etc.) which are inconsistent with the physical process being monitored. The philosophical question of whether or not these suspected extraneous data points should be removed from the raw data is not considered here, but it may be appropriate to exclude an outlier from reduced data. Too many outliers can cause the integrity of the total data set to come under suspicion.

Examine specific reported values to confirm that the isotherm data were collected under reasonable conditions and using reasonable parameters. For example, confirm that the free space values reported are typical for the sample holder and bath in use. A problem with either ambient or analysis free space values may indicate a free space measurement error and affect all calculations of quantity adsorbed.

The raw data should be carefully examined before it is reduced. Errors that occur in raw data will only be exacerbated in reduced data.[1)]

Evaluate the Isotherm Tabular Data Set

Another place to look for reasonableness of the data is the adsorptive uptake by the sample in the BET range (P/P_0 = 0.05 to 0.30). Total uptake is the specific quantity adsorbed (cm^3/g STP) times the sample mass (g). As an example, the level of uncertainty in this range typically is less than 0.1 cm^3 STP for a high performance system. Total uptake quantities should be some multiple of this level of uncertainty. Otherwise, an unfavorable signal-to-noise ratio and unreliable data result. The solution is to use a greater quantity of sample to increase adsorptive uptake.

Another valuable bit of information resides in the tabulated saturation pressure. This pressure is expected to change somewhat over the duration of an analysis, but it is not expected to do so with large or abrupt transitions. Unreasonable saturation pressures or unusual changes may indicate that a gas different from the adsorptive was used in determining P_0, that the level of the cryogen fell too far, or that the cryogen is impure or inappropriate.

With experience, obvious signs of problems can be detected by a quick inspection of the tabular and graphical data. If the data appear satisfactory, the next step is to evaluate the reduced data.[2)]

Review Reduced Data

Isotherm data may be analyzed by any one of several reduction methods depending on the analyzer model and pressure range employed. The quality of the results depends on the quality of the isotherm, the congruity of the data reduction parameters with experimental conditions, the agreement of the theoretical model with the physical gas-solid system, and compliance to the pressure range over which the method is valid. Typically, results can be appraised by examining a few salient areas of the report as described in the following topics.[3)]

1) The information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).
2) Most of the information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).
3) Most of the information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).

PHYSICAL PARAMETERS

The value of physical parameters which are used only in data reduction routines should be reviewed to assure that they agree with experimental conditions. These parameters can be changed and the experimental data recalculated if an error is discovered or if exploring an alternate value is desired. Analysis condition values used in the calculation of quantity adsorbed can be changed also. These are typically the manually entered free space(s), nonideality correction factor, and bath temperature.

The area occupied by a single adsorbed molecule is a required parameter in the calculation of surface area by the BET and Langmuir methods. The software provides a default value, but other values are found in the literature. McClellan and Harnsberger[1] provide a comprehensive review of such values.

The volume of pores of a specific size range is calculated from the gas quantity adsorbed in them by converting the quantity to its liquid equivalent volume. This is achieved through use of a density conversion factor calculated from the ratio of molar densities of the condensed adsorbate at bath temperature to the gaseous phase at STP. The necessary information is found in handbooks. The software contains default values for common adsorptives; values for other adsorptives must be calculated.

The terms for liquid surface tension γ, contact angle between solid and liquid phase θ, molar volume of the adsorbate n, gas constant R, and sample temperature T are treated as one constant, the adsorbate property factor A expressed by:

$$A = \frac{2\gamma\vartheta \cos\theta}{RT}$$

using which, the Kelvin equation[2] reduces to

$$\ln \frac{P^*}{Po} = \frac{A}{r_m}$$

Either surface tension, contact angle, or molar volume can be revised individually to give a new value for the factor A, or A can simply be altered arbitrarily for exploratory purposes.

The thermal transpiration correction requires two parameters which may be adjusted from those of the default values. The first is the inside stem (neck) diameter of the sample holder, and the second is the hard-sphere diameter of the adsorptive molecule. The sample holder inside diameter is available from the documentation provided with it or is measurable. Information on hard-sphere diameters of molecules may be obtained from handbooks.

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1) McClellan, A.L., and Harnsberger, H.F., Journal of Colloid and Interface Science, 23, 577 (1967).
2) Thomson, W., Phil. Mag. S., 42, 448 (1871).

For terms such as the interaction parameter found in the Horvath-Kawazoe calculation[1], the Dubinin affinity coefficient, or Astakhov exponent[2], the default values as provided by the software generally are adequate. A search of the technical literature is required if the analysis involves a gas-solid system other than that covered by the default values.

The t-Plot method plots quantity adsorbed against thickness (t) derived from a thickness equation, and the Dubinin transform plots quantity adsorbed against $\log(P/P_0)n$. All of these data reduction methods were first proposed for specific applications. The user must make a judgment as to the applicability of the method to a gas-solid system.

If applied appropriately, all transform plots will exhibit a linear range and the regression analysis must be applied only over the linear range and within the range of application. Fitting a regression line to surface area transformation plots should yield a correlation coefficient of 0.9999 or better and for t-plots and Dubinin plots the correlation coefficient should be 0.99 or better.

If the data reduction model does not apply to the gas-solid system under examination, then it may be that either no linear range exists within the pressure range of validity, or that solutions derived from the regression line of the linear range are intuitively incorrect, that is, they have no relevance to the physical situation, such as a negative C-value from a BET transform.

BET C-VALUE

BET theory assumes uniform surface coverage with no favored adsorption sites and it also assumes that the gas is more strongly attracted to the surface than to other gas molecules. The typical range of BET C-values is from about 5 to well over 100. Values much less than 5 imply that the gas-to-gas affinity is competing with the gas-to-solid affinity which conflicts with the basic assumptions of BET theory. C-values much greater than 100 indicate very strong attraction for the surface or preferential adsorption.

Provided the isotherm was determined with negligible error and the regression line to the BET transformation data was fit properly, then an out-of-range C-value probably indicates that the gas-solid interaction for the particular sample material does not conform to the BET model. An inappropriate adsorption model may be indicated also by the coefficient of correlation of the regression line, 0.999 being about the minimum value expected with five more or less equally spaced points. In the case of indications of poor conformance to the BET model, the Langmuir data reduction method should be examined.

1) Everett, D.H. and Powl, J.C., J. Chem Soc., Faraday Trans. 1, 72, 619 (1976).

2) Dubinin, M. and Radushkevich, L.V., Proc. Acad. Sci. USSR, 55, 331 (1947).

DATA ANALYSES BY THE BJH METHOD

In general, this method visualizes 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 is accounted for totally. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, ultimately it will predict a larger increment of adsorptive for a given pressure increment than is actually observed. The algorithm must stop since a negative pore volume is nonphysical. Accumulated error results in the calculation of a too large volume of (possibly nonexistent) small pores if the thickness curve used underestimates film thinning.

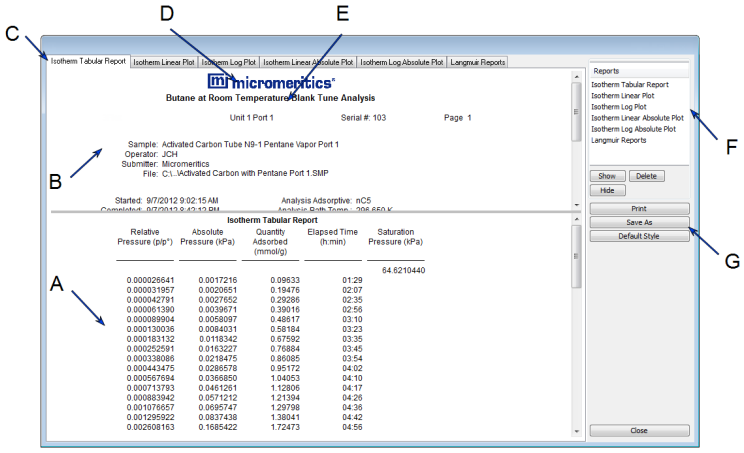
REPORT FEATURES AND SHORTCUTS

CFR Note

In 21CFR11 environments, members of the Analyst group must click **Preview** on the sample file window to access this screen.

Reports can be customized and manipulated using the toolbar, shortcut menus, the zoom feature, or axis cross-hairs.

- After analysis, reports can be viewed, printed, and/or copied and pasted into other documents.
- The report zoom feature provides the viewing of fine graph details and the ability to shift the axes.
- All reports contain a header displaying file statistics.



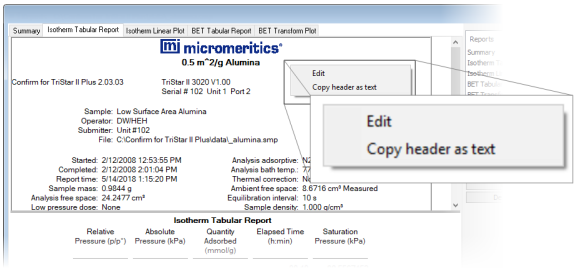
A. Data display (graph or text)
B. Header
C. Generated tabs
D. Graphic
E. Title
F. List box
G. Toolbar

If configured, the report header can also contain a graphic and/or a title.

- Tabular and graphical reports contain sample and analyzer statistics such as analysis date/-time, analysis conditions, etc.
- The headers contain notes of sample file changes occurring after analysis.

REPORT HEADER SHORTCUTS

Right-click in the report header to display header shortcuts.

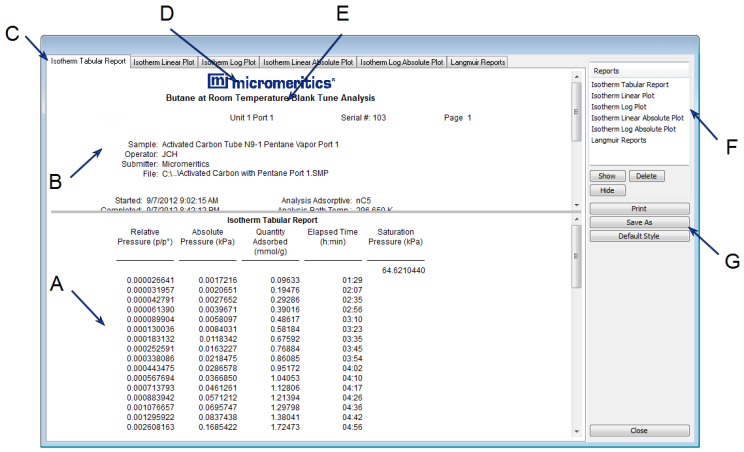


Report Header Shortcuts

Field or Button	Description
Copy header as text	Copies the report header as text. Text is copied to the clipboard and then can be pasted into other documents.
Edit	Opens a dialog box for editing the report title.

REPORT TOOLBAR

The *Report* window has a toolbar on the right portion of the window and selectable tabs at the top of the report header. To view a specific report, either select the tab or the report in the *Reports* list box, then click **Show**.



A. Data display (graph or text)
B. Header
C. Generated tabs
D. Graphic
E. Title
F. List box
G. Toolbar

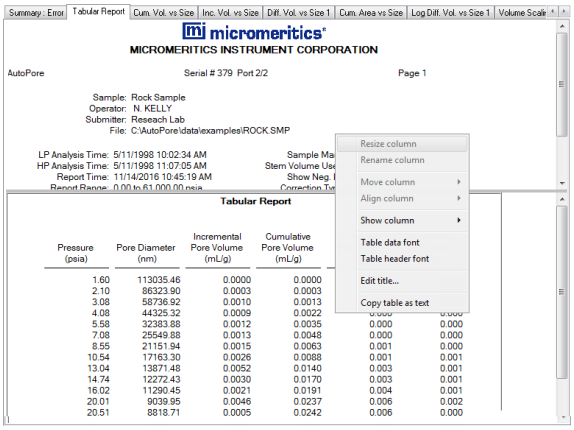
Report Toolbar

Field or Button	Description
Default Style [*button*]	Specifies default report parameters for fonts and curve properties.
Delete [*button*]	Deletes the selected report in the *Reports* list box. Deleted reports will have to be regenerated if deleted in error.
Hide [*button*]	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the *Reports* list box.
Print [*button*]	Displays the *Print* window for report output.
Reports [*group box*]	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the **Hide** button.
Show [*button*]	Displays the selected or hidden report in the *Reports* list box.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

TABULAR REPORT FEATURES AND SHORTCUTS

Display tabular report shortcuts by right-clicking in the body of the tabular report. Column shortcuts require right-clicking on the column to be modified.



Tabular Report Shortcuts

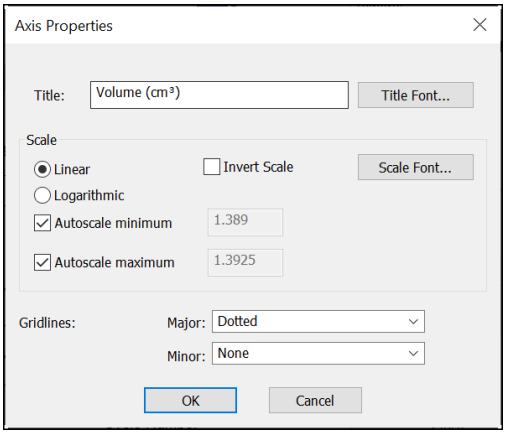
Field or Button	Description
Align column	Changes the column alignment to either left, right, or centered.
Copy table as text	Copies the report contents to the clipboard as tab-delimited text. It can then be pasted into another document.
Edit title	Edits the report title and/or title font attributes. Click **Font** to modify font attributes.
Move column	Right-click the column to be moved. Select *Move column* on the short-cut menu and select *Left* or *Right* for the move.
Rename column	Right-click the column to be renamed. Select *Rename column* on the shortcut menu and enter the new column name.
Resize column	Right-click the column to be resized. Select *Resize column* on the shortcut menu and enter the new column width in inches.
Show column	Displays a list of all columns. Click a column to add a checkmark to show the column or remove the checkmark to hide the column.
Table data font	Right-click in the report data. Select *Table data font* on the shortcut menu.
Table header font	Right-click in the report data. Select *Table header font* on the shortcut menu.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

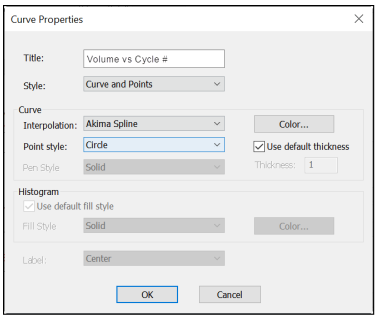
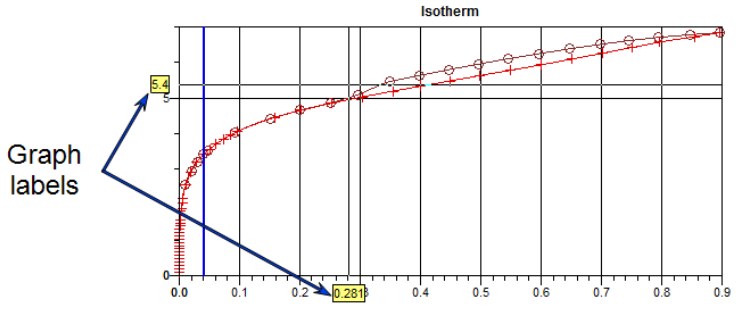
GRAPH FEATURES AND SHORTCUTS

Right-click in the graph area to display graph report shortcuts.

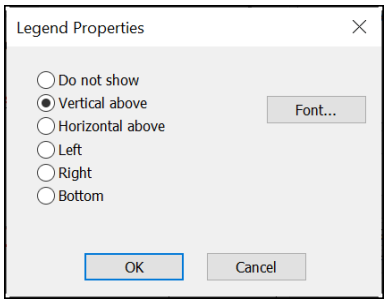
Graph Shortcut Options

Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy data	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns or copied as an overlay onto another graph.
Copy graph	Copies the graph to the clipboard. It can then be pasted into other software programs.
Edit axis	Edits the selected axis properties.  **Gridlines.** Changes how to display major / minor grid lines. **Scale.** ■ **Autoscale minimum/maximum.** To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. ■ **Invert scale.** Inverts the scale. ■ **Linear/Logarithmic.** Scales the graph as linear or logarithmic. ■ **Scale font.** Modifies the font for the scale label. Deselect *Use default font* to enable font options. **Title.** Edits the selected axis label. **Title font.** Modifies the font for the selected axis label. Deselect *Use*

Graph Shortcut Options (continued)

Field or Button	Description
	default font. Select new font attributes for report data. Enable *Use default font* to reset default fonts.
Edit curve	Edits selected curve properties.  **Color.** Changes the curve color. **Curve.** Changes the interpolation, point style, and pen style for the selected curve. These options are disabled if *Use default fill style* is selected in the *Histogram* group box. **Histogram.** Enabled only if *Histogram* is selected in the *Style* drop-down list. Specifies the type of fill, fill color, and label position for the selected curve. **Label.** Designates where the graph point labels will display (left, right, center, etc.) on the SPC report.  **Style.** Selects another style for the collected data curve. **Title.** Changes the title of the selected curve. **Use default thickness.** Uses the default curve thickness. Deselect to enter a new thickness number in the *Thickness* text box.

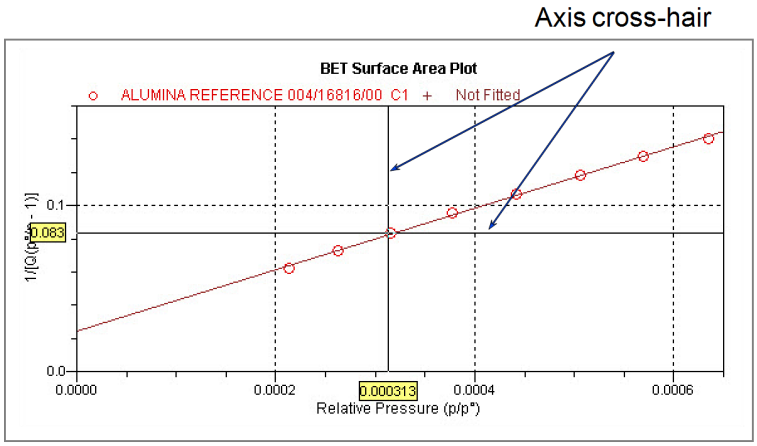
Graph Shortcut Options (continued)

Field or Button	Description
Edit legend	Changes the legend location and font. 
Edit title	Changes the report title.
Enable overlays	If overlays have been selected, this option displays (or hides) the overlays.
Include report	When selected, places a checkmark to the left of the report in the *Select Reports* list box on the *Report Options* tab.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Select overlays...	Displays the option to select files to overlay onto the active graph. To view the overlays, click *Enable Overlays* on the shortcut menu.
Show curve	Displays a list of all curves. Select the curve(s) to display.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

AXIS CROSS-HAIR

Left-click on the graph to view the cross-hair coordinates.



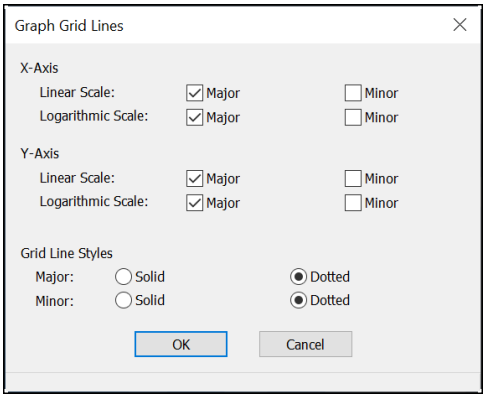
Example of Axis cross-hair feature

ZOOM FEATURE

Use the zoom feature to examine graph details. Click, hold, and drag the left mouse button on the graphical area to be enlarged. A box will display in the area to be enlarged. To return to normal view, right-click in the graph and select *Autoscale all axes*.

