FLEX™ SERIES

SURFACE AND CATALYST CHARACTERIZATION ANALYZER

micromeritics®

OPERATOR MANUAL

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(Rev U)

micromeritics®

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2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.
3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

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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, 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 and 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)
- Operator Manual (PDF)
- Parts and Accessories
- Smart VacPrep Operator Manual (PDF)
- Vacuum Pump Guide (PDF)

All references to 3500, Flex, or Flex Series in this document encompass the Flex, 3Flex, and Flex 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*]. This document can be found on the Micromeritics web page (www.Micromeritics.com).

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

Common Icons

Icon	Refers to
C	Chemisorption and Dynamic Analysis
P	Physisorption only
DA	Dynamic Analysis
SC	Chemisorption only

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).

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

GENERAL SAFETY

Do not modify this instrument without the authorization of Micromeritics Service Personnel.

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 Flex weighs approximately 103 - 106 kg (227 - 234 lb) depending on configuration.
- 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 Flex 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 to 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

Micromeritics Flex Surface Characterization Analyzer is a fully automated one, two or three station instrument capable of high-performance physisorption and chemisorption analyses with superior accuracy, resolution, and data reduction.

The instrument is intended to be operated by trained personnel familiar with the proper operation of the equipment recommended by the manufacturer and as well as relevant hazards involved and prevention methods. Other than what is described in this manual, all use 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 customer's responsibility 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 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.

SYMBOLS THAT MAY APPEAR ON YOUR INSTRUMENT

The following symbols or icons indicate safety precautions and/or supplemental information and may appear on your instrument:

Use extreme caution when working on the instrument where one of these symbols may be displayed. These symbols indicate the part may be hot and cause serious burns.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

When working on the instrument where this symbol is displayed, refer to your Micromeritics' instruction manual for additional information.

When this symbol is displayed, toxic or flammable gases require proper venting of exhaust.

This symbol can also indicate the instrument uses mercury which is an extremely toxic substance. Read the Material Safety Data Sheet (MSDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident

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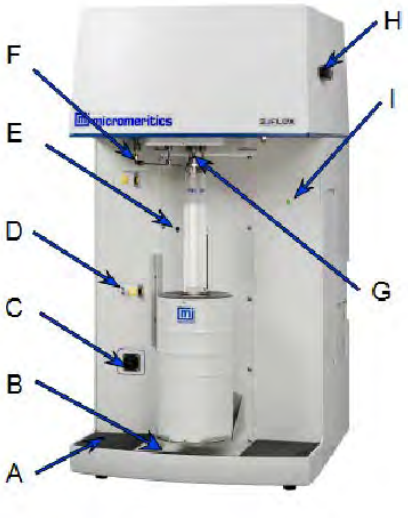
1 Analyzer Components

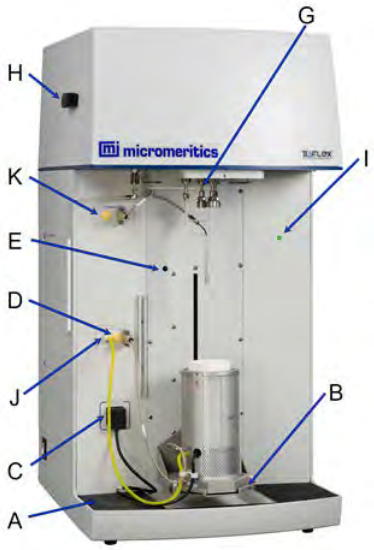
CFR Note	Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

Parts and accessories are located on the Micromeritics web page.

Front Components



Physisorption



Chemisorption

Cold Trap option

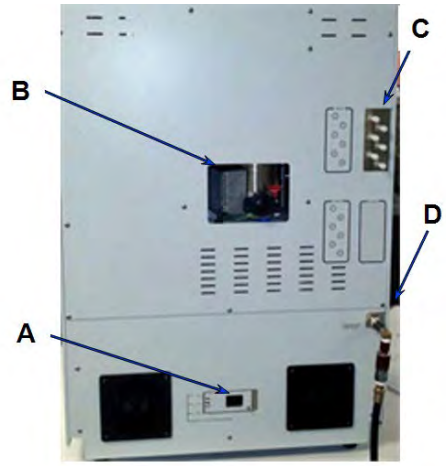
Front Components

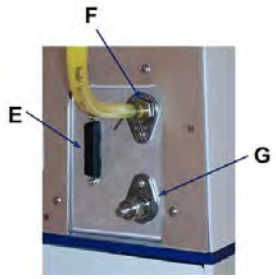
Component		Description
A	Access panels to reset buttons	Lift the pad and panel to access the reset buttons for the mantle, heater, and transformer.
B	Elevator	The elevator raises and lowers automatically when the analysis is started and completed. During analysis, the elevator *optionally* lowers after the free space measurement to allow evacuation, then raises and continues the analysis.
C	Mantle / Furnace power connector	Power connector for the mantle or furnace.
D	Mantle / Furnace thermocouple connector	Connector for the thermocouple.

Front Components (continued)

Component		Description
E	Elevator reset button	Resets the elevator in case of failure. The message *Elevator Circuit Breaker Open* on the analyzer schematic indicates this reset button should be used.
F	P_0 tube	For measuring the saturation pressure.
G	Sample ports	For installing up to three sample tubes.
H	Manifold compartment cover latch	Holds the removable manifold compartment cover.
I	Power indicator light	Blinks when power is applied to the analyzer; illuminates when the analysis program is initiated and ready for operation.
J	Cooling gas port	Forces air into the furnace to cool it down.
K	Thermocouple	For chemical adsorption samples.
	Cold Trap option	Attaches to the left side of the analyzer and removes water of reaction or other reaction products that might interfere with the operation of the thermal conductivity detector or to remove impure vapors in the analysis gas stream, especially water vapor.

BACK AND SIDE COMPONENTS





Chemisorption units only

A. Power cord connector
B. Vacuum pump foreline connector
C. Gas inlet valves
D. Compressed air connector
E. Mass Spec external trigger connector
F. Exhaust port
G. Mass Spec port

Back and Side Components

Components	Description
Compressed air connector	For compressed air supply for the pneumatically actuated, hard seal valves.
Ethernet port *(not shown)*	Located on the side panel. Port for a shielded Ethernet cable allowing communication between the analyzer and the computer.
Exhaust port C	The top port is to vent flowing chemisorption gas. The bottom port is used for the optional mass spectrometer.
Gas inlet valves	Inlet valves 1-6 for analysis gases.
Mass spectrometer external trigger signal C	For connecting the external trigger signal for the optional mass spectrometer.
Mass spectrometer exhaust port C	For connecting the exhaust line for the optional mass spectrometer.
Power switch *(not shown)*	Located on the side panel. For powering the analyzer on and off.
Power cord connector	For setting the power voltage and connecting the analyzer to the power supply.
RS-232 port *(not shown)*	Located on the side panel. connect the Smart VacPrep.
Vacuum pump foreline connector	For attaching the dry diaphragm roughing vacuum pump hose.

EQUIPMENT OPTIONS AND UPGRADES

Parts and accessories are located on the Micromeritics web page.

Option	Description
Chemisorption	Includes a Mass Flow Controller for precise flowing preparation of samples, a furnace to control sample temperature from ambient to 1100 °C, quartz flow-through sample tubes, an exhaust port for venting hazardous gases, a mass spectrometer port and electronic trigger signal, and six additional gas inlets for a total of twelve. The single chemisorption port is port number 2. The micropore option is required for the chemisorption port.
Chiller Dewar	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 is located on the Micromeritics web page.
CryoStat	A closed-cycle CryoCooler based on the Gifford-McMahon (GM) refrigeration principle. It uses helium gas from a helium compressor to generate cryogenic temperatures. The cryostat eliminates the need for liquid nitrogen and can obtain temperatures below the 77 K of liquid nitrogen. The decibel rating of the CryoStat HC-4A Zephyr is 56 dBA at 1 meter.
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 the sample material. The FlowPrep is an independent unit and not controlled by the analyzer.
Micropore Option	Each port on the standard analyzer can be upgraded for high quality micropore analyses with 10 torr and 0.1 torr transducers on each micropore port. Any remaining ports continue to operate in standard mode. The micropore option is required to run krypton.
MKS Cirrus 3	Connects directly to a dedicated port for detection and identification of low concentrations of condensable and/or reactive gases.
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

Option	Description
	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.
Thermal Conductivity Detector (TCD)	With the TCD option, dynamic analyses provide the ability to perform temperature programmed reduction (TPR), oxidation (TPO), desorption (TPD), and reactions (TPRx). Provides the capability to investigate temperature dependence of specific adsorption or desorption process profiles for catalyst and adsorbents, as well as pulse chemisorption.
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 the 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.
Vacuum Pump	The analyzer requires a dry roughing vacuum pump for sample analysis. Appropriate vacuum pumps are available from Micromeritics.
Vapor Adsorption Option	Vapor adsorption allows analyses with hydrocarbon vapors or water vapor. The analyzer allows for dosing from one dedicated sample port to the other two sample ports. A vapor adsorption option provides for dosing from a reservoir attached to the Psat port to all three sample ports. The vapor option includes a stainless steel chamber with a hard seal, manual cutoff valve to be attached in place of the Psat tube, and a heating mantle to control the temperature of the chamber at an operator-specified temperature between ambient and 43 °C.

Gas Requirements and Purity

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to 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 are located on the Micromeritics web page.

All analyzer types		Chemisorption units
(CGA 580) N_2 (CGA 580) He	99.999% 99.999%	All gases listed to the left and: (CGA 350) H_2 99.999% (CGA 350) CO 99.990% Pre-mix 10% CO in helium (required for Dynamic Chemisorption units and TCD units only)

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 to 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 FLEX

Pressure Measurement

Analysis	Physisorption	*Chemisorption*
Analysis Range	1.3×10^{-9} to 1.0 P/P_0	1×10^{-6} to 900 mmHg
Micropore Analysis	Standard on all ports	Standard on all ports
Dosing and Evacuation Control	Proprietary servo	Proprietary servo
Roughing Pump	4 stage diaphragm	4 stage diaphragm
Krypton Analysis	Standard on all ports	Standard on all ports

Analysis Gas Per Sample

One probe per gas port. 3Flex can use to up three gases concurrently.

Minimum Measurable Surface Area

Standard	0.01 m^2/g	0.01 m^2/g
Krypton	0.0005 m^2/g	0.0005 m^2/g

Instrument Control and Features

Temperature Control	45 ± 0.05 °C with 3 strategically placed RTDs	
Adsorptive Gas Inlets	6	12 standard; 16 optional
Vapor Sorption Option	Standard ports 1 and 2, optional heated	
Furnace	N/A	Programmable 0.1–50° C/min 50° C/min to 600° C 20° C/min from 600-800° C 10° C/min from 800-1100° C
Mass Flow Controller	N/A	Standard, flow up to 200 sccm, ±1% of set point

Instrument Control and Features (continued)

TCD with Cold Trap	N/A	Two optional levels of performance available
Standard External Dedicated Port	N/A	Heated port for connection of external detectors including an available Cirrus quadrapole mass spec
Degas	3 in situ for 3Flex 1 in situ for Flex Up to 4 Smart VacPreps can be attached to give a maximum of 24 additional degas ports	
A/D Data Acquisition	29 bit (effective 25)	
Pressure Transducer System	6 for Flex 12 for 3Flex	
Transducer Accuracy	1000 mmHg - 0.12% reading 10 mmHg - 2% reading 0.1mmHg - 0.15% reading	

Environment

Temperature	10 to 35 °C (50 to 95 °F), operating 0 to 50 °C (32 to 122 °F), non-operating
Humidity	20 to 80% relative, non-condensing
Indoor or outdoor use	Indoor only (not suitable for wet locations) Altitude: 2000 m max (6500 ft) Pollution degree of the intended environment: 2

Cryogen

Cryogen Dewar	3.2 L. Greater than 70 hours Virtually unlimited duration with refill during analysis
Cryogen Free Space Control	Isothermal jacket

Electrical

Voltage	100/120/230VAC
Frequency	50/60 Hz
Power	1350 VA
Overvoltage category	II

Physical

Height	112 cm (44 in.)
Width	57.2 cm (23 in.)
Depth	61 cm (24 in.)
Weight	103 - 106 kg (227 - 234 lb.) (depending on configuration)

Computer Requirements

Operating System	Windows 10 or higher operating system is required. 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. Ensure the "Sleep" setting on the desktop is set to "Never" to avoid interruption while running an analysis. If this occurs, the application loses network connectivity with the instrument and a communications error will be reported. A restart of the Windows application may be required if automatic reconnection is not successful.
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 application users 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 - 38

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in a 21CFR11 environment.

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 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.

In addition to customizable standard reports, user-defined calculations and reports can be created through the Advanced reports feature (using Python).

Data can be manipulated and displayed interactively using MicroActive reports.

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

Selections	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 change presentation options, set the method and active metals defaults, configure signal calibration, manage libraries, select units, and create report styles.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Use to access the embedded operator manual, Micromeritics web page, 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

Selections	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 to show all the data. 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 and displays a prompt to either accept or reject changes.
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 to display in the report header about the sample or analysis.
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.

Common Fields and Buttons (continued)

Selections	Description
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	Exits the application. If a file is open with unsaved changes, a prompt displays the option to save the changes and exit or 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 spread-sheet (.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	Indicates the *From* and *To* range for x- and/or y-axes when working with report parameters windows.
Insert	Inserts one row above the selected row in the table.
List	Creates a list of samples or other types of files. The list will contain the 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.

Common Fields and Buttons (continued)

Selections	Description
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.
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. A portion can be saved as a separate, stand-alone file, such as Analysis Conditions or Report Options, when saving sample information.
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. Enabled only in Service Test Mode. **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 the 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 used for analysis.
Complete	Sample files used in an analysis that is completed.
Entered	Sample files containing manually entered data.
No Analysis	Sample files that 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
Sample Tube Properties	.STB
SPC Report	.SPC
Thickness Curve[2])	.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 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.
2) 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** to open 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

Selections	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)

Selections	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 the onscreen reports.

OPTION PRESENTATION

Options > Option Presentation

CFR Note

For 21CFR11 environments, see *Software in 21CFR11 Environments on page 2 - 38*.

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

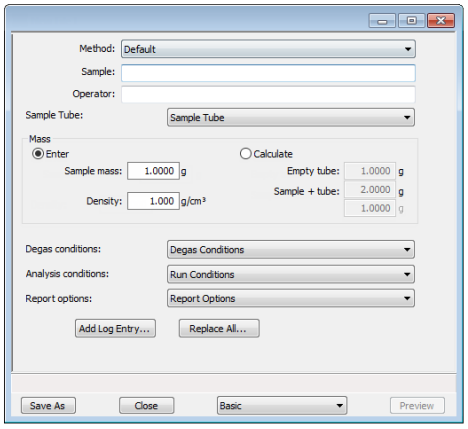
Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample file in a single window like 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.
Show Degas Conditions C	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.
Check Shield	When enabled, checks to ensure the shield is in place around the Dewar or furnace prior to starting an analysis. If this option is selected and the Dewar or furnace shield is not in place prior to starting an analysis, a warning message displays on the analyzer schematic window. An entry is made in the analyzer log regardless of operator choice.
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



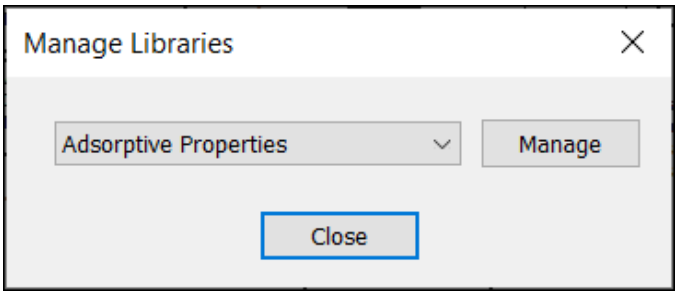
Basic or Restricted view

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 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, and provides 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

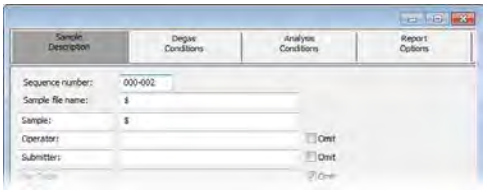
Options > Default Method
File > Open > [.MTH File]

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.

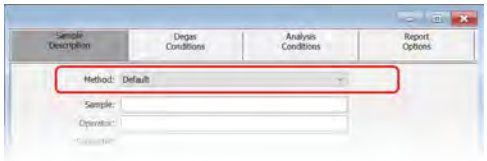
A *Method* determines the default sample identification format and sequence number. A *Method* is a template of specifications that go into a newly created sample file. It allows for the definition of complete sets of parameters for each type of sample commonly analyzed. Only a single selection is required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

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

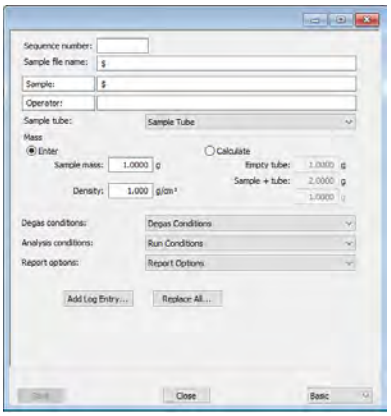
Developer Group view in a 21CFR environment



Default Method Examples



Default Method Examples



Analyst Group view in a 21CFR environment

Default Methods

Selections	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 use as a prefix in the *Sample* field when a new sample file is created. This number increments with each sample file created.

Default Method Files

Method Selected	Default File Modified
Physisorption	3500.SMP
Chemisorption*	3500Chemi.SMP
Dynamic Analysis *	3500DynamicChemi.SMP

* Not applicable in a 21CFR11 environment.

CONFIGURE THE ANALYZER

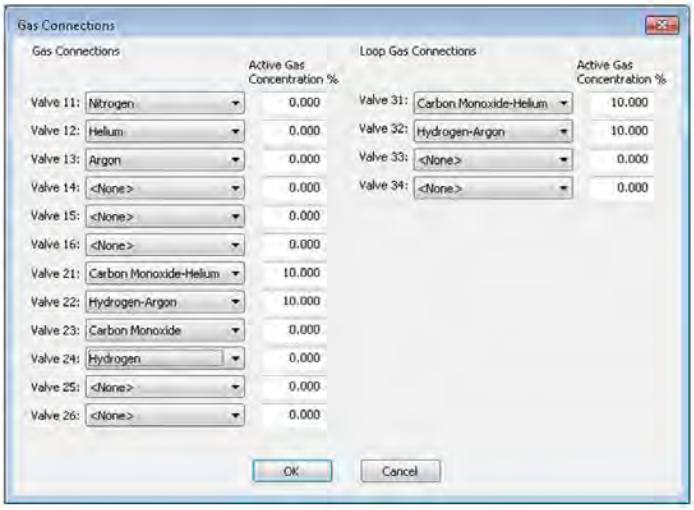
SPECIFY GAS PORTS

Unit [n] > Unit Configuration

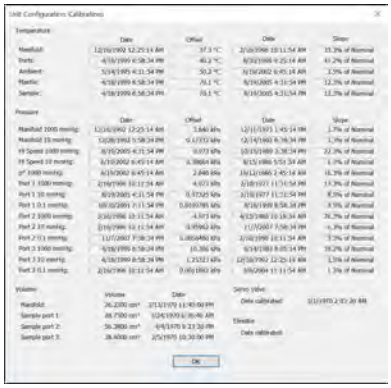
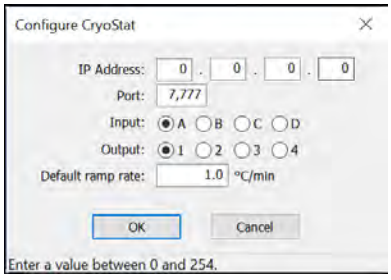
Use to display hardware / software configurations, calibrations, and gas selections of the connected analyzer.

The analyzer has gas inlets for up to six analysis gases (12 with the chemisorption option, plus four loop gas inlets with the TCD Loop option). The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window. The analysis software must be kept informed of any changes in gases.

Enter the gas mnemonic in the gas fields to indicate the type of gas connected to that valve.



Unit Configuration

Selections	Description
Calibrations [*button*]	Displays calibration information for analyzer components. 
Configuration [*group box*]	Displays the IP address used by the analysis program, serial number, and type of analyzer. **IP address.** Displays the IP address of the analyzer. **Change IP.** Displays the Board ID dialog, which describes the circuit boards in the analyzer. Use the Board drop-down list to select a board to view. **Board ID.** Click to display information from the circuit boards in the analyzer. Use the drop-down list to select a board to view. The parameters shown cannot be edited.
CryoStat [*button*]	 **IP Address.** The IP address of the cryostat temperature controller. **Port.** The computer port to which the cryostat temperature controller is attached. **Input.** Temperature sensor input channel. **Output.** Heater output channel. **Default ramp rate.**The rate at which the temperature will change

Unit Configuration (continued)

Selections	Description
	while advancing to the hold temperature.
Gas Selections [*group box*]	Enter the mnemonics for the analysis gases attached to inlet valves. Valves 11-16. PC Valves 21-26. C With chemisorption option. Valves 31-24. DA With loop configuration.
Loop Volume [*text box*] DA	Enter the loop volume for TCD.
Options [*group box*]	Displays options installed on the analyzer.
Software Versions [*group box*]	Displays the software versions of the MIC BIOS, controller, and analysis program.

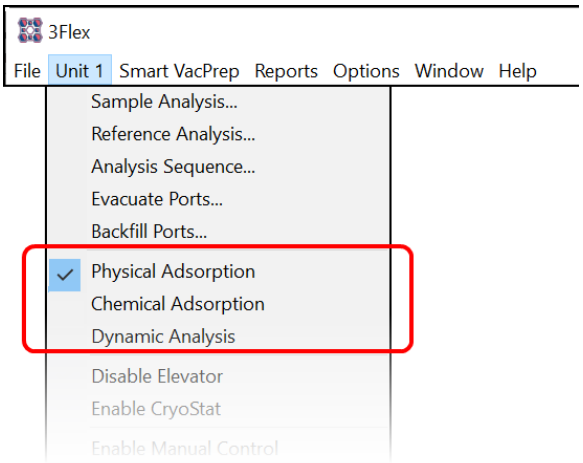
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

SPECIFY THE TYPE OF ANALYZER

Unit [n] > Physical Adsorption
Unit [n] > Chemical Adsorption
Unit [n] > Dynamic Analysis

This topic does not apply if physisorption units only are installed.

Up to four analyzers can be connected, and each analyzer may be either physisorption or chemisorption. When multiple units are installed, the application's title bar reflects the number of analyzers installed and the type of analyzer.



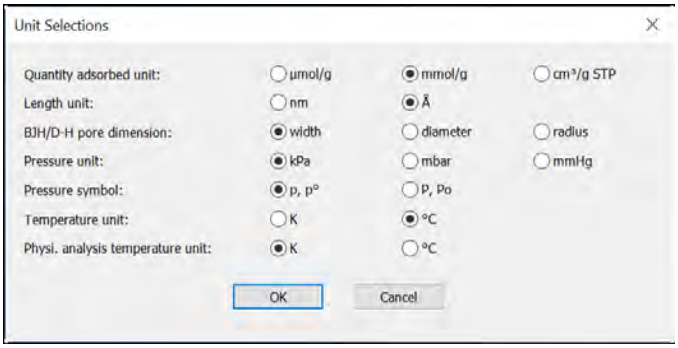
The analysis type affects the appearance of the analysis sample selection window, the analyzer schematic, and the status window. This menu selection is disabled when an analysis is being performed.

When application windows are opened, the title bar reflects both the serial number and the analysis type. The title bar changes when the analysis type changes.

UNIT SELECTION

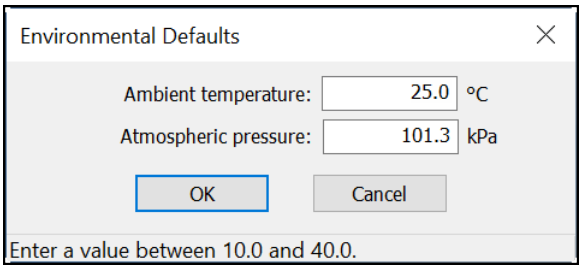
Options > Units

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.



Environmental Defaults for TCD Analyzers

Options > Environmental Defaults



These values are used in some calculations to account for the effect of environmental conditions on the analysis. These parameters are used in the Dynamic Analysis reports. To ensure accurate loop calibration, enter current environmental conditions daily, or whenever there is a significant change in one or more of the environmental conditions.

Saturation pressure (P_o) of the adsorbate used by BET analysis is obtained from the adsorptive properties for the experiment gas specified.

Environmental Defaults

Selections	Description
Ambient temperature [*text box*]	The temperature of the room where the analyzer is located.
Atmospheric pressure [*text box*]	The atmospheric pressure of the room where the analyzer is located.

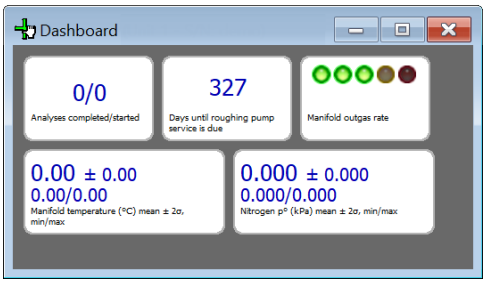
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

INSTRUMENT STATUS

SHOW DASHBOARD

Unit [n] > Show Dashboard

Data for the dashboard comes from the logged diagnostic data. The dashboard remains current as the relevant diagnostic data items are updated. The gauges will be updated even if the dashboard window is not open.



Red numbers on the dashboard require attention. To reset the dashboard numbers, right-click on the dashboard setting, then click **Reset**.

Dashboard Gauges

Selections	Description
Analyses completed / started	Displays *N/M* where *N* is the number of analyses that have finished data collection and *M* is the number of analyses that have been started. Analyses canceled or terminated by errors before the termination stage starts are not counted as completed.
Days until roughing-pump service is due	Annual maintenance is recommended. The number of days until the anniversary of the last pump maintenance is shown. The displayed value is updated at least once per day and when the maintenance time is reset. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.

Dashboard Gauges (continued)

Selections	Description
Manifold outgas rate	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate. The gauge is updated when the *Analysis Manifold Test* is run. See *Schedule Diagnostic Tests on page 9 - 3* and *Start Diagnostic Test on page 9 - 1*. ■ Three green LEDs are lit if outgas rate is below 30% of outgas rate limit. ■ At 30%, the left LED turns off. ■ At 60%, the center LED turns off. ■ At 90%, three green LED lights turn off and the center yellow LED turns on. ■ At 110% and above, only the red LED turns on and attention is required.
Manifold temperature	Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.
Nitrogen P_0	Displays statistics of the saturation pressures measured with nitrogen gas at liquid nitrogen temperatures. The mean, two-sigma, minimum, and maximum values display. The gauge is updated when a P_0 is logged with nitrogen as the adsorptive and a bath temperature of 77±2 K.

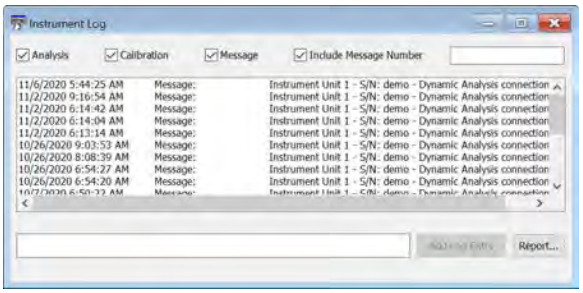
SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

CFR Note

In 21CFR11 environments, see *Software in 21CFR11 Environments on page 2 - 38*.