GRAPH GRID LINES

Options > Graph Grid Lines



Use to select how grid lines appear on reports. This menu option is not available if using *Restricted* option presentation.

Graph Grid Lines

Field or Button	Description
Grid Line Styles [*selection*]	Select if the major and/or minor grid lines should appear as solid or dotted lines.
X-Axis / Y-Axis [*selection*]	Select major and/or minor lines to display in reports for the log-arithmic and linear scales. Deselect this option to remove the grid lines.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

GRAPH AND SAMPLE OVERLAYS

CFR Note

In 21CFR11 environments, this feature is applicable to members of the Developer group only.

Use the graph overlay functions to compare multiple graph options. Graphical lines are differentiated by the use of varying colored symbols outlined on a legend. Overlays may be generated in two ways:

- **Multiple Graph Overlays.** Overlay two different types of graphs from one sample.
- **Multiple Sample Overlays.** Overlay graphs of the same type with that of the current plot. This type of overlay is available only for:
  - BJH Adsorption / Desorption
  - DFT Pore Size / Surface Energy
  - Dollimore-Heal Adsorption / Desorption
  - Horvath-Kawazoe
  - M-P Method

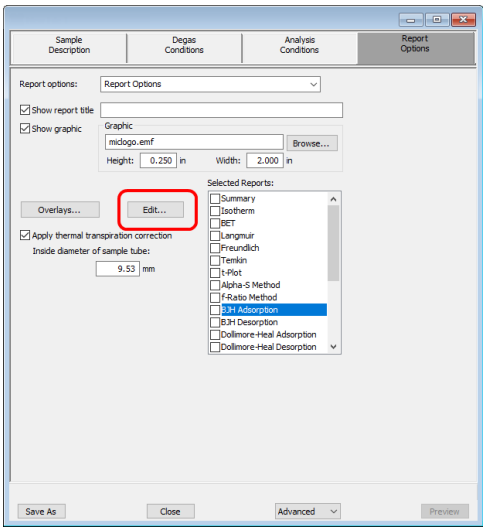
This feature is available only when using *Advanced* option presentation. Go to ***Options > Option Presentation > Advanced***.

Overlay Multiple Sample Files

CFR Note	In 21CFR11 environments, this feature is applicable to members of the Developer group only.

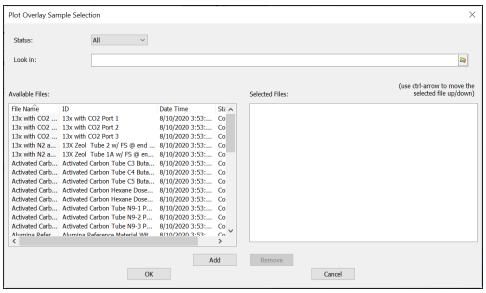
To overlay the same type of graph on multiple samples:

1. Go to ***File > Open***.
2. Select a .SMP file, then click **Open**. If the Isotherm plot displays, select *Advanced* from the view selector drop-down list at the bottom of the window to display the tabbed window view.
3. Click the *Report Options* tab.
4. In the *Selected Reports* list box, highlight a report then click **Edit**. Use the following table to complete the process for the selected report.



If overlaying this type of report...	Then...
■ Isotherm	a. On the *Isotherm Report Options* window, select one or more plots in the *Selected Reports* group box, then click **Options** to the right of the selected plot. b. On the *Plot Options* window, select *Plot curve* and/or *Plot points* if they are to be included in the overlay. If the x- and/or y-axes are to be autoscaled, enable *Autoscale*; otherwise, enter the *From* and *To* points for the axes. Click **OK**. c. On the *Isotherm Report Options* window, in the *Plot Options* group box, select *Plot overlays*. Click **OK**. d. Continue to Step 5.
■ Alpha-S Method ■ BET Surface Area ■ *f*-Ratio Method ■ Freundlich ■ Langmuir Surface Area ■ *t*-plot ■ Temkin	a. On the pop-up window, select *Overlay samples*. Verify other fields. Click **OK**. b. Continue to Step 5.
■ BJH ■ Dollimore-Heal ■ MP-Method	a. Select the report variable from the *Selected Reports* group box, then click **Edit**. b. Click the down arrow on the *Overlay* field, then select the *Samples* option. c. Verify other fields. d. Click **OK**. e. Click **OK** again.

5. On the *Report Options* tab, click **Overlays**.
6. On the *Plot Overlay Sample Selection* window, move up to 25 files from the *Available Files* box to the *Selected Files* box:



7. Click **OK**.
8. To view the report, click **Preview**.

Overlay Sample Selection

Field	Description
Available Files [*selection*]	Lists files that meet the selected criteria. Select the files to be combined, then click **Add**. The selected files are moved to the *Selected Files* list box.
Look in [*button*]	Click the **Browse** icon to change the file folder location.
Selected Files [*selection*]	Lists the files selected to be combined. Click **OK** to combine the files.
Status [*drop-down box*]	Select the status of files to be combined.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

REPORT EXAMPLES

BJH ADSORPTION REPORT

micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

Sample: SiAl Reference Material 004/16821/00 - Full
Operator: SV
Submitter: Micromeritics
File: C:\Gemini VII\data\sialfull.smp

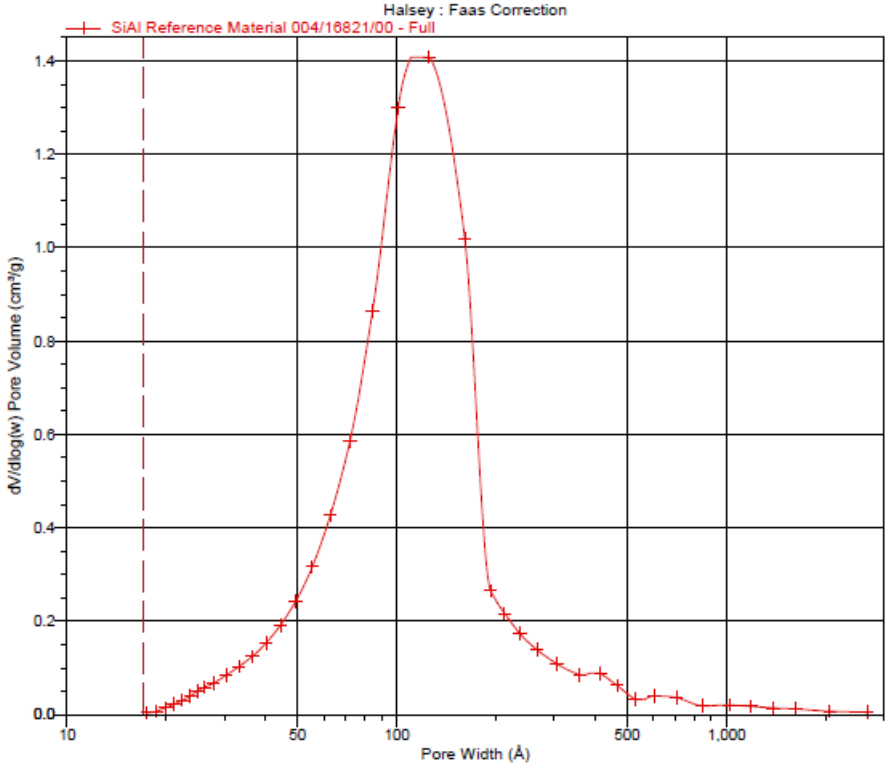
Started: 1/5/2009 9:21:04 AM
Completed: 1/6/2009 6:50:21 AM
Report Time: 3/13/2018 7:04:34 AM
Sample Mass: 0.3054 g
Free Space Type: Measured
Evac. Rate: 133.32 kPa/min

Analysis Adsorptive: N2
Equilibration Time: 10 s
Sat. Pressure: 99.8223 kPa
Free Space Diff.: -0.2625 cm³
Sample Density: 1.000 g/cm³
Gemini Model: Not Gemini

Validation errors exist for this report. Review the validation report for details.

BJH Adsorption dV/dlog(w) Pore Volume

Halsey : Faas Correction



HORVATH-KAWAZOE REPORT

micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

Sample: SiAl Reference Material 004/16821/00 - Full
Operator: SV
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Free Space Type: Measured
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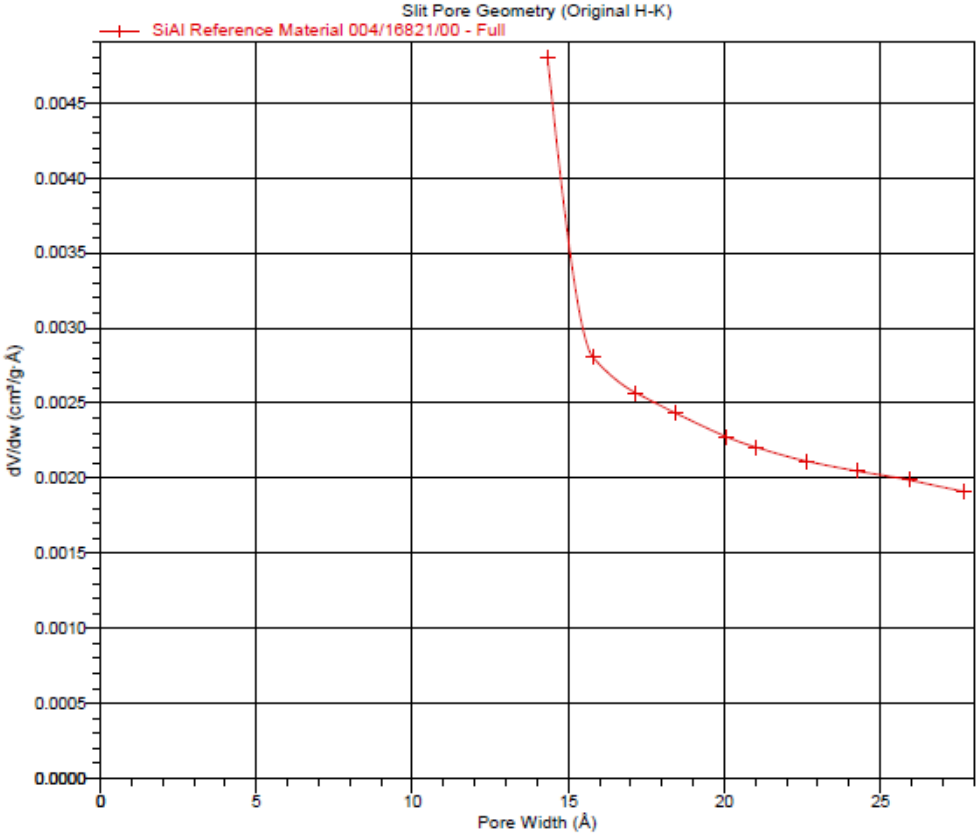
Analysis Adsorptive: N2
Equilibration Time: 10 s
Sat. Pressure: 99.8223 kPa
Free Space Diff.: -0.2625 cm³
Sample Density: 1.000 g/cm³
Gemini Model: Not Gemini

Horvath-Kawazoe Differential Pore Volume Plot

Slit Pore Geometry (Original H-K)



ISOTHERM LINEAR PLOT REPORT

micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

Sample: SiAl Reference Material 004/16821/00 - Full
Operator: SV
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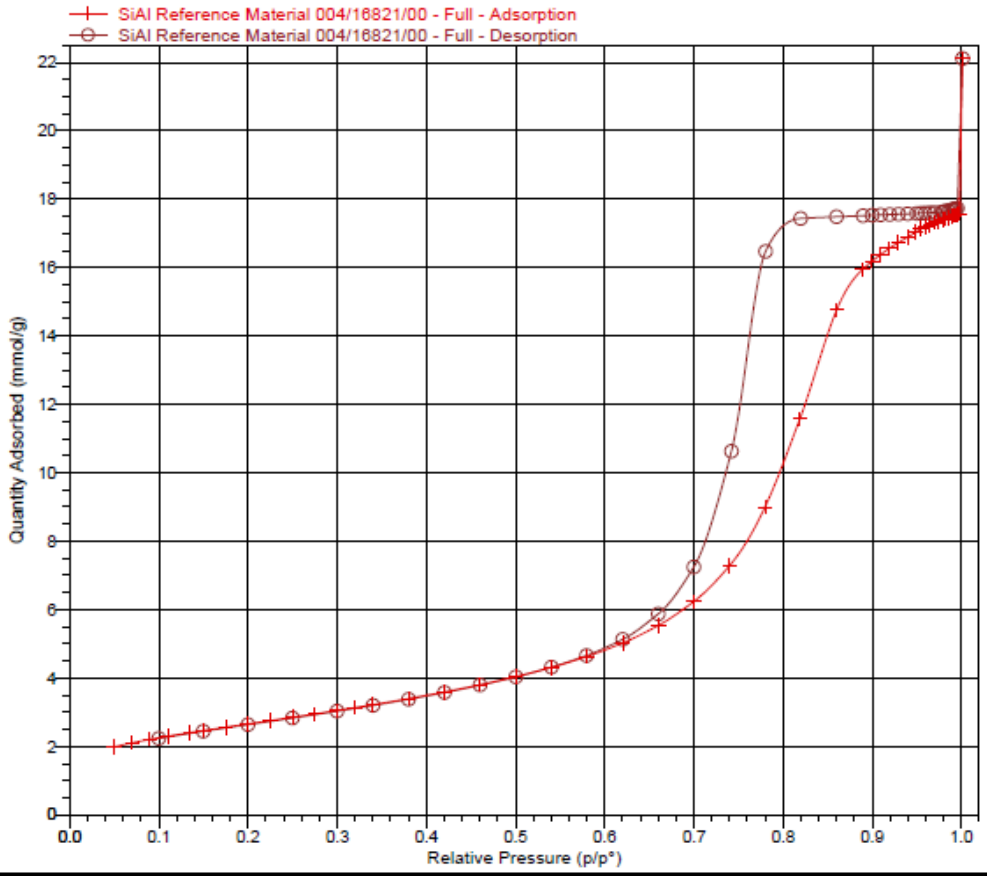
Report Time: 3/13/2018 7:04:34 AM
Sample Mass: 0.3054 g
Free Space Type: Measured
Evac. Rate: 133.32 kPa/min

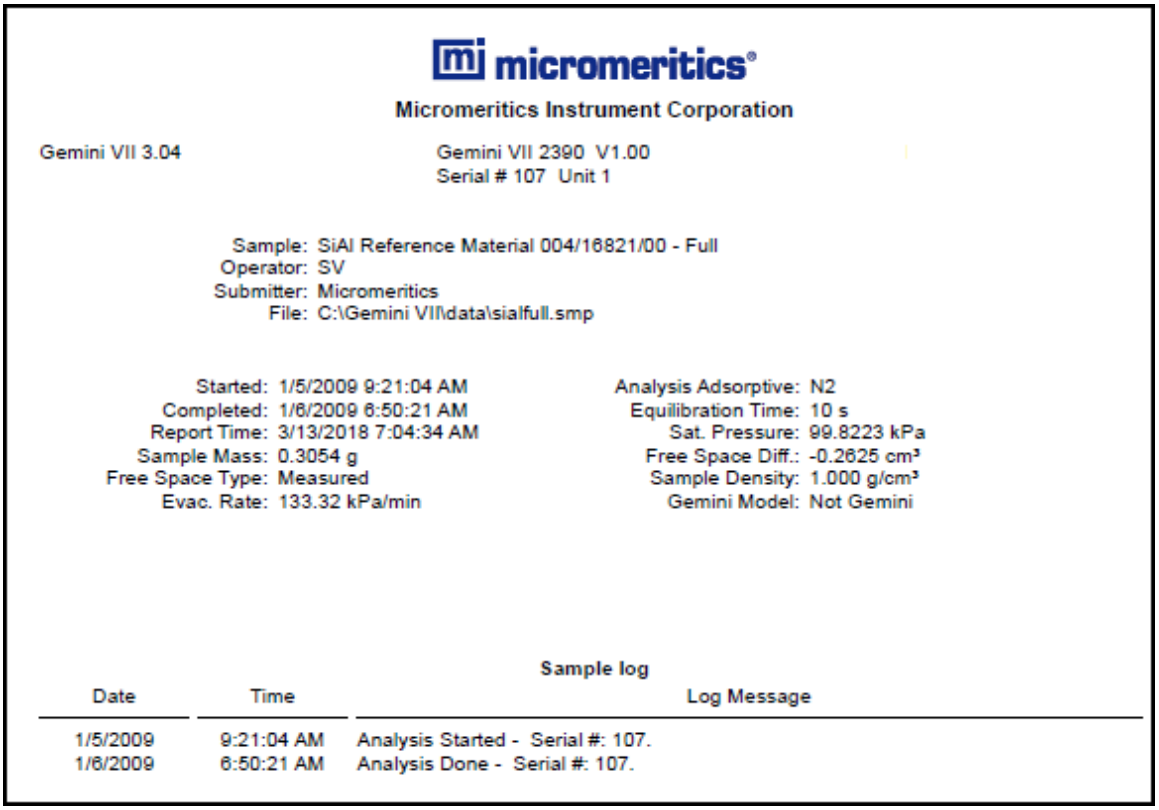
Analysis Adsorptive: N2
Equilibration Time: 10 s
Sat. Pressure: 99.8223 kPa
Free Space Diff.: -0.2625 cm³
Sample Density: 1.000 g/cm³
Gemini Model: Not Gemini

Validation errors exist for this report. Review the validation report for details.

Isotherm Linear Plot



SAMPLE LOG REPORT



micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

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Evac. Rate: 133.32 kPa/min

Analysis Adsorptive: N2
Equilibration Time: 10 s
Sat. Pressure: 99.8223 kPa
Free Space Diff.: -0.2625 cm³
Sample Density: 1.000 g/cm³
Gemini Model: Not Gemini

Sample log

Date	Time	Log Message
1/5/2009	9:21:04 AM	Analysis Started - Serial #: 107.
1/6/2009	6:50:21 AM	Analysis Done - Serial #: 107.

t-Plot Report

micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

Sample: SiAl Reference Material 004/16821/00 - Full
Operator: SV
Submitter: Micromeritics
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Equilibration Time: 10 s
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Free Space Diff.: -0.2625 cm³
Sample Density: 1.000 g/cm³
Gemini Model: Not Gemini

t-Plot Report

Micropore Volume: 0.002373 cm³/g
Micropore Area: 9.0368 m²/g
External Surface Area: 205.5550 m²/g
Slope: 0.592888 ± 0.001717 mmol/g·Å
Y-Intercept: 0.068458 ± 0.007276 mmol/g
Correlation Coefficient: 0.999971
Surface Area Correction Factor: 1.000
Density Conversion Factor: 0.0015468
Total Surface Area (BET): 214.5918 m²/g
Thickness Range: 3.5000 Å to 5.0000 Å
Thickness Equation: Harkins and Jura

Thickness Curve

t = [13.99 / (0.034 - log(p/p°))] ^ 0.5

t-Plot Report - Data

Relative Pressure (p/p°)	Statistical Thickness (Å)	Quantity Adsorbed (mmol/g)	Fitted
0.050127557	3.2385	1.98507	
0.069963864	3.4300	2.10309	
0.090012579	3.5996	2.20441	*
0.109862177	3.7532	2.29511	*
0.134871644	3.9337	2.40065	*
0.149810730	4.0369	2.46094	*
0.174903377	4.2050	2.55921	*
0.199872020	4.3680	2.65619	*
0.224829880	4.5287	2.75168	*
0.249831907	4.6888	2.84903	*
0.275004731	4.8504	2.94747	*
0.300001339	5.0122	3.04804	
0.320086875	5.1439	3.13155	
0.339990182	5.2763	3.21636	
0.380129171	5.5507	3.39563	
0.420146569	5.8371	3.58895	
0.460260414	6.1408	3.80047	
0.500427370	6.4656	4.03753	
0.540155815	6.8121	4.30652	
0.579603932	7.1867	4.62301	
0.620178490	7.6114	5.02313	
0.660380872	8.0815	5.53708	
0.699950841	8.6061	6.23388	
0.740017237	9.2157	7.26041	

VALIDATION REPORT

micromeritics®

Micromeritics Instrument Corporation

Gemini VII 3.04

Gemini VII 2390 V1.00
Serial # 107 Unit 1

Sample: SiAl Reference Material 004/16821/00 - Full
Operator: SV
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Evac. Rate: 133.32 kPa/min

Analysis Adsorptive: N2
Equilibration Time: 10 s
Sat. Pressure: 99.8223 kPa
Free Space Diff.: -0.2625 cm^3
Sample Density: 1.000 g/cm^3
Gemini Model: Not Gemini

Validation Report

Isotherm Reports

Free Space: Low free space values may be observed when using liquid argon or ice baths.

p° Passed

Pressure/Quantity adsorbed: Passed

Desorption Not within limits. Increase the sample mass, increase the equilibration interval, or check the free space.

BJH Adsorption Reports

Pressure range: Passed

Desorption pressures .45 to .40: Phantom 40-angstrom pores may be observed due to the properties of nitrogen at relative pressures 0.4 to 0.45.

Minimum Pore Width: Passed

Thickness curve: Passed

7 SELECTED REPORT OPTIONS

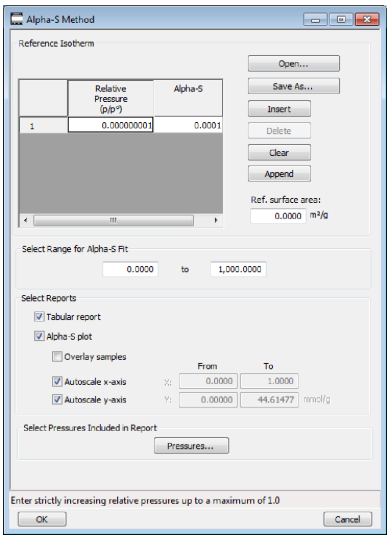
CFR Note

In 21CFR11 environments, this feature is applicable to members of the Developer group only.

To edit reports, open the *Sample* file then select the *Report Options* tab. Highlight the report name in the *Selected Reports* list box and click **Edit**.

ALPHA-S METHOD REPORT

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

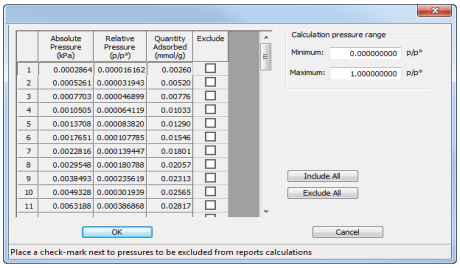


One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the Alpha-S values.

Alpha-S Method Report

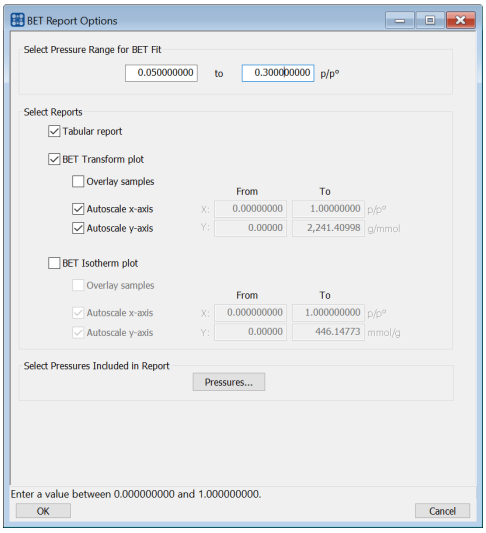
Field or Button	Description
Open [*button*]	Use to import values from an existing thickness curve (.ALS). The table to be imported must be saved as ASCII text with a .ALS file extension. It must have a two-column format with the relative pressures in the first column and the alpha-s values in the second column. Columns must be separated by a space or a tab.

Alpha-S Method Report (continued)

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Ref. surface area [*text box*]	Enter the surface area from the reference curve. This value is used to calculate the sample surface area.
Select Range for Alpha-S Fit [*group box*]	Enter minimum and maximum relative pressures to determine the fit.
Selected Reports [*group box*]	**Alpha-S Plot.** Use to plot data in graph format. ■ **Autoscale x-axis.** The x-axis field shows the relative pressure. ■ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the plot. **Tabular Report.** Use to have a tabular report of data generated.

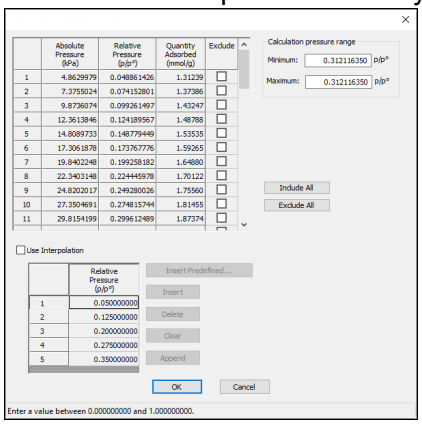
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

BET Surface Area Report



The BET calculation obtains the sample surface area value by determining the monolayer capacity of adsorbed gas from the isotherm data. BET uses a multilayer model.

BET Surface Area Report

Field or Button	Description
Pressures [*button*]	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table if not using the *Use Interpolation* option. **Exclude All.** Select to exclude all pressure points in the table.

BET Surface Area Report (continued)

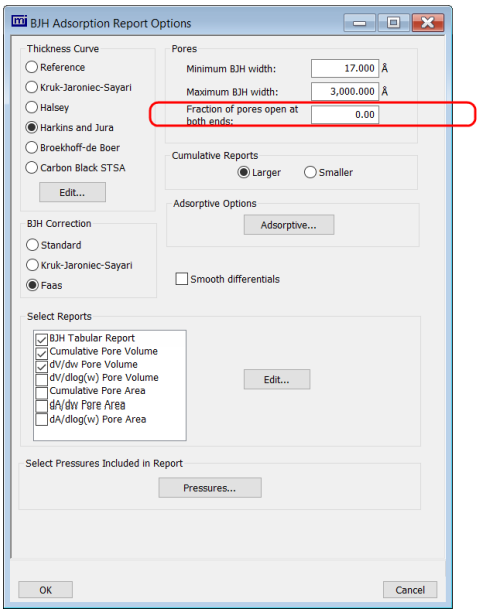
Field or Button	Description
	Include All. Select to include all pressure points in the table. **Insert Predefined.** Click to insert a predefined (default) set of points into the report. *Use Interpolation* must be selected to enable this button. This button displays for BET reports only. **Use Interpolation.** Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.
Select Pressure Range for BET fit [*text box***]**	Enter values to indicate the fitted pressure range.
Selected Reports [*group box***]**	**BET Isotherm plot.** Uses BET monolayer capacity and constant to produce an isotherm. ■ **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET. ■ **Autoscale y-axis**. The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the BET isotherm plot. **BET Transform plot.** Use to generate a traditional BET surface area plot used to determine monolayer volume and BET C constant. ■ **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET. ■ **Autoscale y-axis**. The y-axis field shows BET transformation. ■ **Overlay samples.** Use to overlay sample files on the BET transform plot. **Tabular report.** Use to have a table of measured and calculated values generated.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

BJH Adsorption/Desorption Report

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. Reports can be generated from both adsorption and desorption data. The fields for both *BJH Adsorption Report Options* and *BJH Desorption Report Options* are identical unless otherwise specified.

An incomplete pore distribution may be generated if a thickness curve selection is not a good match for the sample being analyzed.

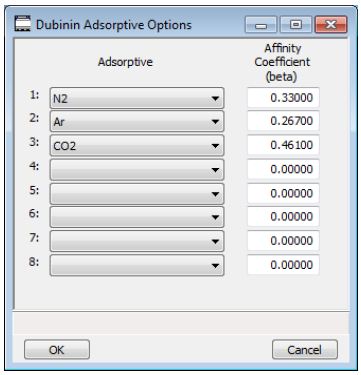


Circled selection is applicable to BJH Adsorption only

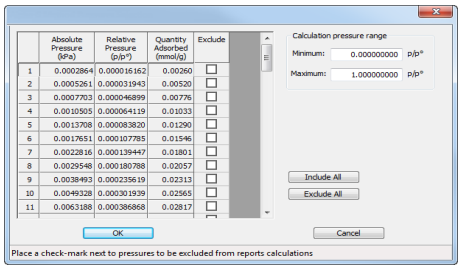
BJH Adsorption/Desorption Report

Field or Button	Description
Adsorptive [*button*]	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.

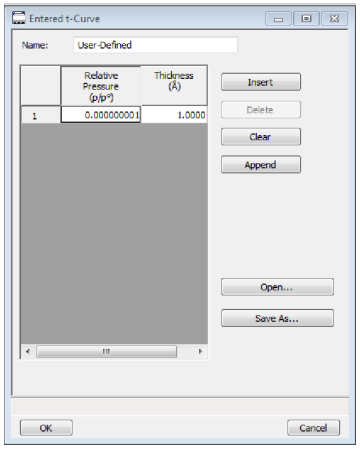
BJH Adsorption/Desorption Report (continued)

Field or Button	Description
	
BJH Correction [*group box*]	Select the type of correction to apply to calculations. The selected type will display in the report header. **Faas.** Good for statistical thickness curves. **Kruk-Jaroniec-Sayari.** Good for reference thickness curves. **Standard.** Uses original BJH models.
Cumulative Reports [*group box*]	**Larger.** Use to report the total volume found in pores larger than the current pore size. **Smaller.** Use to report the total volume found in pores smaller than the current pore size.
Pores [*group box*]	Enter the minimum and maximum diameter (radius or width) of pores to include in the BJH reports. **Fraction of pores open at both ends.** This field is not available for the *BJH Desorption Report Options* window. During adsorption calculations, the analysis program assumes that all pores are closed at one end. Occasionally, 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 the fraction of pores open at both ends to compensate for this error.

BJH Adsorption/Desorption Report (continued)

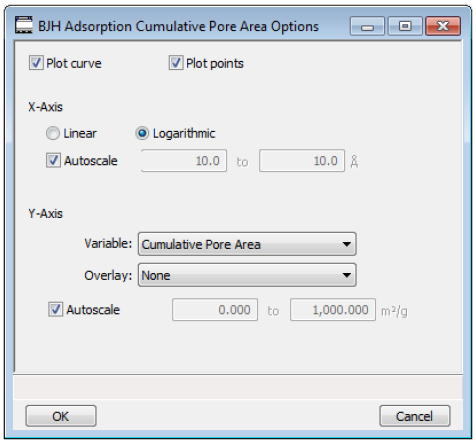
Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Select Reports [*group box*]	Select the report names to include in the report. Highlight the report name, then click **Edit** to modify report parameters.
Smooth differentials [*check box*]	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve [*group box*]	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select **Reference**, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.

BJH Adsorption/Desorption Report (continued)

Field or Button	Description
	 To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have 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.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

BJH PLOT



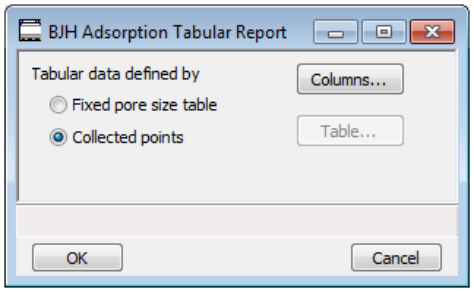
The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click **Edit**.

BJH Plot Report

Field or Button	Description
Autoscale [*check box*]	When enabled on the report parameters windows, allows the x- and y- axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data is shown. If Autoscale is not selected, the entered range is used.
Plot curve / Plot points [*check box*]	Select to plot points on the graph.
X-Axis [*group box*]	Use to have the x-axis on a logarithmic or linear scale.
Y-Axis [*group box*]	**Overlay**. Select an option to overlay onto the current report. **Variable.** Select a variable.

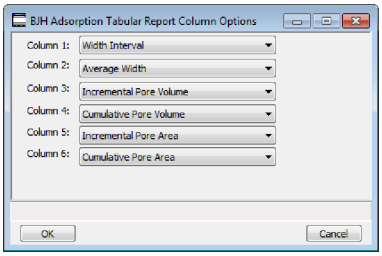
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

BJH TABULAR REPORT



Highlight *BJH Tabular Report* in the *Selected Reports* list box on the *BJH Adsorption Report Options* window, then click **Edit** to specify the method of data reduction.

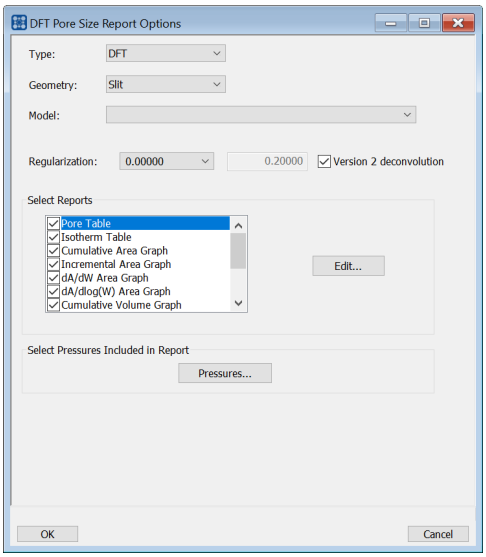
BJH Tabular Report

Field or Button	Description
Collected points [*selection*]	Use to include all relative pressure points collected. Refer to the **Columns** button below.
Columns [*button*]	Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report. 
Fixed pore size table [*selection*]	Use to specify exact pore sizes for volume or area data. Click **Table** to modify the fixed pore size table. Refer to **Table** and **Columns** buttons elsewhere in this table.
Table [*button*]	The fixed pore size table must contain a minimum of two points. The points must be strictly decreasing. Enabled only when *Fixed pore size table* is selected.

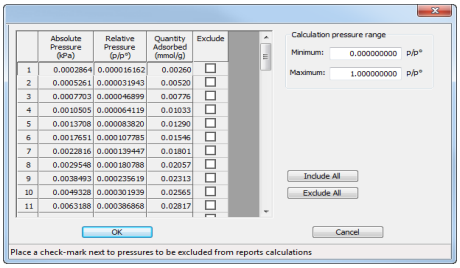
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DFT Pore Size Report

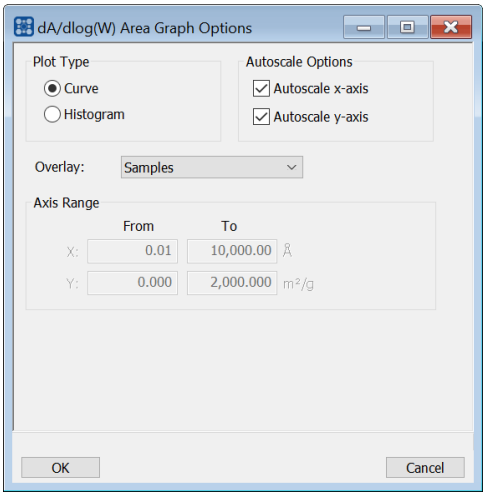
The *DFT Pore Size* report contains the results of pore size distribution analyses using a non-local DFT range of micro and mesopore ranges.



DFT Pore Size Report

Field or Button	Description
Geometry [*drop-down box*]	Select the pore shape.
Model [*drop-down box*]	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*.

DFT Pore Size Report (continued)

Field or Button	Description
	Exclude All. Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Regularization [*drop-down box*]	Select the extent of smoothing to apply to the data. If *0.20000 (user)* is selected, enter a number in the text box giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.
Select Reports [*group box*]	Select the reports to generate. To edit graph details, highlight the graph option and click **Edit**. The *Log Goodness of Fit* and *Goodness of Fit* graphs cannot be edited.  **Autoscale Options.** Use to autoscale the x-axis and/or y-axes. **Axis Range.** *From*/*To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes. ■ **x-axis.** Shows the pore size. ■ **y-axis.** Shows the area. **Overlay.** Select an overlay for the report. **Plot Type.** Select the method for data display.

DFT Pore Size Report (continued)

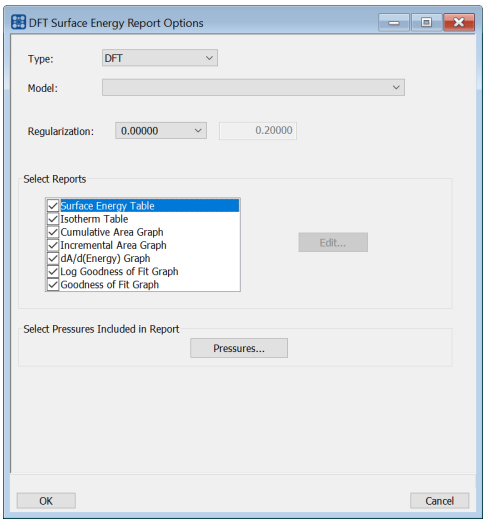
Field or Button	Description
Type [*drop-down box*]	**Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See *DFT Models on page B - 1*. **DFT.** Model based on the density functional theory.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DFT Surface Energy Report

DFT Pore Size Report on page 7 - 12

The *DFT Surface Energy* report contains the results of surface energy distribution analyses.

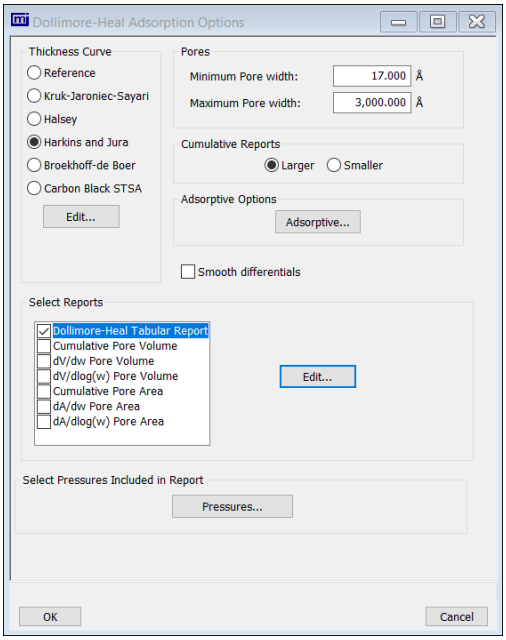


DFT Surface Energy Report Options fields and buttons are identical to the *DFT Pore Size Report Options*.

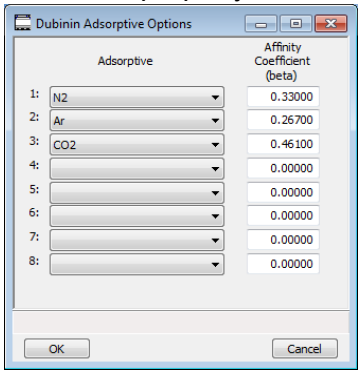
DOLLIMORE-HEAL ADSORPTION/DESORPTION REPORT

BJH Adsorption/Desorption Report on page 7 - 6 for additional information on field and buttons for this report.