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



Instrument Log

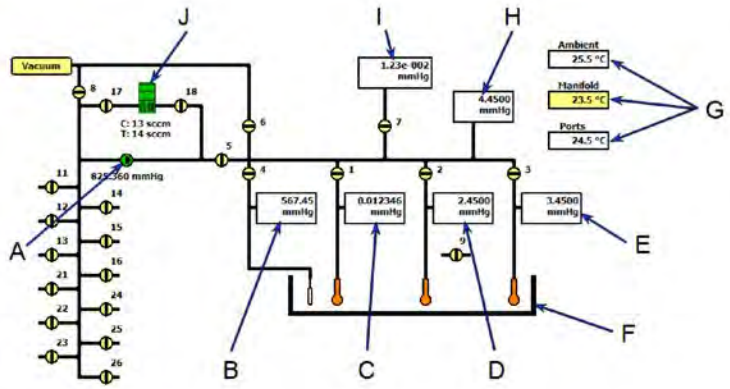
Selections	Description
Add Log Entry [*button*]	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Analysis/ Calibration/ Message [*check box*]	Select the logs to display.
Include Message Number [*text box*]	Enter any of the following information in the message text box to generate a log report that includes the following: ■ Message number in the text box to view all occurrences of the entered message, ■ Asterisk in the message box to see all numbered messages, or ■ Several message numbers separated by commas to include only messages with those numbers. Numbered messages contain more detailed information about analyzer operation.
Report [*button*]	Click to select the print destination and the report start date.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3***.**

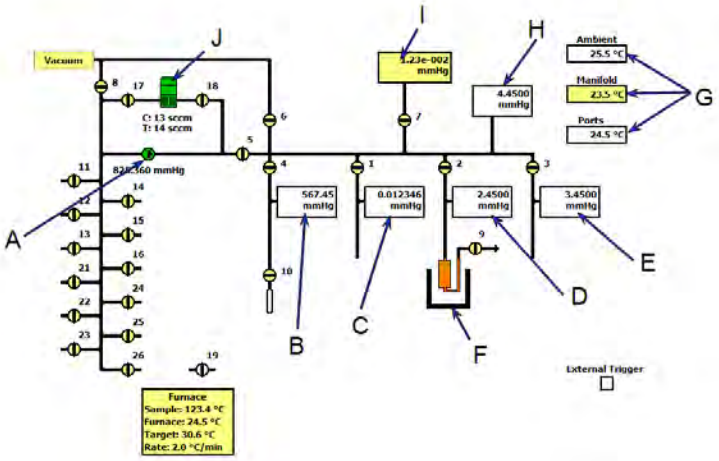
SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic

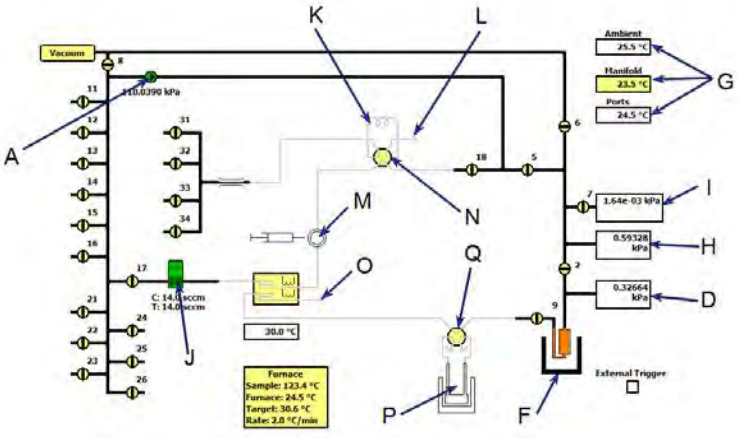
Use to display an analyzer schematic. To operate the valves and elevator from this window, manual control must be enabled (***Unit [n] > Enable Manual Control***).



Physisorption



Chemisorption



Dynamic Analysis with Loop option

A. Servo valve
B. P_0 pressure
C. Port 1 pressure
D. Port 2 pressure
E. Port 3 pressure
F. Elevator
G. Temperature sensors
H. Manifold pressure
I. Vacuum gauge
J. Mass flow controller C
K. Injection loop DA *
L. Loop gas exhaust port DA *
M. Septum DA *
N. Loop rotary valve DA *
O. Chemical exhaust port DA
P. Cold trap
Q. Cold trap rotary valve

* Displays only if TCD Loop Valve option is installed.

Analyzer Schematic Components

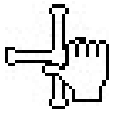
Schematic Components	Description
1-3	Sample ports
4	Po port
5	Servo isolation valve
6	Manifold vacuum
7	Vacuum gauge isolation valve
8	Inlet vacuum
9	Exhaust valve for chemisorption only
10	Reference volume (shown in Service Test mode only)
11-16 and 21-26	Inlet valves
31-34	DA Loop gas inlet valves (displays only if the TCD Loop Valve option is installed)

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.
	Elevator.
	C **Sample Tube** and **Furnace Elevator.** The sample tube icon is white when the sample and furnace temperatures are 50 °C or lower. If either the sample or furnace temperature exceeds 50 °C, the sample tube icon turns orange. Temperature readings and ramp rate are displayed below and to the left of the icon. The furnace icon resides on the elevator.

Icon or Symbol	Description
	C Mass Flow Controller (MFC). Controls the flow of gas into the sample port. The current (C) rate and the target (T) rate are shown to the right of the MFC icon. Applicable only for the gas used in the flowing operations. The mass flow controller constant is preset for gases provided with the application. See *Chemisorption Tasks on page 4 - 13*.
	Sample Tube. Cannot be manually controlled.
	P SC A warning that indicates the safety shield is not in place.
	Vapor Source with Heating Mantle. Also displays the current temperature, the target vapor source temperature, and rate of temperature increase.

Instrument Schematic Shortcut Menus

Schematic Shortcuts

Icon or Symbol	Description
Valve options 	**Automatic.** Automatically operates the servo valve during dosing or evacuation. Enter the target pressure. **Close.** Closes the selected valve. **Direct.** Used in Service Test mode only under the direction of a Micromeritics Service Representative. **Open.** Opens the selected valve. **Pulse.** Use to quickly turn the valve on and off allowing the operation to proceed in small increments.
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.

Schematic Shortcuts (continued)

Icon or Symbol	Description
Temperature control options	**On.** Enables the temperature control. **Off.** Disables the temperature control. **Set.** Select to set the following: ■ Enable or disable temperature control ■ Control sample temperature ■ Control furnace temperature ■ Cool the sample to less than 50 °C ■ Set heater power percent

PRESSURE TRANSDUCERS

The schematic shows the pressure transducers present in the system.

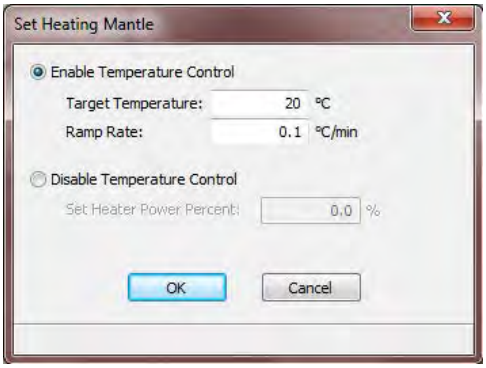
- All systems have 1000 mmHg pressure transducers on the manifold and the analysis ports.
- Optional 10 mmHg pressure transducers on the manifold and one or more analysis ports may be present.
- Optional 0.1 mmHg pressure transducers on one or more analysis ports may be present.

When multiple pressure transducers are present in a port or the manifold, the display automatically changes to show the pressure reading from the lowest range transducer currently on the scale.

HEATING MANTLE

When samples are being degassed on the analysis ports, the following icons appear beneath the elevator. When the mantle temperature is above 50 °C, the sample tube icons (not shown below) turn orange. If using a GlasCol heating mantle, the sample temperature shows in the yellow *Degas Mantle* box.

Right-click the *Degas Mantle* box to set the target temperature or the rate of temperature increase.

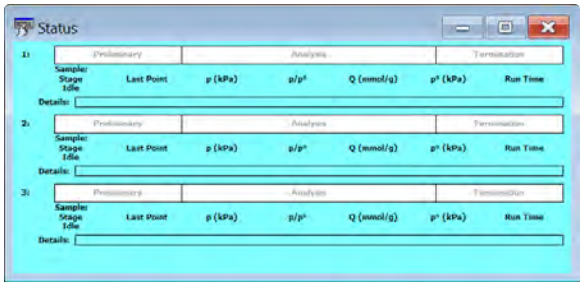


Select *Enable Temperature Control*, then enter both the *Target Temperature* and *Ramp Rate*. The *Set Heater Power Percent* field (shown when *Disable Temperature Control* is selected) is enabled in Service Test Mode only.

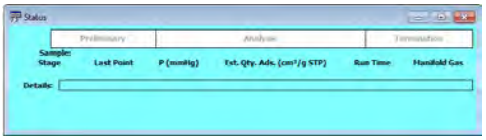
SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



Physisorption



Chemisorption

Dynamic Analysis

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

ANALYSIS TYPES FOR TCD ANALYZERS

The basic concept for all analyses is the same: the filament detects changes in the gas mixture flowing past it. The sample, gas selection, and analysis conditions determine what changes occur.

TEMPERATURE PROGRAMMED DESORPTION ANALYSIS

Temperature Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows through the sample and adsorbs on the active sites. Programmed desorption begins by raising the temperature linearly with time while a steady stream of inert carrier gas flows through the sample.

At a certain temperature, the heat overcomes the activation energy; therefore, the bond between the adsorbate and adsorbent will break, and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperatures. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which detects changes in gas composition. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

Temperature Programmed Desorption	**Temperature Programmed Decomposition**
■ Heated pretreatment ■ Active sites to probe ■ Active gas to probe sites (NH_3, CO_2, H_2, etc.) ■ Material (sample) does not change during temperature ramp	■ No pretreatment ■ No active sites to probe ■ Only inert gas is used ■ Material changes throughout temperature ramp
Observe changes of gas phase species during temperature ramp.	

TEMPERATURE PROGRAMMED REDUCTION ANALYSIS

Temperature Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperatures at which the reduction occurs.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) through the sample, usually at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

When using any mixture of gases for TPR or TPO analyses, ensure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity. See ***Gas Charts on page I - 1*** .

TEMPERATURE PROGRAMMED OXIDATION ANALYSIS

Temperature Programmed Oxidation (TPO) examines the extent to which a catalyst can be oxidized or was previously reduced.

Usually the sample is pretreated and the metal oxides are reduced to the base metal, typically with a gas mixture of hydrogen with either nitrogen or argon. Then the oxidizing gas, typically 2-5% oxygen with helium, is applied to the sample in pulses or, alternatively, as a steady stream.

The furnace heats the sample tube and sample according to the user-selected temperature program. The oxidation reaction occurs at a specific temperature. The analyzer measures the uptake of oxygen.

When using any mixture of gases for TPR or TPO analyses, ensure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity. See ***Gas Charts on page I - 1***.

PULSE CHEMISORPTION ANALYSIS

A Pulse chemisorption analysis determines the amount of gas irreversibly adsorbed and subsequently calculates active surface area, percent metal dispersion, and the average active particle size by applying measured doses of reactant gas to the sample.

The active gas interacts with each active site until all active sites are covered. Once the active sites have been completely covered, the discretely injected gas volumes elute from the sample tube unchanged. The amount chemisorbed is the difference between the total amount of active gas injected and the amount that eluted from the system. The quantity of each pulse of reactant gas is determined by the loop volume on an automatically controlled valve. A 0.5 cm^3 loop is provided with the analyzer and an optional 1 cm^3 loop is available.

ADDITIONAL USES OF THE TCD ANALYZER

The analyzer may also be used for temperature programmed reactions, catalyst pretreatment, and isothermal reactions. The tremendous flexibility of the analyzer allows the use of custom applications.

Temperature Programmed Reaction

A temperature programmed reaction monitors the products from the reaction between gases and a catalyst at a specified temperature. The analyzer can be programmed to raise the temperature of a catalyst bed at a constant ramping rate as the gases flow through the catalyst. At the optimal temperature, the gases react in the presence of the catalyst, creating products. The products of the reaction and excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

Catalyst Pretreatment

Catalyst pretreatment usually consists of activating a catalyst before its use in a chemical reaction. For example, a temperature programmed oxidation reaction may require reduction of the catalyst under a flow of H_2 at a specific temperature.

Isothermal Reaction

An isothermal reaction is similar to a temperature programmed reaction except that the catalyst is kept at a constant temperature (isothermal) to perform the catalytic reaction. Both the product of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

APPLICATIONS

Catalytic processes that benefit from TPD/TPR analyses include:

- Polymerization
- Hydrogenation
- Catalyst cracking
- Hydrocracking
- Isomerization
- Oxidation
- Dehydrogenation
- Hydrotreating
- Alkylation reforming

PEAK EDITOR FOR DYNAMIC ANALYSIS

Peak Editor for Dynamic Analysis on page 7 - 10

The TCD (Thermal Conductivity Detector) features a Peak Editor, which allows the evaluation of results, peak editing, and reports. Adjusting peak boundaries can be used to eliminate baseline noise or other undesirable effects. The Peak Editor also allows the separation of composite peaks.

EXPORT FILES

File > Export

Exported Data Example on page G - 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. The data can be imported into other applications that read these file formats when exported to a file.

1. Click ***List*** and open an .SMP file.
2. Select an experiment and the applicable options.
3. Click **OK**.

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 UNINSTALL

Uninstall Software in 21CFR11 Environments on page 2 - 43

The software can be uninstalled in two ways. Either method removes only the files required to run the software, not the 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 UPDATES

A User Account Control in the Windows operating system must be enabled to ensure all components of the Micromeritics application are correctly installed. 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.

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 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

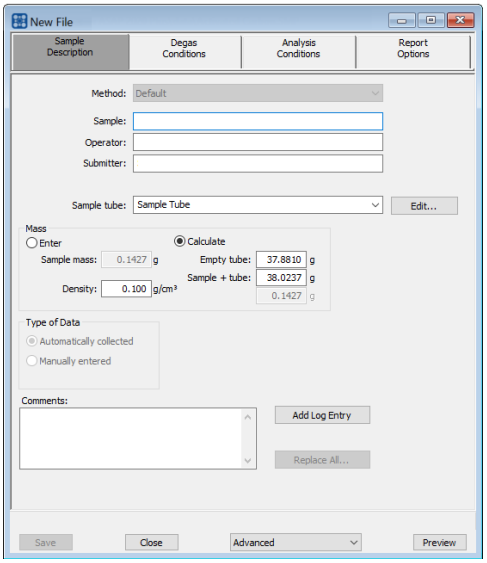
Options > Option Presentation

Option Presentation Display

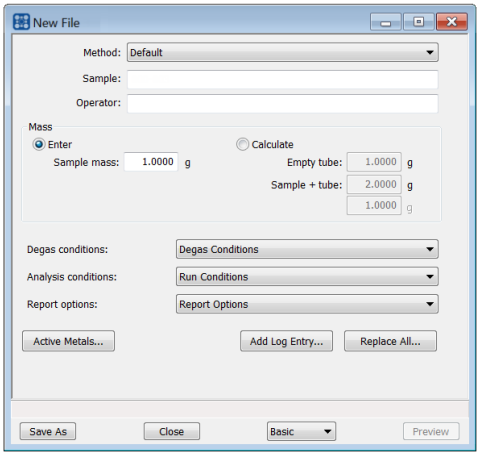
Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Show 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.
Check Shield	When enabled, checks to ensure the shield is in place around the dewar or furnace prior to starting an analysis. If this option is selected and the dewar or furnace shield is not in place prior to starting an analysis, a warning message displays on the analyzer schematic window. An entry is made in the analyzer log regardless of operator choice.
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.

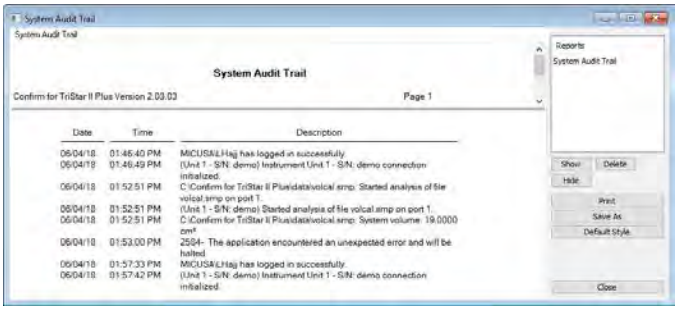
CREATE A NEW FOLDER

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.

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3 SAMPLE FILES

Option Presentation on page 2 - 10
Software in 21CFR11 Environments on page 2 - 38

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

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

A sample file consists of parameter sets; however, parameter sets can also stand alone. A sample file may be created either before 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 P

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.

Analysis condition defaults for metal stoichiometry factors can be set using the sample information metal table editor. Stoichiometry factors can also be set for each pulse chemisorption experiment using the analysis conditions experiment step editor. If required, stoichiometry factors for a completed pulse chemisorption experiment can be viewed and modified using the peak editor's stoichiometry settings window.

For dynamic analyses, the *Loop Volume* and *Environmental Default* values must be correct before starting an analysis.

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.

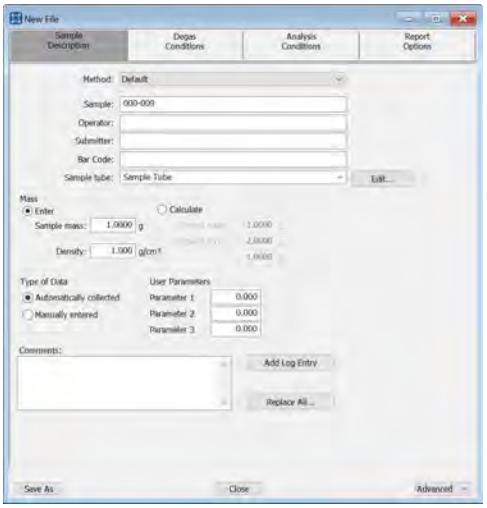
Specify or change the option presentation by selecting ***Options > Option Presentation*** or use the view selector drop-down list at the bottom of the window.

Sample files created in the *Basic* option presentation are 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.

Sample Tube parameters are edited on the *Sample Description* tab.

For physisorption, the *Degas Conditions* tab displays only if enabled in ***Options > Option Presentation > Show Degas Conditions***.

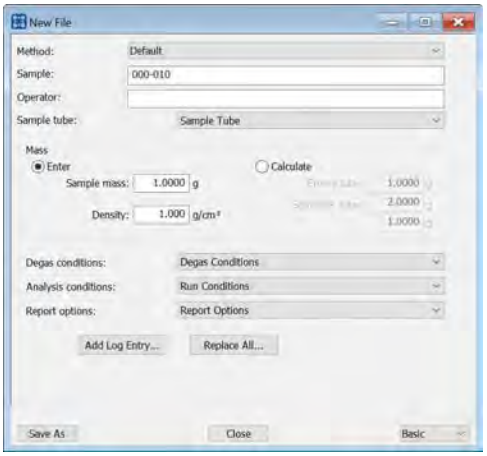
Advanced Option Presentation



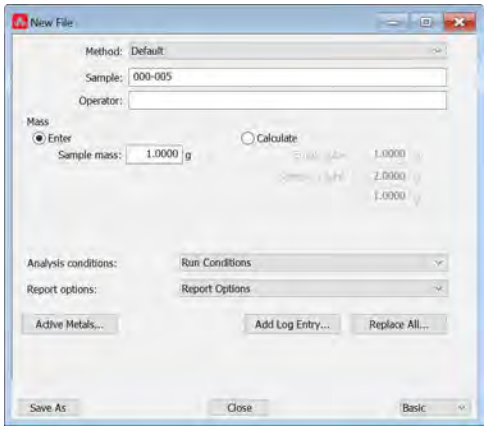
Physisorption

Chemisorption or Dynamic Analysis

Basic Option Presentation

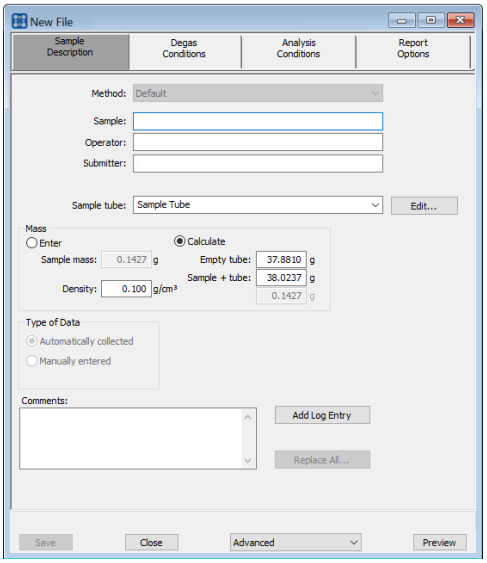


Physisorption

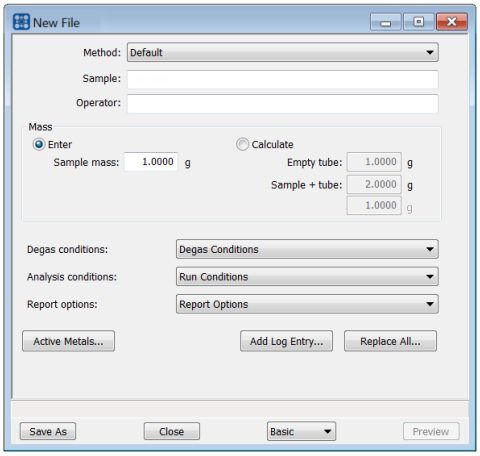


Chemisorption or Dynamic Analysis

File Editor Example for 21CFR environments



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

Selections	Description
Active Metals [*button*] C	Displays a list of active metals. See ***Active Metals for Chemisorption Analyzers on page 3 - 12***.
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 to display in the report header about the sample or analysis.
Mass [*group box*]	If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If an accurate mass is entered, the reported surface area is normalized per gram of sample. Choose whether to enter mass manually or have the system automatically calculate mass. 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.

Sample Files (continued)

Selections	Description
	Calculate. Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$ **Density.** P Value is used for the calculated free space method only. Use 0.000 for a blank analysis.
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.
Sample Tube [*drop-down box*] P	Select a sample tube file from the drop-down list, or click **Edit** to modify or create a new sample tube file.
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 - 8*.
User Parameters [*group box*] P *	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*.

For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 3*****.**

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

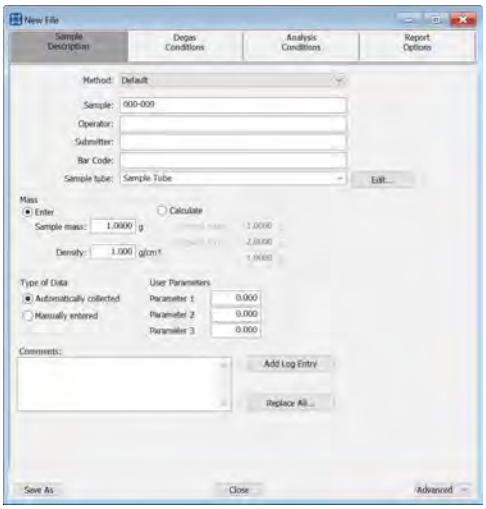
OPEN A SAMPLE FILE

File > Open > [.SMP File]

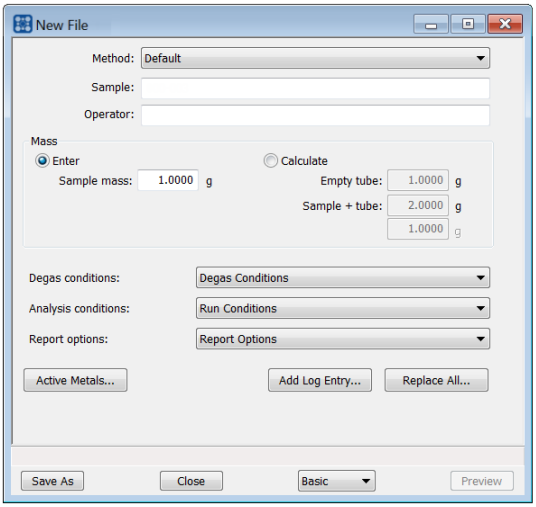
When working with an existing sample file, consider copying the sample file to maintain the original configuration options.

File Type	File Status	Displays
Physisorption or Chemisorption	Preparing Prepared No Analysis	Tabbed file editor
	Complete Analyzing Entered	MicroActive report window

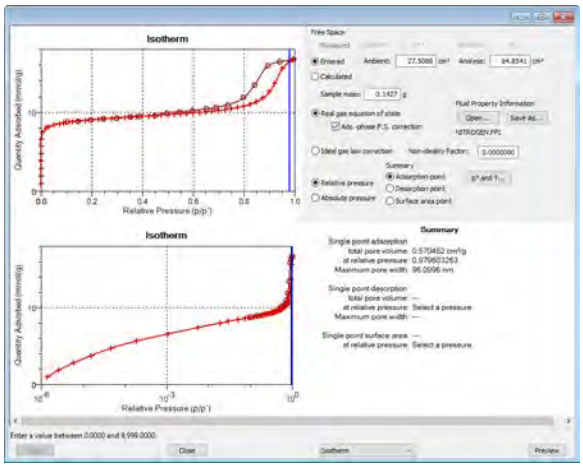
File Editor Example for CFR21



Advanced or Developer view



Basic or Analyst view



Example of a Report window

To view the tabbed file editor for a sample file with a *Complete* status, select *Advanced* 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.

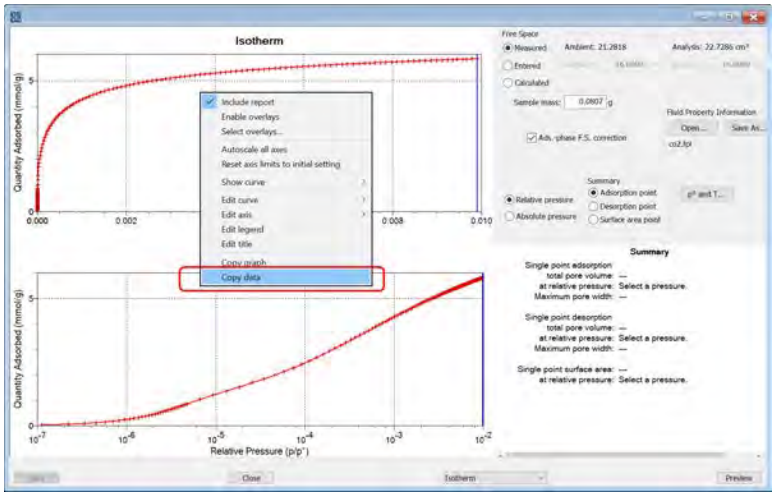
This process allows the manual entry of pressure data from a sample file with a *Complete* status. There are two methods for manually entering data into a sample file:

- 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

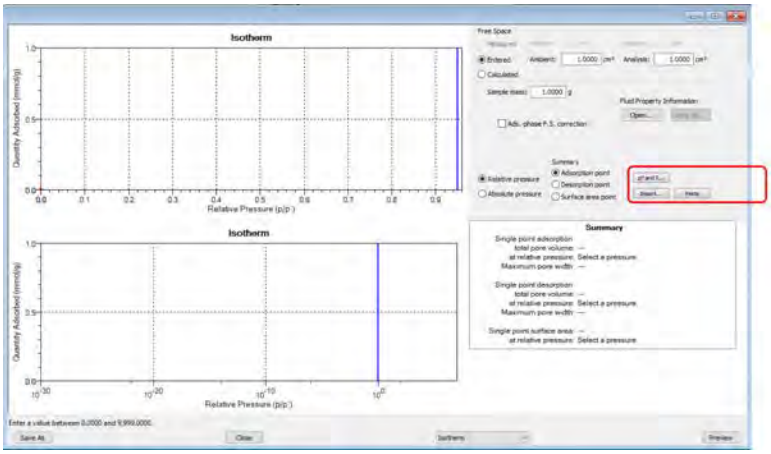
1. Open a sample file with a *Complete* status. The file will open in the interactive reports window.
2. Right-click in the graph area of the interactive reports window, then select *Copy data*.