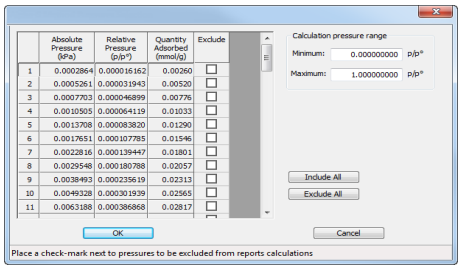
The *Dollimore-Heal Adsorption Report Option* and the *Dollimore-Heal Desorption Report Option* generate reports from both adsorption and desorption data.



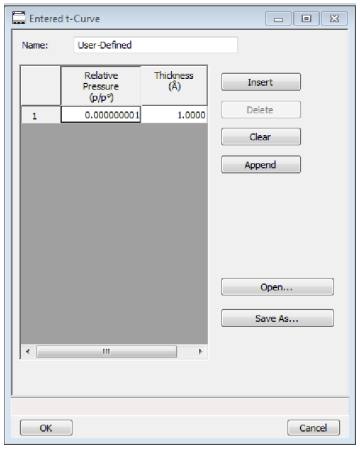
Dollimore-Heal Adsorption/Desorption Report

Field or Button	Description
Adsorptive [*button*]	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified. 

Dollimore-Heal Adsorption/Desorption Report (continued)

Field or Button	Description
Cumulative Reports [*group box*]	**Larger.** Use to report the total volume found in pores larger than the current pore size. **Smaller.** Use to report the total volume found in pores smaller than the current pore size.
Pores [*group box*]	Enter the minimum and maximum diameter (radius or width) of pores to include in the BJH reports.
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Select Reports [*group box*]	Select the report names to include in the report. Highlight the report name, then click **Edit** to modify report parameters.
Smooth differentials [*check box*]	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve [*group box*]	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select **Reference**, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.

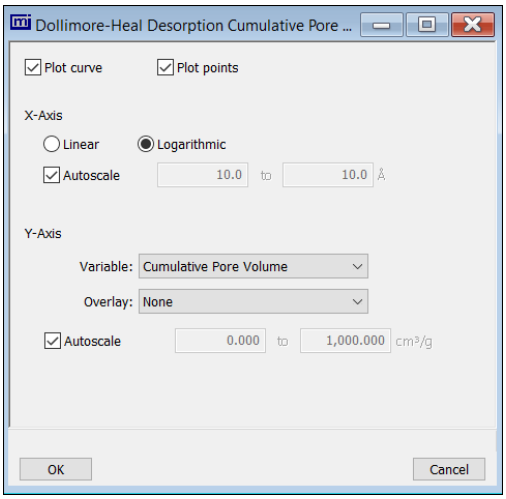
Dollimore-Heal Adsorption/Desorption Report (continued)

Field or Button	Description
	 To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have 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.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DOLLIMORE-HEAL DESORPTION PLOT OPTIONS

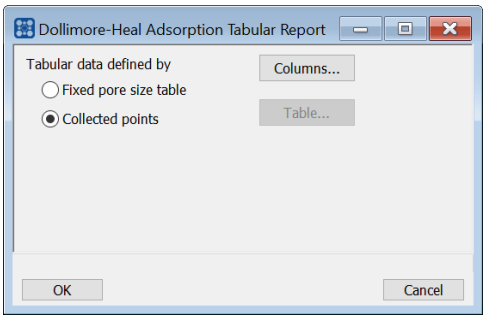
BJH Plot on page 7 - 10



The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *Dollimore-Heal Report Options* window, then click **Edit**. The fields and buttons for these reports are identical to the *BJH Plot Report Options*.

DOLLIMORE-HEAL TABULAR REPORT OPTIONS

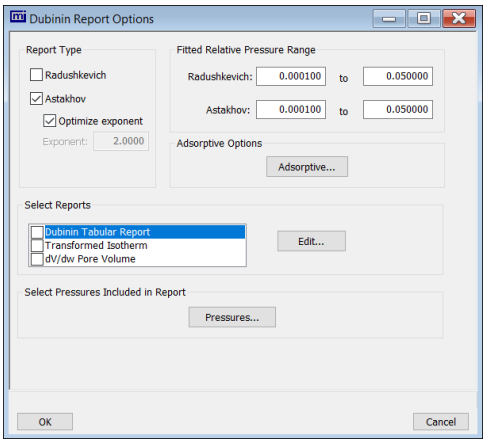
BJH Tabular Report on page 7 - 11



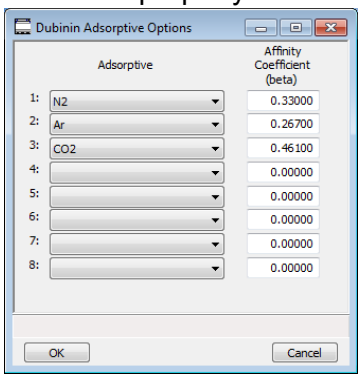
Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*.

DUBININ REPORT

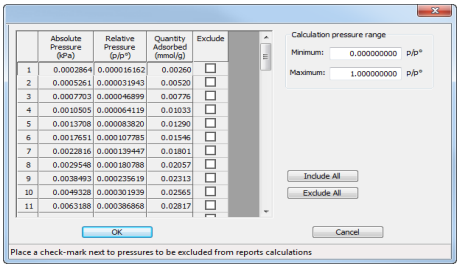
The *Dubinin* method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential.



Dubinin Report

Field or Button	Description
Adsorptive [*button*]	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified. 
Fitted Relative Pressure Range [*group box*]	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.

Dubinin Report (continued)

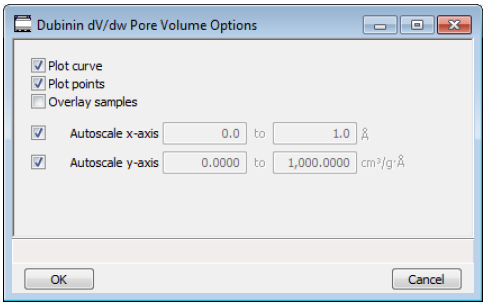
Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Report Type [*group box*]	Select report types. If *Astakhov* is selected, either select *Optimize exponent* or enter an appropriate exponent value in the text box.
Select Reports [*group box*]	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DUBININ PORE VOLUME REPORT OPTIONS

In the *Dubinin Report Options* window, highlight *dV/dw Pore Volume* in the *Selected Reports* list box, then click **Edit**.

This option plots differential pore volume as a function of pore width.



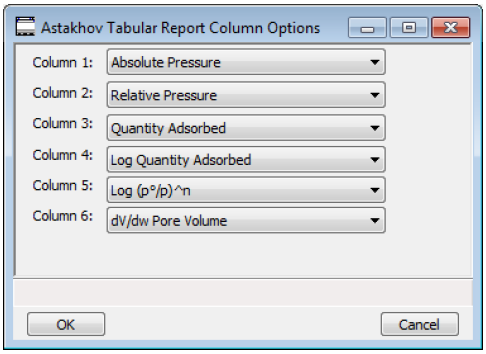
Dubinin Pore Volume Report

Field or Button	Description
Autoscale x-axis / Autoscale y-axis [*check box*]	Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Enable to enter beginning and ending values manually.
Overlay samples [*selection*]	Use to overlay sample files on the plot.
Plot curve / Plot points [*selection*]	Select to plot points on the graph.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

DUBININ TABULAR REPORT OPTIONS

In the *Dubinin Report Options* window, highlight *Dubinin Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.

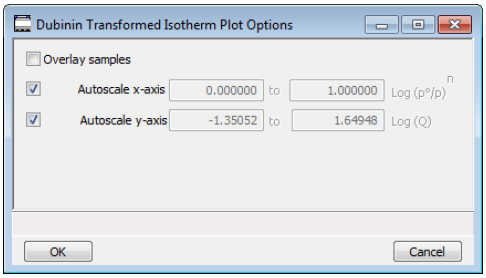


Log (p^o/p)^n. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

DUBININ TRANSFORMED ISOTHERM PLOT OPTIONS

Highlight *Transformed Isotherm* in the *Selected Reports* list box in the *Dubinin Report Options* window, then click **Edit**.

The transformed Dubinin isotherm is the logarithm of quantity adsorbed as a function of the log of relative pressure raised to a power. Isotherms for which the Dubinin method is applicable produce straight lines when transformed in this way.



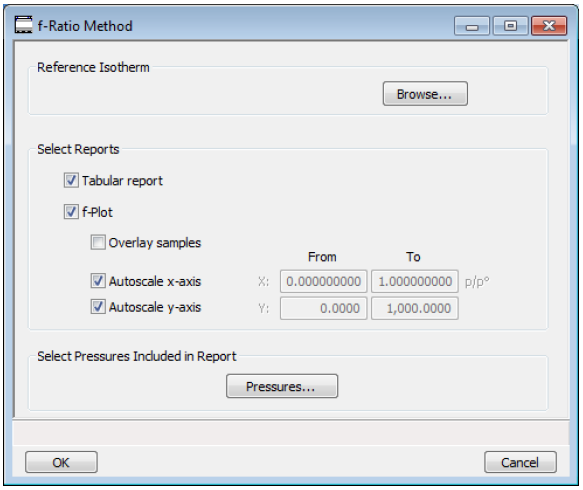
Dubinin Transformed Isotherm Plot Report

Field or Button	Description
Autoscale x-axis / Autoscale y-axis [*check box*]	Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Deselect to enter beginning and ending values manually. **Autoscale x-axis.** Shows the quantity of gas adsorbed at standard temperature and pressure. **Autoscale y-axis.** Shows the log of relative pressure.
Overlay samples [*check box*]	Use to overlay sample files on the plot.

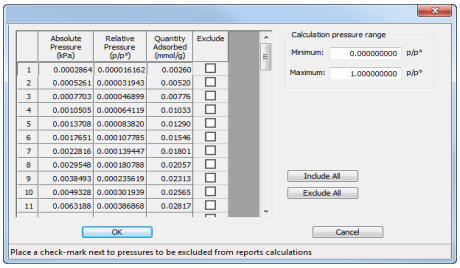
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

f-Ratio Method Report

The *f*-Ratio report uses the measured isotherm and normalizes it using a reference isotherm.



f-Ratio Report

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

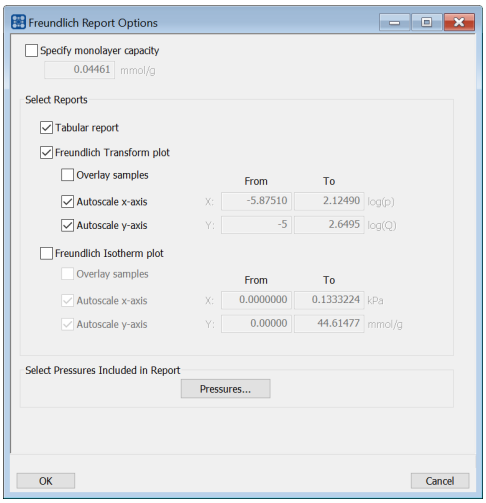
f-Ratio Report (continued)

Field or Button	Description
Reference isotherm [*group box*]	Browse to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample.
Selected Reports [*group box*]	**Tabular Report.** Use to have a tabular report of data generated. **f-Plot.** Use to generate a normalized isotherm. ■ **Autoscale x-axis.** The x-axis field is dimensionless in units of f-ratio. ■ **Autoscale y-axis**. The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the f-plot.

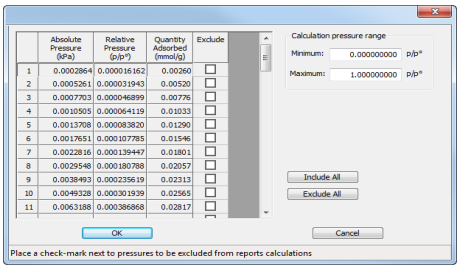
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

FREUNDLICH REPORT

The *Freundlich Isotherm* is an empirical isotherm used to model low pressure adsorption data. It can also be applied to model some micropore isotherms. In the *Selected Reports* list box, highlight *Freundlich*, then click **Edit**.



Freundlich Report

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

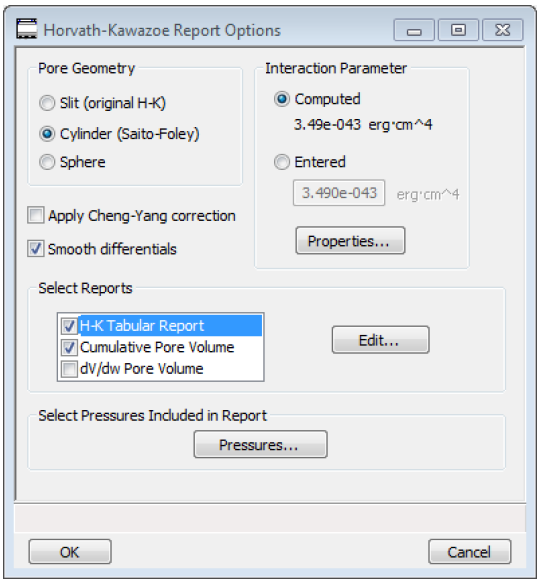
Freundlich Report (continued)

Field or Button	Description
Select Reports [*group box*]	**Freundlich Isotherm plot.** Plots the absolute pressure vs quantity adsorbed. Shows best fit line. ■ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure. ■ **Autoscale y-axis.** y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the Freundlich isotherm plot. **Freundlich Transform plot.** Plots the log(P) vs log(Q) and the best fit. ■ **Autoscale x-axis.** The x-axis field shows the absolute pressure. ■ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the Freundlich transform plot. **Tabular report.** Select to include pressure points included in the report.
Specify monolayer capacity [*selection*]	Select and enter the monolayer capacity of the sample.
Tabular report [*selection*]	Use to have a report of the pressure points generated.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

HORVATH-KAWAZOE REPORT

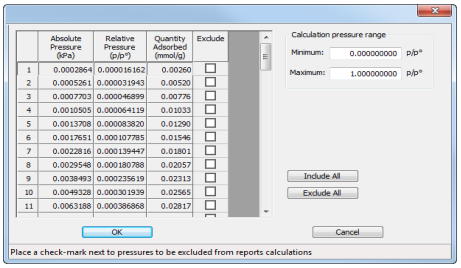
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.



Horvath-Kawazoe Report

Field or Button	Description
Apply Cheng-Yang correction [*selection***]**	Use 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.
Interaction Parameter [*group box***]**	Use to determine which interaction parameter will be used in the report. These options are disabled if *Sphere* is selected in the *Pore Geometry* group box. **Computed.** Use to calculate using the parameters on the *Horvath-Kawazoe Physical Properties* window (click **Properties** to display the *Physical Properties* window). The interaction parameter is recalculated each time a parameter in the *Physical Properties* window is edited. **Entered.** Calculates using the value entered in the text box.
Pore Geometry [*group box***]**	Select the option that best represents the physical geometry of the micropores in the sample material. When *Sphere* is selected, options in the *Interaction Parameter* group box are disabled.

Horvath-Kawazoe Report (continued)

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Properties [*button*]	Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive. **Adsorbent**. Contains the parameters for the sample. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. ■ **Density.** Enter the density per unit area of the sample. * ■ **Description.** Select the name of the sample used in the analysis. ■ **Diameter.** Enter the diameter of the sample atom. ■ **Diameter at zero energy.** Enter the diameter of an atom at zero interaction energy: $(2/5)^{1/6}$ × diameter. ■ **Magnetic susceptibility.** Enter the magnetic susceptibility of the sample. * ■ **Polarizability.** Enter the polarizability of the sample. * **Adsorptive.** Contains the parameters for the adsorptives. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. ■ **Density.** Enter the density per unit area of the adsorptive. * ■ **Diameter.** Enter the diameter of the gas phase atom.

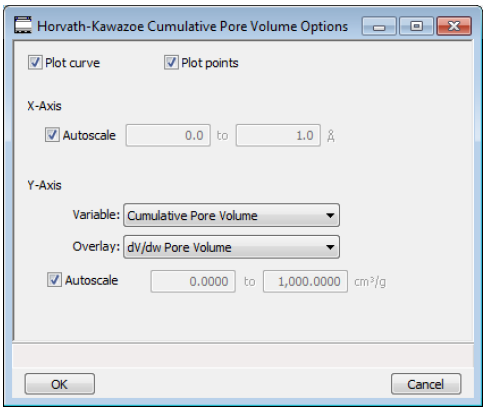
Horvath-Kawazoe Report (continued)

Field or Button	Description
	■ **Diameter at zero energy.** Enter the diameter of an atom at zero interaction energy: $(2/5)^{1/6}$ × diameter. ■ **Magnetic susceptibility.** Enter the magnetic susceptibility of the adsorptive. * ■ **Mnemonic.** Select the mnemonic of the adsorptive gas in use. ■ **Polarizability.** Enter the polarizability of the adsorptive. * * Option is disabled if *Entered* is selected in the *Interactions Parameter* group box.
Select Reports [*group box*]	Select the types of reports to generate. Highlight the report, then click **Edit** to modify report parameters.
Smooth differentials [*selection*]	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

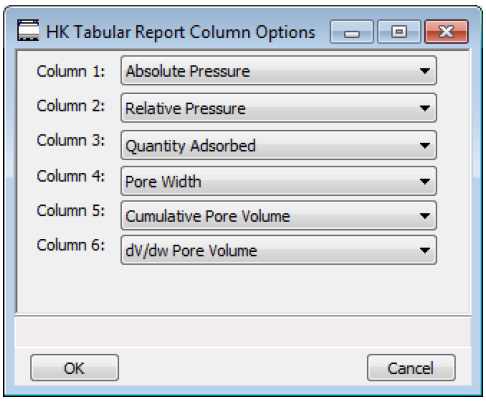
HORVATH-KAWAZOE PLOT

See *BJH Plot on page 7 - 10* for additional information on fields and buttons for this report.



Highlight a plot option in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit** to customize the plotting method.

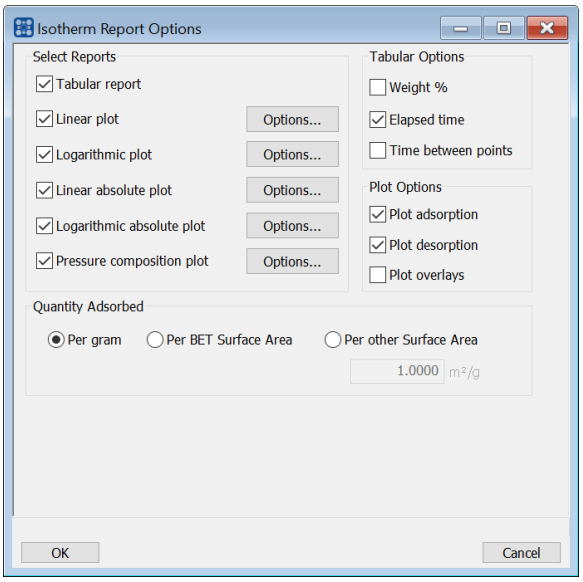
HORVATH-KAWAZOE TABULAR REPORT



Highlight *H-K Tabular Report* in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit**. Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.

ISOTHERM REPORT

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.



Isotherm Report

Field or Button	Description
Options [*button*]	Click to display related linear plot options. All plot windows contain identical fields. **Autoscale x-axis.** Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. **Plot curve / Plot points.** Select to plot points on the graph.
Plot Options [*group box*]	Select the types of isotherm to plot.
Quantity Adsorbed [*group box*]	Select how to report the quantity adsorbed. ■ per gram (cm^3/g) STP ■ per BET Surface Area (cm^3/m^2) STP or mmol/g ■ per other Surface Area (cm^3/m^2) STP or $mmol/m^2$

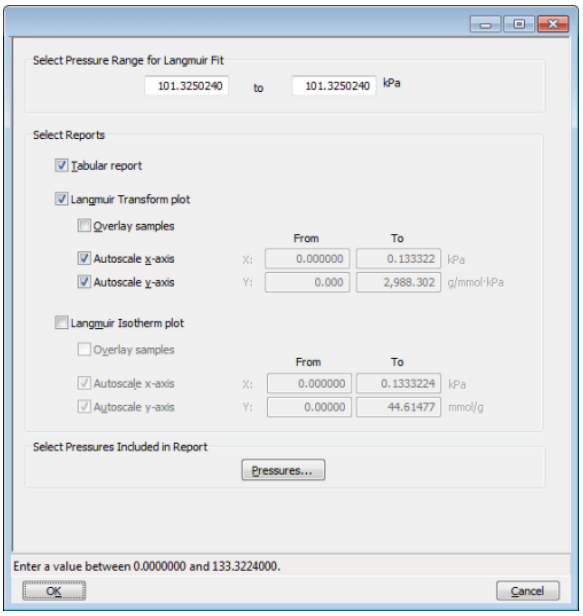
Isotherm Report (continued)

Field or Button	Description
Selected Reports [*group box*]	Select each option to include on the final report. Click the Options button of a selected item to include plot curve, plot points, and to autoscale x- and y-axes.
Tabular Options [*group box*]	Select the options to include on the report. **Elapsed time.** Time elapsed during the analysis. **Time between points.** Time elapsed between points during the analysis. **Weight %.** Enter the mass percentage when plotting pressure composition.

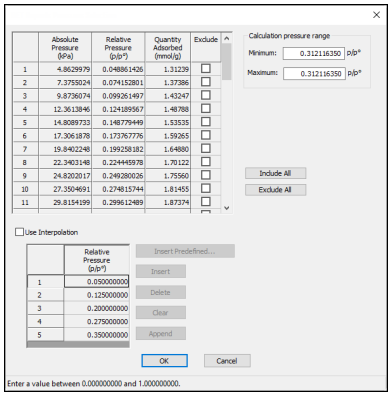
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

LANGMUIR REPORT

The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer. Langmuir uses a single layer model.



Langmuir Report

Field or Button	Description
Pressures [*button*]	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*.

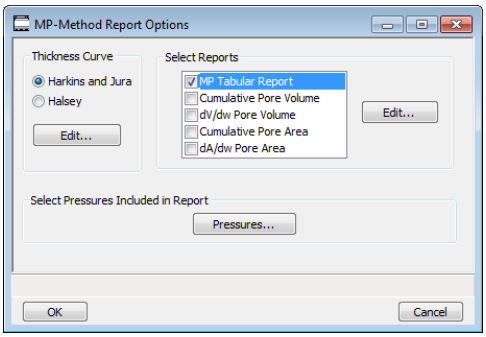
Langmuir Report (continued)

Field or Button	Description
	Exclude All. Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Use Interpolation.** Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.
Select Pressure Range for Langmuir fit [*group box*]	Enter values to indicate the fitted pressure range.
Select Reports [*group box*]	**Langmuir Isotherm Plot.** Uses the Langmuir monolayer volume and constant to produce an isotherm. ■ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ■ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the Langmuir isotherm plot. **Langmuir Transform Plot.** Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant. ■ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ■ **Autoscale y-axis.** The y-axis field shows Langmuir transformation. ■ **Overlay samples.** Use to overlay sample files on the Langmuir transform plot.

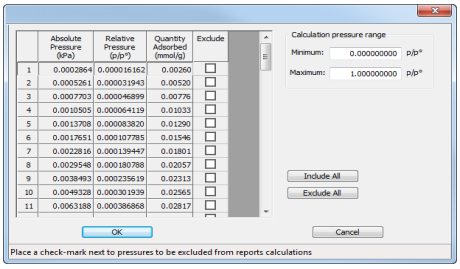
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

MP-METHOD REPORT

The *MP-Method Report* provides pore volume distributions for microporous materials by correlating quantity adsorbed with the thickness of the adsorbed layer as determined from a user-selected thickness curve. Pore size can be expressed in angstroms or nanometers. Go to ***Options > Units*** to specify the unit.



MP-Method Report

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

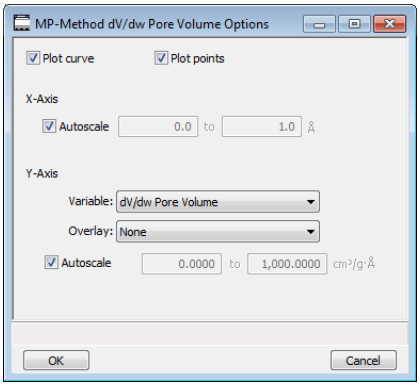
MP-Method Report (continued)

Field or Button	Description
Select Reports [*group box*]	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.
Thickness Curve [*group box*]	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

MP-METHOD PLOT REPORT

In the *MP-Method Report Options* window, highlight a plot option in the *Selected Reports* list box, then click **Edit** to customize the plotting method.



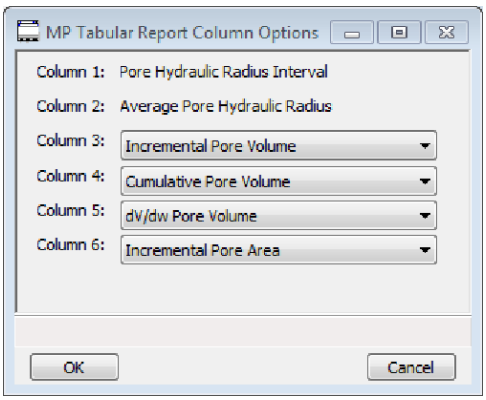
MP Method Plot Report

Field or Button	Description
Overlay [*drop-down box*]	Select an option to overlay on the current report.
Plot curve / Plot points [*selection*]	Select to plot points on the graph.
Thickness Curve [*group box*]	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve.
X-Axis [*check box*]	Use to have the x-axis autoscaled or enter beginning and ending values.
Y-Axis [*group box*]	**Autoscale.** Use to have the y-axis autoscaled or enter beginning and ending values. **Overlay.** Select an option to overlay on the current report. **Variable.** Select a variable.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

MP-METHOD TABULAR REPORT

In the *MP-Method Report Options* window, highlight *MP Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.



The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reported.

OPTIONS REPORT

Lists the conditions used to perform the analysis such as:

- Adsorptive properties
- Analysis conditions
- Analysis method
- Degas conditions
- Free space
- Saturation pressure (P_0) and temperature

Options reports cannot be edited.

SAMPLE AUDIT TRAIL REPORT

CFR Note

For 21CFR11 environments only.

This report lists all changes and comments that have been applied to sample files with a *Complete* status.

SAMPLE LOG REPORT

Sample Log reports cannot be edited.

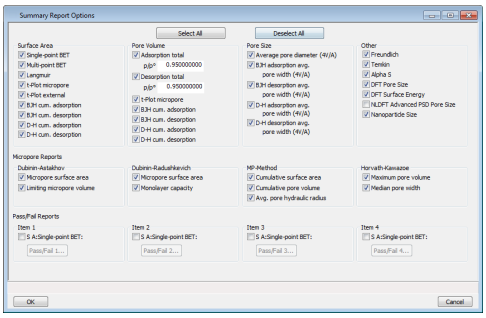
Inserts a log of sample operations in the reports.

This report provides information on:

- 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.

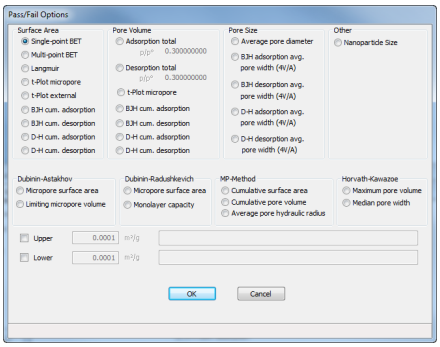
SUMMARY REPORT

The *Summary Report* for physisorption analyses provides a condensed summary of selected data results.



In the *Pore Volume* group box, if *Adsorption total* or *Desorption total* is selected, the *p/po* field is enabled. Enter the relative pressure used to calculate the total pore volume.

Summary Report

Field or Button	Description
Item [n] [*selection*]	Use to enable the first *Pass/Fail* item. Until the *Summary Report* is selected, *S A Single-point BET* will be displayed by default. When selected, click **Pass/Fail**, then select pass/fail criteria options. **Pass/Fail [n].** Click to display the *Pass/Fail Options* window for selection of pass/fail criteria.  **S A: Single-point BET.** Use to enable **Pass/Fail [*n*]** in the *Item [n]* group box. **Upper/Lower.** Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of *Upper / Lower*, enter operator instructions to be displayed if a failure is encountered.

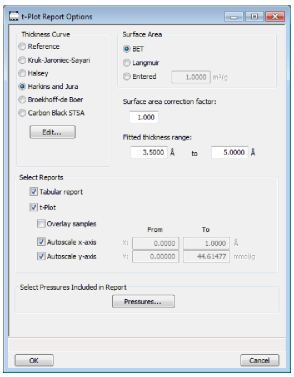
Summary Report (continued)

Field or Button	Description
Select All / Deselect All [*button*]	Selects (or deselects) all options.

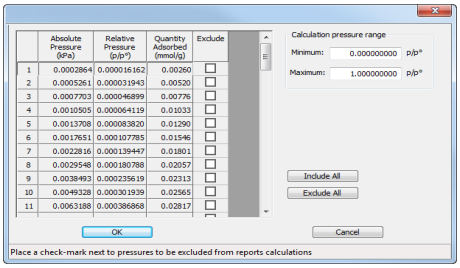
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

t-Plot Report

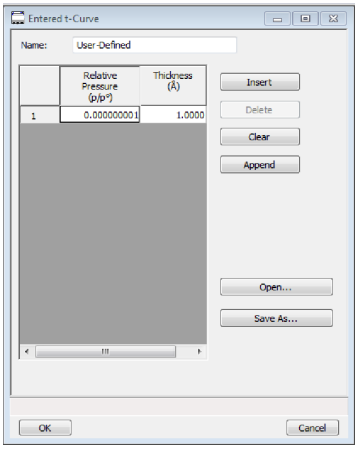
The *t*-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials.



t-Plot Report

Field or Button	Description
Fitted thickness range [*text box*]	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to ***Options > Units*** to specify default units.
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

t-Plot Report (continued)

Field or Button	Description
Selected Reports [*group box*]	**Tabular Report.** Use to have a tabular report of data generated. ***t*-Plot.** Use to have a graphical representation of data generated. ■ **Autoscale x-axis.** The x-axis field shows the statistical thickness of the adsorbed film. ■ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the *t*-plot.
Surface area correction factor [*text box*]	Enter the value to correct for surface areas that are not smooth. This brings the values for BET surface area and micropore surface area into accordance. For most samples, the default value of 1.000 is adequate.
Surface Area [*group box*]	Select the surface area value used for thickness calculations. BET is the most commonly used option.
Thickness Curve [*group box*]	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select **Reference**, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory. 

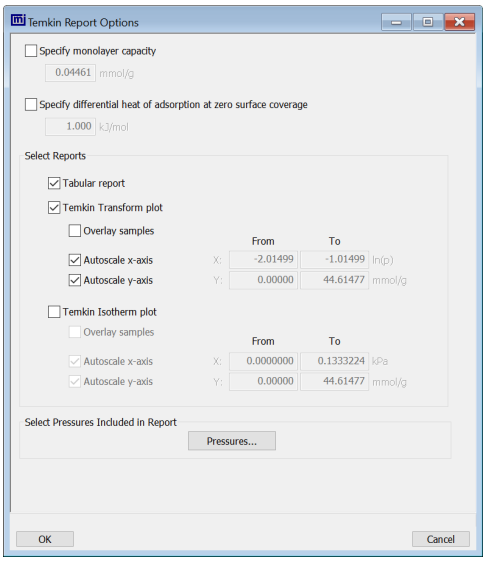
t-Plot Report (continued)

Field or Button	Description
	To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have 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.
t-Plot [*check box***]**	Use to have a graphical representation of data generated. **Autoscale x-axis.** The x-axis field shows the statistical thickness of the adsorbed film. **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. **Overlay samples.** Use to overlay sample files on the *t*-plot.

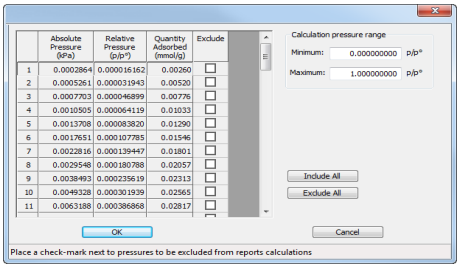
For fields and buttons not listed in this table, see ***Common Fields and Buttons on page 2 - 2*****.**

TEMKIN REPORT

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



Temkin Report

Field or Button	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

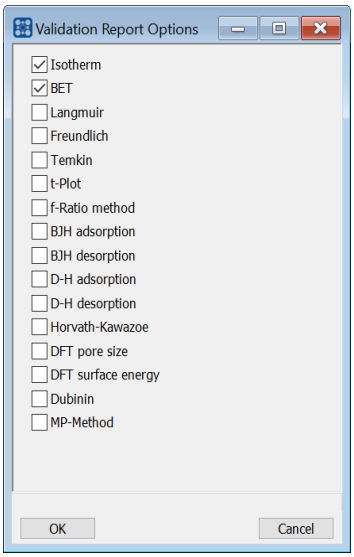
Temkin Report (continued)

Field or Button	Description
Select Reports [*group box*]	**Tabular Report**. Generates a tabular report of the included samples that contains the numeric values contributed by each sample. **Temkin Isotherm plot.** Overlays the Temkin isotherm with the analysis data. ■ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure. ■ **Autoscale y-axis.** y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the isotherm plot. **Temkin Transform plot.** Plots a linear form of the Temkin transform plot. ■ **Autoscale x-axis**. The x-axis field shows the logarithm of pressure (ln). ■ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. ■ **Overlay samples.** Use to overlay sample files on the transform plot.
Specify differential heat of adsorption [*check box*]	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
Specify monolayer capacity [*check box*]	Select and enter the monolayer capacity of the sample.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2*.

VALIDATION REPORT

This report allows data to be examined by the analysis program to determine if the results are within typical ranges. If the data for any reports selected for validation are determined to be out of range, a warning will display and suggestions are given for corrective action.



8 DIAGNOSTICS

Unit [n] > Diagnostics

Use to display diagnostic readings, start diagnostic tests, and open saved diagnostic reports. Each test generates a file to the default directory name and path of *...\...\Service\userdiag* unless another directory name was specified. These reports can be sent to a Micromeritics Service Representative for examination.

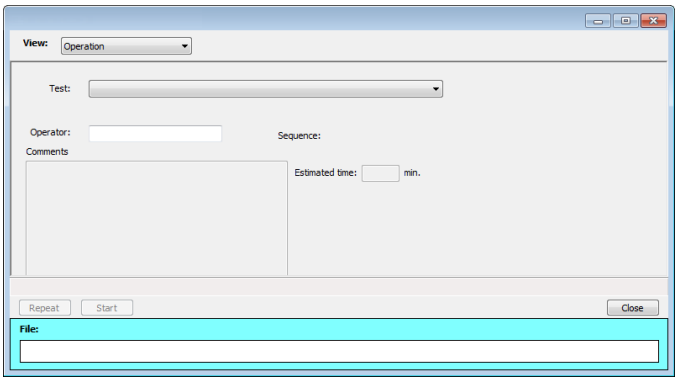
START DIAGNOSTIC TEST

Unit [n] > Diagnostics

Provides a method to start a diagnostic test immediately. Upon completion of the diagnostic test, the file is saved as a .REP file which can be retrieved by going to ***Reports > Open Report*** and selecting the report file.

Use to clean and verify gas lines when connecting or changing a gas. See ***Clean and Verify the Gas Line on page 10 - 8*** for detailed instructions. Also used to perform diagnostic tests requested by a Micromeritics Service Representative.

This option is typically used and /or requested by a Service Representative. These tests create and save files to the *...\...\Service\userdiag* directory. The Micromeritics Service Representative may request an emailed or faxed copy of the file for diagnostic purposes.



Start Diagnostic Test

Field or Button	Description
Comments [*text box*]	Displays comments from the selected diagnostic test.
Estimated time (min.)	Approximate time for test completion.
File [*group box*]	Shows a status bar of steps complete once the test begins.
Next [*button*]	Starts the next test.
Operator [*text box*]	Enter information to identify the person running the service test.
Repeat [*button*]	Repeats the selected diagnostic test.
Report after test [*check box*]	Automatically generates reports to the selected destination when the test is complete.
Sequence	Sequence number assigned to the test.
Start [*button*]	Starts the diagnostic test.
Test [*drop-down box*]	Select the diagnostic test to be performed.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 2***.**

9 CALIBRATION

Unit [n] > Calibration

A calibration file was created specifically for the analyzer and included with the accessories. It is not necessary to recalibrate the system unless it seems out of calibration.

Disabled calibration menu options can be accessed only with the assistance of an authorized Micromeritics Service Representative. Calibrations can be saved to a file and reloaded later.

To review calibration details of the analyzer, go to ***Unit [n] > Unit Configuration***.

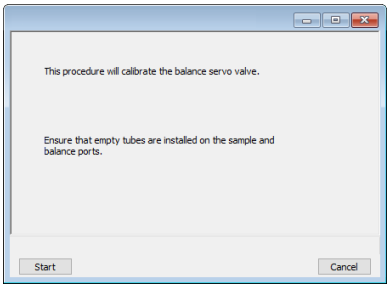
Generally, it will not be necessary to change the data in the calibration file. However, if a condition occurs during the operational verification that requires changes to the calibration data, changes should be saved in a file. Calibration data files are retained in the analyzer history file and can be reloaded in the event that calibration data becomes corrupt.

BALANCE SERVO VALVE

Unit [n] > Calibration > Balance Servo

Use 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 pressure transducer should be calibrated before starting this calibration procedure.

Ensure the pressure transducer has been calibrated before performing this procedure. Go to ***Unit [n] > Unit Configuration*** and view the calibration information. Contact your Micromeritics Service Representative if calibration dates are not listed.



Click **Start**. The window closes when the calibration is complete. Click **Cancel** to stop the calibration process.

MATCH TRANSDUCERS

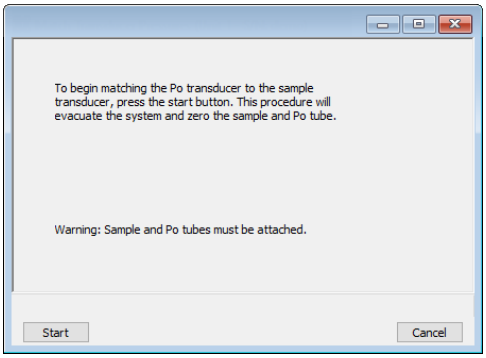
Unit [n] > Calibration > Match Transducers

This process should not be performed when the main transducer reading is abnormal.