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:

For Physisorption or Chemisorption:

- Relative Pressure
- Absolute Pressure (mmHg)
- Absolute Pressure (kPa)
- Absolute Pressure (mBar)
- Quantity Adsorbed (mmol/g)
- Quantity Adsorbed (cm^3/g STP)
- Quantity Adsorbed (cm^3/g STP)

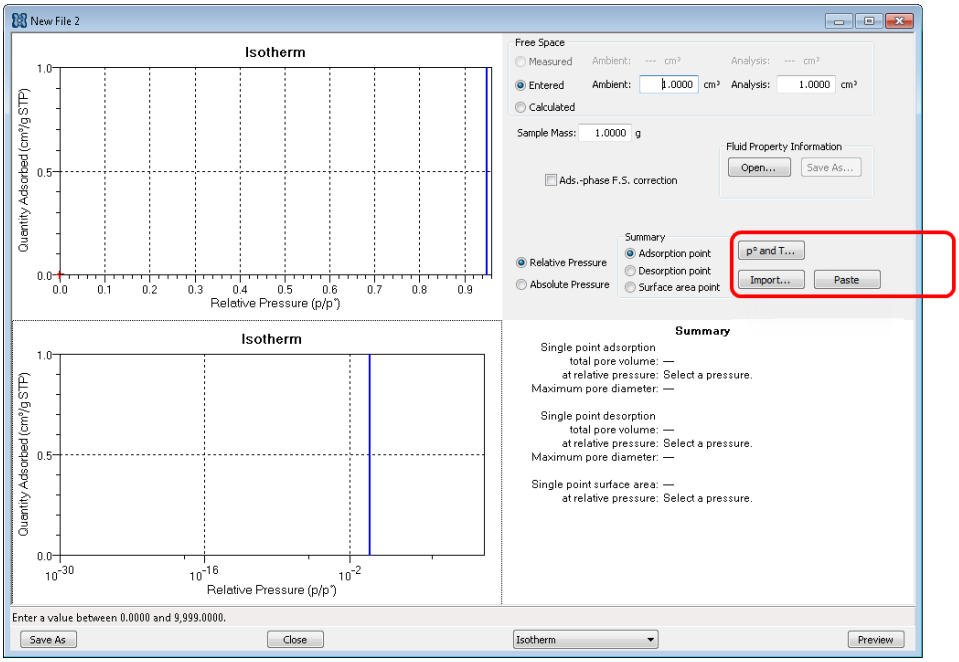
Sample Physisorption ASCII Text File

```
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.

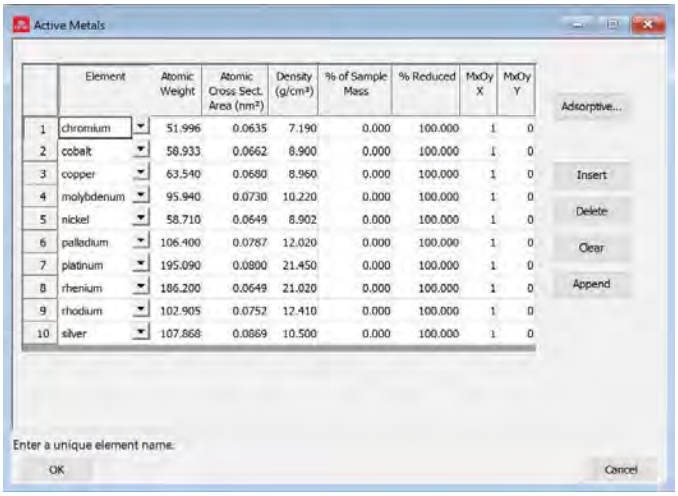
ACTIVE METALS FOR CHEMISORPTION ANALYZERS

Options > Active Metals Defaults

Or, click **Active Metals** on the *Sample Description* tab when using the *Advanced* presentation option.

Atomic Weights and Cross-Sectional Areas on page B - 1

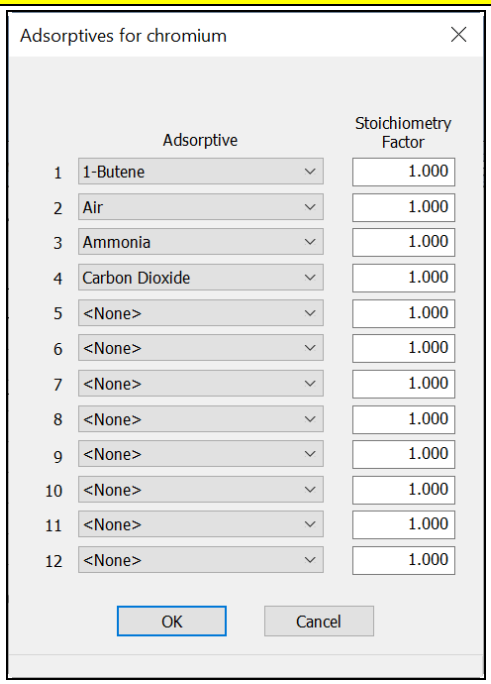
Up to 20 elements can be specified. At least one element must have a non-zero % of the sample weight.



Active Metals

Selections	Description
% of Sample Weight * [*column*]	Percentage, by weight, of each element contained in the sample.
% Reduced * [*column*]	The percent of metal reduced during preparation.
Adsorptive [*button*]	Click to display and modify both the adsorptives and their associated stoichiometry factor. Stoichiometry factors for each gas are metal-specific.

Active Metals (continued)

Selections	Description
	 **Stoichiometry Factor.** The stoichiometry factor is defined as the moles of metal species covered per mole of adsorptive.
Atomic Cross Sect. Area (nm^2) [*column*]	Atomic cross-sectional area of the element.
Atomic Weight [*column*]	Atomic weight of the element.
Density g/cm^3 [*column*]	Density of the element.
Element [*drop-down box*]	Select or enter the active metal.
MxOy, X * MxOy, Y * [*column*]	X and Y values specify the empirical formula for metal and oxygen, respectively, in a metal oxide.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

* Options are shown only when using the **Active Metals** button on the *Sample Description* tab.

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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.

CFR Note	Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

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 created.

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

Parameter File Types

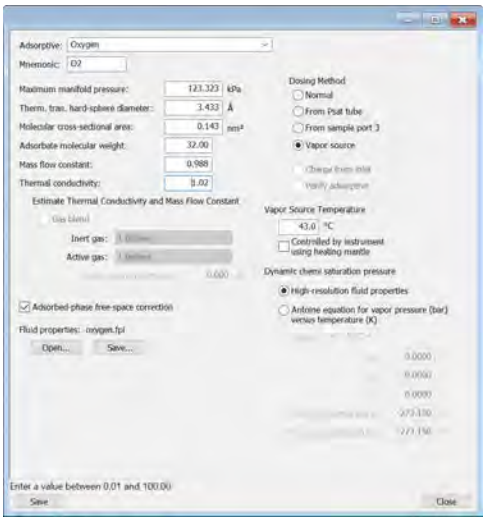
File Type	File Extension
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Degas Conditions P	.DEG
Method	.MTH
Report Options	.RPO
Sample Tube P	.STB

ADSORPTIVE PROPERTIES

File > Open > [.ADP File]

Or, for physisorption and chemisorption, click **Edit** next to the *Adsorptive* selection on the *Analysis Conditions* tab when in *Advanced* option presentation. For a dynamic analysis, there is no *Adsorptive Properties* tab; use ***File > Open > [.ADP file]***.

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



Adsorptive Properties

Selections	Description
Adsorbate molecular weight [*text box***]**	The molecular mass is used for the weight % column of the isotherm tabular report and the pressure composition isotherm plot.
Adsorbed phase free-space correction [*check box***]**	Adsorbed molecules occupy volume in the sample tube reducing the analysis free space. Select *Adsorbed-phase free space correction* to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
Adsorptive [*drop-down box***]**	Name of the adsorptive gas whose properties are being defined.
Dosing Method [*group box***]**	**Normal.** Dose from a pressurized tank of gas attached to a gas inlet port. **From Psat tube.** Select if the Psat tube is to be filled with condensed adsorptive and dosed from the Psat tube. Select this option if using krypton.

Adsorptive Properties (continued)

Selections	Description
	From sample port 3. P Select if the tube attached to sample port 3 is to be filled with condensed adsorptive and dosed from port 3. **Vapor source.** P SC Select if a container of condensed vapor is to be attached to the Psat port in place of the Psat tube and is dosed from the Psat port. **Charge from inlet.** P Use to have the tube automatically charged with condensate from a gas inlet port after the Dewar is raised. **Purify adsorptive.** P Use to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If *Charge from inlet* is selected, select *Purify adsorptive* to have noncondensing contaminants automatically removed from the dosing tube prior to analysis. After the adsorptive has condensed in the selected Psat tube or port 3, the remaining gas in the tube will be evacuated to remove noncondensing contaminants. A small amount of the purified adsorptive condensate will then return to gas phase to restore equilibrium pressure in the tube.
Dynamic chemi saturation pressure	Select if vapor is going to be used and enter the vapor defaults. **High-resolution fluid properties.** Use to import parameters from a Fluid Properties (.FPI) file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided. Contact Micromeritics Scientific Services if new fluid properties are required. **Antoine equation.** Enter the Antoine constants. ▪ **A, B, C.** Enter the Antoine constants used in the vapor TCD calibration. ▪ **Min/Max temperature.** Enter the temperature range where the constants are valid.
Fluid properties [*button***]**	Use to import parameters from a Fluid Properties (.FPI) file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided. Contact Micromeritics Scientific Services if new fluid properties are required.

Adsorptive Properties (continued)

Selections	Description
Gas Blend [*check box*] DA	Select to specify a preblended mixture of a chemically insert gas and a chemically active gas. The blend's *Mass Flow Constant* and *Relative Thermal Conductivity* are automatically calculated from those for the two gases in the blend.
Mass flow constant [*text box*] C	Scaling factor for the Mass Flow Controller measured flow rate. Applicable only for the gas used in the flow prep tasks. The default is preset for gases provided with the application.
Maximum manifold pressure [*text box*]	The highest pressure to which the manifold will be dosed. To avoid damage to the analyzer, this number is limited to 925 mmHg. Low pressure sources will require lower numbers. For gases to be used for dosing after charging a tube from a gas inlet, enter the maximum pressure for dosing from the inlet, not from the tube of condensate.
Mnemonic [*text box*]	Enter the mnemonic name for the adsorptive. See *Specify Gas Ports on page 2 - 15*.
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.
Relative Thermal conductivity DA	See *Gas Charts on page I - 1*.
Therm. tran. hard-sphere diameter [*text box*]	Estimate of the molecular size used in calculating the thermal transpiration correction.
Vapor Source Temperature [*group box*]	Select if the vapor source temperature is to be controlled by the analyzer. This field is enabled only if *Vapor Source* is selected.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3***.**

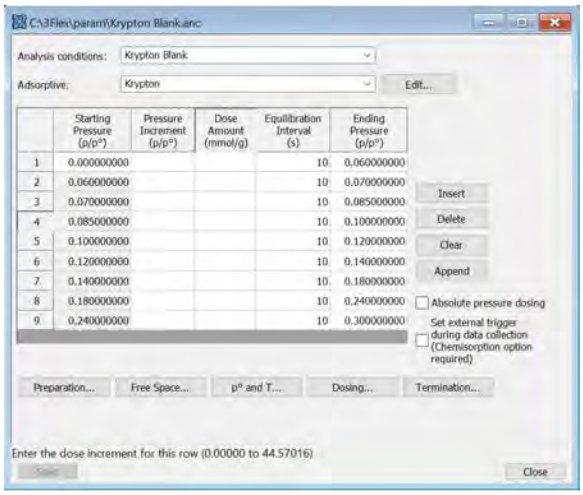
ANALYSIS CONDITIONS FOR PHYSISORPTION AND CHEMISORPTION

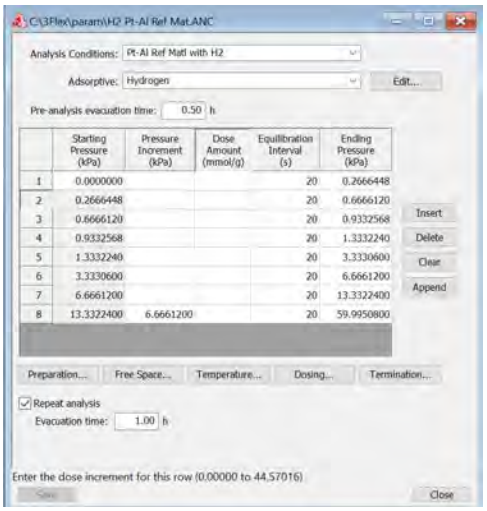
CFR Note	In 21CFR11 environments, this section is applicable only to members of the Developer group; however, members of the Analyst group may find information in this section helpful. Parameter file information that is available to Analysts is created by a member in the Developer group using information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

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.

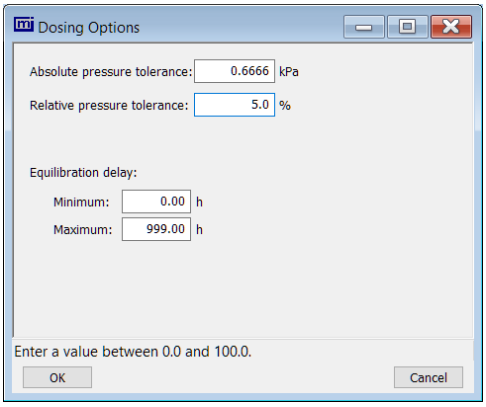




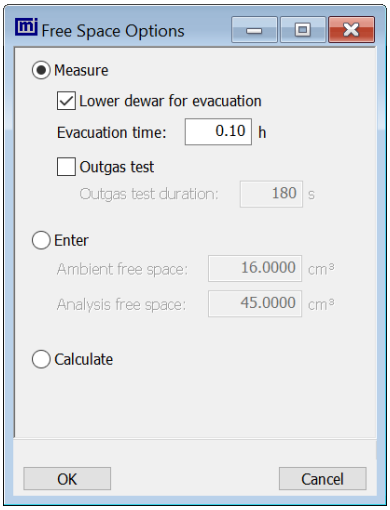
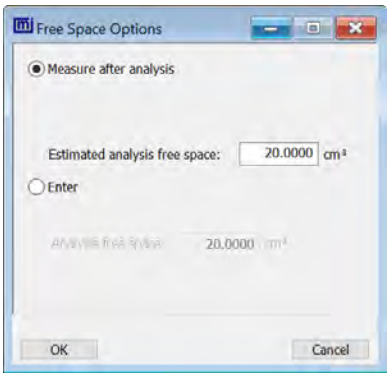
Physisorption ***Chemisorption***

The Pressures tables have a *Starting Pressure* and an *Ending Pressure* column. Each table row represents a pressure interval. The *Starting Pressure* column is for information only and cannot be edited.

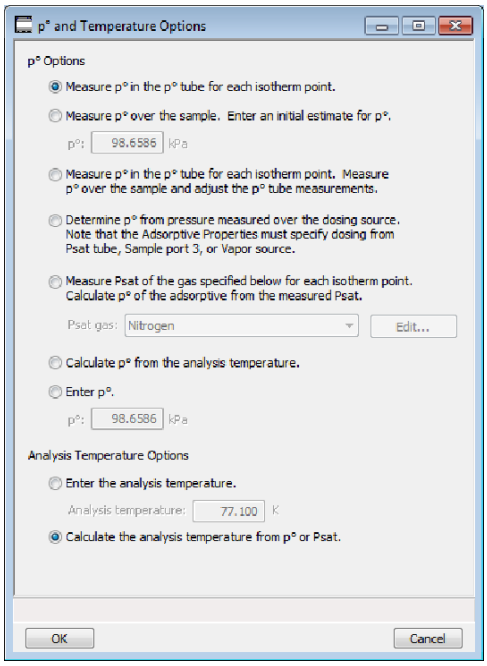
Analysis Conditions

Selections	Description
Absolute pressure dosing [*check box*] P	Specifies pressure targets in mmHg or mbar instead of relative pressure. If this option is selected, the *Relative Pressure* labels and entries change to *Absolute Pressure* in the selected pressure units.
Adsorptive [*drop-down 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.
Dosing [*button*]	Options for dosing tolerance, low pressure dosing, and dosing near saturation pressure.  **Absolute / Relative pressure tolerance.** Values used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is lower. At higher pressures, the absolute tolerance value is lower. For example: **Experiment 1.** There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg. **Experiment 2.** There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be

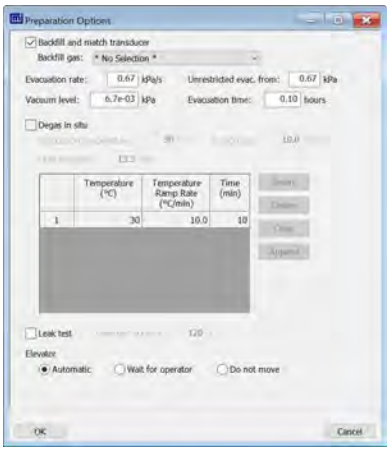
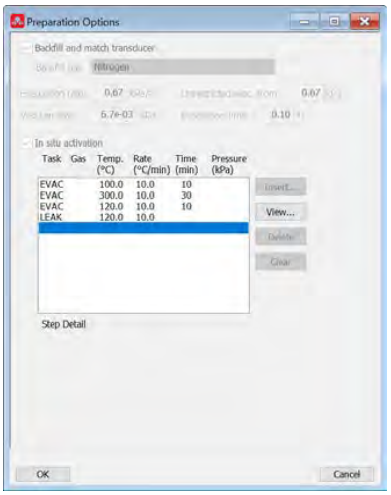
Analysis Conditions (continued)

Selections	Description
	attained to collect data for a target pressure of 200 mmHg. Normally, surface area measurement points are widely spaced and the resulting measurement is not very sensitive to the precise location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis. **Minimum equilibration delay at p/po > = 0.995.** P The minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995. This field is not available if *Absolute pressure dosing* is selected on the *Analysis Conditions* tab. **Low pressure equilibration delay.** Delays to be used until the first pressure in the table is reached.
Free Space [*button*]	How the free space is to be measured.  ***Physisorption***  ***Chemisorption*** **Calculate.** P Use to have the free space measurement calculated using the sample and tube parameters. **Enter.** Allows manual entry of free space. **Measure after analysis.** Measures free space after analysis ends. Enter the estimated free space measurements. **Measure before analysis.** P Select if the free space is to be measured before the analysis begins.

Analysis Conditions (continued)

Selections	Description
	■ **Lower Dewar for evacuation.** If the Dewar is to be lowered for evacuation, select this option and enter the length of time for evacuation after the free space measurement in the *Evacuation time* field. If using a cryostat, the operator must manually move the cryostat assembly when prompted. ■ **Outgas test.** Checks for system leaks or sample outgassing. After free space is measured, the Dewar is lowered and the sample evacuated for the specified amount of time. The leak test is performed after evacuation. If the pressure rises more than 0.025 mmHg within the time specified in the *Outgas test duration* field, outgassing is present. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails,the analysis stops and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak occurs.
p° and Temperature Options [*button*] P	How the saturation pressure (P_0) is to be measured or calculated and the analysis bath temperature.  **Analysis Temperature Options.** Select an option to enter analysis temperature manually, or choose to have it automatically calculated from p° or P_0.

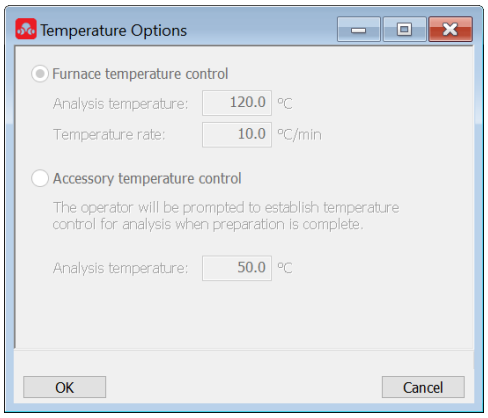
Analysis Conditions (continued)

Selections	Description
	p° Options. Select one option indicating how P_0 is to be measured or calculated. If using a cryostat, *Calculate p0 from the analysis temperature* should be selected. **Psat Gas.** If choosing to measure the P_0 for each isotherm point using a gas other than the adsorptive, select the P_0 gas from the drop-down list, then click **Edit** to modify the P_0 adsorptive properties. Refer to the *Adsorptive* drop-down list earlier in this table for details on editing this window.
Preparation [*button*]	Evacuation rate / time level, leak test and time values, elevator prompts, and in situ degassing or activation. To insert chemisorption tasks, see *Chemisorption Tasks on page 4 - 13*.   ***Physisorption*** ***Chemisorption*** **Backfill and match transducer.** Backfills the sample tube to 760 mmHg at the beginning of the analysis and to recalibrate the sample port pressure transducer scale to match the manifold pressure transducer. Select the backfill gas to be used. **Degas in situ.** P Degases the sample on the analysis port prior to analysis. ■ **Evacuation Temperature.** Temperature of the sample during evac-

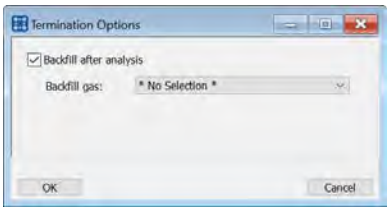
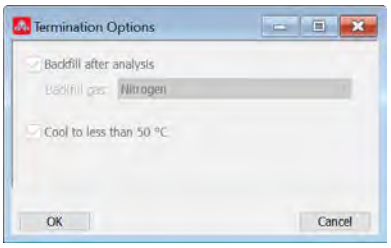
Analysis Conditions (continued)

Selections	Description
	uation. ■ **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold pressure*. This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity. ■ **Ramp Rate.** Rate at which the temperature is to change when advancing to the target temperature. **Elevator.** P Select the appropriate elevator control option. ■ **Automatic.** The elevator is raised and lowered automatically. ■ **Wait for operator.** Prompts the operator to set the elevator or analysis bath to the preferred height. When the prompt is acknowledged, the analysis will continue. This option should be used if the analysis bath must be placed manually in the preferred position, or the elevator must be raised to a height other than the standard analysis height. ■ **Do not move.** Use to have the analysis proceed without pausing or moving the elevator. This option should be used when the analysis bath is already in position and should not be moved during analysis. If ***Unit [n] > Enable CryoStat*** is enabled, the elevator option is automatically set to *Do not move*. **Evacuation rate.** The rate for restricted evacuation. **Evacuation time.** The length of time for preliminary evacuation. If this field is blank, the time entered in the *Pre-analysis evacuation* field on the *Analysis Conditions* window is used. **In situ activation.** SC When selected, preparation steps will be done. If not selected, the task table is disabled and analysis starts after the preliminary evacuation. **Leak Test.** P Enables the system to check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists. **Leak test duration.** P Enter the duration of the leak test.

Analysis Conditions (continued)

Selections	Description
	Vacuum level. The pressure for unrestricted evacuation. **Unrestricted evac. from.** The pressure at which unrestricted evacuation is to begin. This option is enabled when *Fast evacuation* is not selected.
Pre-analysis evacuation time [*text box*] C	Evacuation is required prior to analysis. The default setting is 30 minutes. **Preparation.** Use to set the evacuation rate, unrestricted pressure, and setpoint. **Temperature.** Use to set the temperature and ramp rate.
Repeat analysis [*selection*] SC	Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold pressure*. This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity.
Set external trigger during data collection	If selected, the contact closure used to trigger an external mass spectrometer or Calorimeter will be activated. If deselected, the contact closure will be deselected. Chemisorption option is required.
Temperature [*button*] SC	Provides access to furnace and accessory temperature control.  **Furnace temperature control.** Enter the analysis temperature rate. **Accessory temperature control.** *For user supplied temperature control only*. Enter the intended analysis temperature. The operator will be prompted to establish the analysis temperature before analysis begins.

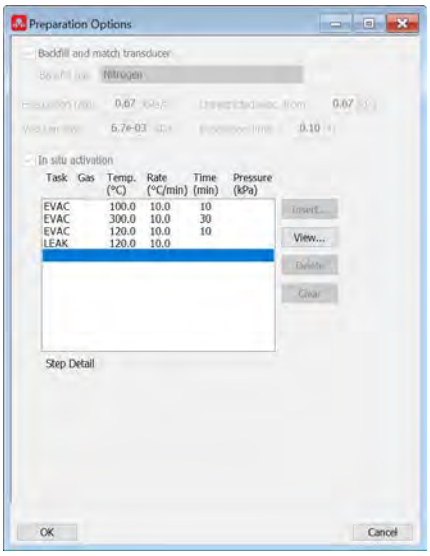
Analysis Conditions (continued)

Selections	Description
Termination [*button*]	Select the backfill gas if backfill is to be done after the analysis. If using a cryostat, deselect *Backfill after analysis*.  ***Physisorption***  ***Chemisorption*** **Cool to less than 50 °C.** SC Select to enable the cool down option.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

CHEMISORPTION TASKS

File > Open > [.SMP File] > Analysis Conditions tab > Preparation > Insert

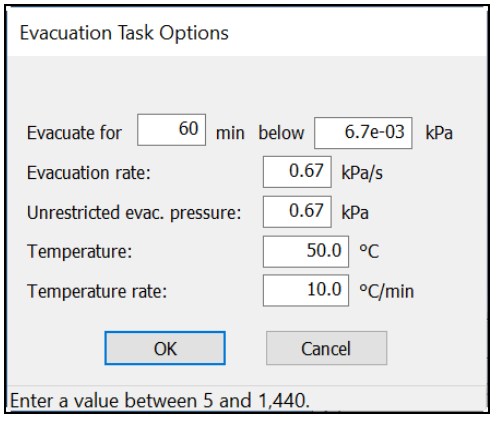


To ensure safe operation and reliable results, an evacuation task should be included:

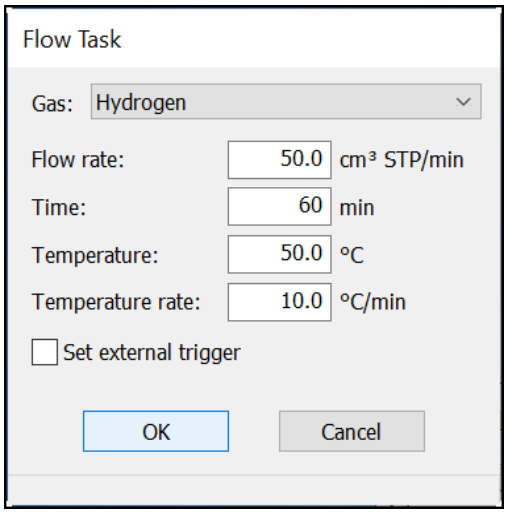
- between tasks using different gases.
- preceding a leak test.

An evacuation will be performed at the analysis temperature for the *Pre-analysis Evacuation Time* after the last task and before analysis.

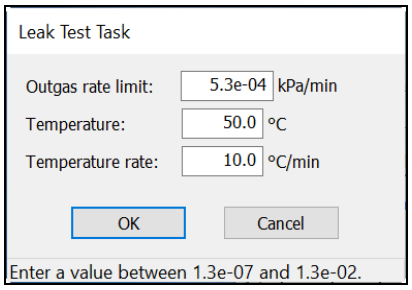
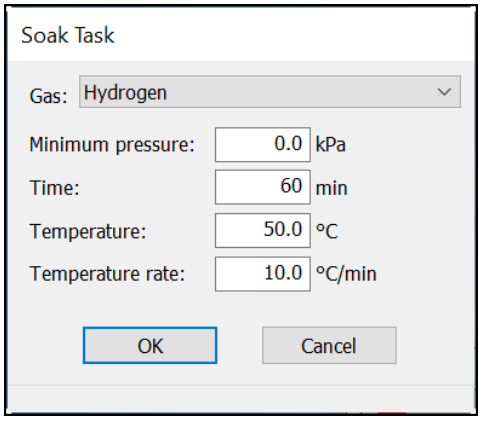
Chemisorption Tasks for Gas Adsorption

Task	Description
Evacuation [*selection*]	 **Evacuate for [*n*] min below [*n*] kPa or Pa.** The minutes and pressure

Chemisorption Tasks for Gas Adsorption (continued)

Task	Description
	for preliminary evacuation. **Evacuation rate [*n*] kPa/s.** The rate for restricted evacuation. **Temperature [*n*] °C or K**. The temperature to reach during evacuation. **Temperature rate [*n*] °C/min or K/min**. Enter the analysis temperature rate. **Unrestricted evac. pressure [*n*] kPa**. The pressure at which unrestricted evacuation is to begin. This option is enabled when *Fast evacuation* is not selected.
Flow Task [*selection*]	 **Flow rate [*n*] cm^3 STP/min.**The rate at which gas is to flow. **Gas.** Gas used for the flow task. **Set external trigger.** [*check box*] If selected, the contact closure used to trigger an external mass spectrometer or Calorimeter will be activated. If deselected, the contact closure will be deselected. **Temperature [*n*] °C.** The temperature at which the gas will flow for the specified time. **Temperature rate [*n*] °C/min.** Enter the analysis temperature rate. **Time [*n*] min.** The duration of time the sample should remain at the specified temperature.

Chemisorption Tasks for Gas Adsorption (continued)

Task	Description
Leak Test Task [*selection*]	 **Outgas rate limit [*n*] kPa/min**. If a measured leak or outgas rate exceeds the entered value, the test will be reported as failed. Analysis will not be canceled. **Temperature [*n*] °C.** The target temperature for the leak test. **Temperature rate [*n*] °C/min.** Enter the analysis temperature rate.
Soak [*selection*]	 **Gas.** Gas used during the soak task. **Minimum pressure [*n*] kPa.** The minimum pressure to be maintained over the sample during the soak. **Temperature [*n*] °C.** The temperature at which sample is to be soaked. **Temperature rate [*n*] °C/min.** Enter the analysis temperature rate. **Time [*n*] min.** The duration of time the sample is to soak at the specified temperature.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

Evacuation Rules for Chemisorption

Evacuation parameters apply to all four stages of evacuation with the exception of evacuation time. Evacuation time is set using the fields specified in the following evacuation rules.

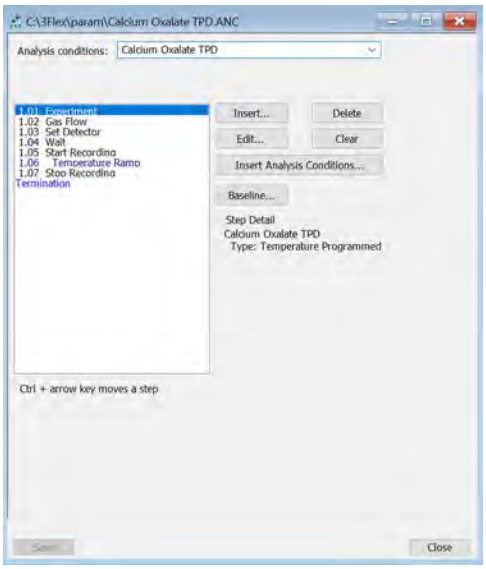
When an analysis starts, evacuation begins at:

1. Ambient temperature before the first preparation step. This step uses the *Evacuation time* field on the *Preparation Options* window.
2. Analysis temperature *after* preparation and prior to the start of the analysis stage. This step uses the *Pre-analysis evacuation time* field on the *Analysis Conditions* window.
3. Analysis temperature *before* the repeat analysis if *Repeat analysis* is selected on the *Analysis Conditions* window. This step uses the *Repeat Analysis / Evacuation time* field.
4. Analysis temperature before the free space measurement. This step uses the *Evacuation time* field on the *Free Space Options* window. *Measure after analysis* must be selected to enable the *Evacuation time* field.

ANALYSIS CONDITIONS FOR DYNAMIC ANALYSIS

File > Open > [.ANC File]

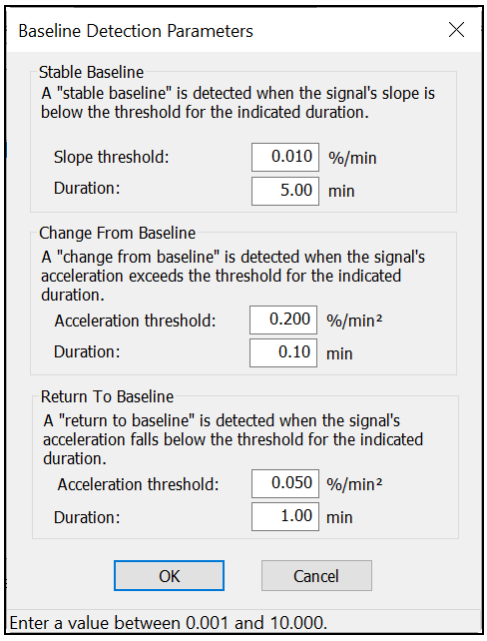
Analysis conditions specify the parameters used to guide an analysis.



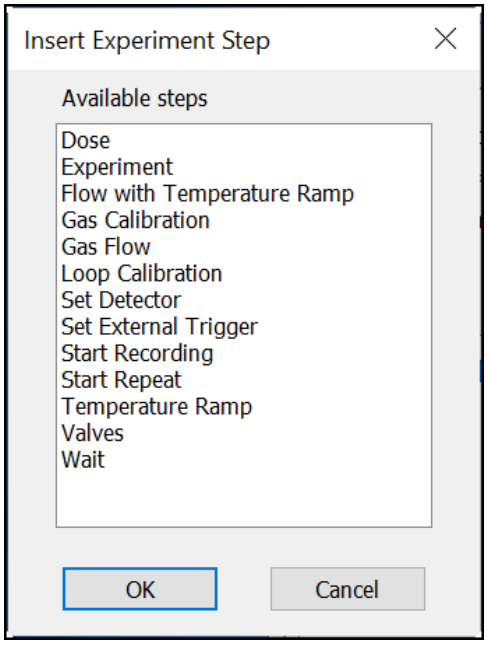
Analysis Conditions for Dynamic Analysis

Selections	Description
Analysis Conditions [*drop-down box*]	Use to browse for an *Analysis Conditions* file that contains analysis condition parameters to be used in the analysis.
Baseline [*button*]	Specifies baseline settings if a *Wait* step depends upon the *Baseline*.

Analysis Conditions for Dynamic Analysis (continued)

Selections	Description
	 Establishes the *Slope* or *Acceleration* threshold and *Duration* for determining what constitutes a *Stable Baseline*, a *Change From Baseline*, and a *Return To Baseline*. These values control whether a particular change in the signal is significant to the current experiment, such as defining a stable baseline. Some *Wait* steps are contingent upon the values selected, such as if the experiment contains a *Wait until Baseline is stable* step, the signal is compared to these values to determine if a stable baseline has been established. Lower slope/acceleration values and longer durations create a more rigorous definition of these factors than higher values and shorter durations. **Stable Baseline.** [*group box*] Detected when the signal slope is below the threshold for the indicated duration. **Change from Baseline.** [*group box*] Detected when the signal acceleration exceeds the threshold for the indicated duration. **Return to Baseline.** [*group box*] Detected when the signal acceleration falls below the threshold for the indicated duration.

Analysis Conditions for Dynamic Analysis (continued)

Selections	Description