This option is disabled for the Gemini 2390a model.

Use to evacuate the system and zero the pressure transducers, then adjust the scale to match them to the manifold transducer near full scale pressure.

A blank sample tube or small plug must be installed on each selected port prior to starting this process.

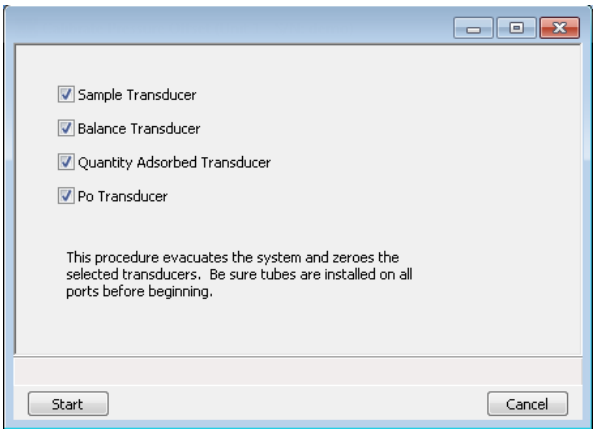


1. Install a blank sample tube or small plug on each port.
2. Click **Start**. The window closes when the operation is complete. Click **OK** when the process is complete.

ZERO PRESSURE

Unit [n] > Calibration > Zero Pressure

This procedure evacuates the system and zeroes the pressure transducers. This calibration should only be performed by qualified service personnel. In order to perform this procedure, sample tubes must be attached to each port.



1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. Click **OK** when the process is complete. The current pressure readings and operation status messages display.

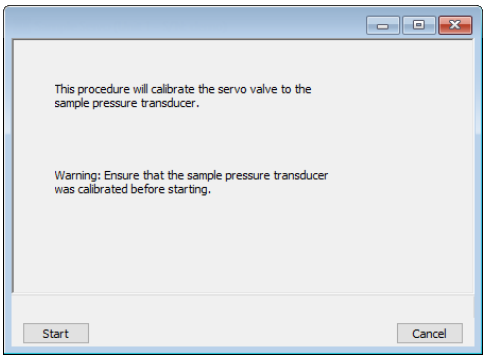
SAMPLE SERVO VALVE

Unit [n] > Calibration > Sample Servo

Unit [n] > Calibrate Analysis > Sample Servo

Use to calibrate the servo valve to the sample transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The pressure transducer should be calibrated before starting this calibration procedure.

Ensure the pressure transducer has been calibrated before performing this procedure. Go to ***Unit [n] > Unit Configuration*** and view the calibration information. Contact your Micromeritics Service Representative if calibration dates are not listed.



Click **Start**. The window closes when the calibration is complete.

Click **Cancel** to stop the calibration process.

Load Calibration from File

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

It is recommended that the current calibration settings be saved using ***Unit [n] > Calibration > Save to File*** prior to loading another calibration file. When loading a previously saved calibration file, a backup of the current file is created and saved as *[SN]last.cal*. The backup file is overwritten each time a new one is created.

Changing the calibration may affect the analyzer's performance.

Save Calibration to File

Unit [n] > Calibration > Save to File

Use to save the current calibration settings to a backup file which can later be reloaded using the ***Unit [n] > Calibration > Load from File*** menu option.

The default file naming convention for calibration files can be used or the file name can be changed. The default file name of 0217-2013-04-25.CAL is interpreted as:

0217	Analyzer serial number
2013-04-25	Date the calibration file was saved
.CAL	File name extension

10 TROUBLESHOOTING

The analyzer has been designed to provide efficient and continuous service; however, certain maintenance procedures should be followed to obtain the best results over the longest period of time. When unexpected results occur, some common operational problems not indicated on the window and their respective causes and solutions are provided.

The following can be found on the Micromeritics web page (www.Micromeritics.com).

- Error Messages document (PDF)
- Parts and Accessories
- Smart VacPrep Operator Manual (PDF)
- Vacuum Pump Guide (PDF)

Most operational problems are caused by:

- Leaks (commonly found at 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
- Impure gas supply

When unexpected analysis results occur, check the above first. Some common operational problems not indicated on the window and their respective causes and solutions are provided below:

Elevator cannot be raised or lowered.

Cause: Dewar elevator stuck.

Action: Check for possible obstruction to elevator movement.

Elevator is noisy.

Cause: The elevator screw may need greasing.

Action: Contact your Micromeritics Service Representative.

Expected results are not within range.

Cause A: Sample improperly degassed.

Action A: Verify that the degassing temperature and degas time were set properly.

Cause B: Gas cylinder(s) may be almost empty - causing gas impurity.

Action B: Replace gas cylinder. See ***Replace a Gas Cylinder on page 10 - 11***.

Cause C: Undetermined.

Action C: Do the following:

1. Perform an empty tube analysis. See ***Perform a Leak Test on page 10 - 15*** and print the results.
2. Perform a reference material analysis. See ***Perform a Reference Material Analysis on page 5 - 16*** . Print the results.
3. Contact your Micromeritics Service Representative.

Specified pressure not reached.

Cause A: Sample or balance tube not properly attached.

Action A: Reattach sample and balance tubes. Ensure both are securely attached to ports. Replace O-rings if defective. See ***Sample Tube O-ring Replacement on page 10 - 25***.

Cause B: Leak in gas line.

Action B: Perform the Adsorptive and Helium Line tests to determine if a leak exists. See ***Diagnostics on page 8 - 1***.

Unable to reach a satisfactory vacuum.

Cause A: Vacuum pump oil level is low or needs to be changed.

Action A: Inspect the oil to see if it is low or needs changing.

Cause B: Vacuum pump is leaking.

Action B: Locate and repair the source of the leak or replace pump.

Cause C: Centering ring has become too flat and unable to hold a vacuum.

Action C: Check the centering ring at the pump intake port. Also check the centering ring at the top of the oil vapor trap. Replace if necessary.

Nitrogen or helium drained from tank, or depleted in a short period of time.

Cause: Leak in the gas line connection.

Action: Perform the Adsorptive (or Helium) Line test to determine the location of the leak. See ***Diagnostics on page 8 - 1***.

Unit does not work when powered ON.

Cause A: Power cord not fully inserted at one end.

Action A: Insert power plug firmly into power source and analyzer power connecter.

Cause B: No power at outlet.

Action B: Plug in another electrical device to test the outlet. If there is no power, contact an electrician.

Cause C: Plug prongs bent so that contact not made at outlet.

Action C: Gently move power plug at outlet while watching power indicator light. If the indicator light comes on, have electrician replace outlet or plug.

Cause D: Power cord damaged.

Action D: Have electrician check cord using a test meter. Replace the cord if defective.

Cause E: Loose internal connection or broken wire.

Action E: Call a Micromeritics Service Representative for repair or replacement information.

Valves cannot be operated.

Cause: Cable from computer to the instrument is loose.

Action: Reconnect the cable.

Vacuum pump is noisy.

Cause A: Sample tube connector is loose.

Action A: Tighten fitting. Replace O-ring.

Cause B: Sample tube O-ring is worn or cracked.

Action B: Replace O-ring. See ***Sample Tube O-ring Replacement on page 10 - 25***.

Cause C: Sample tube is cracked.

Action C: Replace with new sample tube.

Cause D: No sample tube loaded on a selected port.

Action D: Install plug or empty sample tube.

Cause E: Gas inlet valve open while vacuum valve open.

Action E: With manual control enabled, use the instrument schematic to close gas inlet valve.

PARTS AND ACCESSORIES

Parts and accessories can be found online at www.Micromeritics.com.

POWER

The Gemini VII 2390 is designed to operate with a universal input power supply (100-240VAC) at 50/60Hz. Noise-free power of the correct voltage and frequency, with a safety earth ground, should be available through a standard wall receptacle. The power outlet should be able to supply 15 amps @ 100 or 110VAC ±10% or 7.5 amps @ 230VAC ±10%. These requirements can be checked by using a voltage meter (available at most hardware or electronic supply houses) or a multimeter. There should also be sufficient outlets for the computer, monitor, printer, and any other peripheral devices.

The analyzer and peripheral devices **must** be installed on their own dedicated power line. Other devices — such as motors, generators, or ovens — **should not** be placed on the same power line.

Replacement power supply cords must be rated for the specifications stated above.

SAFE SERVICING

Do not modify this instrument without the authorization of a Micromeritics service personnel.

To ensure safe servicing and continued safety of the instrument after servicing, service personnel should be aware of the following risks:

Product specific risks that may affect service personnel:

- **Electrical**. Servicing or repair could require opening the outer panels and exposing energized electrical components.
- **Liquid nitrogen**. See *Dewar Precautions on page 5 - 1*.
- **Elevator.** Could pose a pinching hazard when lowering. Maintenance may require the elevator screw to be greased. The service engineer can use a manual switch on the elevator assembly to cycle the elevator to distribute the grease to permit safe servicing and continued safety of the equipment after servicing.

Protective measures for these risks:

- **Electrical**. The majority of electrical components operate at low voltage (24V or less) and pose low risk when energized. Maintenance, troubleshooting, and repairs should be performed with the instrument de-energized whenever possible, in accordance with standard electrical safety guidelines.
- **Elevator**. Moves very slowly. It is normally operated from the computer attached to the instrument so the operator is physically distanced from the instrument. The safety shield door of the instrument should be closed during elevator operation. Use of the manual switch on the instrument requires particular attention and care by the service personnel.

Verification of the safe state of the instrument after repair:

- Elevator must be in the down position.
- Sample tubes must be removed to prevent accidental breakage. Ports should be capped (recommended).
- Safety shield door is closed (recommended).

GUIDELINES FOR CONNECTING GASES

These instructions refer to the installation of a gas line, regulator, and gas cylinder for each type of gas used. Expansion kits or other accessories may be used in the lab. If so, special consideration should be given to these configurations when installing the gas lines.

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

- Place gas cylinders within 6 feet (2 m) of the gas inlets of the analyzer. Place the cylinders close enough to allow for proper connection at the analyzer inlet.

  Using gas line extenders on gas cylinders located in remote areas may degrade gas quality and reduce pressure. Gas lines are typically five to six feet long.

  Long gas lines, such as those used with gas cylinders placed in remote areas, must be evacuated for an extended period of time to remove ambient gases. When possible, avoid placing gas cylinders in remote locations. It is always best to have gas cylinders located near the analyzer
- Use a retaining strap (or other appropriate tether) to secure the gas cylinder.
- Always use the gas lines provided with the analyzer. It is very important that proper gas lines are used with the analyzer.

  - **Do not use** polymer tubing for the gas line.
  - **Do not use** flexible gas lines. Some flexible lines may appear to be appropriate, such as those with a herringbone covering, but the line may be coated internally with a polymer.

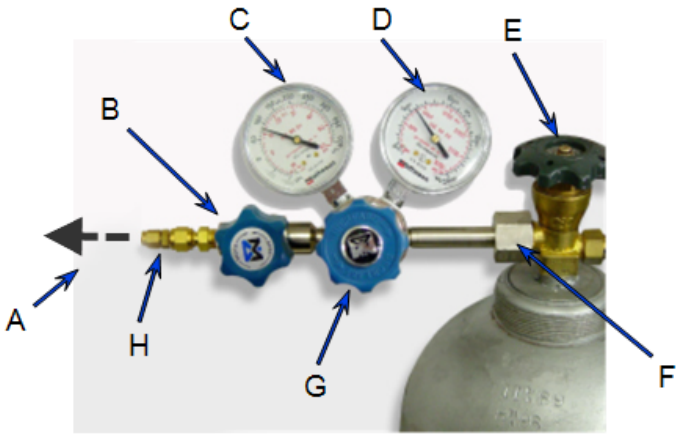
- Carefully route the gas lines from the cylinder to the analyzer avoiding overlapping or entangling gas lines. This will help avoid confusion when maintenance is required.
- Label the gas line at the analyzer inlet for proper identification and maintenance.
- Replace gas cylinders before gas is depleted. It is best to replace a gas cylinder when the pressure reads approximately 500 psi (3500 kPa) on the high pressure gauge. Contaminants absorbed to the walls of the cylinder will desorb as the pressure decreases.
- Ensure the gas cylinder is closed before connecting to the analyzer.

CLEAN AND VERIFY THE GAS LINE

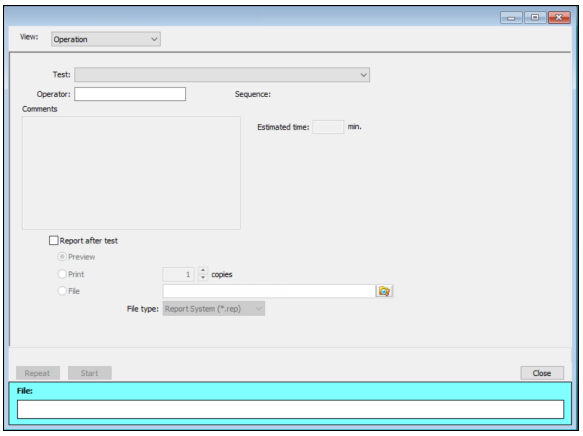
Unit [n] > Diagnostics

Always clean the gas lines and verify there are no leaks at the connections after a gas cylinder is connected. This test examines the gas line from the analyzer to the gas cylinder, then from the analyzer to the regulator shut-off valve. A report is generated at the completion of the test to verify that it has passed or failed. Causes and corrective action for a failure are provided.

Before beginning, confirm that the state for valves and the low pressure gauge are as follows:

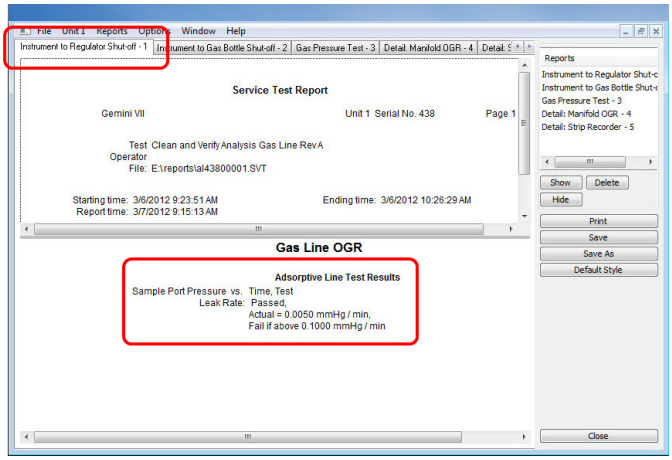


A. Gas tubing to instrument
B. Gas regulator shut-off valve - OPEN
C. Low pressure gauge - 14-15 psig
D. High pressure gauge
E. Gas cylinder shut-off valve - CLOSED
F. Regulator connecter nut
G. Regulator control knob - OPEN
H. Brass reducer fitting



1. Click the down arrow to the right of the *Test* field and select *Gas Line Clean and Verify # Rev [n]*. This procedure uses the analysis gas as an example. Select *Clean and Verify Helium Gas Line Rev [n]* if the helium cylinder is being verified.
2. Select *Report after test* and select *Preview* as the destination. Resize the window if this option does not display.
3. Click **Start**. From the *View* drop-down list, select either *Operation*, *Instrument Log*, or *Instrument Schematic*.

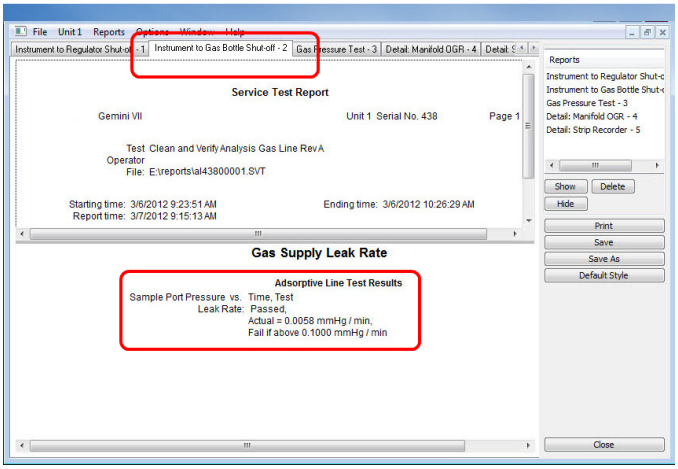
4. A series of prompts display requiring operator response. The length of time a test will run is also indicated.
5. A popup window indicates the test is complete. Click **OK**.
6. Click the *Instrument to Regulator Shut-off Valve -1* tab to display its report.



The *Leak Rate field* should read *Passed* indicating the gas line from the analyzer to the regulator is clean and leak-free.

If the *Leak Rate field* indicates *Failed*, a leak is indicated. Check the connections from the analyzer to the gas regulator valve. Tighten as necessary, then run the test again.

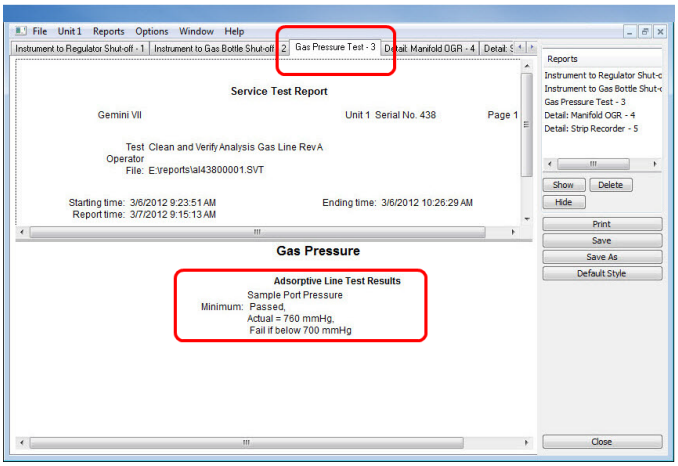
7. Click the *Instrument to Gas Bottle Shut-off Valve - 2* tab to display its report.



The *Leak Rate* field should read *Passed* indicating the gas line from the analyzer to the regulator is clean and leak-free.

If the *Leak Rate* field indicates *Failed*, a leak is indicated. Check the connections from the regulator shut-off valve to the gas cylinder shut-off valve. Tighten as necessary, then run the test again.

8. Click the *Gas Pressure Test - 3* tab to display its report.



On the report, the *Minimum field* should read *Passed* indicating all valves are in the proper state for operation.

If the *Minimum* field indicates *Failed*, one or more valves is not in the proper position. Set the valves as shown at the beginning of this section and ensure the appropriate pressure is displayed on the low pressure gauge.

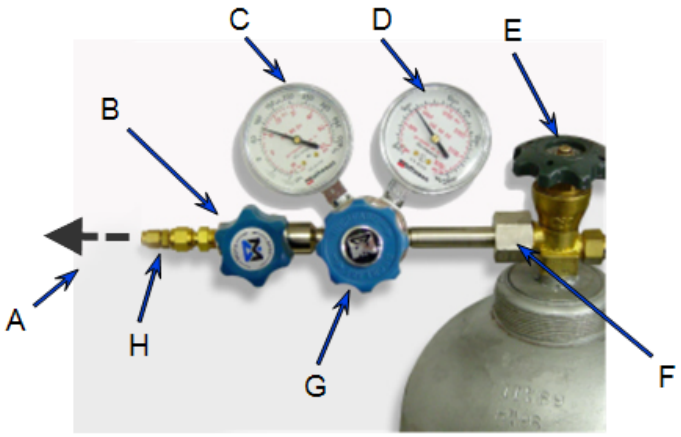
If running the test again, close the gas cylinder valve before starting the test.