Insert [*button*]	Inserts a new step into the experiment. Steps can be inserted after the analysis has started by suspending the analysis. See ***Insert Experiment Steps on the next page***.  To ensure safe operation and reliable results, a chemically inert gas flow should be inserted between flows of two chemically reactive gases such as hydrogen and oxygen.
Insert Analysis Conditions [*button*]	Click to load an entire list of steps, baseline parameters, and peaks parameters from the selected dynamic analysis conditions file.
Step Detail [*group box*]	Displays summary information about the highlighted experiment step.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

INSERT EXPERIMENT STEPS

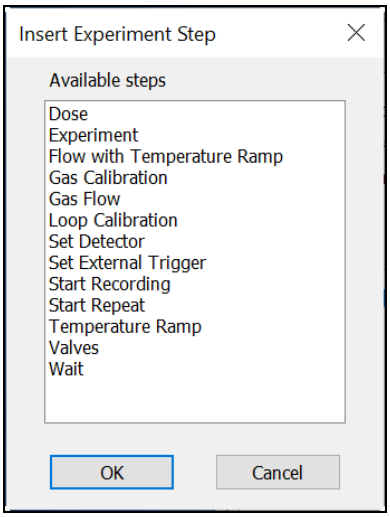
Experiments can be customized to control the analysis reaction. When an experiment is inserted, the initial conditions are specified first, then the individual steps.

1. On the *Analysis Conditions* tab, click **Insert** to insert an experiment.
2. Select the experiment step in the box.
3. Click **Edit** to modify settings. An analysis set is created by inserting up to 99 experiments in the sample file.

To edit steps that have not started, the analysis must be suspended.

- Upcoming steps - display as black or blue
- Current step - step is highlighted and displays as light blue
- Completed steps - display as light green

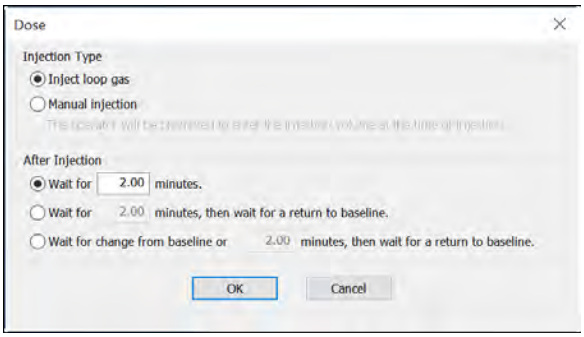
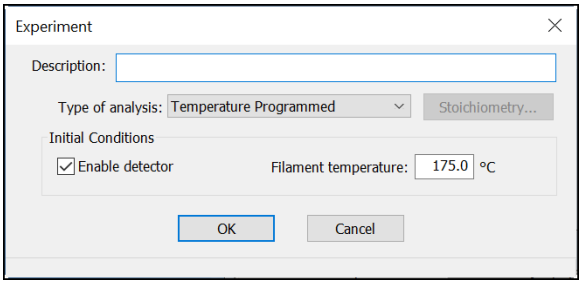
The *DDE Command* step and *Wait for synchronization message* option in the Wait step are no longer supported.



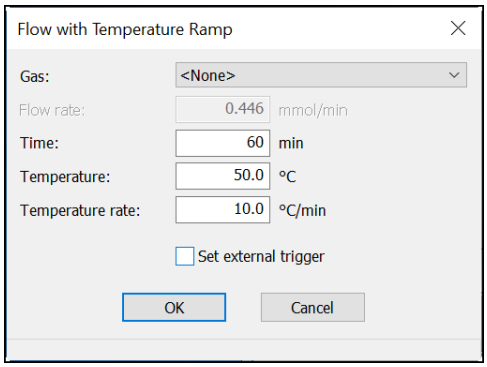
Note that Loop Calibration, Loop Gas, Inject loop gas, and Loop Valve selections are only applicable to analyzers that have the Loop option. Do not select these for analyses to be performed on an instrument without the Loop option.

Data from one experiment are not available for editing until the next experiment in the analysis has begun recording.

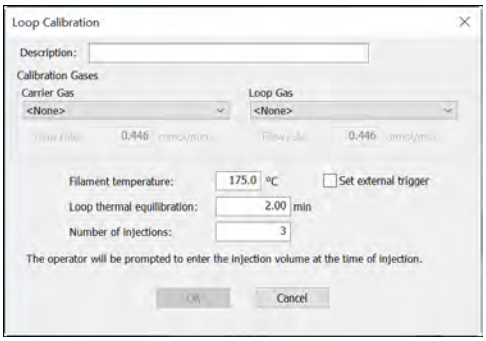
Experiment Fields

Selections	Description
Dose	 **Injection Type:** ■ **Inject loop gas [***button***].** Automatically injects the contents of the loop into the path that leads to the sample. The contents of the loop are pushed out of the loop by the carrier gas. ■ **Manual injection [***button***].** Prompts the user to inject a dose of gas into the septum using a syringe. **After Injection.** Specifies the conditions for completion of this step.
Experiment	 **Description [***text box***].** Description of the experiment. **Enable Detector [***check box***].** Enables the *Filament temperature* field and enables the thermal conductivity detector (TCD). **Filament temperature [n] °C [***text box***].** Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used. **Type of analysis [***drop-down box***].** See ***Analysis Types for TCD Analyzers on page 2 - 31***.If *Pulse Chemisorption* is selected, the **Stoichiometry** button is enabled. When modifying the *Active Metals* table during the insertion of a *Pulse Chemisorption* experiment, changes to the *Active Metals* table on the *Sample Description* tab are not affected. This is useful when sequencing

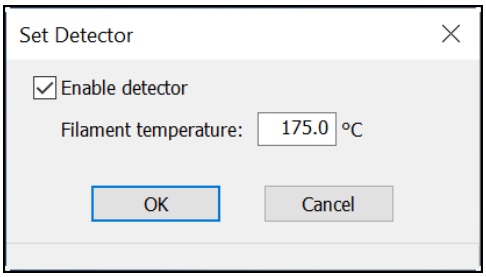
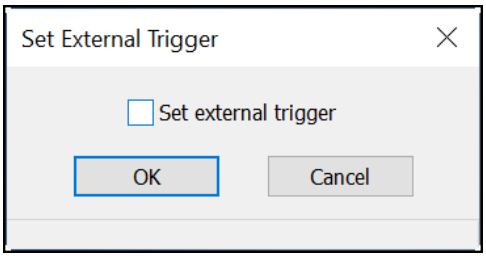
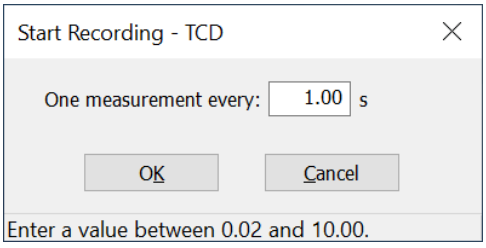
Experiment Fields (continued)

Selections	Description
	multiple experiments and Stoichiometry is different from one experiment to the next.
Flow with Temperature Ramp	 **Flow Rate [*n*] mmol/min** [*text box*]. The rate at which gas is to flow. **Gas** [*drop-down box*]. Gas used for the flow task. **Set external trigger** [*check box*]. If selected, the contact closure used to trigger an external mass spectrometer will be activated at the beginning of the experiment. **Temperature [*n*] °C** [*text box*]. The temperature at which the gas will flow for the specified time. **Temperature rate [*n*] °C/min**. Enter the analysis temperature rate. **Time [*n*] min [*text box*]**. The duration of time the sample should remain at the specified temperature.
Gas Flow	**Carrier Gas / Loop or Injection Gas [*drop-down box*].** Select the gas for each set of inlet ports. The gases listed are those with defined adsorptive properties. If manual injections are to be programmed, the gas to be injected must be selected as *Loop* or *Injection* gas. If loop injections are to be programmed, this gas must be connected to a loop gas inlet and assigned in ***Unit [n] > Unit Configuration***. See ***Configure the Analyzer on page 2 - 15***. When an analysis is started, the application verifies that the selected gases are connected to the appropriate ports. If there is a discrepancy between a gas selected for the current sample file and the gases indicated in the *Unit Configuration* window, an error message is displayed. **Flow rate [*n*] mmol/min [*text box*].** The rate at which gas is to flow.

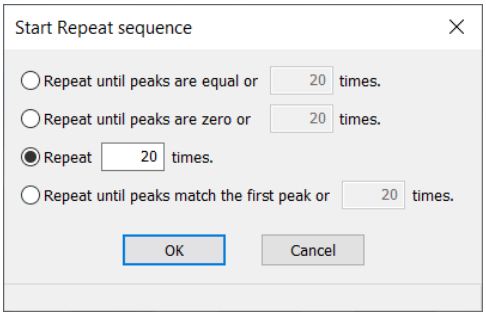
Experiment Fields (continued)

Selections	Description
	Cold trap valve. Select the status of the cold trap valve: *Bypass* or *Trap*. **Loop valve.** Select the status of the loop valve: *Fill* or *Inject*.
Loop Calibration	Use to calibrate the gas injection loop. This experiment verifies the exact volume of the loop for use in calculations on analyses in which the loop is used. Sample analysis data yield signal vs. time or temperature data and peak areas. After the analysis, select the *Loop Calibration* report and enter the *Loop volume* from the report on the *Unit Configuration* window. Entering the calibrated *Loop volume* on the *Unit Configuration* window makes it possible for the application to convert sample data to volume values. See *Loop volume* located in ***Specify Gas Ports on page 2 - 15***.  **Description [*text box*].** Description of the experiment. **Calibration Gases.** Select the *Carrier* and *Loop* gases to be used in this calibration. The gases available are those specified in an Adsorptive Properties [.ADP] file. . See ***Adsorptive Properties on page 4 - 2*** and ***Gas Charts on page I - 1*** for gas combinations. **Flow rate [*n*] mmol/min [*text box*].** The rate at which gas is to flow. **Filament temperature [*n*] °C [*text box*].** Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used. **Loop thermal equilibration [*n*] min [*text box*].** Specify the duration of the equilibration delay. This delay allows extra time for the instrument to stabilize thermally after the baseline has fully stabilized.

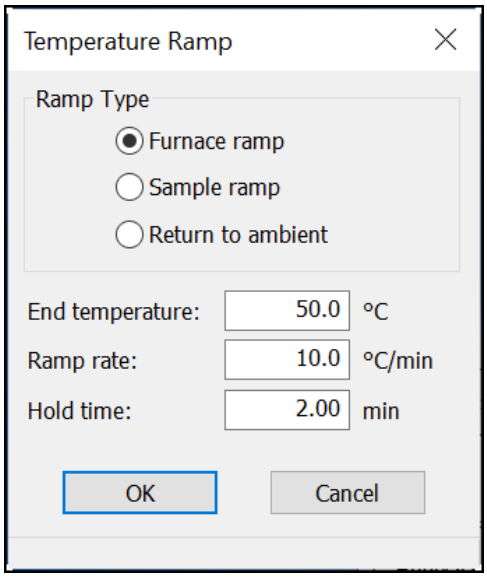
Experiment Fields (continued)

Selections	Description
	Number of injections [*n*] [*text box*]. Indicate the number of gas injections to be made into the gas stream. **Set external trigger [*check box*].** If selected, the contact closure used to trigger an external mass spectrometer will be activated.
Set Detector	 **Enable detector [*check box*].** Enables the *Filament temperature* field and enables the thermal conductivity detector (TCD). **Filament temperature [*n*] °C [*text box*].** Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used.
Set External Trigger	 **Set external trigger [*check box*].** If selected, the contact closure used to trigger an external mass spectrometer or Calorimeter will be activated. If deselected, the contact closure will be deselected.
Start Recording	 Specifies how frequently the signal reading is recorded. A *Stop Recording* step is inserted in the steps automatically when a *Start Recording* step is inserted. Multiple steps can be inserted between

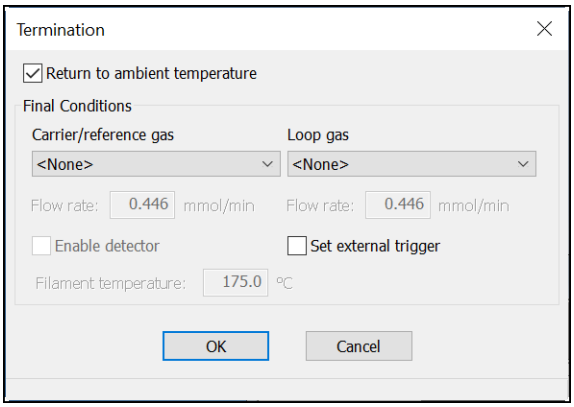
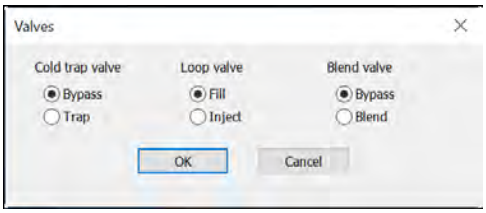
Experiment Fields (continued)

Selections	Description
	the *Start Recording* and *Stop Recording* steps. **One measurement every [*n*] seconds.** [*text box*] Specify the frequency of measurements. If a *Start Recording* step is immediately followed by a step that prompts an immediate peak, peak data are recorded before any baseline readings can be collected. To collect some baseline data before the first peak, insert a *Wait for [n] minutes* step after the *Start Recording* step but before the step which causes the peak.
Start Repeat	 Specifies the duration of the repeat sequence. Automatically inserts a *Start Repeat* and a *Stop Repeat* in the list of steps. Multiple experiment steps can be inserted within the *Repeat* loop. **Repeat until peaks are equal or [*n*] times.** [*button*] Stops repeating the steps within the loop when the last two peaks are equal to within 5% of the area, or when the maximum number of repeats is reached. This option is useful when performing H_2 or CO pulse chemisorption on supported metal catalysts. **Repeat until peaks are zero or [*n*] times.** [*button*] Stops repeating the steps within the loop when the last two peaks are each less than 10% of the area of the first peak, or when the maximum number of repeats is reached. This option is useful when performing an N_2O decomposition for characterizing copper catalysts. **Repeat [*n*] times.** [*button*] Stops repeating the steps within the loop when the specified number of times is reached. **Repeat until peaks match the first peak or [*n*] times.** [*button*]

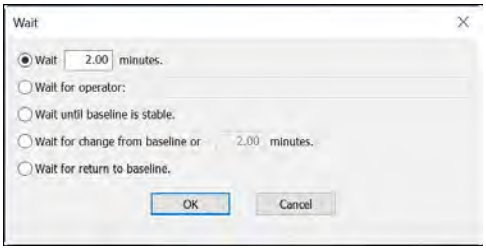
Experiment Fields (continued)

Selections	Description
	Stops repeating the steps within the loop when the last two peaks each match the first peak to within 10% of its area or when the maximum number of repeats is reached.
Temperature Ramp	Changes the sample temperature.  **End temperature [*n*] °C.** [*selection*] The ending temperature for the ramping procedure. If the CryoCooler is installed, it is automatically enabled if an ending temperature below 20 °C is used. **Furnace ramp.** [*button*] Ramps the furnace temperature directly to the *End Temperature*, ignoring the sample temperature. **Hold time [*n*] min.** [*selection*] How long the sample is to be held at the specified temperature before beginning to cool down. **Ramp rate [*n*] °C/min.** [*selection*] The rate at which the temperature will change while advancing to the hold temperature. **Return to ambient.** [*button*] Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature to below 45 °C. **Sample ramp.** [*button*] Ramps the sample temperature to the *End* temperature. The actual furnace temperature is adjusted to meet this target.

Experiment Fields (continued)

Selections	Description
Termination	 **Carrier / Reference / Loop Gas.** [*drop-down box*] Select the gas to be used during the termination stage of the analysis. **Enable detector. [***check box***]** Enables the *Filament temperature* field and enables the thermal conductivity detector (TCD). **Filament temperature [*n*] °C.** [*selection*] Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used. **Flow rate [*n*] mmol/min. [*selection*]** The rate at which gas is to flow. **Return to ambient temperature. [***check box***]** Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature between 14 °C and 50 °C. **Set external trigger. [***check box***]** If selected, the contact closure used to trigger an external mass spectrometer or Calorimeter will be activated. If deselected, the contact closure will be deselected.
Valves	Sets the cold trap valve and the loop valve as specified. 

Experiment Fields (continued)

Selections	Description
Wait	Specify a waiting routine.  **Wait [*n*] minutes.** [*selection*] Specify the time to wait. **Wait for change from baseline or [*n*] minutes.** [*button*] Specify the time to wait, click **OK**, then click **Baseline** to specify the settings. **Wait for operator. [*button*]** Enter a description of the operator task. During an analysis, the entered message displays at the appropriate time. The analysis continues after the operator clicks **OK**. **Wait for return to baseline. [*button*]** Waits for a return to baseline. If enabled, click **OK**, then click **Baseline** to specify the settings. **Wait until Baseline is stable. [*button*]** Specify if the analysis should wait until the baseline becomes stable, then click **Baseline** to specify the settings.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

DEGAS CONDITIONS

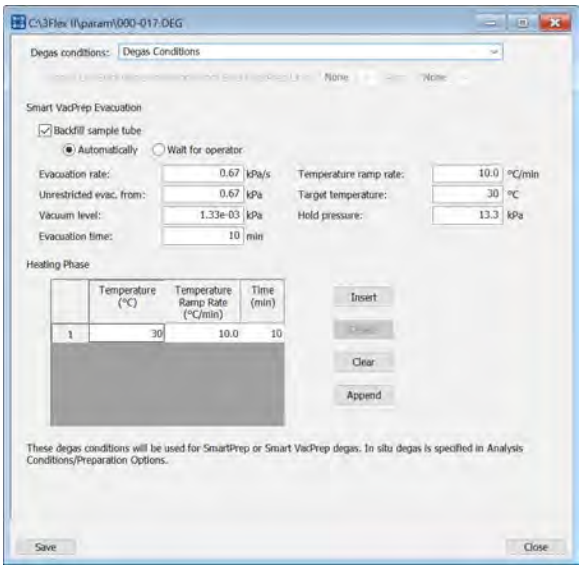
File > Open > [.DEG File]

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.

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

Selections	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)

Selections	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 - 3*.

REPORT OPTIONS

File > Open > [.RPO File]

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

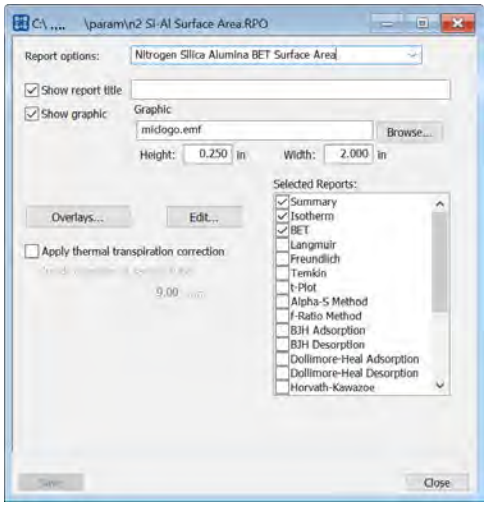
Selected Reports on page 8 - 1

Additional reports are available using the *Reports* menu.

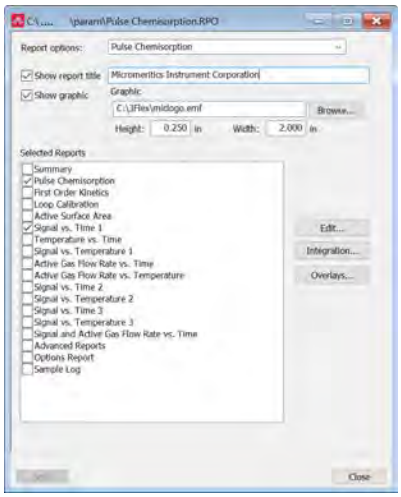
Use to specify report options for data collected from an analysis or manually entered data. *Report Options* files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations. These files may contain tabular reports, plots, or both,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



Physisorption and Chemisorption



Dynamic Analysis

Report Options

Selections	Description
Apply thermal transpiration correction [*check box*] P SC	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analyses. **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. Refer to the *Thermal Transpiration Correction* section of the *Calculations* document.
Overlays [*button*] P SC	See *Graph and Sample Overlays on page 7 - 32*.
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. For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R), or pore diameter (D). Go to ***Options > Units*** to specify default calculations.
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's appearance on the generated report.
Show report title [*check box*]	Select then enter a report title to appear on the report header.

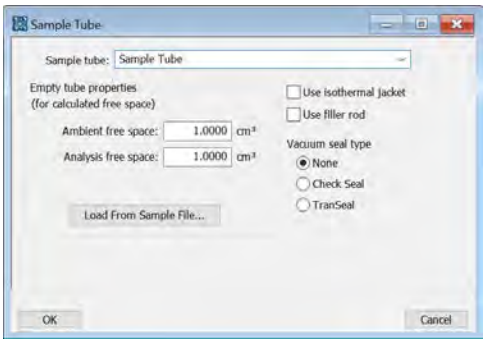
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3***.**

SAMPLE TUBE

File > Open > [.STB File]

Or, click **Edit** next to the *Sample Tube* selection on the *Sample Description* tab when in *Advanced* option presentation.

Sample Tube files specify information about the sample tube.



Sample Tube

Selections	Description
Ambient free space [*text box*]	Empty sample tube gas capacity measured at room temperature.
Analysis free space [*text box*]	Empty sample tube gas capacity measured with the Dewar raised.
Load from Sample File [*button*]	Loads parameters from the selected sample file.
Sample tube [*drop-down box*]	It is a good practice to label each sample tube with a unique identification. Enter that information here. This information will also appear in the *Sample Tube* drop-down list on the *Sample Description* tab.
Use filler rod [*check box*]	Select if a filler rod is to be used in the sample tube. A filler rod reduces the stem free space volume resulting in reduction of free space error. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area.

Sample Tube (continued)

Selections	Description
Use isothermal jacket [*check box*]	Select if an isothermal jacket is to be used. An isothermal jacket maintains a constant temperature profile along the sample tube stem during an extended analysis of more than 1 or 2 hours.
Vacuum seal type [*group box*]	Select the seal type to be used.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

5 DEGASSING

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

After the sample has been weighed, degas the sample on:

- the analysis port (see ***Degas in Situ on page 5 - 3***),
- a Smart VacPrep. The *Smart VacPrep Operator Manual* can be found on the Micromeritics web page (www.Micromeritics.com), or
- a user supplied degasser.

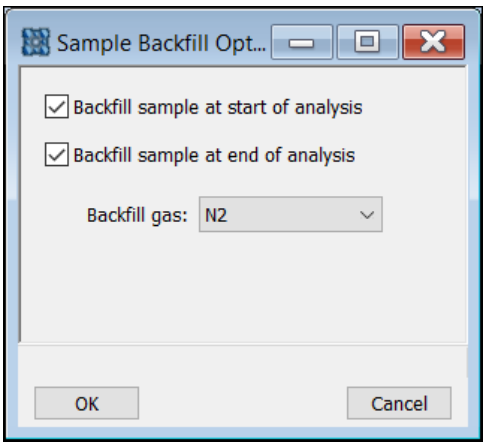
The Check Seal or TranSeal sample tube closures can be used with the Smart VacPrep to minimize sample contamination when transferring the sample tube from the Smart VacPrep to the analyzer port.

- If using the TranSeal, refer to the instructions included with the TranSeal [*part number 350-42803-00*].
- If using the Check Seal, see the instructions included with the Check Seal [*part number 350-42802-00*].

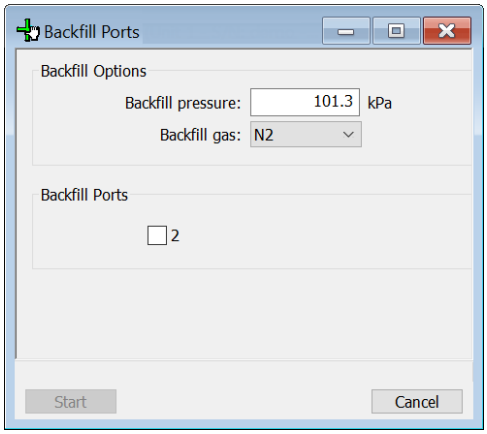
BACKFILL PORTS

Unit [n] > Backfill Ports

Use to backfill ports with gas.



Physisorption



Chemisorption or Dynamic Analysis

DEGAS IN SITU

Unit [n] > Sample Analysis

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The degas process heats the sample to remove the moisture and contaminants.

Physisorption samples can be degassed on either the analyzer's analysis port (in situ) or on a separate device such as a Smart VacPrep. Degassing should be performed prior to starting analysis.

Samples containing excessive amounts of moisture or significant amounts of other contaminants must be degassed on a separate degas system before attaching to the analyzer to prevent contamination of the analyzer high vacuum system.

Microporous samples should receive a secondary in situ degas on the analyzer to remove any moisture readmitted during transfer from the separate degas system to the analyzer.

When the degas is completed, observe the temperatures on the analyzer schematic. When the mantle has cooled, the *Sample Analysis* window will display a prompt to remove the degas heating mantle, properly position the isothermal jackets and Dewar cover, then install the Dewar.

If using the heating mantle option, remove the heating mantle (it is not necessary to unplug the mantle), support the bottom of the tubes, then remove the mantle cover.

To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled below 45 °C.

TRANSFER A DEGASSED SAMPLE TO AN ANALYSIS PORT

Sample Data Worksheet for Gas Adsorption on page O - 2

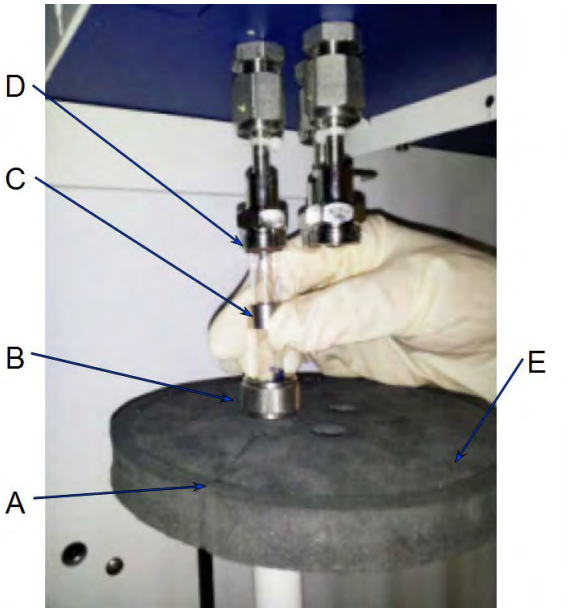
When degassing on a separate degasser such as a Smart VacPrep, the sample tube must be removed from the degas port, weighed, and then installed onto the analysis port for analysis.

If the sample tube is not mounted on the analysis port immediately, leave it on the degas port. If it is necessary to remove the sample tube and a Check Seal or TranSeal was not used, insert a rubber stopper into the sample tube.

1. Allow the sample tube to cool to room temperature (approximately 15 minutes).

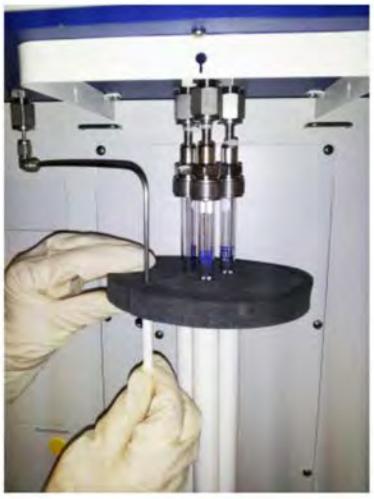
Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube, heating mantle, or heating mantle clip before they have cooled could result in burns.

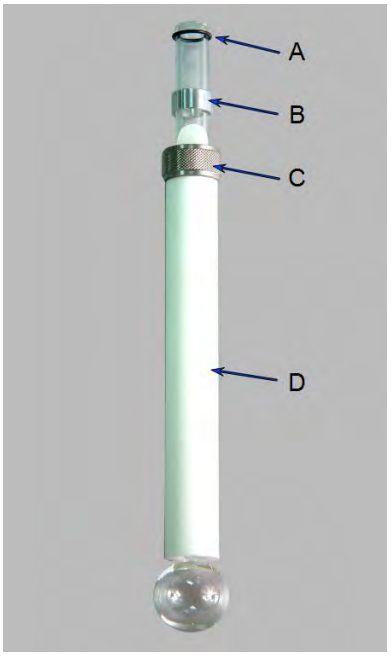
2. Carefully remove the heating mantle clip and the heating mantle from the sample tube.
3. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If a Check Seal or TranSeal was not inserted prior to degassing, immediately insert a rubber stopper into the sample tube.
4. Complete the *Sample Data Worksheet*.
5. If using a Check Seal, ensure that the Check Seal opener is installed in the analyzer sample port. If a rubber stopper was used, remove the stopper from the sample tube.
6. bulb
7. Place the connector nut, ferrule, and O-ring onto the sample tube stem.
8. On the analyzer, loosen the connector nut on the Psat tube and rotate it out of the way.
9. Position the Dewar cover so that the slot for the Psat tube is on the left between port 1 and port 2.



A. P_0 slot
B. Connector nut
C. Ferrule
D. O-ring
E. Dewar cover

10. Insert the sample tube through one of the holes in the Dewar cover.
11. Place the sample port nut, ferrule, and O-ring onto the sample tube stem.
12. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the sample port nut onto the analysis port.
13. Repeat all previous steps for each sample tube.
14. Position the Dewar cover approximately 3/4 in. (19 mm) below the sample port nut.
15. Slide the Psat tube into the Psat slot in the Dewar cover and retighten the Psat tube connector nut.





A. O-ring (1/4 in. from top)
B. Ferrule
C. Connector nut
D. Isothermal jacket

16. If degassing a sample on the sample port, see ***Degas in Situ on page 5 - 3***. Otherwise, place the Dewar cover over the sample tube stem just above the isothermal jacket. Ensure the P_0 tube is next to the sample tube. Refer to the image in step 9.
17. Install the Dewar onto the elevator and place the safety shield over the sample tube and Dewar.
18. Begin the analysis.

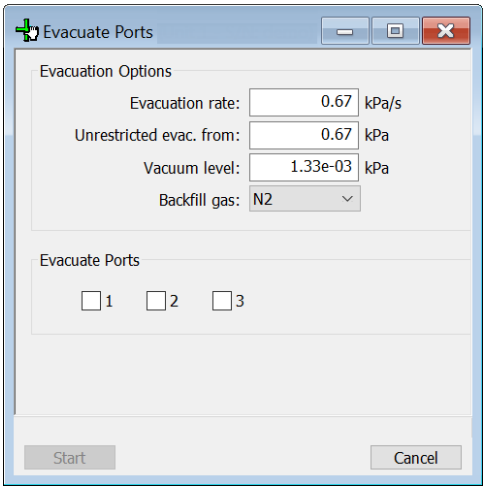
DEGAS ON THE SMART VACPREP

Equipment Options and Upgrades on page 1 - 4

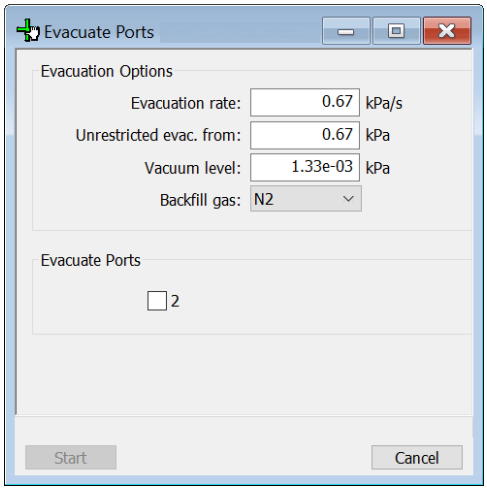
The *Smart VacPrep Operator Manual* can be found on the Micromeritics web page (www.Micromeritics.com).

EVACUATE PORTS

Unit [n] > Evacuate Ports



Physisorption



Chemisorption or Dynamic Analysis

Evacuate Ports

Selections	Description
Evacuation Options [*group box*]	**Evacuation rate.** The maximum rate of change of pressure when evacuating the sample tube. **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. **Vacuum level.** Evacuation time starts when the vacuum level is reached. **Backfill Gas.** Select the backfill gas from the drop-down list.
Evacuate Port[s] [*group box*]	Select the ports to evacuate.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

6 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.

CAUTION	If using a cryostat for analysis, see *CryoStat on page D - 1* prior to use.

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 to 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.

MIXING AN IPA/LN$_2$ SLURRY

An isopropyl alcohol (IPA) /liquid nitrogen (LN_2) slurry is used to maintain cold trap temperatures of approximately -80 °C. Improperly mixing an IPA/LN_2 slurry could cause injury. If the mixture is not stirred continuously, gas may build up under the surface, causing the liquids to splash out of the Dewar.

1. Chill a 600 mL Dewar by rinsing it with LN_2. Allow a small amount of LN_2 to remain in the bottom of the Dewar (approximately 1 cm deep).
2. Stirring constantly, slowly add approximately 500 mL of IPA. For greatest safety, use a laboratory squirt bottle. Squirt the stream of IPA along the inside edge of the Dewar, close to the top of the Dewar, allowing the IPA to flow down the inside wall of the Dewar. Do not stop stirring the mixture in the bottom of the Dewar, even if stirring becomes difficult. As stirring and adding IPA continues, the mixture will loosen and become easier to stir.
3. When all the IPA has been placed in the Dewar, slowly begin pouring LN_2 into the Dewar. Approximately 1 liter is needed. Continue to stir the mixture as LN_2 is added. As ice chunks form, break them up and stir them down into the mixture. Avoid splashing. Gently knock ice chunks away from the sides of the Dewar and continue stirring. Add liquid nitrogen until the slurry is within 25 mm (1 in.) of the top of the Dewar.
4. Lift the stirrer out of the slurry and observe as the slurry drips into the Dewar; the stirrer should be thickly coated with slurry.

   The goal is to achieve a slurry that has a thick, syrupy consistency without large chunks. When the slurry is cold enough, small pieces of ice may be present (10 - 20% of the mixture). A little ice accumulation along the sides of the Dewar is acceptable.

DYNAMIC ANALYSIS

To surround the cold trap, the Dewar must rest on a small stand. Hold the Dewar beneath the cold trap, then raise it to slide the stand underneath the Dewar. Lower the Dewar until it rests on the stand. Ensure the cold trap is immersed.

PREPARE FOR ANALYSIS

It is recommended to perform the tasks in the provided order.

Dewar Precautions on page 6 - 2

Step 1 - *Clean and Label Sample Tubes on the facing page*
Step 2 - *Create Sample Files on page 3 - 2*
Step 3 - *Determine the Sample Mass for Physisorption on page 6 - 8* or *Determine the Sample Mass for Chemisorption on page 6 - 13*
Step 4 - *Degas the Sample on page 6 - 20*
Step 5 - *Sample Tube Installation for Physisorption on page 6 - 10*
Step 6 - *Fill and Install the Dewar on page 6 - 21*

DA For dynamic analyses that include manual or loop injections of a chemically active gas, selection of an active gas/inert gas pair is required. The gas to be injected may be a blend of chemically active gas and inert gas, or a pure active gas. If a blend is used for injections, the inert gas in the blend should be the same as the carrier gas. See *Adsorptive Properties on page 4 - 2* and *Gas Charts on page I - 1*. The inert carrier gas should be selected to have a very different thermal conductivity from the active gas so as to provide a large change in the thermal conductivity detector signal, for instance helium (inert, T.C.=5.84) with carbon monoxide (active, T.C.=0.97) or argon (inert, T.C.=0.68) with hydrogen (active, T.C.=7.07). The selected carrier gas must be connected to one of the 12 primary gas inlets on the rear panel of the instrument. For manual injections, the gas to be injected must be connected to an external septum (provided in the accessory kit) to allow filling the syringe. For loop injections, the gas to be injected must be connected to one of the four loop gas inlets on the side of the instrument. Be sure to enter the mnemonics for the connected gases under *Specify Gas Ports on page 2 - 15*.

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 (required for chemisorption sample tubes) ■ Stopper for sample tube (Check Seal, rubber stopper, or TranSeal)	■ 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, Check Seal, or TranSeal.

DETERMINE THE SAMPLE MASS FOR PHYSISORPTION

Sample Data Worksheet for Gas Adsorption on page O - 2

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

There are several different surface area ranges that require different protocol for optimal results.

- If there is between 0.1 m^2 and 1 m^2 in the tube, follow the recommendations for balancing the free space using glass beads, along with filler rods, and measure the resulting free space difference.
- If there is between 1 m^2 and 5 m^2 of total surface under test, measure the free space difference between the two tubes as a part of each analysis.
- If there is between 5 m^2 and 30 m^2, use the option to calculate the free space difference between the two tubes using the density of the sample in the tube, along with the mass entered. There are some special considerations when using the tubes with the cylindrical bulb on the end as opposed to the straight-walled tubes.
- If there is more than 30 m^2 in the tube total, it may not be necessary to determine the free space difference between the two tubes as a part of the analysis.

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 mass. 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.

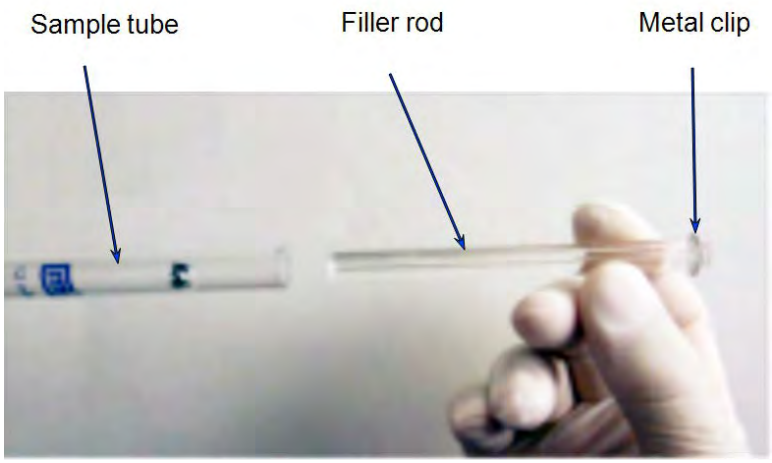
5. Place a sample container on the balance. Tare the balance and allow it to stabilize to zero.
6. Slowly pour the specified amount of sample into the sample container.
7. Remove the rubber stopper, Seal Frit, Check Seal, or TranSeal from the sample tube.
8. Use the sample tube funnel (provided in the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

SAMPLE TUBE INSTALLATION FOR PHYSISORPTION

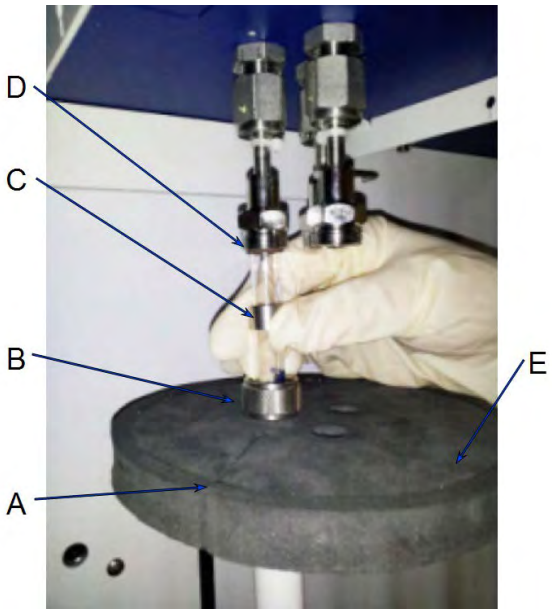
Sample Tube Installation for Chemisorption on page 6 - 16
Sample Tube O-ring Replacement on page 12 - 21

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

Repeat the following steps for each sample to be installed.

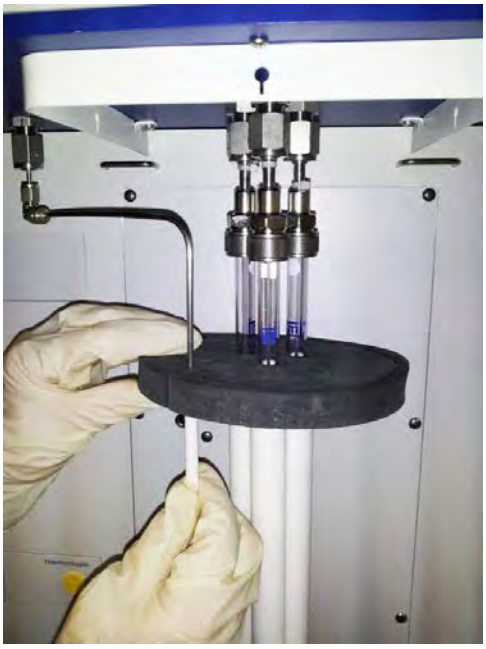
If using...	Then...
A rubber stopper	Remove it.
An isothermal jacket	Slide the jacket down over the stem of the sample tube until it touches the sample tube bulb. The top of the isothermal jacket should be aligned with the mark on the sample tube. If using sample material, insert it into the sample tube.
A filler rod	Hold the sample tube horizontally and carefully slide the filler rod into the tube until the metal clip touches the end of the tube. If using a hanging filler rod, the rod attaches to the Seal Frit.  CAUTION Do not hold the rod vertically and drop the rod into the tube as this could break the rod and/or tube.

1. Loosen the connector nut on the Psat tube and rotate it out of the way.
2. If using a Check Seal, verify that the port has the Check Seal opener installed or if using a TranSeal, install it at this time.
3. Position the Dewar cover so that the slot for the Psat tube is on the left between ports 1 and 2.



A. P_0 slot
B. Connector nut
C. Ferrule
D. O-ring
E. Dewar cover

4. Insert the sample tube through one of the holes in the Dewar cover.
5. Place the sample port nut, ferrule, and O-ring onto the sample tube stem.
6. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the sample port nut onto the analysis port.
7. Repeat for each sample tube.
8. Position the Dewar cover approximately 3/4 in. (19 mm) below the sample port nut.
9. Slide the Psat tube into the Psat slot in the Dewar cover and retighten the Psat tube connector nut.
10. Insert the jacket onto the Psat tube and ensure that the Psat tube jacket is below the Dewar cover.



- If using the TranSeal, refer to the instructions included with the TranSeal [*part number 350-42803-00*].
- If using the Check Seal, see the instructions included with the Check Seal [*part number 350-42802-00*].

DETERMINE THE SAMPLE MASS FOR CHEMISORPTION

Use Quartz Filter Discs for Chemisorption on the next page
Sample Data Worksheet for Chemisorption on page O - 3

Bulb sample tubes are for pellets and other samples without loose particles. Using powder samples in bulb tubes may cause the loose particles to go into the analyzer's exhaust.

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

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.
3. If analyzing a powder or sample made of fine particles, push a piece of quartz wool all the way down into the sample tube.
4. If using *quartz wool*, put a second piece of quartz wool just inside the sample tube.
   If using *filter discs*, push a filter disc down into the tube until it sits on top of the quartz wool. Place a second filter disc just inside the sample tube.
5. Place the sample tube set (sample tube with quartz wool or filter discs and stoppers) on the sample support. Record the stabilized mass on the *Sample Data Worksheet*.

6. Remove the sample weighing support and sample tube set from the balance.
7. Place the sample container on the balance and allow the balance to stabilize at zero.

Do not touch the sample with bare hands. Oil from hands could affect the accuracy of results.

8. Slowly add approximately 0.5 to 1.0 gram of sample to the sample container.
9. If a second piece of quartz wool or filter disc was inserted, remove the top portion of the quartz wool or the filter disc from the sample tube.
10. Use a funnel to slowly pour sample from the container into the sample tube on top of the quartz wool in the tube.

Ensure all sample in the container is placed in the sample tube to avoid errors caused by incorrect sample mass.

11. If using *quartz wool*, insert the top portion of quartz wool into the tube and press it down. If using *filter discs*, insert the filter disc into the tube and press it down.

Ensure the disc is flat on top of the sample. A seal must be created around the edge to prevent the sample from escaping.

12. Wipe the top of the sample tube with a clean, lint-free cloth, such as a Kimwipe®, to remove any quartz wool that may have adhered to the surface.
13. Weigh the sample tube set containing the sample and the stoppers. Record this mass as the *Sample + tube.*

Use Quartz Filter Discs for Chemisorption

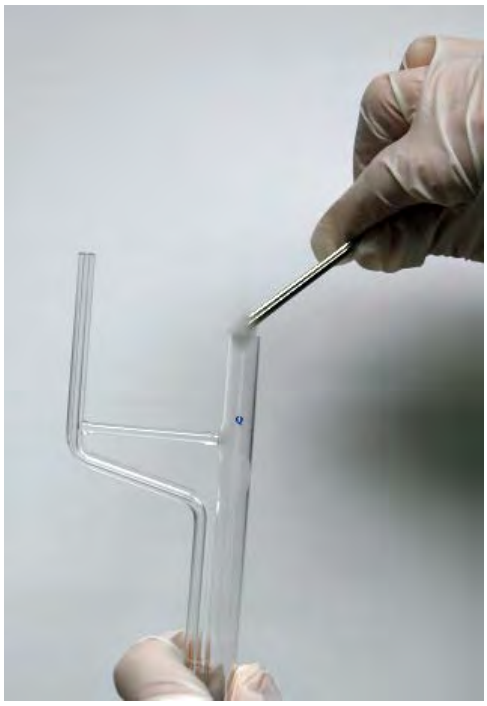
The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

The use of quartz wool is not mandatory; however, it can provide extra protection for light powdered samples.

Wear latex gloves when handling the quartz sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and/or components, preventing a proper seal of the sample tube.

Use quartz filter discs or quartz wool to aid in chemisorption sample preparation. Quartz filter discs (placed both below and above powdered samples) not only provide a more uniform sample surface but also keep the analyzer free of sample debris. The filters can be used up to 900 °C.

1. Insert a small portion of quartz wool into the sample tube to serve as a support for the powdered sample. Use a filler rod or smaller sample tube to push the quartz wool to the bottom of the sample tube.



Insert quartz wool and disc

Top disc, powdered sample, bottom disc, quartz wool

2. Insert a quartz disc into the sample tube and push it into the tube until it rests on top of the quartz wool. Inspect the disc to ensure that there is a good seal and that the sample will not go past the filter. An additional filter can be inserted if needed.
3. Insert a second filter disc on top of the quartz wool. Ensure that the filter is placed high enough into the sample tube for easy retrieval.
4. Take the initial tube weight (with both filters).
5. Remove the top filter disc. Place it on a clean surface, then use a funnel to add the powdered sample on the bottom filter disc.
6. Reinsert the top filter disc into the sample tube, then use a rod or smaller sample tube to push it down until it reaches the top of the sample.
7. To remove the quartz wool and disc after analysis, use the quartz wool extractor tool.

SAMPLE TUBE INSTALLATION FOR CHEMISORPTION

Sample Tube Installation for Physisorption on page 6 - 10

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

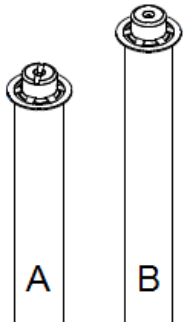
Wear latex gloves when handling the quartz sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and/or components, preventing a proper seal of the sample tube.

Remove the plug from the exhaust port and sample tube port #2

1. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
2. Use a clean, lint-free swab moistened with IPA and wipe the interior rims of the sample and exhaust ports.
3. Use a clean, lint-free tissue moistened with IPA and wipe the O-ring, ferrule, and connector nuts for the sample and exhaust tubes. Place on a clean, lint-free tissue.

Sample and exhaust ports, as well as all components that contact the sample and exhaust ports, must be clean, therefore it is recommended that the previous steps be repeated each time a sample tube is installed onto a port.

4. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
5. If using a hanging filler rod (recommended), hold the sample tube slightly tilted and carefully place the filler rod into the tube.

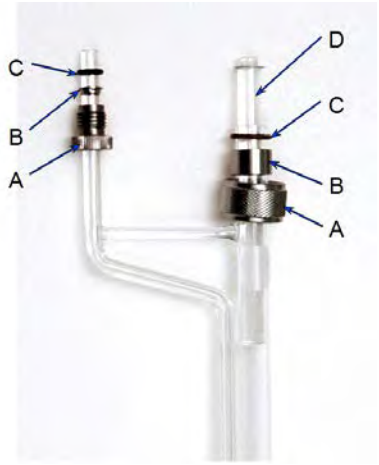


A. Quartz filler rod (for chemisorption analyses
B. Borosilicate glass filler rod (for physisorption analyses)

The quartz filler rod can be used for both physisorption and chemisorption analyses; however, the borosilicate glass filler rod can only be used with physisorption analyses. The quartz filler rod can be identified by the notch across the top of the rod.

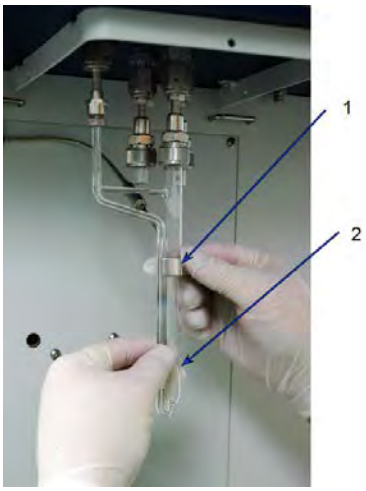
Use of the borosilicate glass filler rod in a chemisorption analysis can cause damage to the instrument.

6. Assemble and install the sample and exhaust tube components.



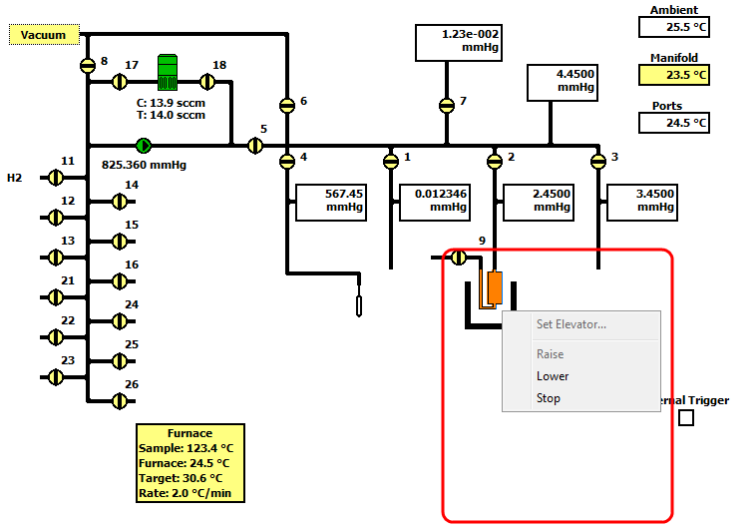
A. Connector nut
B. Ferrule
C. O-ring
D. Filler rod

7. Insert the assembled sample tube into the exhaust port and analysis port #2.
8. Slide the connector nuts up the stems and screw the nuts clockwise to secure the tube in place.
9. Hand tighten both connector nuts until snug.



10. Install clip and slide it up the sample tube.
11. Ensure the thermocouple rests on the lip on the back of the sample tube.

12. Rest the sample thermocouple tip on the lip on the back of the sample tube and install the sample tube clip around the sample tube and thermocouple. Ensure the clip is high enough on the sample tube to clear the furnace disk when the elevator and furnace are raised.
13. Ensure the furnace is on the elevator shelf and manually raise the elevator. To raise the elevator, go to ***Unit [n]*** and ensure there is a check mark to the left of the chemisorption option. If not, select ***Unit [n] > [Chemical Analysis]*** to select it.
14. 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.***



15. On the schematic, right-click the furnace icon and select *Raise* to raise the elevator. If it is necessary to stop the elevator, right-click the elevator icon again and select *Stop*.
16. When the elevator reaches the top, insert the two furnace disk halves on top of the furnace opening.

17. Place the first disk behind the sample tube and the second disk in front of the sample tube, ensuring the clip remains above the furnace disks.

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.
2. Record the mass on the Sample Data Worksheet as *Mass of Sample tube set plus sample (After Degas)*.
3. 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)*.

FILL AND INSTALL THE DEWAR

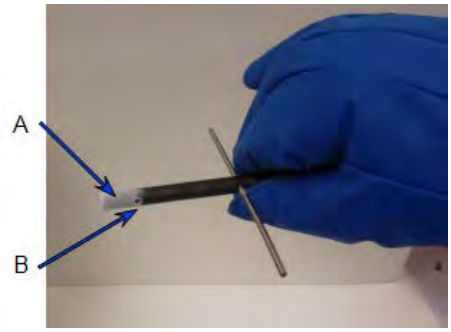
Dewar Precautions on page 6 - 2

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

1. Fill the Dewar with the analysis bath liquid (such as liquid nitrogen) to no higher than 2 1/4 in. (5.7 cm) from the top. Filling the Dewar higher than this will cause an error in the free space measurement.

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

2. Insert the dipstick and check the level of the analysis bath liquid. Condensation should not exceed the level indicator mark.



A. Wetness or frozen condensation indicates bath liquid level
B. Level indicator mark

3. 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.
4. Slide the Dewar cover to approximately 3/4 in. (19 mm) from the sample port nuts to ensure a proper seal on the top of the Dewar.
5. Place the Dewar on the elevator.
6. Attach the safety shield to the brackets on the front of the analyzer.

PERFORM AN ANALYSIS SEQUENCE

Unit [n] > Analysis Sequence

Refill the Dewar during Analysis on page 6 - 43.

If using a cryostat for analysis, see *CryoStat on page D - 1* prior to use.

After running a chemisorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physisorption tubes until the system vacuum level is at 9×10^{-6} mmHg or lower.

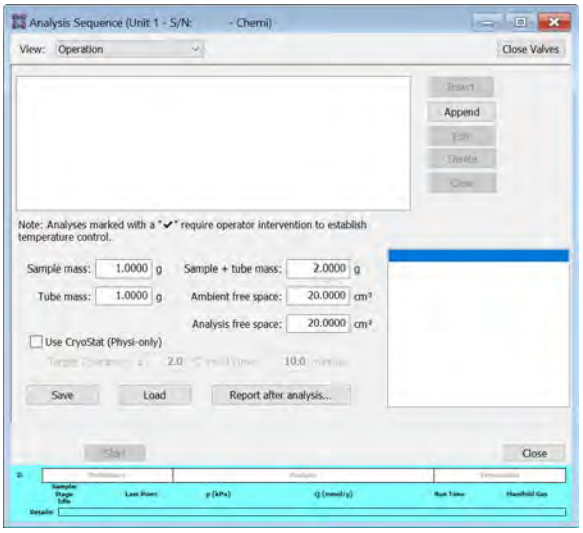
Use to perform a sequence of analyses on port 2. If the *Analysis Sequence* option is selected and a sequence analysis is already in progress, the program continues to run the four steps for the analysis in progress. Files can be added and removed from the sequence while it is in progress.

The analysis sequence can run a:

- sequence of only physisorption files.
- physisorption analysis as a part of the chemisorption analysis sequence.
- physisorption file in any position in the sequence.

The physisorption live graph will be used for the physisorption analysis within a chemisorption sequence.

Operator intervention will be required between analyses in the sequence if the temperature control type changes. Intervention will be required if at the beginning of the analysis.



1. Click **Close Valves**.
2. Click **Insert** and select a sample file.
3. Edit the fields below the sample file selections.
4. Click **Save** to save the selected files as an analysis sequence file (.SEQ) for future analyses.
5. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
6. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

Use caution when removing the sample tube if using a hanging filler rod. The sample tube O-ring or Dewar cover may snag the filler rod retaining ring. Loosen the snag gently; excessive force may break the tip of the filler rod.

Analysis Sequence

Selections	Description
Close Valves [*button***]**	Closes all valves on the unit.
Report after analysis [*button***]**	Generates reports to the selected destination when the analysis is complete.
Sample mass / Sample + tube mass / Tube mass / Analysis free space [*text box***]**	Enter the values for the sample mass and free space.

Analysis Sequence (continued)

Selections	Description
Use CryoStat [*check box*] P	**For cryostat use only**. If using a cryostat, select and enter the *Target Tolerance* and *Hold Time*. The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

PERFORM A BLANK ANALYSIS

Unit [n] > Reference Analysis

Perform a Reference Material Analysis on page 6 - 30
Refill the Dewar during Analysis on page 6 - 43

After running a chemisorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physisorption tubes until the system vacuum level is at 9×10^{-6} mmHg or lower.

The *Blank Analysis* is run in the same manner as a *Reference Material* analysis except a blank method is selected and no sample material is placed in the sample tube.

PERFORM A PULSE CHEMISORPTION ANALYSIS

This topic provides an example of Pulse chemisorption being performed on a sample of Pt/Al_2O_3 with CO. Make the appropriate modifications for the material being analyzed. Platinum Alumina Reference Material can be ordered from Micromeritics. Parts and accessories are located on the Micromeritics web page.

A Pulse chemisorption analysis determines the quantity of active gas irreversibly adsorbed. By applying pulses of known quantity of active gas to the sample, calculations of active surface area, percent metal dispersion, and active particle size can be made for supported metals. The sample is dosed with the analysis gas using the injection loop or a syringe until all accessible active sites are covered. The amount chemisorbed is the difference between the total amount of active gas injected and the amount that does not interact irreversibly with the sample.

The number of injections depends on the quantity in each pulse and total number of active sites present.

LOOP VOLUME

Environmental Defaults for TCD Analyzers on page 2 - 20

The gas quantity in each pulse is determined by active gas concentration, the loop or syringe volume, temperature, and pressure. The loop temperature is controlled by the instrument. The ambient (syringe) temperature and atmospheric pressure are entered by the operator. If a syringe is used, the volume injected is determined by the operator. In general, it is desirable for the sample to require at least two doses of gas, but no more than ten doses, before the reaction ends (although this may vary from lab to lab). Some factors that influence the number of doses required are sample size, the density of active sites, the concentration of active gas, and the size of the loop.

A 0.5 cm^3 loop is provided with the analyzer. There are also 1 cm^3 and 5 cm^3 loops available.

LOOP CALIBRATION

Loop Calibration for TCD Analyzers on page 10 - 15