9. Click **Close** to close the test report. Click **Close** again to close the test.

REPLACE A GAS CYLINDER

These instructions apply to working with inert gases only. When working with hazardous gases, follow the safety procedures established by your lab.

A power failure or loss of cryogen can result in dangerous pressures in the sample chamber. When using toxic or flammable gases, additional venting of the cabinet may be required.



A. Gas tubing to instrument
B. Gas regulator shut-off valve
C. Low pressure gauge
D. High pressure gauge
E. Gas cylinder shut-off valve
F. Regulator connecter nut
G. Regulator control knob
H. Brass reducer fitting

Disconnect a Depleted Gas Cylinder

1. Close the regulator shut-off valve and gas cylinder shut-off valve by turning the knobs clockwise.
2. Disconnect the gas line from the regulator. Gas will be vented from the line. It is not necessary to disconnect the gas line from the analyzer inlet if the cylinder will be replaced immediately with one of the same type.
3. Open the gas regulator shut-off valve by turning the knob counter-clockwise. Gas will be vented from the regulator.
4. Turn the regulator control knob clockwise to open and vent any remaining gas. Both gauges should read at or near zero. If not, make sure the gas regulator shut-off valve is open.
5. Close the regulator by turning the control knob counter-clockwise.
6. Use an appropriate wrench to loosen the nut at the regulator connector nut then remove the regulator from the cylinder.
7. Replace the protective cap on the depleted cylinder. Disconnect the retaining strap and move the cylinder to an appropriate location.

Connect a Gas Cylinder

Regulator Pressure Settings

Analyzer	Gauge should indicate
Gemini	15-18 psig (103 - 124 kPag)

Exceeding the maximum recommended air pressure could cause personal injury or damage the instrument.

Move the replacement cylinder close to the analyzer and tether it into place.

1. Use an appropriate cylinder wrench to remove the protective cap from the replacement gas cylinder.
2. Place the protective cap in a secure location. It will be needed to recap the gas cylinder when it is depleted and replaced.
3. Attach the gas regulator to the gas cylinder connector. Hand tighten the nut, then use an appropriate wrench to tighten an additional 3/4 turn.

Over-tightening the fitting may cause a leak.

4. Check for leaks at the high pressure side of the regulator and in the connector.
   a. Turn the regulator control knob fully counter-clockwise.
   b. Slowly open the gas cylinder shut-off valve, then quickly close it.
   c. Observe the pressure on the high pressure gauge for approximately one minute.
      - If the pressure is stable, proceed with the next step.
      - 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.
5. Purge the air from the lines by doing the following:

Purge the regulator before proceeding to prevent contamination of the analysis gas supply.

   a. Open the gas cylinder valve to pressurize the regulator, then close the valve.
   b. Adjust the *Pressure Control* knob to approximately 5 psi.
   c. Turn the regulator *Shut-off* valve counter-clockwise to open. Allow gas to flow until both gauges read approximately zero.
   d. Close the regulator *Shut-off* valve to stop gas flow.

e. Reconnect the gas line to the regulator.
f. Use two 7/16 in. (11 mm) wrenches to tighten the gas line connection. Hold one wrench fitting steady and the other to tighten the connector nut.

6. Set the analyzer pressure by doing the following:
   a. Turn the *Regulator Control* knob clockwise until the low pressure gauge indicates the appropriate pressure. See the *Regulator Pressure Settings* table in ***Connect a Gas Cylinder on the previous page***.
   b. Open the regulator *Shut-off* valve.
   c. Open the gas cylinder *Shut-off* valve and flow gas for 10 to 30 seconds.
   d. Close the gas cylinder *Shut-off* valve.
   e. Close the gas cylinder valve.
7. If the gas line to the instrument inlet was previously disconnected, reconnect it now.

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

Show Instrument Schematic on page 2 - 17

Use to enable the manual control of certain system valves and pump components on the analyzer schematic.

When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***.

If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.

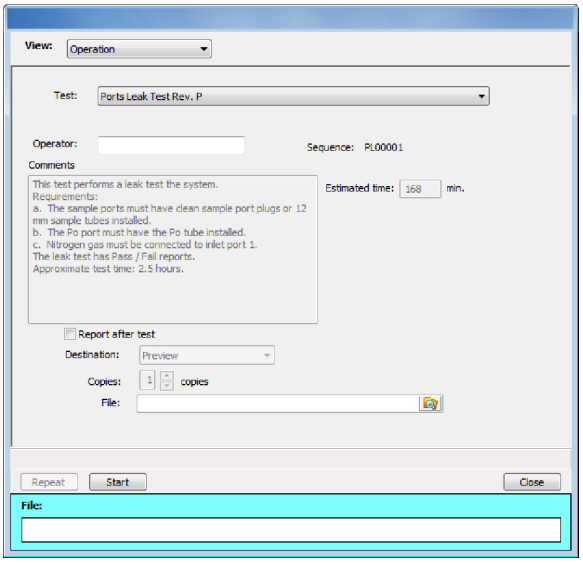
PERFORM A LEAK TEST

Unit [n] > Diagnostics

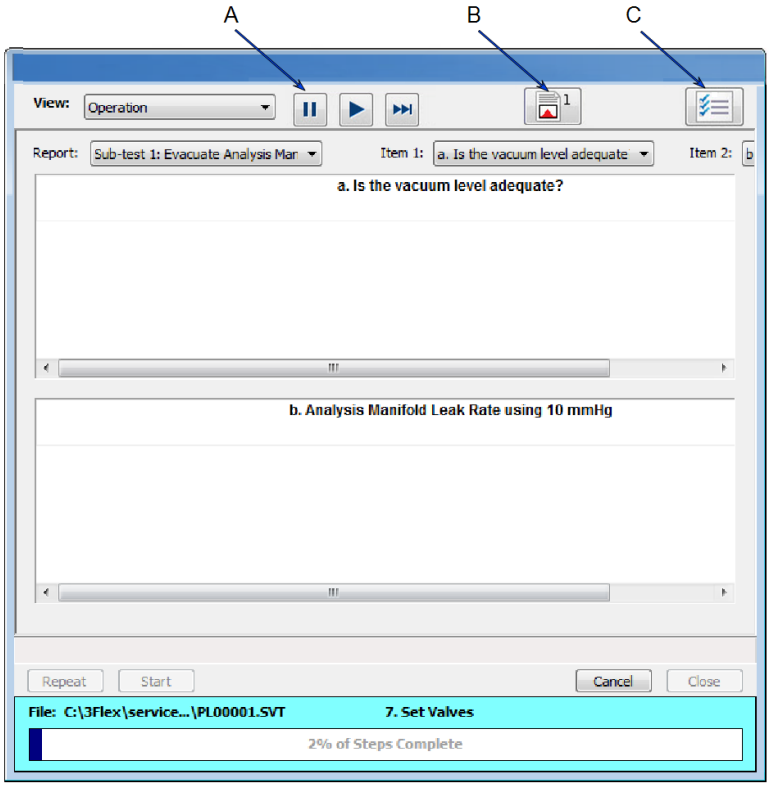
A Micromeritics Service Representative may request that a leak test be performed to determine if there is a system leak and may also require a copy of the report generated by this test.

The test provides:

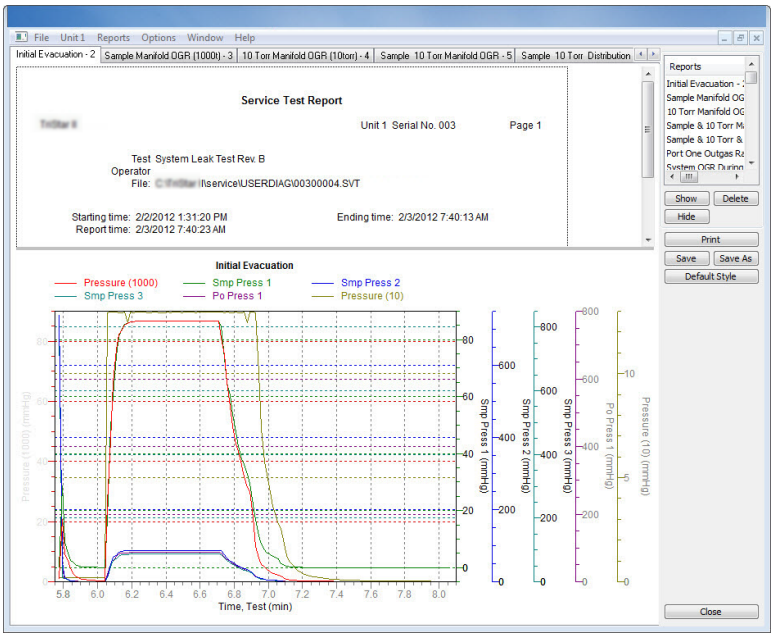
- Prompts on preparing the analyzer for the test.
- Approximate time period of the test.
- Prompts in which an operator response is required.



1. Select the test to run.
2. *Select Report After Test* and choose *Preview as the destination.*
3. Click **Start**.
4. Verify all tests have a *Passed* status by selecting the tabs and looking for the *Passed* status for each test run.
5. Click **Save As** to save the test file results.



A. Suspend/Resume/ Skip/Play buttons
B. Port report buttons
C. Live graph settings



PERFORM REFERENCE MATERIAL ANALYSIS

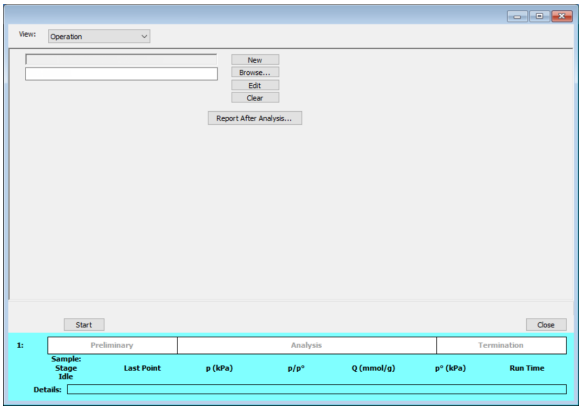
Perform a Reference Material Analysis on page 5 - 16

PERFORM AN EMPTY TUBE ANALYSIS FOR DIAGNOSTIC PURPOSES

An empty tube analysis is a means of pinpointing operational problems in the analyzer. Leaks, as well as electronic problems, will result in an unsatisfactory blank analysis. This test should be performed prior to detailed troubleshooting. The data produced during the blank analysis can be helpful in identifying the cause of a problem and should be performed before contacting a Micromeritics Service Representative.

Helium is required for performing an empty tube analysis since a measured free space is required. If helium is unavailable, contact a Micromeritics Service Representative for an alternative method of performing this procedure.

1. Install empty, straight-wall sample tubes (free of cracks or chips) of the same size in the sample and balance ports. Ensure the O-rings are in good condition.
2. Place the Dewar support on the elevator, then place a Dewar of water at ambient temperature on the Dewar support. (A Dewar support is not necessary for the larger Dewar used with the Gemini VII 2390t. Place the Dewar directly on the elevator.)
3. Go to ***Unit [n] > Empty Tube Analysis***.



4. Click **Browse** and select a file or to create a new one. Click **Open**.
5. Click **Edit** to make modifications or replace all parameters in the open file using the **Replace All** button. Click **Save**, then click **Close**.
6. Click **Report after analysis** to generate reports to specify the report destination when the analysis is complete. Click **OK**.
7. Click **Start** to begin the analysis. A window displays data as they are collected. A short delay is encountered before the port status changes from the Idle state at the bottom of the screen. Review the analysis results to see if they are within the tolerances shown in the following table. If these results are not achieved, call a Micromeritics Service Representative.

P/P_0	Tolerance in cm^3 STP
0.1	±0.008
0.2	±0.010
0.3	±0.012
0.4	±0.014
0.5	±0.016
0.6	±0.018
0.7	±0.020
0.8	±0.022
0.9	±0.024

PREVENTIVE MAINTENANCE

Perform the following preventive maintenance procedures to keep the analyzer operating at peak performance. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

Maintenance Required	Frequency
Alumina in vapor oil traps *	Replace as required.
Analyzer exterior	Clean as required or every 3 to 6 months.
Dewar	Check and clean weekly.
Diaphragm vacuum pump **	If an oil-free pump is used, replace diaphragm(s) every 12 months.
Dry forepumps	As required or every 24 months.
Port filters and O-rings	Replace every 3 to 6 months.
Sample tube O-ring	Replace as required or every 3 to 6 months.
Test for leaks	As required or every 12 months.
Vacuum pump exhaust filter	Replace as required or every 12 months.
Vacuum pump fluid *	Inspect and change as required or every 3 to 6 months.

* Oil sealed vacuum pumps only.

** For oil-free pumps only: After 12 to 18 months of operation, the diaphragm(s) in the pump will wear out and the pump becomes completely inoperable. To prevent instrument downtime due to an inoperable pump, it is recommend that the diaphragm(s) be replaced by a Micromeritics Service Representative every 12 months.

CHECK AND CLEAN THE DEWAR

When handling Dewars, follow the precautions outlined in *Dewar Precautions on page 5 - 1*.

Always handle glass Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. If in doubt, contact your safety officer.

Ice and suspended frost particles may accumulate in the bottom of the analysis port Dewar. Particles or deposits exceeding 1/4 in. in depth may jam between the bottom of the sample tube and the bottom of the Dewar.

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.

Accumulated ice is likely to melt and form a pool of water in the Dewar if all liquid nitrogen evaporates. The water must be removed, otherwise it will solidify when liquid nitrogen is added and could press on the bottom of the sample tube causing breakage.

To ensure problems do not develop due to ice accumulation, check the Dewar after each use. Clean on a weekly basis.

1. Remove the Dewar from the analyzer.
2. Pour out liquid nitrogen into an appropriate cryogenic container. Do not re-use liquid nitrogen.

CAUTION

Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.

3. Rinse the Dewar with warm water to melt any remaining ice accumulation which may remain. Dry thoroughly.
4. Replace the Dewar.

CLEAN THE INSTRUMENT

The exterior casing of the instrument may be cleaned using a clean, lint-free cloth dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution. Do not use any type of abrasive cleaner. It is not necessary to remove knobs, screws, etc. while cleaning.

- Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.
- Use only a mild detergent in water to clean safety shields. The use of isopropyl alcohol can damage the shield surface.

CLEAN THE GAS DELIVERY TUBES

If using a FlowPrep, gas delivery tubes should be wiped with a clean, lint-free cloth after each use to remove any particles of sample that may have adhered to the tube. If a gas delivery tube becomes clogged or damaged, it should be replaced.

1. Remove the gas delivery tube from the flexible tubing.
2. Attach a new gas delivery tube by pressing the flexible tubing over the gas delivery tube. Ensure that the flexible tubing completely covers the ferrule at the end of the gas delivery tube.

LUBRICATE THE ELEVATOR DRIVE ASSEMBLY

The elevator screw is lubricated before it leaves the factory and should not require lubricating. If the elevator starts to vibrate or becomes noisy when traveling, contact a Micromeritics Service Representative for disposition.

Should lubrication become necessary, apply a light coat of Teflon Magnalube-G grease [*Micromeritics part number 004-16163-00*] to the elevator screw. Do not grease the guide rods.

POWER INSTRUMENT ON AND OFF

DO NOT connect or disconnect cables when the instrument is powered ON.

If a Smart VacPrep is used, it is recommended that the power to the Smart VacPrep remain ON when the analyzer is powered on. If it does become necessary to power off the Smart VacPrep, exit the analyzer program first. Restart the analyzer program, then power on the Smart VacPrep.

Power ON the equipment in the following order:

1. Computer, monitor, and printer.
2. Analyzer.
3. External vacuum pump (the pump must warm approximately two hours before performing analyses).
4. Degasser.

Power OFF the equipment in the following order

1. Exit the analysis program. Failure to do so could result in loss of data. If an analysis is in progress when closing the application, the following message is displayed:

   2459 - An Instrument is busy. A delay in restarting this application could result in loss of new data. Continue program exit? Yes / No

   Yes. Closes the program. The analysis continues and data continue to be collected. The data will be restored when the application is restarted. Reports queued in the print manager will print. If a power failure occurs and an uninterruptible power supply (UPS) is not attached to the analyzer, the data collected after exiting the analysis program are lost.

   No. The program remains open and the analysis continues to run.

2. Computer, monitor, and printer.
3. Analyzer.
4. External pump.
5. Smart VacPrep.

SAMPLE PORT FRIT REPLACEMENT

The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

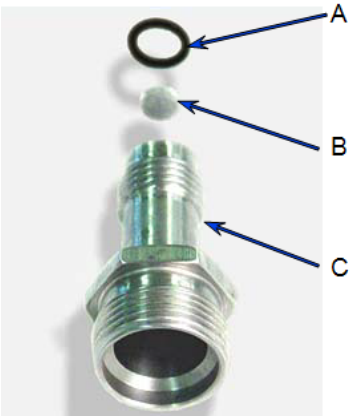
A frit is located in the connecting nut attached to each analysis port. If the frit becomes contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated frit on the analysis port may be indicated as a leak or a free space reading much lower than normal.

Use the proper size frit for the sample port. The analyzer will not operate properly if an incorrect size is used.

1. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. If the analyzer schematic does not display, go to ***Unit [n] > Show Instrument Schematic.***
2. Right click on the valve of the appropriate port. If the valve is open, click **Close** to close the valve.
3. Use a wrench to remove the connecting nut from the sample port. Pry out the filter (frit) and O-ring.

To avoid degassing problems, the frit should be clean and should not be touched with bare hands.

4. Place a new frit into the connecting nut.



A. O-ring
B. Filter (frit)
C. Sample tube fitting

5. Replace the filter and O-ring. Carefully reassemble the sample tube fitting and reinstall on the sample port. Tighten by hand, then with a wrench to prevent leaks.

SAMPLE TUBE O-RING REPLACEMENT

The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

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.

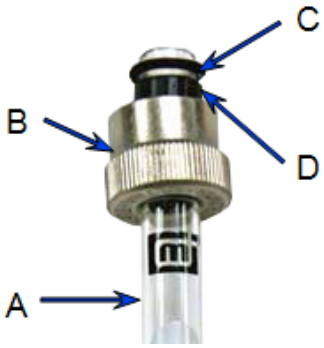
Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the analyzer schematic to verify valve status.

CAUTION

Do not allow the sample tube connector nut drop onto the bottom of the tube as it may break the tube.

Use the proper size frit for the sample port. The analyzer will not operate properly if an incorrect size is used.

1. Carefully remove the Dewar from the analyzer. Take care not to bump the sample tube bulbs with the Dewar during this process. Place the Dewar aside.
2. Hold the sample tube firmly with one hand, loosen the sample tube connector nut by turning counter-clockwise.
3. Carefully pull the sample tube down until it is free from the port. It may be necessary to grasp the sample tube with both hands.



A. Sample tube or balance tube
B. Connector nut
C. O-ring
D. Ferrule

4. Remove the O-ring from the top of the sample tube and replace it with a new one.

If the O-ring remains inside the sample port, use the O-ring removal tool from the accessory kit *[tool part number 004-54618-00*] or use a pair of tweezers or needle-nose pliers to remove it.

5. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.
6. Slide the sample tube connector nut up the tube until it comes in contact with the port fitting (the ferrule and O-ring will move along with the connector nut). Then, turning clockwise, hand tighten the connector nut to the sample connector.