For the most accurate determination of the injected gas quantity, a loop calibration experiment should be performed. An independent loop calibration can be performed before or after the analysis.

The following example assumes that the loop calibration will be performed after the analysis. A *Loop Calibration* step can be included in a pulse chemisorption analysis.

Preparation	
Pretreatment	Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature. Hydrogen of at least 10% concentration is generally used for reduction.
Analysis	Pulse the loop gas over the sample until the peak area remains constant.
Cold or Sorption Trap	The cold trap does not need to be cooled during the reference material analysis. This may vary depending on application or analysis method. The cold trap tube should remain on the instrument. Including the cold trap in the flow path is optional.
Pressure regulator	Gas cylinders should be set to a level between 10 and 15 psig (69 and 103.5 kPag).
Furnace temperature	Select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Ensure the *Termination* step is set to return the sample temperature to ambient.

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See *Prepare for Analysis on page 6 - 4*.

PROCEDURE

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select either *Pt-Al CO Pulse Chemisorption Loop* or *Pt-Al CO Pulse Chemisorption Syringe* and modify the steps as needed depending on the analysis to be performed.
3. On the *Report Options* tab, select the required report options for the analysis.
4. Save and close the file.
5. Go to ***Unit [n] > Sample Analysis*** and select the sample file saved in the previous step. Chemisorption can only be performed on port 2, therefore Port 2 is the only selection available.
6. Click **Start** to start the analysis.
7. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*. See ***Peak Editor for Dynamic Analysis on page 7 - 10***.

When the analysis ends, the furnace begins to lower the sample temperature to room temperature. When the sample has cooled, open the furnace and remove the sample tube.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

GENERATE THE REPORT

Peak Editor for Dynamic Analysis on page 7 - 10

Open the Peak Editor and ensure that peaks are properly marked.

There are three possibilities for each dose of gas injected during:

- all of the gas is taken up by the sample,
- some of the gas is taken up by the sample, or
- none of the gas is taken up by the sample.

When Pulse chemisorption is properly performed, there will be some injections of each type. When the data is viewed using the Peak Editor, however, only those injections in which some or none of the gas is taken up will appear as peaks. When all of the gas is taken up by the sample, none of it reaches the detector and, therefore, the peak area is zero. These types of peaks are detected automatically by the application and do not require marking.

To generate the report, go to ***Reports > Start Report> [.SMP file*** created above***]*** or click **Preview** and select the signal view.

PERFORM A REFERENCE MATERIAL ANALYSIS

Unit [n] > Reference 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.

Refill the Dewar during Analysis on page 6 - 43

CAUTION

If using a cryostat for analysis, see *CryoStat on page D - 1* prior to use.

After running a chemisorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physisorption tubes until the system vacuum level is at 9×10^{-6} mmHg or lower.

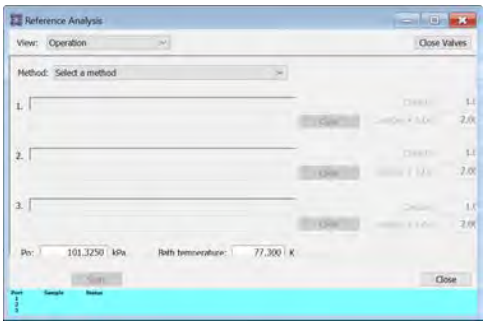
A reference material analysis is used to verify the analyzer is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether quantities are in or out of specification.

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.

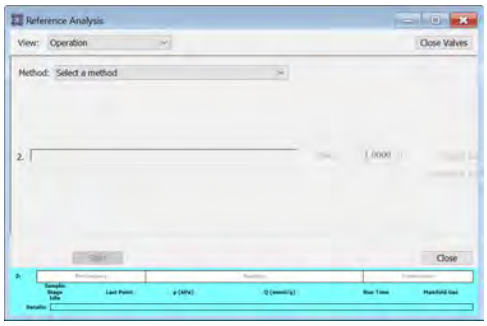
P If using a cryostat, select *Use CryoStat*, enter a *Target Tolerance* and a *Hold Time*. The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.

Flex Physisorption

Flex Chemisorption or Dynamic Analysis

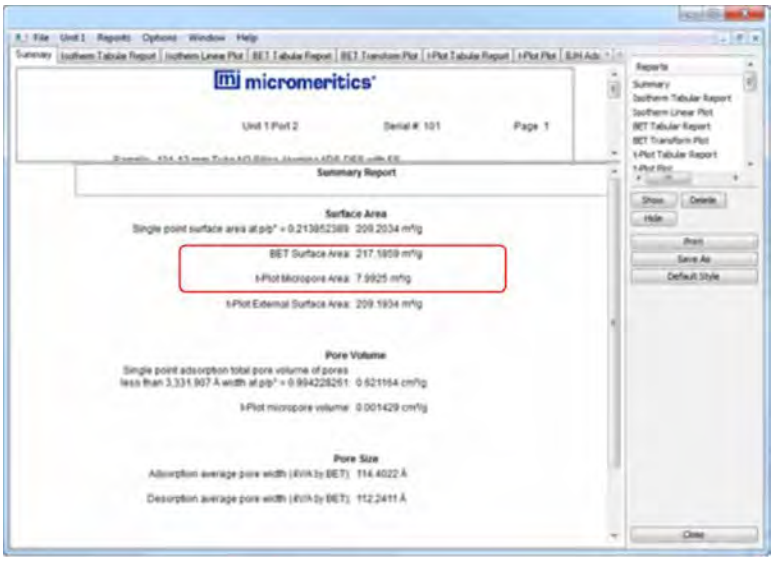


Flex Physisorption



Flex Chemisorption or Dynamic Analysis

If a *Blank Analysis* was selected, look at the *Blank Analysis Report*. The isotherm points should all fall between the minimum and maximum specification lines.



- If the results are within tolerance, the analyzer is operating properly. Click **Close**.
- If the results are not within tolerance, refer to the following *Cause and Action* table. After performing the action, perform the reference material analysis again.

Cause and Action

Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Perform the procedure for cleaning and verifying gas lines, then try again.
The measured free space is too high.	This indicates the helium is not pure enough. Use helium that is 99.999% pure, then try again.

PERFORM A SAMPLE ANALYSIS

Unit [n] > Sample Analysis

Prepare for Analysis on page 6 - 4
Refill the Dewar during Analysis on page 6 - 43

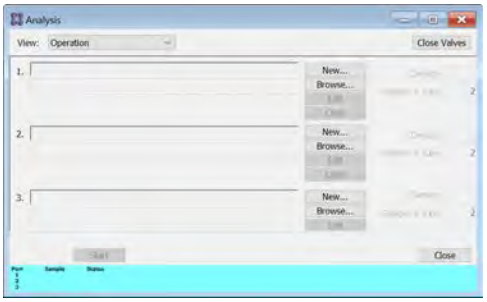
CFR Note

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.

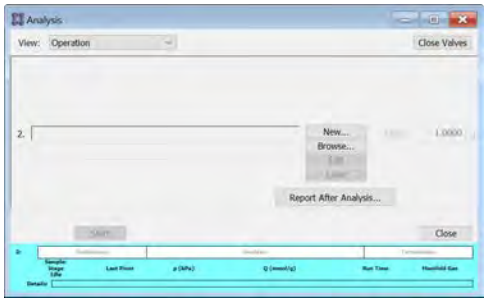
CAUTION

If using a cryostat for analysis, see *CryoStat on page D - 1* prior to use.

After running a chemisorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physisorption tubes until the system vacuum level is at 9×10^{-6} mmHg or lower.



Physisorption



Chemisorption or Dynamic Analysis

P Use to perform up to three analyses with different analysis conditions and/or report options. Sample files can be loaded into ports 1, 2, and 3 allowing one analysis using different analysis conditions to run on each port.

C Use to perform an analysis on port 2.

When **Start** is selected, the selected sample file's analysis conditions will be compared with the port's hardware configuration to verify that the specified analysis is supported by the hardware:

- The minimum target pressure must be no less than the *Minimum Record Pressure* for the minimum range transducer present on the port.
- The selected sample files will be checked for matching adsorptive gases, matching Psat or P_0 gases if measured, and matching backfill gases. All selected gases, except those in a precharged vapor source, must be connected to gas inlets and identified on the *Unit [n] > Unit Configuration > Gas Selections* window. See ***Specify Gas Ports on page 2 - 15***.
- If any selected sample file specifies an *Adsorptive Dose Method* from port 3 and a sample file is selected for port 3, an error message displays indicating the problem and the *Start* window will remain active.

- If *in-situ Degas* is selected for any samples, the operator is prompted to raise the isothermal jackets, then connect and install the degas heating mantle on the sample tubes. If this occurs, the operator will be prompted after degas to remove the heating mantle and properly position both the isothermal jackets and Dewar cover.
- If *Vapor Source Temperature Control* is selected and the vapor heating mantle is not connected, the operator is prompted to install and connect the vapor heating mantle. If *Degas* is selected for any samples, this will occur after the prompt to remove the degas heating mantle. Otherwise, this will occur immediately at the start of analysis.

Click **Next** to schedule additional analyses after the completion of the first series of analyses. The **Next** button displays after the first set of analyses is complete. Samples cannot be removed from or added to ports until the full set of analyses has completed.

1. Click **Close Valves** to manually close all analyzer valves.
2. For a selected port, either click **Browse** and select a sample file, or click **New** to create a new sample file.

P On port 1, up to three sample files may be selected. The files will be loaded into ports 1, 2, and 3 in the order they appear in the file selector.

P A sample cannot be attached to port 3 if port 3 is being used as the vapor source.

C Port 2 is the only available port.

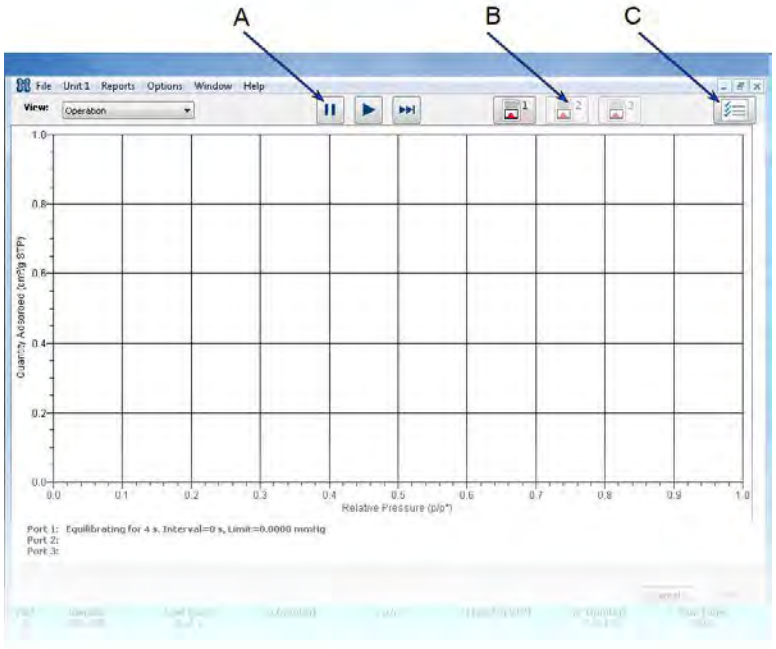
3. Verify the information populated into the sample identification. This information is pulled from the selected or newly created sample file. The *Density* value is applicable only if using the *Calculate* method for the free space determination.
4. P Edit the P_0 and *Bath temperature* fields, if necessary.
5. If using a cryostat, select *Use CryoStat* and complete the *Target tolerance* and *Hold time* fields.

6. Click **Report after analysis** to generate reports automatically when the analysis is complete.
7. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
8. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.

DA When running a dynamic analysis, after starting the analysis several windows display with various warnings, messages, and prompts for the user to perform a specific task. Tasks will depend on the experiments in the current analysis — such as: wait status, manual injections, or calibration file selection.

9. **P** When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

P Use caution when removing the sample tube if using a hanging filler rod. The sample tube O-ring or Dewar cover may snag the filler rod retaining ring. Loosen the snag gently as excessive force may break the tip of the filler rod.



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

Sample Analysis Graph

Selections	Description
Port [*button*]	Generates a report on data being collected . The reports are displayed on the computer monitor only.
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.
Skip [*button*]	Moves to the next step. This button is visible only when an analysis is in progress. Select the ports to skip. In 21CFR11 environments, steps cannot be skipped.
Resume [*button*]	Restarts the suspended application.
Status window	Displays the last point pressure and relative pressure for each port.
Suspend [*button*]	Suspends an analysis in progress.

For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 3*****.**

TEMPERATURE PROGRAMMED DESORPTION ANALYSIS

This topic provides an example of how to perform a Temperature Programmed Desorption analysis using NH_3 on calcium oxalate under helium with a 10 °C/min temperature ramp. Make the appropriate modifications for the material being analyzed.

Temperature Programmed Desorption (TPD) analyses determine the quantity, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows over the sample and reacts with the active sites. (Alternatively, Pulse chemisorption can be used to react with active sites.) Programmed desorption begins when the temperature is ramped linearly over time while a constant stream of inert carrier gas passes over the sample.

At a certain temperature, the heat will overcome the activation energy, breaking the bond between the adsorbate and adsorbent. The adsorbed species will then desorb. If different active metals are present, they usually will desorb the reacted species at different temperatures. The desorbed molecules enter the stream of inert carrier gas and are swept to the detector where the detector response is proportional to the gas concentrations. The quantity of desorbed species, combined with the stoichiometry factor, and the temperature at which pre-adsorbed species desorb, yield the quantity and strength of active sites, respectively.

If TPD is performed after coverage of the active sites by flow or pulse chemisorption, additional information about the distribution of active sites and the strength of active sites is collected.

PREPARATION

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See ***Prepare for Analysis on page 6 - 4***.

PROCEDURE

1. For NH_3 on zeolite, a sample mass of 50 mg ±10 mg should be used. Install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select *Y Zeolite with nitrogen, measure freespace* and modify the steps as needed depending on the analysis to be performed.
3. On the *Report Options* tab, select the required report options for the analysis.
4. Save and close the file.
5. Go to ***Unit [n] > Sample Analysis*** and select the sample file saved in the previous step.
6. Click **Start** to start the analysis.
7. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. See ***Peak Editor for Dynamic Analysis on page 7 - 10***.

When the analysis ends, the furnace begins to lower the sample temperature to room temperature. When the sample has cooled, remove the sample tube when the analysis is complete.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

8. Verify that the peak temperatures are consistent with specifications in the *Reference Material* booklet.

TEMPERATURE PROGRAMMED OXIDATION ANALYSIS

This topic provides an example of performing a TPO on WO_3. Because TPR is often used as the preparation for TPO, the TPR process was included in this example.

Temperature Programmed Oxidation (TPO) examines the extent to which a catalyst can be re-oxidized. Generally, TPO analyses are used to measure the degree of reduction of certain oxides.

Usually the sample is pretreated and the metal oxides are reduced to the base metal. Then the reactant gas is applied to the sample in pulses or (alternatively) as a steady stream. The analyzer measures the uptake of the reactant gas.

TPO is often performed after TPR is performed. When the TPR experiment concludes, the sample is returned to room temperature. Then, the analysis gas is changed to 2-5% O_2 + He. This gas mixture is flowed after the sample is at ambient temperature, then the temperature is ramped up to the same maximum temperature used for the preceding TPR analysis. The portion of the sample that had been reduced is re-oxidized, and the degree of reduction can be calculated.

If the TPR and TPO results are different, there are several possible causes: the sample material sintered such that only a surface oxide (and not a bulk oxide) is formed, or part of the sample was re-oxidized at room temperature while the TCD baseline was stabilizing.

When using any mixture of gases for TPR or TPO analyses, make sure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity, See *Gas Charts on page I - 1*.

Preparation	
Pretreatment	TPR
Analysis	2 to 5% oxygen/helium is flowed through the sample while temperature is ramped, beginning at ambient temperature.
Cold Trap	The cold trap does not need to be cooled during the reference material analysis. This may vary depending on application or analysis method. The cold trap tube should remain on the instrument. Including the cold trap in the flow path is optional.

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See *Prepare for Analysis on page 6 - 4*.

PROCEDURE

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select the analysis conditions file and modify the steps as needed depending on the analysis to be performed.
4. On the *Report Options* tab, select the required report options for the analysis.
5. Save and close the file.
6. Go to ***Unit [n] > Sample Analysis*** and select the sample file saved in the previous step.
7. Click **Start** to start the analysis.
8. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*. See ***Peak Editor for Dynamic Analysis on page 7 - 10***.

When the analysis ends, the furnace begins to lower the sample to room temperature. When the sample has cooled, remove the sample tube when the analysis is complete.

9. As the temperature increases, the sample is oxidized, and the application calculates the volume of oxygen taken up.
10. Allow the TCD signal to return to the initial baseline after the peak has been displayed.
11. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

TEMPERATURE PROGRAMMED REDUCTION ANALYSIS

This topic provides an example of how to perform a TPR analysis of silver oxide. Silver Oxide Reference Material can be ordered from Micromeritics. Parts and accessories are located on the Micromeritics web page.

Temperature Programmed Reduction (TPR) determines the quantity of reducing gas consumed during reaction with the catalyst and reveals the temperature at which reduction occurs. For the reference material analysis, hydrogen (5-10%) is the reducing gas. The following method provides an overview of the steps to successfully analyze the reference material only. For other materials, the sample mass, gas flow rate, temperature ramp, active gas, or active gas concentration may need to be changed. Refer to the reference material booklet and the reference material bottle label for the expected results.

Preparation	
Pretreatment	(Optional step.) Not required for reference material.
Analysis	Flow 5-10% hydrogen/argon or 5-10% hydrogen/nitrogen, while ramping the temperature. The analyzer records hydrogen consumption as a function of temperature. Nitrogen is sometimes used because it may be more economical than argon.
Cold Trap	A cooling of the cold trap is generally needed for the entire analysis. A mixture of liquid nitrogen or dry ice and acetone or isopropanol provides the best performance. A cooling bath with dry ice will last longer than a cooling bath with liquid nitrogen. See step 2 in the following procedure for preparation details.

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See ***Prepare for Analysis on page 6 - 4***.

PROCEDURE

Create Sample Files on page 3 - 2
Peak Editor for Dynamic Analysis on page 7 - 10

1. Obtain the sample mass (refer to the *Silver Oxide Reference Material* booklet for proper mass range), then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Place the isopropyl alcohol in a cold trap Dewar and slowly pour LN_2 into the Dewar while stirring the mixture. Continue to add and stir until the mixture becomes slushy. The mixture must be capable of achieving a temperature of approximately -90 ºC.
3. Place the cold trap Dewar filled with cooling fluid around the cold trap tube. Ensure that the cold trap Dewar contains enough coolant to immerse the lower part of the cold trap tube.

Extreme caution should be used when mixing the IPA/LN_2. See *Dewar Precautions on page 6 - 2*.

4. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select *AgO TPR with H2Ar* .
5. On the *Report Options* tab, select *Summary* in the *Selected Reports* list box.
6. Save and close the file.
7. Go to ***Unit [n] > Sample Analysis*** and select the sample file saved in the previous step.
8. Click **Start** to start the analysis.
9. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*.

   As the temperature increases, the silver oxide is reduced, the water produced by the reaction is collected in the cold trap, and the amount of hydrogen consumed is detected.

   A hydrogen consumption peak which corresponds to the reduction capacity of silver oxide is displayed. Refer to the *Silver Oxide Reference Material* booklet for the maximum peak temperature.
10. When the analysis concludes, remove the sample tube.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

PERFORM A VAPOR ANALYSIS

This option is for physisorption or chemisorption only.

File > Open > [.SMP File]

After running a chemisorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physisorption tubes until the system vacuum level is at 9×10^{-6} mmHg or lower.

A vapor analysis requires that a vapor source container be installed.

Micromeritics offers two methods of installing a vapor source container - one method for analyzers with a shelf support and another method for analyzers without a shelf support. See ***Vapor Source Container Installation on page 11-16***.

1. On the *Adsorptive Properties* window, select *Vapor source* in the *Dosing Method* group box. If running the vapor analysis on port 3, select *From sample port 3* and proceed to Step 3.
2. Set the *Vapor Source Temperature* to the correct value if *Vapor Source* is selected. If sample port 3 is selected, the analysis bath temperature is assumed.
3. Select *Controlled by instrument using heating mantle* if the vapor source is to be automatically heated to this temperature.

If this option is selected for a chemisorption analysis, sample temperature control must be set to *Accessory temperature control*. See *Temperature button* in ***Analysis Conditions for Physisorption and Chemisorption on page 4 - 5***.

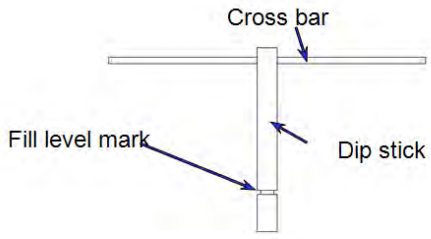
REFILL THE DEWAR DURING ANALYSIS

CAUTION

Always use caution when handling Dewars. See *Dewar Precautions on page 6 - 2*.

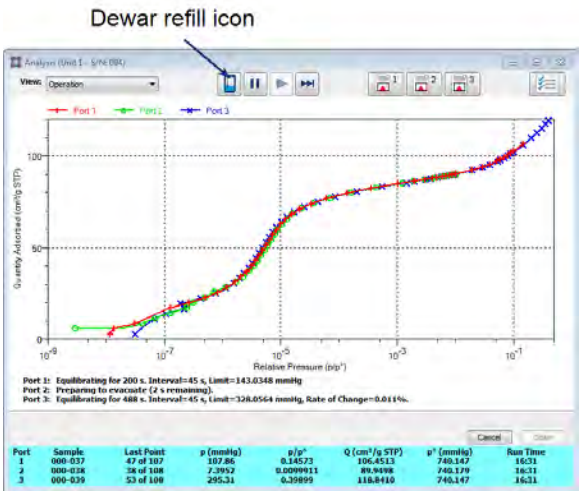
Ensure cryogen is readily available in a suitable delivery system when using this feature. The Dewar will be lowered 2.5 cm, so space for refilling will be limited.

Recommended Tools

Tool	Description
Liquid Nitrogen Transfer Unit [*part number 021-00000-00*]	
Dip Stick [*part number 350-25901-00*] This dipstick is specially designed to easily fit within a 2.5 cm space when the Dewar is lowered.	
Parts and accessories are located on the Micromeritics web page.	

The Dewar refill icon allows the checking and refilling of the Dewar during an analysis. This icon is only enabled during data collection. It is important that the cryogen level always cover the bottom portion of the isothermal jacket during analysis, therefore, the level should be checked within 48 hours of starting an analysis. When using this icon, the operator is prompted when the analysis is paused, the Dewar lowered, and when the analysis resumes. The application automatically ensures that analysis dosing and equilibration will not be disrupted by the refilling procedure.

For ease of use, Micromeritics recommends the use of their Liquid Nitrogen Transfer Unit. This unit has a small delivery tube that easily inserts between the Dewar cover and the top of the Dewar.



1. To lower the Dewar elevator 2.5 cm, click the *Dewar* icon. The following prompt will display:

   The instrument will pause the analysis and lower the Dewar for refilling. This may take up to a minute or so. Have sufficient cryogen prepared to refill the Dewar before continuing.

   [Continue Refill] [Cancel Refill]

   Continue Refill. Click to lower the Dewar elevator 2.5 cm. Continue to the next step for instructions to refill the Dewar.

   Cancel Refill. Click to close the prompt and cancel the elevator movement. Analysis will continue. The remaining steps in this section can be skipped if this button is clicked.

2. If a sample is being dosed with adsorptive, the analysis will pause prior to elevator movement. This pause may take a minute or so then the elevator will lower automatically. The following prompt will display:

   Waiting for analysis to pause. This may take up to a minute or so. When the analysis is paused, the Dewar will be lowered 2.5 cm to allow refill.

   [Cancel Refill]

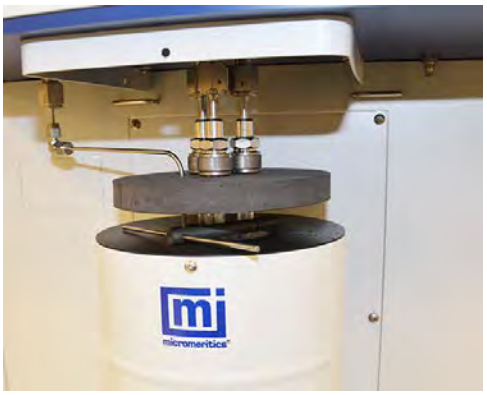
Cancel Refill. Click to close the prompt and cancel the pause. Analysis will continue after equilibration. The remaining steps in this section can be skipped if this button is clicked.

3. When the analysis pauses, the Dewar elevator will lower 2.5 cm. The following prompt will display:

   The analysis is paused. Remove the safety shield and refill the Dewar. When complete, reposition the Dewar cover and safety shield. Then press [Resume Analysis]. The analysis will wait 20 minutes after the Dewar is raised for thermal equilibration.

   [Resume Analysis]

   a. With the elevator lowered, remove the safety shield.
   b. Check the cryogen level using the recommended dip stick. If necessary, slide the Dewar cover upwards slightly to allow a little more room for the dip stick and cryogen delivery tube.
      i. Lay the dip stick horizontally along the top of the Dewar and to the right of the sample tubes on ports 2 and 3.



Slide dip stick against the right side of sample tubes on ports 2 and 3 then pivot dipstick downwards to place in Dewar

      ii. Pivot the dip stick so that the end of the dip stick slides into the Dewar against the right sides of sample tubes on ports 2 and 3. Do not try to insert the dip stick in the middle of the sample tubes. The isothermal jackets will be in the way.
   c. Remove the dipstick and recheck the cryogen level.
   d. To refill the Dewar, insert the cryogen delivery tube into the opening between the Dewar and Dewar cover and fill with cryogen. Check the cryogen level often. When the level is correct, slightly lower the Dewar cover so that it is approximately ½ in. below the top of the sample tubes. It should be low enough so that when the Dewar rises, the lid will rest on the top of the Dewar opening.
   e. When the cryogen level is correct, reinstall the Dewar safety shield.
4. In the application, click **Resume Analysis** on the analysis window. The analysis will resume after a delay for thermal equilibration. Any equilibration that was in process originally will restart automatically.

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7 ABOUT REPORTS

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

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 > Start Report

Generates a report on a sample analysis.

Reports > Close Reports

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

Reports > Open SPC Report

Opens the file selector to open or create an SPC report. See *SPC Report for Physisorption on page 7 - 5*.

Reports > Heat of Adsorption

Opens the file selector to open or create a Heat of Adsorption report. See *Heat of Adsorption Report on the next page*.

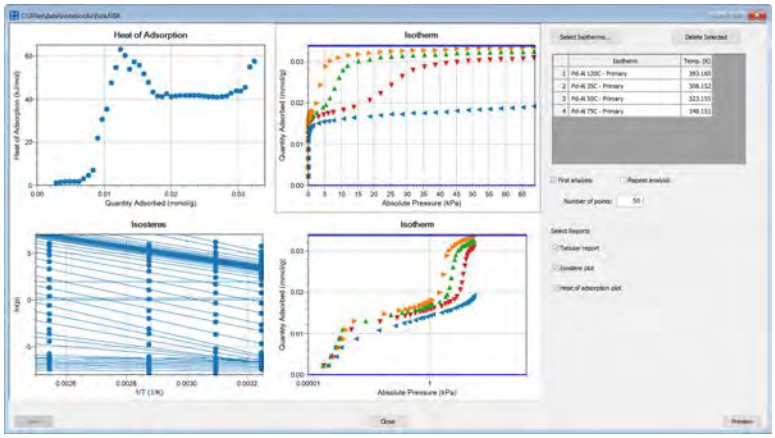
Reports > Open Notebook

Opens the file selector to open or create a Notebook report. See *Open a Report Notebook on page 7 - 7*.

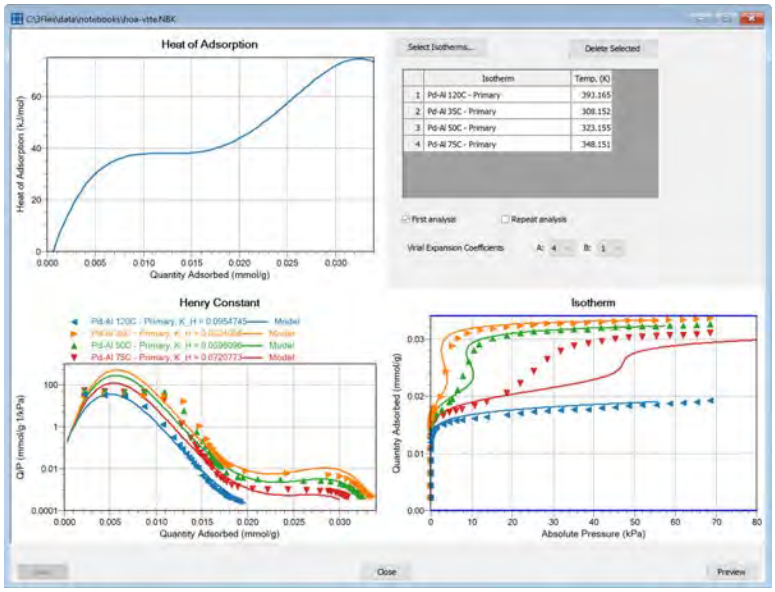
HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption
Reports > Open Notebook

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



Virial Type Thermal Expansion Heat of Adsorption report

Move the blue slider bars in the Isotherm sections of the report to display different values.

When opening an existing *Heat of Adsorption* file, go to ***Reports > Heat of Adsorption*** and select the file with a .HOA file extension.

When a *Heat of Adsorption* file with a .HOA file extension is saved, the saved file will have a .NBK (Notebook) file extension.

Not all items listed in the following table are applicable to every report.

Heat of Adsorption Report

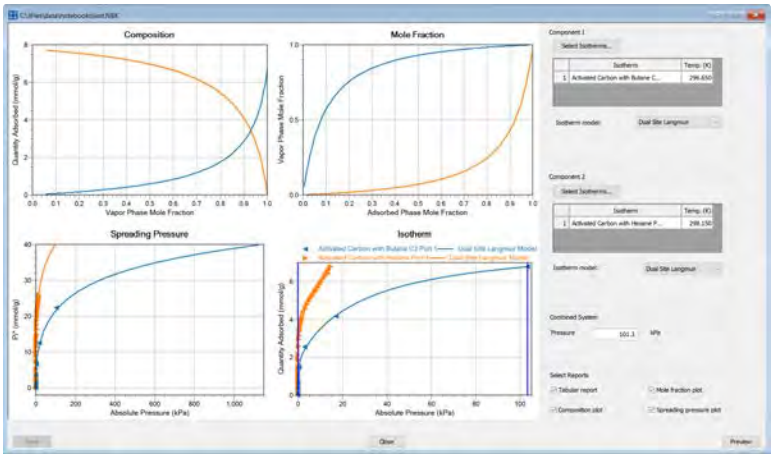
Selection	Description
Combined System [*group box*]	Select to report on mixed gas isotherms.
Delete Selected [*button*]	Deletes the selected Isotherm in the table.
First analysis [*check box*]	Select if this is a first analysis.
Isotherm model [*drop-down box*]	Select the isotherm model to be used in the report.
Repeat analysis [*check box*]	Select if this is a repeat analysis.
Select Isotherms [*button*]	Click to add or remove Isotherms in the report.
Select Reports [*group box*]	Select the reports to include in the analysis.
Virial Expansion Coefficients [*drop-down box*]	Select the A and B virial expansion coefficients to be used in the report.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

IDEAL ADSORBED SOLUTION THEORY (IAST) COMPOSITION REPORT

Reports > Open Notebook > [*.NBK file]

This report applies two separate gas adsorption analyses to a sample material. Each analysis involves a different adsorbing gas species resulting in an experimental isotherm for each gas. The calculation then uses Raoult's Law to predict the behavior of these two gas components when they are adsorbing together in a mixed gas system. The primary prediction of the report is quantity adsorbed of both species of gas as a function of the relative concentrations of the two gases in the gas phase when in equilibrium.



Move the blue slider bars in the Isotherm sections of the report to display different values.

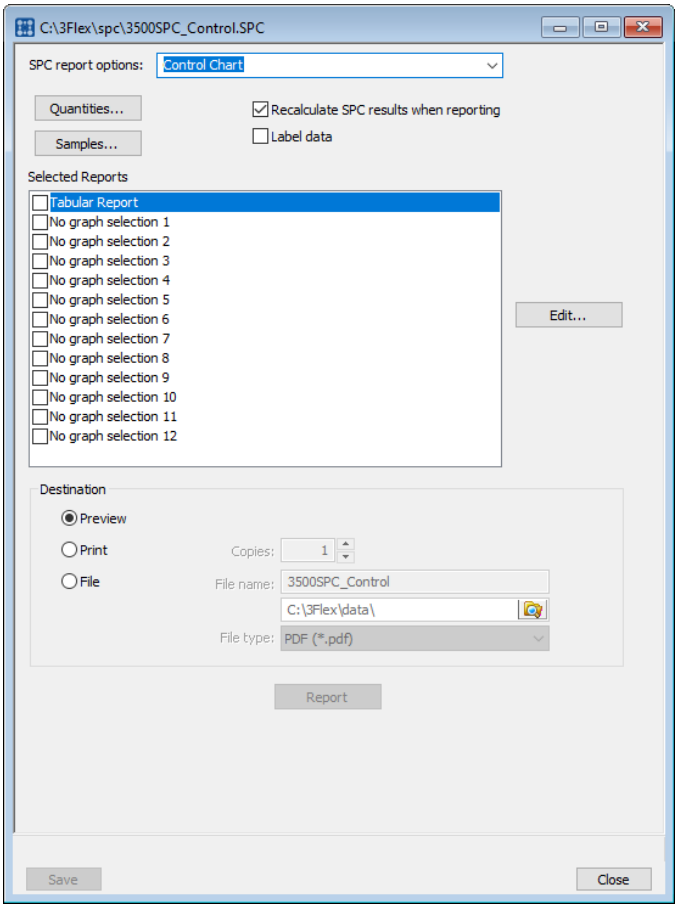
Ideal Adsorbed Solution Theory (IAST) Composition Report

Selection	Description
Combined System [*group box*]	Select to report on mixed gas isotherms.
Isotherm model [*drop-down box*]	Select the isotherm model to be used in the report.
Select Isotherms [*button*]	Click to add or remove Isotherms in the report.
Select Reports [*group box*]	Select the reports to include in the analysis.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

SPC REPORT FOR PHYSISORPTION

Reports > SPC Report Options



- SPC (Statistical Process Control Report)

  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.

- 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.

- Control Chart Report

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

SPC Report Table

Selection	Description
Quantities [*button***]**	Click to select SPC quantities to include in the report.
Samples [*button***]**	Click to select sample files to include in the report.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

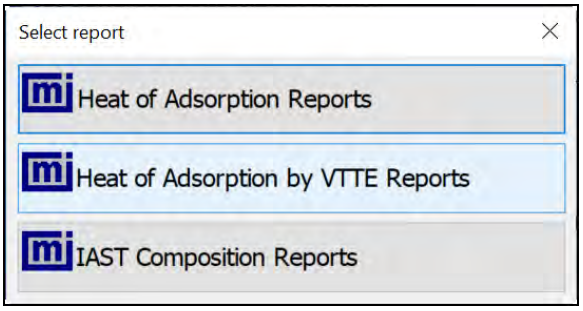
OPEN A REPORT NOTEBOOK

Reports > Open Notebook

File > Open Notebook

Opens the file selector to open or create a *report Notebook.*

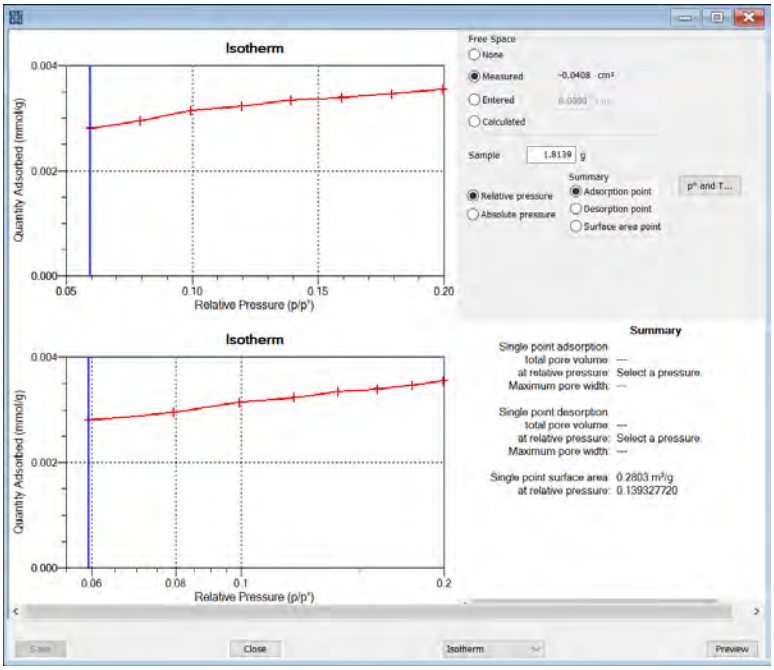
When creating a new report Notebook file using ***File > Open Notebook***, enter the name of the new file and select the type of report from the popup window.



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.
   - Edit the Isotherm Linear Plot to include or omit the data point from the BET 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.

PEAK EDITOR FOR DYNAMIC ANALYSIS

Analysis condition defaults for metal stoichiometry factors can be set using the sample information metal table editor. Stoichiometry factors can also be set for each pulse chemisorption experiment using the analysis conditions experiment step editor. If required, stoichiometry factors for a completed pulse chemisorption experiment can be viewed and modified using the peak editor's stoichiometry settings window.

The *Peak Editor* feature provides the viewing and editing of up to 16 dynamic analysis experiments. The *Peak Editor* options are accessed by opening a Completed sample file and selecting a "Peak Editor" entry from the drop-down menu at the bottom of the report window. Peaks can be defined, edited, or deleted.

Peaks are defined by a baseline. If the **Find All Peaks** button is clicked (enabled when *Edit Peaks* is selected), the *Peak Editor* will define baselines for all positive peaks detected according to the *Integration* window accessed from the **Integration** button. For Pulse experiments and Loop calibrations, peaks are found using events that occurred during the analysis — such as the time an injection started — instead of peak detection options. The baseline can be manually defined by double left clicking in the signal graph on the starting baseline point (this places the Peak Editor into baseline creation mode). The ending baseline point is then defined by left clicking in the signal graph on the ending baseline point. Baseline creation mode can be exited by right clicking in the signal graph.

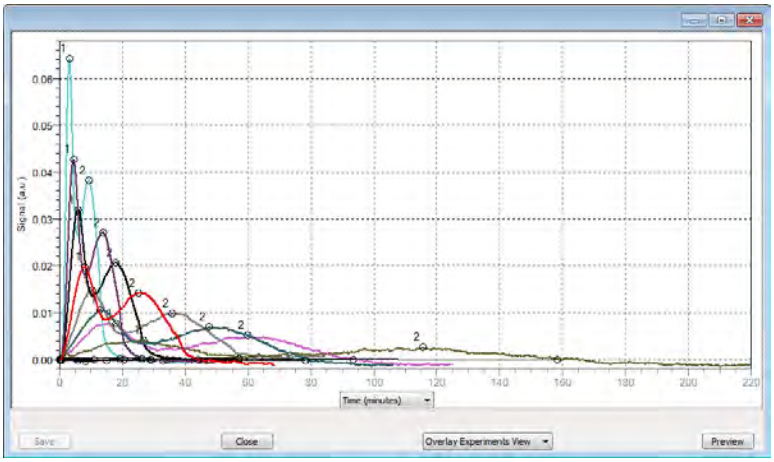
PEAK EDITOR VIEW

Open a Sample File on page 3 - 6

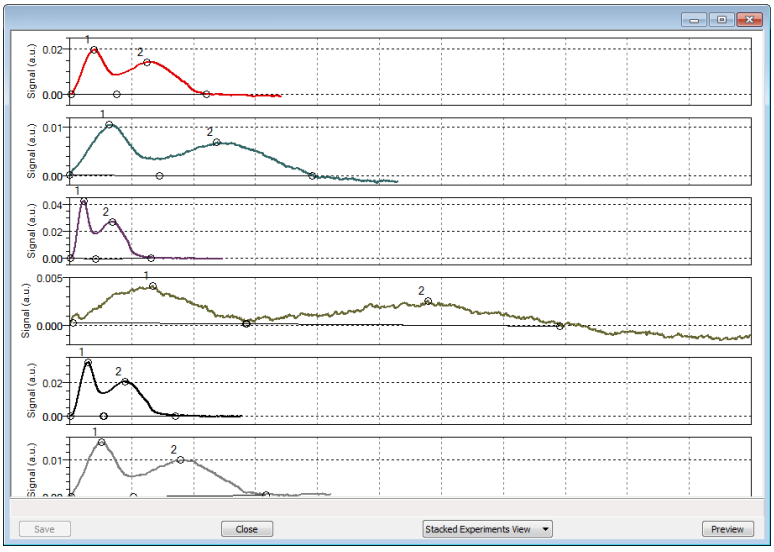
When a sample file with a *Complete* status is opened, three views are available:

- Overlay Experiments View
- Stacked Experiments View
- Peak Editor

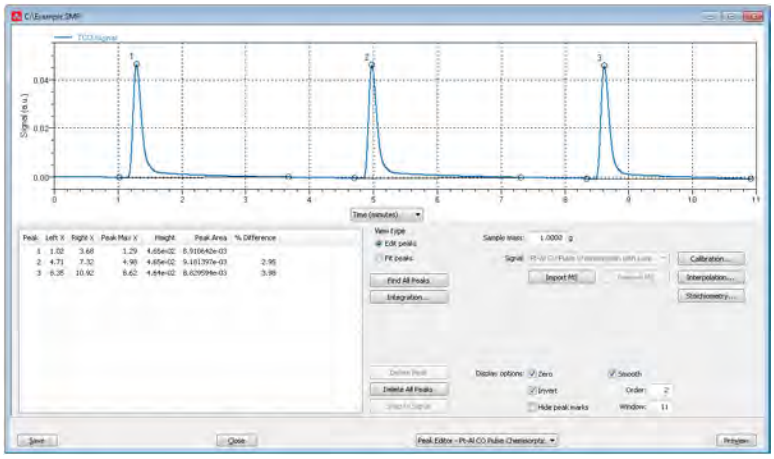
To change the view, select the view from the drop-down list at the bottom of the graph window. Only the *Peak Editor* view allows editing of the experiment.



Example of Overlay Experiments view

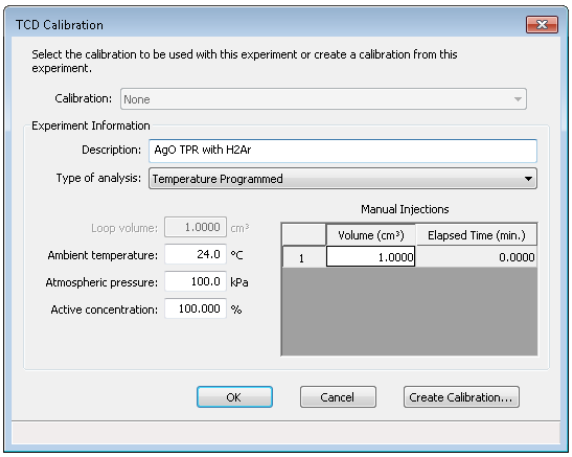


Example of Stacked Experiments view



Example of Peak Editor view

Peak Editor

Selections	Description
Calibration [*button*]	

Calibration. Select a previously defined calibration file. If the experiment and calibration gases differ, a warning displays. The system also compares the experiment flow rate and calibration flow rate. If they differ, a warning displays.

Experiment Information

- **Description.** Enter a description of the current experiment.
- **Type of analysis.** Select the analysis type that most closely describes the current experiment. This will affect what is reported as part of the Summary report data and the available reports.
- **Vapor.** Select a vapor before creating or applying a vapor calibration. This field is only visible for vapor experiments.
- **Loop volume.** Displays the volume of the gas injection loop.

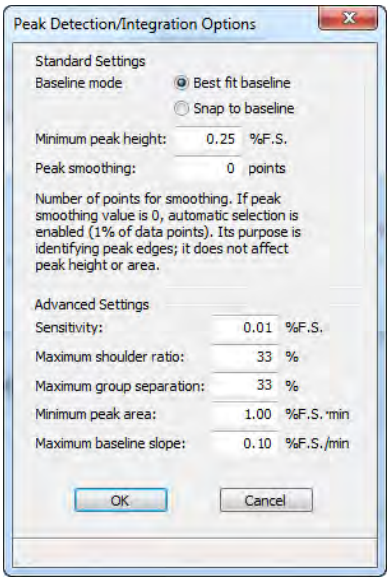
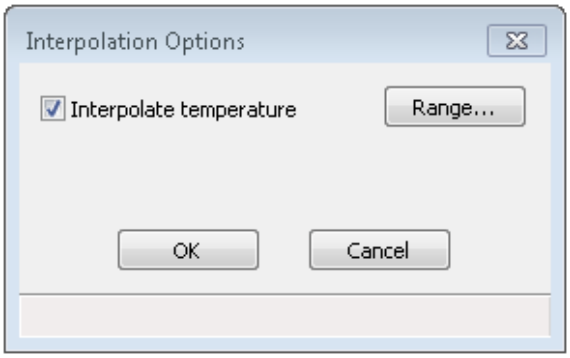
Peak Editor (continued)

Selections	Description
	■ **Ambient temperature**. Displays the ambient temperature entry from the ***Options > Environmental Defaults*** setting. This field may be edited. ■ **Atmospheric pressure.** Displays the atmospheric pressure entry from the ***Options > Environmental Defaults*** setting. This field may be edited. ■ **Active concentration.** Displays the percent of the gas mixture which is composed of the reactive gas versus an inert filler. This value is used for data reduction, including pulse chemisorption. ■ **Adsorbate cross-sectional area.** Displays the area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations. ■ **Manual Injections.** Displays the injection settings entered during analysis. This table may be edited. **Create Calibration.** Create a signal calibration from the currently selected experiment.
Delete All [*button***]** C **Delete All Peaks [***button***]** **Delete Peak [***button***]**	Clears all peaks from the table and removes all markings from the peaks. To delete a single peak, select the peak from the peak table or left click the peak to enable the **Delete Peak** button. **Delete All** is available only when the *Fit Peaks* selection is enabled. **Delete All Peaks** is available only when the *Edit Peaks* selection is enabled. **Delete Peak** is available only when the *Fit Peaks* selection is enabled.
Delete Baseline [*button***]** C	Removes the baseline from the graph. Available only when the *Fit Peaks* selection is enabled.
Display Options [*group box***]**	Use to change how the data are displayed. **Zero.** Select to zero the signal (starting baseline). **Invert.** Select to invert the signal (peak). **Hide Peak Marks.** Select to hide all marks from peaks. **Smooth.** Select to smooth the signal on the display.

Peak Editor (continued)

Selections	Description
	■ **Order** and **Window**. Enabled when the *Smooth* checkbox is selected. The smoothing process uses the Savitzky-Golay filter to fit a polynomial order *n* into size of the specified window [*m*].
Find All Peaks [*button*]	Defines baselines for all positive peaks detected according to the *Integration* window, accessed via the **Integration** button. Pulse experiments and Loop calibrations do not use **Integration** button settings when finding peaks. **Find All Peaks** automatically detects the peaks and draws the baseline for detection. Place the cursor over one of the baseline end points and double left click to grab the baseline. Move the cursor to the new position and right click. Available only when the *Edit Peak* selection is enabled.
Gaussian [*drop-down box*] C	Standard Gaussian curve. Available only when the *Fit Peaks* selection is enabled.
Import M.S. [*button*] **Remove M.S.** [*button*]	Mass spectrometer data can be imported and overlaid in the *Overlay Experiment* view. In the *Peak Editor* view, it is saved as a separate signal. **Import M.S.** Click to import mass spectrometer data. In the bottom right-hand corner of the pop-up window, select the type of mass spectrometer file to import (Quadera, Quadstar, MKS, or TAMS). Select the file, then click **Open** to import the signals. A popup window prompts the user to sync the temperature data with the current experiment temperature data. **Remove M.S.** Click to remove all previously imported mass spectrometer signals from the current experiment.
Import standard peaks [*button*] C	Imports the saved peak parameters from the *Edit Peaks* view. Available only when the *Fit Peaks* selection is enabled.

Peak Editor (continued)

Selections	Description
Integration Options [*button*] C	 See *Peak Detection / Integration Options on page K - 1*.
Interpolation [*button*]	**Interpolate Temperature.** Select to interpolate the temperature. Click **Range** to specify a temperature range. Slide the blue bars on the graph to indicate the range. 
Log Normal Skewed [*button*] C	A 4-parameter, log-normal shape that allows for skewed peaks. This is the default peak. Available only when the *Fit Peaks* selection is enabled.
Peak fit model [*button*] C	Select the peak shape. Available only when the *Fit Peaks* selection is enabled. Options include: Gaussian, Lorentzian, Pearson type VII, Pseudo-Voight, and Log Normal Skewed (3 and 4).
Sample mass [*text box*]	Calculates the mass-specific quantities. Initial value is taken from the sample information at the start of the analysis.

Peak Editor (continued)

Selections	Description
Snap to Signal [*button*] C	Places the selected baseline point on the signal curve.
Stoichiometry [*button*] C	See *Active Metals for Chemisorption Analyzers on page 3 - 12*.
View Type [*group box*] C	**Edit Peak.** Locates peaks via signal integration over a baseline. **Fit Peaks.** Locates peaks via function fitting to minimize residual over a baseline.
x-axis [*drop-down box*]	Change the x-axis to display as time or temperature. This feature is available in *Stacked Experiment* view, *Overlay Experiment* view, and *Peak Editor* view. In *Peak Editor* view, the *Edit Peak* selection must be enabled. **Time (minutes).** Select to display the x-axis as minutes. **Temperature.** Select to display the x-axis as temperature.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

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' static volumetric physisorption analyzers and suggests ways by which the merit of the reported information may be evaluated.

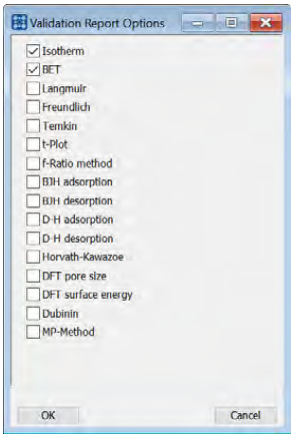
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the 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.

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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

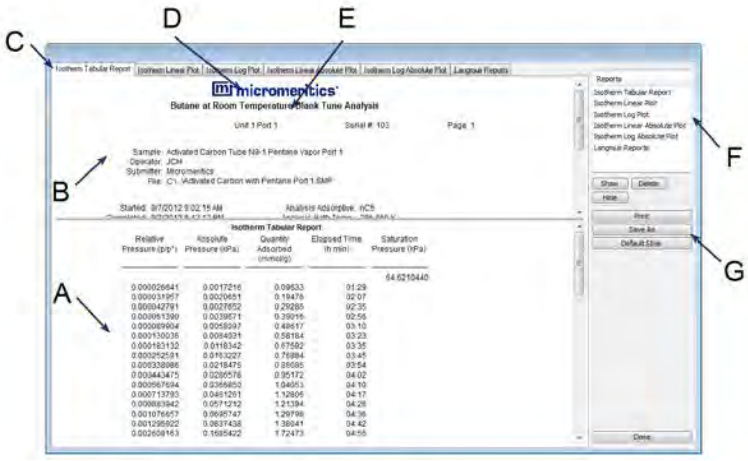
In general, this method visualizes the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step the quantity of adsorptive involved is divided between pore-emptying and film-thinning processes and is accounted for totally. This computational 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.



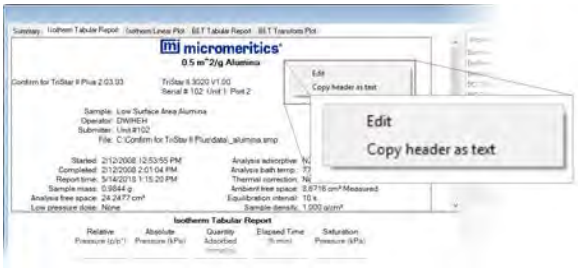
A. Data display (graph or text)
B. Header
C. Generated tabs
D. Graphic
E. Title
F. List box
G. Toolbar

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.

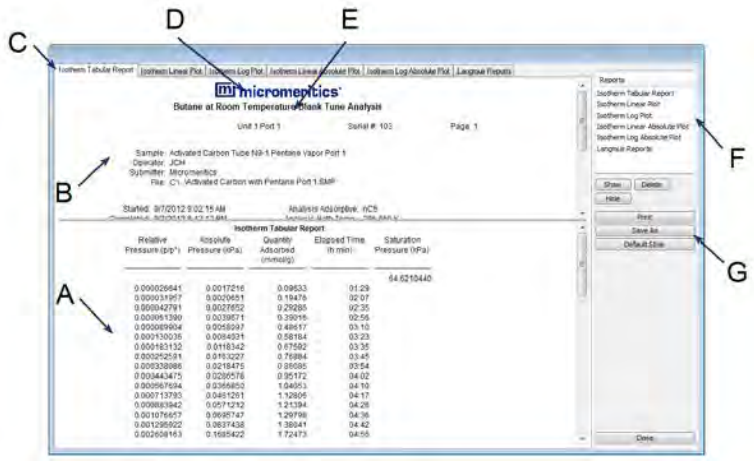


Report Header Shortcuts

Selections	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

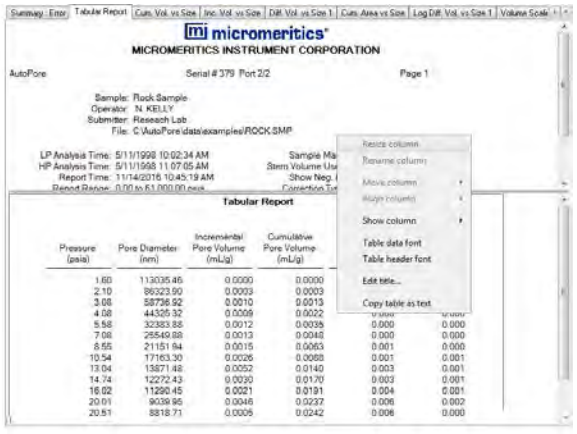
Report Toolbar

Selections	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 - 3*.

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

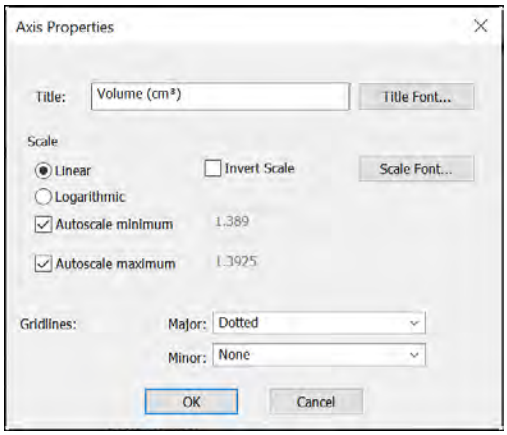
Selections	Description
Align column	Changes the column alignment to either left, right, or centered.
Copy table as text	Copies the report contents to the clipboard as tab-delimited text. It can then be pasted into another document.
Edit title	Edits the report title and/or title font attributes. Click **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 - 3*.

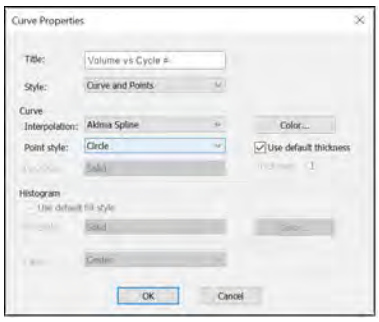
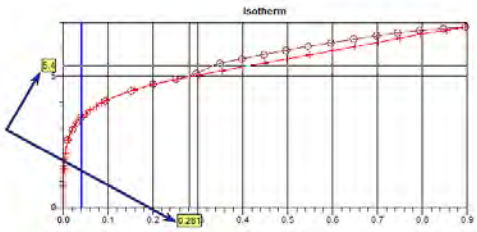
GRAPH FEATURES AND SHORTCUTS

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

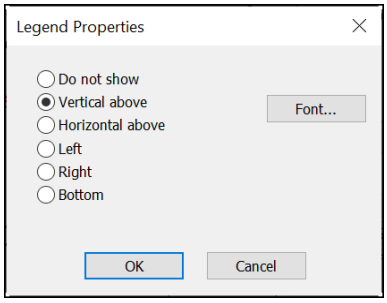
Graph Shortcut Options

Selections	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.
Delete baseline DA	Deletes the baseline from the graph
Delete peak DA	Deletes the selected peak from the table.
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. **Signal (a.u.).** DA Converts the signal from calibrated units to arbitrary units.

Graph Shortcut Options (continued)

Selections	Description
	Title. Edits the selected axis label. **Title font.** Modifies the font for the selected axis label. Deselect *Use 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.

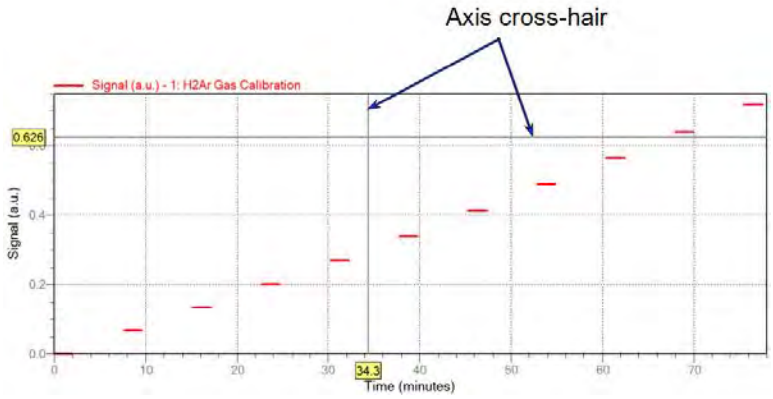
Graph Shortcut Options (continued)

Selections	Description
Edit legend	Changes the legend location and font. 
Edit title	Changes the report title.
Enable overlays PSC	If overlays have been selected, this option displays (or hides) the overlays.
Hide Peak Marks DA	Removes all peak marks from the graph.
Include report PSC	When selected, places a checkmark to the left of the report in the *Select Reports* list box on the *Report Options* tab.
Insert Peak DA	Inserts a peak into the graph at the selected point.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Select overlays... PSC	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 - 3*.

Axis Cross-Hair

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



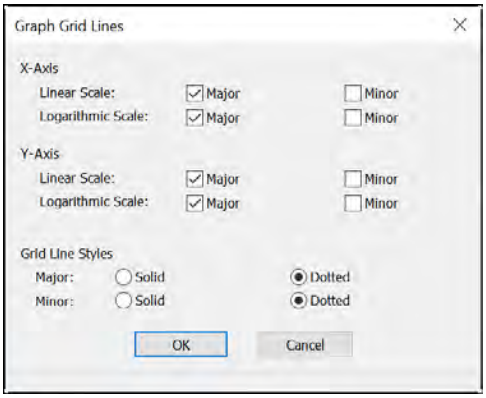
Example of Axis cross-hair feature

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

Selections	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 logarithmic 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 - 3*.

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

GENERATE PORE SIZE DISTRIBUTION GRAPH OVERLAYS

The following reports in the physisorption applications can produce graphical results for a sample material's pore size distribution:

Two methods can be used to import and overlay report data into another interactive graph using shortcut menu options:

- **Import ASCII text data.** Data can be imported from an ASCII text file into the interactive graph. The ASCII text file must follow certain rules.
- **Copy/paste.** Data can be copied from one sample file (source) and pasted into another sample file (target).

Pore size distribution report overlays menu selections are:

- Copy data
- Paste data
- Edit Imported Data
- Display Imported Data

IMPORT ASCII TEXT DATA

ASCII text file format rules

- The header must consist of one line to include title, two unit specifications, and distribution type:
  - Accepted pore dimension units are: A, nm, um
  - Accepted pore volume units are: cm^3/g, cm^3/g, ml/g
  - Accepted distribution types are: cumulative, incremental

  Two examples of a header format:

  My Title (A, cm^3/g, incremental)
  My Title (A, cm^3/g, cumulative)

- The data must be in two columns and should be separated by a comma or white space.
- The data lines must be ordered so that pore dimensions are monotonically increasing or decreasing.

Sample ASCII Text File