Recover from a Power Failure

The analyzer saves entered and collected data in case of power failure. File parameters and any other data entered will still be present when power is restored. If an analysis was in progress when the power failure occurred, it will be canceled when the analyzer restarts. Any data collected during the analysis will still be present, but the analysis should be restarted in order to produce complete results.

Oil-based Vacuum Pump

The *Vacuum Pump Guide* can be found on the Micromeritics web page (www.Micromeritics.com).

A ANALYZE SAMPLES WITH A TOTAL SURFACE AREA OF 1.0M^2 OR LESS

The unique balanced measurement method used in the Gemini permits small amounts of surface area to be measured with nitrogen gas that otherwise would be measurable only with krypton. Low surface area samples often displace many times more nitrogen than they adsorb, especially if composed of low-density materials of large particle size. The standard, built-in, helium free-space difference measurement and mathematical compensation routine typically removes the effects of more than 99% of this sample displacement, but the small amount remaining uncompensated can still be significant.

A technique for adding a compensating volume with negligible surface area into the balance tube has been developed. It can reduce the size of the initial imbalance to low levels and allow precise measurement of adsorbed gas.

This technique can be performed by the following methods:

- Using glass beads in the balance tube
- Using filler rods in the sample and balance tubes

Some users find that a combination of the two methods — using glass beads and filler rods — produces even better results. This, however, will depend on the type of sample you are analyzing.

Filler rods and glass beads are included in the accessories kit. Either straight-wall or bulb sample tubes may be used.

USING GLASS BEADS

This method typically produces the best results.

1. Place an appropriate quantity of sample in a clean sample tube.
2. Load a second sample tube of the same size with glass beads that have a total volume approximately the same as the sample volume.
   a. Determine the volume (v) of the sample in cm^3:

$$v = \frac{m}{\rho}$$

where

m	=	mass of sample (g)
ρ	=	density of sample (g/cm^3); if density is unknown, refer to your laboratory handbook.

   b. Determine the number (n) of glass beads needed to equal the sample volume:

$$n = \frac{v}{0.014 cm^3}$$

where

$0.014 cm^3$ = approximate volume of one bead

3. Outgas the sample in the sample tube at an appropriate temperature for an appropriate amount of time.
4. Install the sample tube (containing the outgassed sample) onto the analysis port and the sample tube (containing the glass beads) onto the balance port.
5. Specify a one-point measurement (P/P_0 = 0.05 to 0.1) so that the initial free-space measurement can quickly be determined; then perform the measurement.
6. Using the measured free space absolute value and the following relationship, determine the mass of glass beads to remove from (or add to) the balance tube to reduce the free-space imbalance:

$$\frac{\text{free space } cm^3 \times 2.515 g/cm^3}{3.53} = \text{mass of glass beads(g)}$$

where

$2.515 g/cm^3$ = density of glass beads

3.53 = thermal correction (no units)

Note that the volume of one glass bead is approximately 0.014 cm^3. Therefore, if the measured free space is less than 0.02 cm^3, it is unnecessary to correct the free space.

7. Use a beaker of warm water to bring the balance tube to room temperature before removing it from the balance port of the Gemini to remove (or add) glass beads. This prevents condensation of moisture from the laboratory atmosphere onto the cold glass beads.
8. Remove the balance tube:
   - If the measured free space is negative (–), add the calculated mass of glass beads into the balance tube.
   - If the measured free space is positive (+), remove the calculated mass of glass beads from the balance tube.
9. Reinstall the balance tube onto the balance port of the Gemini analyzer.

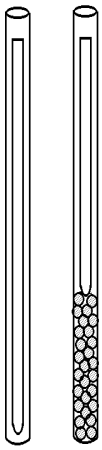
For subsequent samples of the same material, use the same mass of sample that was used for the initial sample so that the original bead quantity may be left undisturbed on the balance port.

10. Prepare the analysis Dewar and place it on the elevator.
11. Close the sample compartment door and start the analysis.

USING FILLER RODS

1. Clean the sample tube, balance tube, and filler rods; label your sample and balance tubes.
2. Prepare the sample and place it into the sample tube; insert the shorter filler rod into the sample tube.

   If using bulbous sample tubes, use the same size filler rod as used in the balance tube.
3. Ensure that the filler rods are equidistant from the top of each tube. If they are not, add or remove sample until they are the same distance from the top of the tube.



CAUTION

Packing of some powders may restrict gas access to the powder and cause slower equilibration and/or lower results.

4. Attach the sample tube to the analysis port and the balance tube to the balance port.
5. Prepare the analysis Dewar and place it on the elevator.
6. Close the sample compartment door and start the analysis.

Using Glass Beads and Filler Rods

This method may further improve results, depending on the sample material being analyzed.

1. After determining and loading the amount of glass beads to use, insert a filler rod into the balance tube.
2. Insert a filler rod into the sample tube. Ensure that the filler rods are equidistant from the top of each tube. If they are not, add or remove sample until they are the same distance from the top of the tube.
3. If using bulbous sample tubes, use the same size filler rod as used in the balance tube.
4. Attach the sample tube to the analysis port and the balance tube to the balance port.
5. Prepare the analysis Dewar and place it on the elevator.
6. Close the sample compartment door and start the analysis.

B 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 physisorption 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 (Q_m), 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:

$$U(s) = 4\epsilon(\frac{\sigma}{s})^{12} - (\frac{\sigma}{s})^{6}$$

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 considerably 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[1)] 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.

1) 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).

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 calculations using these methods still require 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 ***DFT Model References on page B - 17***.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure 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.

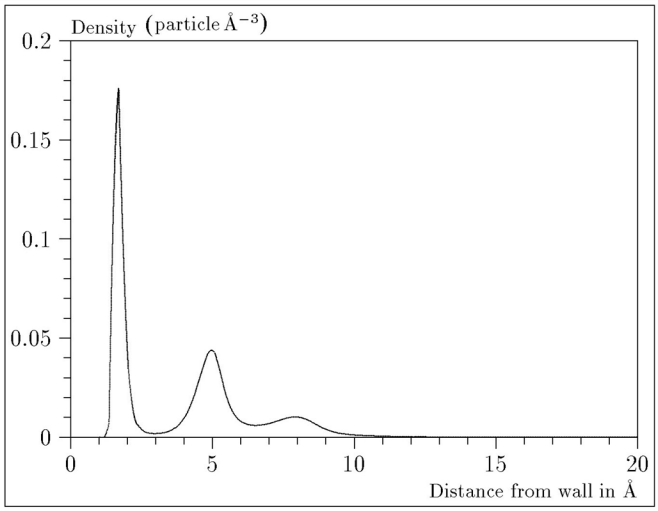


Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

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 2 shows how the profiles change with pressure for a model pore with H = 40 angstroms. The inset shows the density profiles for the corresponding points of the isotherm.

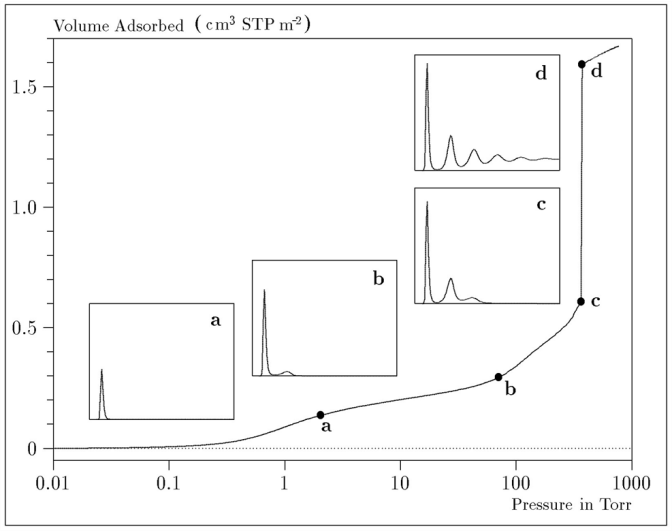


Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

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 the previous graph 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 the Figure 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.

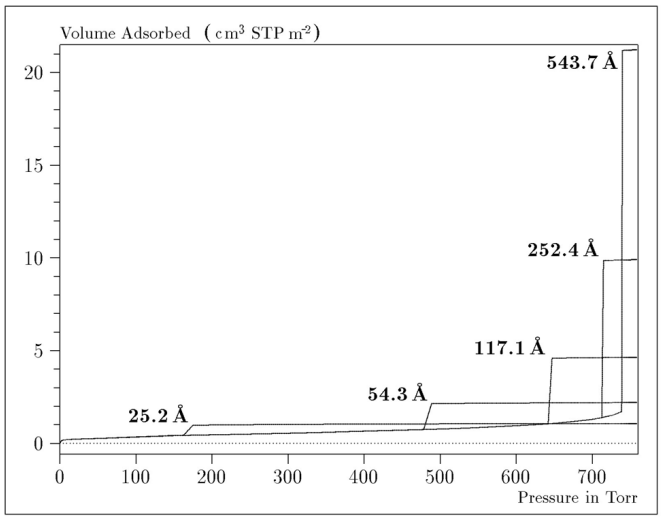


Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure 4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

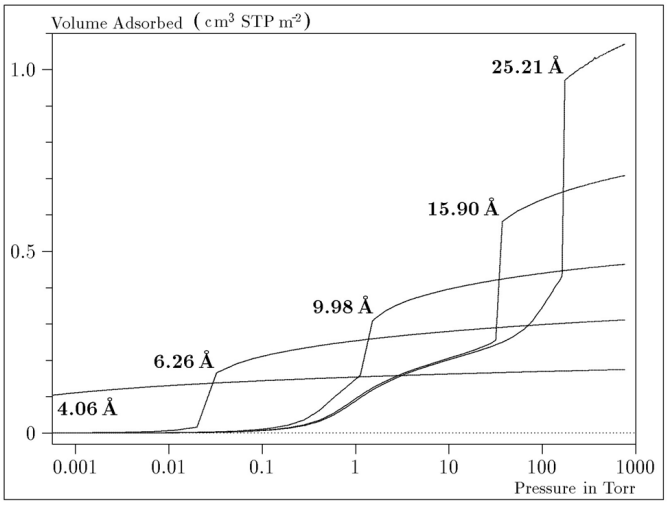


Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

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 1×10^{-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.

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 (see *DFT Model References on page B - 17* [items 3 and 4]), 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* window) 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, i.e., for the adsorptive / adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface
AR - Cylindrical Pores - Oxide Surface

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

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. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

References: 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 77K

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. Occelli, “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.

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, 9816-9823.9b.

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 Model

Geometry: Free surface
Substrate: Any
Category: Surface energy
Method: Argon at 87K

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 77K

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.

References: 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).

N2 - Carbon Slit Pores by NLDFT
Ar - Carbon Slit Pores by NLDFT

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 angstroms.

References: 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).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT
Ar - Carbon Finite Pores, As=6, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 angstroms.

References: Jacek Jagiello and James P. Olivier. “A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.,” The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Finite Pores, As=12, 2D-NLDFT
Ar - Carbon Finite Pores, As=12, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and/or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

References: Jacek Jagiello and James P. Olivier. “A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.,” The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Cylinder, single-wall nanotube by NLDFT
Ar - Argon Cylinder, single-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: 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).

N2 - Carbon Cylinder, multi-wall nanotube by NLDFT
Ar - Argon Cylinder, multi-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: 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)

Ar - Zeolites H-Form by NLDFT

Geometry: Cylinder
Substrate: Zeolite
Category: Porosity
Method: Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H+ and (NH4)+ exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Ar - Zeolites Me-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Models Based on Classical Theories

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory. The *Calculations* document can be found on the Micromeritics web page (www.Micromeritics.com). Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

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-de Boer 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 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{-5.00}{ln(P/P_0)}\right)^{1/3}$$

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 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 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = 3.54\left(\frac{13.99}{0.034 - log(P/P_0)}\right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 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: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log\left(p/p^0\right) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 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).

References: 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).

DFT Model 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: Simple 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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C EXPORTED DATA EXAMPLE

This exported data has been truncated for this manual.

```
Sample Information

                     Sample:    Carbon Black ASTM SRB C7
                   Operator:    AWT
                  Submitter:    Micromeritics
                  Mass type:    Entered
                Sample mass:    0.2388 g
                    Density:    1.900 g/cm³
               Type of data:    Automatically collected
            Instrument type:    2390
  Original instrument type:    2390
                   Comments:


Degas Conditions


  Degas conditions:    Degas Conditions

             Smart VacPrep evacuation

     Backfill sample tube:    Automatic
           Evacuation rate:    0.67 kPa/s
  Unrest. evacuation from:    0.67 kPa
              Vacuum level:    1.333224e-03 kPa
```

```
          Evacuation time:    0 min
    Temperature ramp rate:    1.0 °C/min
       Target temperature:    30 °C
            Hold pressure:    13.3 kPa

                Heating Phase

  Sample                      Ramp
  prep:     Temperature       Rate      Time
  Stage    (°C)  (°C/min)    (min)
----
       1             20          10       10
       2            300          20       60


Analysis Conditions


  Analysis conditions:    Carbon Run Conditions

              Pressure Table

   Starting      Pressure       Ending
   Pressure      Increment     Pressure
    (p/p°)        (p/p°)        (p/p°)
---
  0.000000000                 0.050000000
  0.050000000                 0.075000000
  0.075000000                 0.100000000
  0.100000000                 0.125000000
  0.125000000                 0.150000000
```

0.150000000	0.175000000
0.175000000	0.200000000
0.200000000	0.225000000
0.225000000	0.250000000
0.250000000	0.275000000
0.275000000	0.300000000
0.300000000	0.325000000
0.325000000	0.350000000
0.350000000	0.375000000
0.375000000	0.400000000
0.400000000	0.425000000
0.425000000	0.450000000
0.450000000	0.475000000
0.475000000	0.500000000
0.500000000	0.525000000
0.525000000	0.550000000
0.550000000	0.575000000
0.575000000	0.600000000
0.600000000	0.625000000
0.625000000	0.650000000
0.650000000	0.675000000
0.675000000	0.700000000
0.700000000	0.725000000
0.725000000	0.750000000
0.750000000	0.762500000
0.762500000	0.775000000
0.775000000	0.787500000
0.787500000	0.800000000
0.800000000	0.812500000
0.812500000	0.825000000
0.825000000	0.837500000
0.837500000	0.850000000

```
0.850000000                    0.862500000
0.862500000                    0.875000000
0.875000000                    0.887500000
0.887500000                    0.900000000
0.900000000                    0.910000000
0.910000000                    0.920000000
0.920000000                    0.930000000
0.930000000                    0.940000000
0.940000000                    0.950000000
0.950000000                    0.960000000
0.960000000                    0.970000000
0.970000000                    0.980000000
0.980000000                    0.990000000

            Preparation

Evacuation rate:    40.00 kPa/min
Evacuation time:    2.00 min




   Free Space

Calculated

                    p° and Temperature

          p° type:    Measured in Psat tube for each point
Temperature type:    Entered
     Temperature:    77.150 K
```

```
            Analysis Method

       Analysis mode:    Equilibration
  Equilibration time:        2 s


Adsorptive Properties


                      Adsorptive:    Nitrogen (N2)
       Non-condensing adsorptive:    No
       Maximum manifold pressure:    139.989 kPa
  Molecular cross-sectional area:    0.162 nm²
     Adsorbate molecular weight:    28.01
            Thermal conductivity:    1.00
             Non-ideality factor:    0.0000620
       Density conversion factor:    0.0015468


Report Options


    Report options:    Carbon Black
  Show report title:    Yes
       Report title:    Micromeritics Instrument Corporation
       Show graphic:    Yes
       Graphic file:    miclogo.emf
     Graphic height:    0.250 in
      Graphic width:    2.000 in
```

```
Summary: Yes

Surface Area

Single-point BET:    Yes
Multi-point BET:     Yes
Langmuir:            No
t-Plot Micropore:    Yes
t-Plot External:     Yes
BJH Cumulative Adsorption:    Yes
BJH Cumulative Desorption:    No
D-H Cum. Adsorption:    No
D-H Cum. Desorption:    No

Pore Volume

Adsorption Total:    Yes
Relative Pressure:    -1.000000000 p/p°
Desorption Total:    No
t-Plot Micropore:    Yes
BJH Cum. Adsorption:    Yes
BJH Cum. Desorption:    No
D-H Cum. Adsorption:    No
D-H Cum. Desorption:    No

Pore Size

Average pore diameter:    Yes
BJH adsorption avg.:    Yes
BJH desorption avg.:    No
D-H adsorption avg.:    No
```

```
   D-H desorption avg.:    No

           Other

         Freundlich:    No
             Temkin:    No
     Alpha-S method:    No
      DFT Pore Size:    No
DFT Surface Energy:    No
 Nanoparticle Size:    No

      Horvath-Kawazoe

Maximum pore volume:    No
  Median pore width:    No

     Dubinin-Radushkevich

Micropore surface area:    No
    Monolayer capacity:    No

         Dubinin-Astakhov

   Micropore surface area:    No
Limiting micropore volume:    No

              MP-Method

       Cumulative surface area:    No
        Cumulative pore volume:    No
Average pore hydraulic radius:    No
```

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D O-RING COMPATIBILITY

Viton or Buna-N may also be suitable for analyses similar to the reference material example file. The ability to re-use Buna-N or Viton O-rings may be limited, while the re-use of Kalrez O-rings should be more broad. Frequency of use and, potentially, several other factors affect the duration of O-ring use, so rigid rules cannot be specified for these materials. Leak rate and ultimate vacuum levels may be used as indicators for O-ring performance.

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E WORKSHEETS

Worksheets in this section may be copied as needed.

Sample Data Worksheet for Gas Adsorption on the next page

Sample Data Worksheet for Gas Adsorption

Sample tube identification: _____

Sample Mass (g)				
		Before Degas	After Degas	After Analysis
1.	Mass of empty sample tube set	g		
2.	Mass of sample tube set plus sample	g	g	g
3.	Mass of sample (step 2 minus step 1)	g	g	g

Degas Information	
Degas apparatus	
Temperature (°C)	
Time (hours)	
Actual time started	
Actual time finished	

Degas Notes:

micromeritics®

EU DECLARATION OF CONFORMITY

This declaration of conformity is issued under the sole responsibility of the manufacturer:

Micromeritics Instrument Corporation
4356 Communications Drive
Norcross, GA 30093, USA

Hereby declares that the product:

Gemini™ Surface Area and Porosity Analyzer
Models Gemini VII 2390a, Gemini VII 2390p and Gemini VII 2390t

is in conformity with the following **EU harmonization legislation**:

2014/35/EU - LVD Directive
2014/30/EU - EMC Directive
2011/65/EU - RoHS Directive

and that the equipment is in conformity with the following harmonized and other appropriate standards;

2014/35/EU (LVD)
EN 61010-1:2010/A1:2019 - *Safety requirements for electrical equipment for measurement, control, and laboratory use — Part 1: General requirements.*
EN 61010-2-081:2020 - *Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes*

2014/30/EU (EMC)
EN 61326-1:2013 - *Electrical equipment for measurement, control and laboratory use — EMC requirements — Part 1: General requirements*
EN 61000-3-2:2014 - *Part 3-2: Limits — Limits for harmonic current emissions (equipment input current ≤ 16 A per phase)*
EN 61000-3-3:2014 - *Part 3-3: Limits — Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current <= 16 A per phase and not subject to conditional connection*

2011/65/EU (RoHS)
EN 63000:2018 - *Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances*

Name: John McCaffrey, Ph.D.

Title: Vice President, R & D

Signature:

Date of issue: 04/30/2021

Location: Norcross, GA USA

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