```
silica alumina bjh (A, cm3/g,
cumulative)

456.657          0.0133559
444.847          0.0546427
429.168          0.0869924
425.419          0.119721
419.629          0.132681
360.634          0.156611
340.859          0.197672
326.601          0.233092
```

Window appearance will vary depending on the selected report. This function can be performed on samples files with a *Complete* status or during an analysis.

1. Create the ASCII text file using the proper format as indicated above.
2. In the analyzer application, go to ***File > Open***.
3. Select a sample file to overlay graphs on to.
4. Click **Open** (or double-click the file name).
5. Right-click in the graph area and select *Edit imported data*.

6. In the *Select Imported Overlays* window, if the ASCII text file does not display, click **Import** to locate the file.
7. Select the ASCII text file in the *Select Imported Overlays* window, then click **OK**. If an error message will display instead, verify that the .TXT file format is in the correct format.
8. To include the overlay data in a printed report, see *Print Pore Size Distribution Overlay Data in Reports*.

OVERLAY PORE SIZE DISTRIBUTION DATA USING COPY/PASTE

1. Open a source sample file and a target sample file; both should have a *Complete* status. The report will open to the interactive reports window.
2. In the source sample file, right-click on the graph and select *Show Curve*.
3. Deselect any differential curve data to hide them in the graph.
4. Right-click in the graph area again and select *Copy Data*.
5. Change to the target sample file, right-click the graph, and select *Paste data*. The graph now displays overlayed data from the source sample file.

   Typically, one new graph will appear showing both the cumulative and differential curves. To show or hide individual curves, right-click the graph and select (or deselect) *Display imported data*.

6. Ensure that all parameter fields are set appropriately, then click **Paste**.
7. To include the overlay data in a printed report, see *Print Pore Size Distribution Overlay Data in Reports.*

Print Pore Size Distribution Overlay Data in Reports

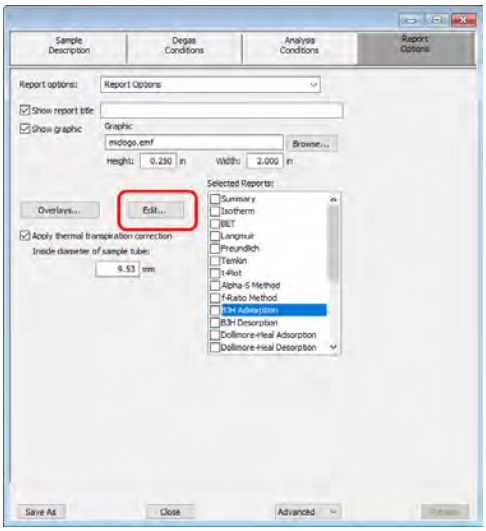
1. Open the sample file containing the overlay data and select *Advanced* from the view selector drop-down list at the bottom of the window.
2. Click the *Report Options* tab.
3. In the *Selected Reports* list box, select the cumulative, differential, or incremental intrusion graph to show the imported distribution data, then click **Edit** (or double-click the selected report).
4. In the *Overlay* drop-down box, select *Imported*.
5. Click **OK** to close the window.
6. Click **Preview** on the *Report Options* tab.
7. Click **Print** in the reports toolbar section to display print options.

OVERLAY MULTIPLE PHYSISORPTION 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.



For Physisorption Reports

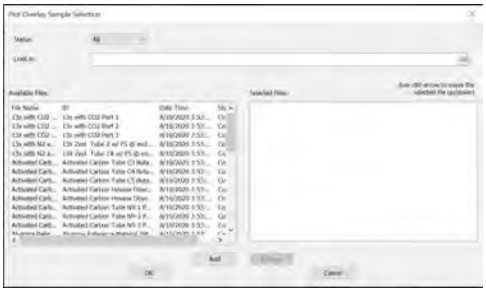
If overlaying this type of report...	Then...
■ Isotherm	a. On the *Isotherm Report Options* window, select one or more plots in the *Selected Reports* group box, then click **Options** to the right of the selected plot. b. On the *Plot Options* window, select *Plot curve* and/or *Plot points* if they 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.

For Chemisorption Reports

If overlaying this type of report...	Then...
■ Difference Method ■ Freundlich ■ Langmuir ■ Sinfelt Method ■ Temkin	a. On the pop-up window, select *Overlay samples*. b. Verify other fields. c. Click **OK**. d. Continue to Step 5.

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

Selections	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*]	Changes the file folder location. Click the **Browse** icon.
Selected Files [*selection*]	Lists the files selected to be combined. Click **Remove** to move a file back to the *Available Files* list box. Click **OK** to combine the files.
Status [*drop-down box*]	Selects the status of files to be combined.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

Import ASCII Pore Distribution Data

Manually Enter Data on page 3 - 8

Import an ASCII Text File using Graph Shortcuts

1. Create an ASCII text file.
2. Open a report with a *Complete* status.
3. Select a pore-size distribution report from the view selector drop-down list at the bottom of the window.
4. Right-click on the graph and select *Edit imported data* on the shortcut menu.
5. If the ASCII text file does not display on the *Selected Imported Overlays* window, click **Import**.
6. Locate and select the file, then click **Open**. Header information from the ASCII text file will appear in the *Select Imported Overlays* window.
7. Select the entry, then click **OK**. If an error message appears, verify that the .TXT file format is correct.
8. To hide or show imported data, right-click in the graph area and use the *Display imported data* option on the shortcut menu.

Copy/Paste an ASCII Text File using Graph Shortcuts

1. Create an ASCII text file.
2. Copy the ASCII text data to the clipboard.
3. Open a report with a *Complete* status.
4. Select a pore-size distribution report from the view selector drop-down list at the bottom of the window.
5. Right-click on the graph and select *Paste data* on the shortcut menu.
6. To hide or show imported data, right-click in the graph area and use the *Display imported data* option on the shortcut menu.

Copy/Paste Graph Data from Another Graph

1. Open a source pore distribution data report with a *Complete* status.
2. Right-click on the graph and select *Copy Data* on the shortcut menu.
3. Open the target pore distribution data report.
4. Right-click on the graph and select *Paste Data* on the shortcut menu.
5. To hide or show imported data, right-click in the graph area and use the *Display imported data* option on the shortcut menu.

REPORT EXAMPLES

BET SURFACE AREA

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Unit 1 Port 2 Serial #: 101 Page 9

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started: 8/17/2012 8:44:16 PM
Completed: 8/18/2012 6:33:42 PM
Report Time: 8/30/2012 6:34:58 AM
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³
Low Pressure Dose: None
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm³ Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm³

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

BET Surface Area Report

BET Surface Area: 217.1859 ± 0.2311 m²/g
Slope: 0.019839 ± 0.000021 g/cm³ STP
Y-Intercept: 0.000205 ± 0.000003 g/cm³ STP
C: 97.887751
Qm: 49.8911 cm³/g STP
Correlation Coefficient: 0.9999977
Molecular Cross-Sectional Area: 0.1620 nm²

Relative Pressure (p/p°)	Quantity Adsorbed (cm³/g STP)	1/[Q(p°/p - 1)]
0.077824186	48.2830	0.001748
0.106574940	51.3824	0.002322
0.135877231	54.2113	0.002901
0.163237219	56.6908	0.003441
0.188595088	58.9330	0.003944
0.213652389	61.1303	0.004450

BET Surface Area Plot

micromeritics®

Unit 1 Port 2 Serial #: 101 Page 10

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started: 8/17/2012 8:44:16 PM
Completed: 8/18/2012 6:33:42 PM
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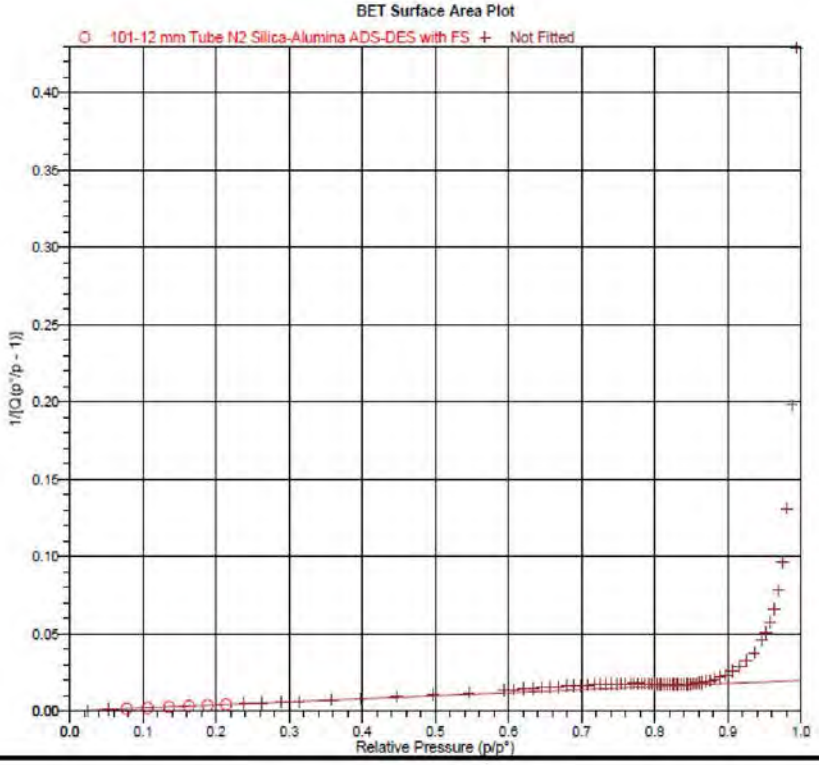
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³
Low Pressure Dose: None
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm³ Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm³

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



BJH DESORPTION: CUMULATIVE PORE VOLUME

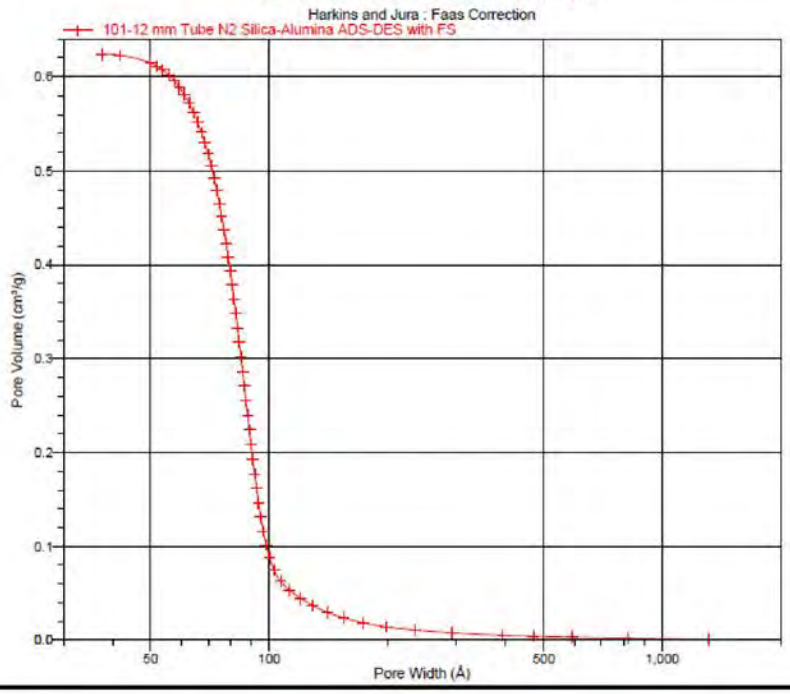
micromeritics®

Unit 1 Port 2 Serial #: 101 Page 24

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



BJH Adsorption: Cumulative Pore Volume

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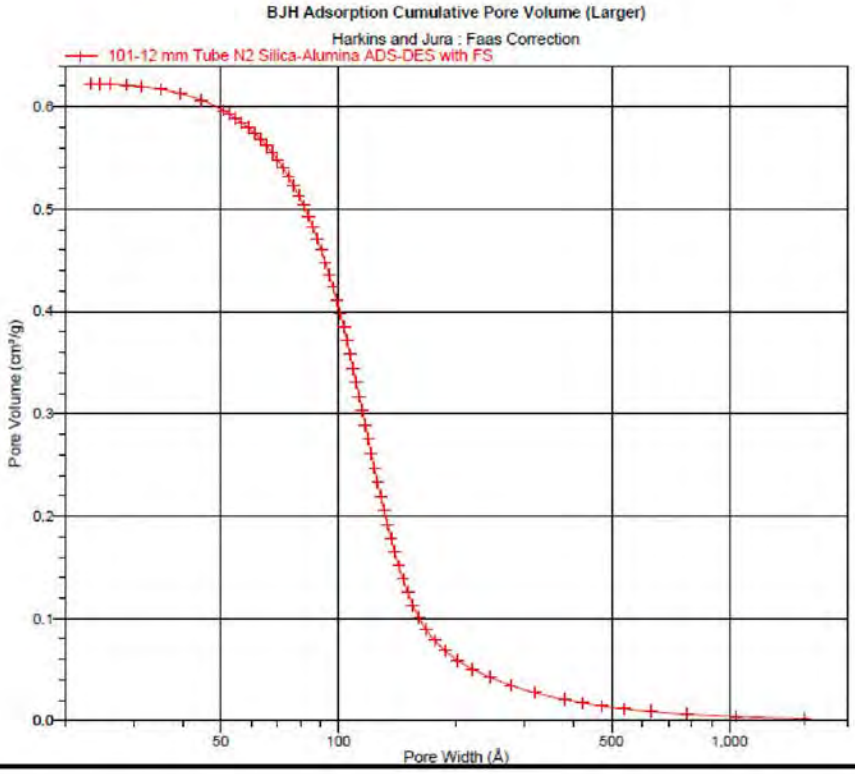
Unit 1 Port 2 Serial #: 101 Page 16

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started: 8/17/2012 8:44:16 PM
Completed: 8/18/2012 6:33:42 PM
Report Time: 8/30/2012 6:34:58 AM
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³
Low Pressure Dose: None
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm³ Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm³

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



ISOTHERM LINEAR PLOT

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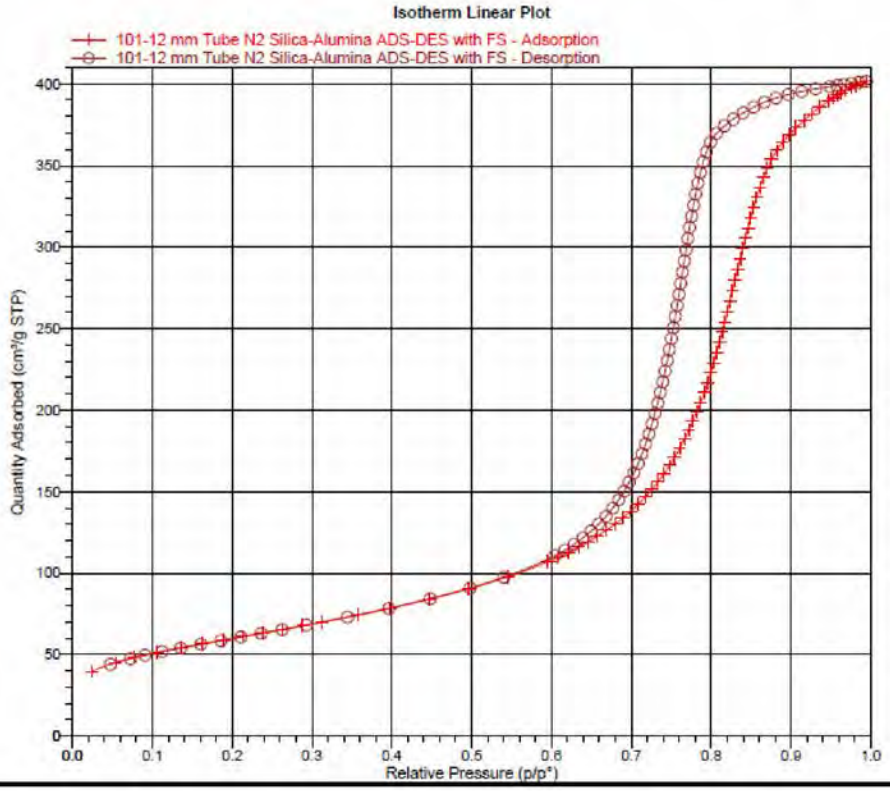
Unit 1 Port 2 Serial #: 101 Page 8

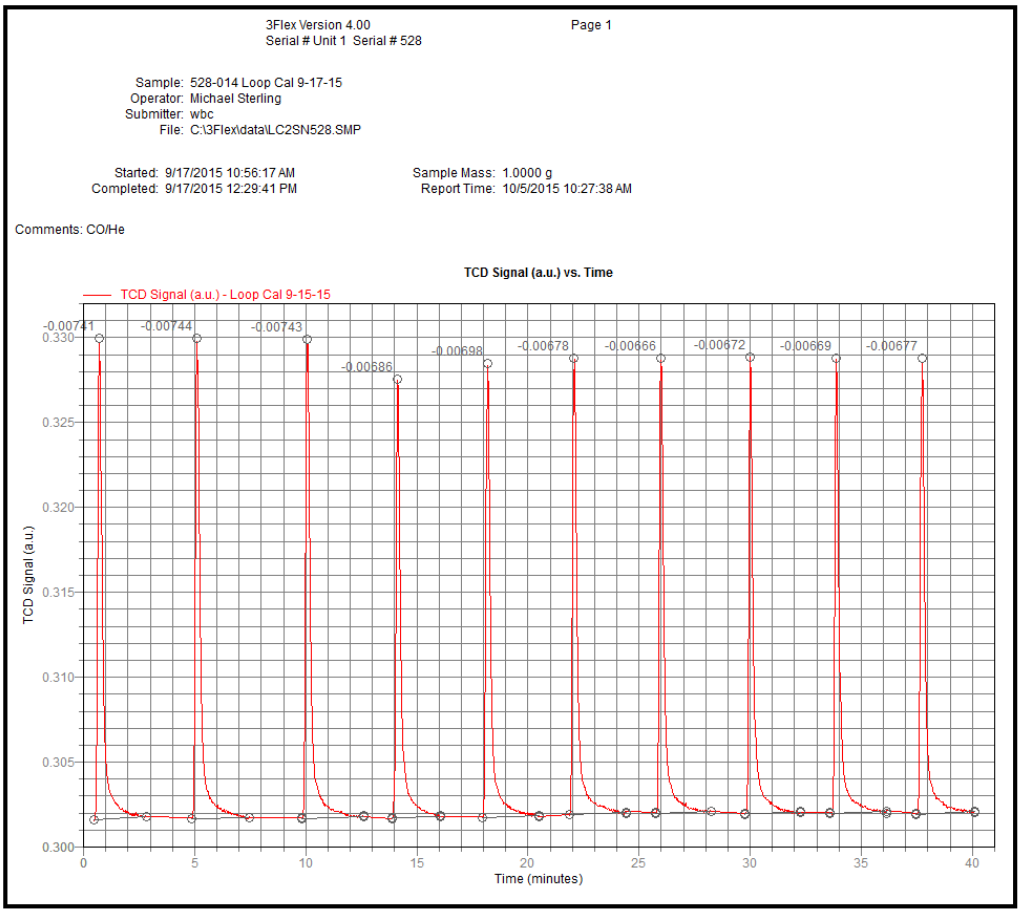
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



LOOP CALIBRATION



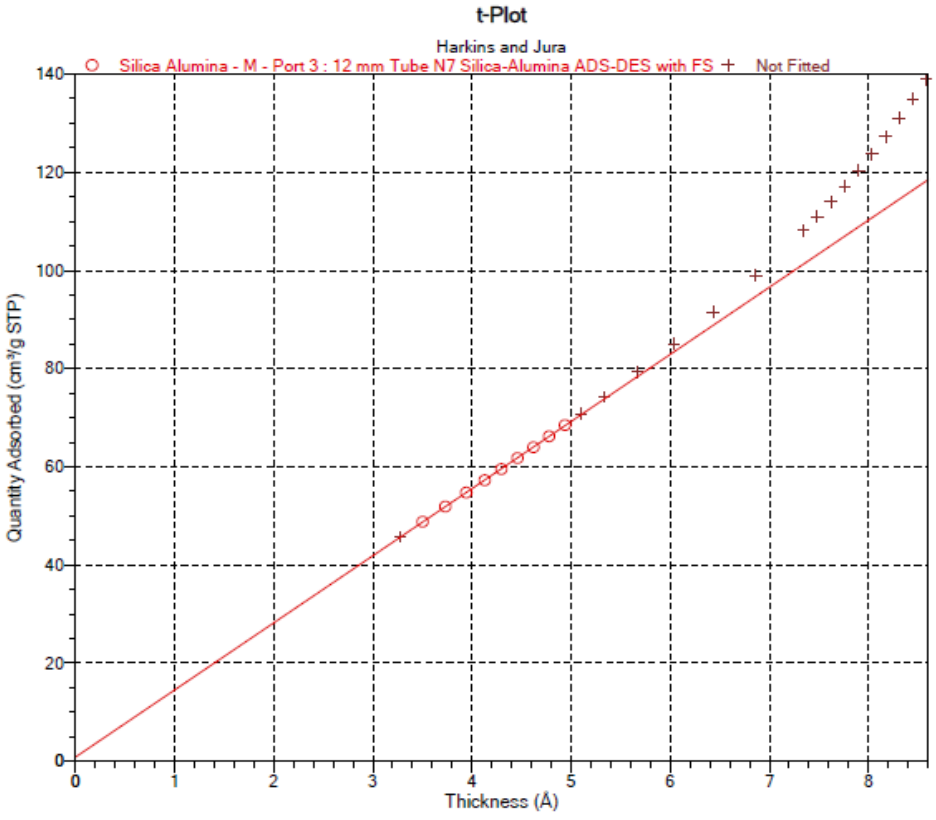
t-Plot Report

micromeritics®

3Flex 4.03 | 3Flex Serial # 104 Unit 2 Port 3 | Page 10 of 22

Sample: 12 mm Tube N7 Silica-Alumina ADS-DES with FS
Operator: JMK
Submitter: Micromeritics
File: C:\3Flex\data\examp...\Silica Alumina - M - Port 3.SMP

Started:	8/22/2012 3:58:36 PM	Analysis adsorptive:	N2
Completed:	8/23/2012 12:38:38 PM	Analysis bath temp.:	77.147 K
Report time:	7/6/2017 10:16:12 AM	Thermal correction:	No
Sample mass:	0.2471 g	Ambient free space:	15.8813 cm³ Measured
Analysis free space:	56.5480 cm³	Equilibration interval:	10 s
Low pressure dose:	None	Sample density:	1.000 g/cm³
Automatic degas:	No		



TCD SIGNAL VS TEMPERATURE

micromeritics®

Micromeritics Instrument Corporation

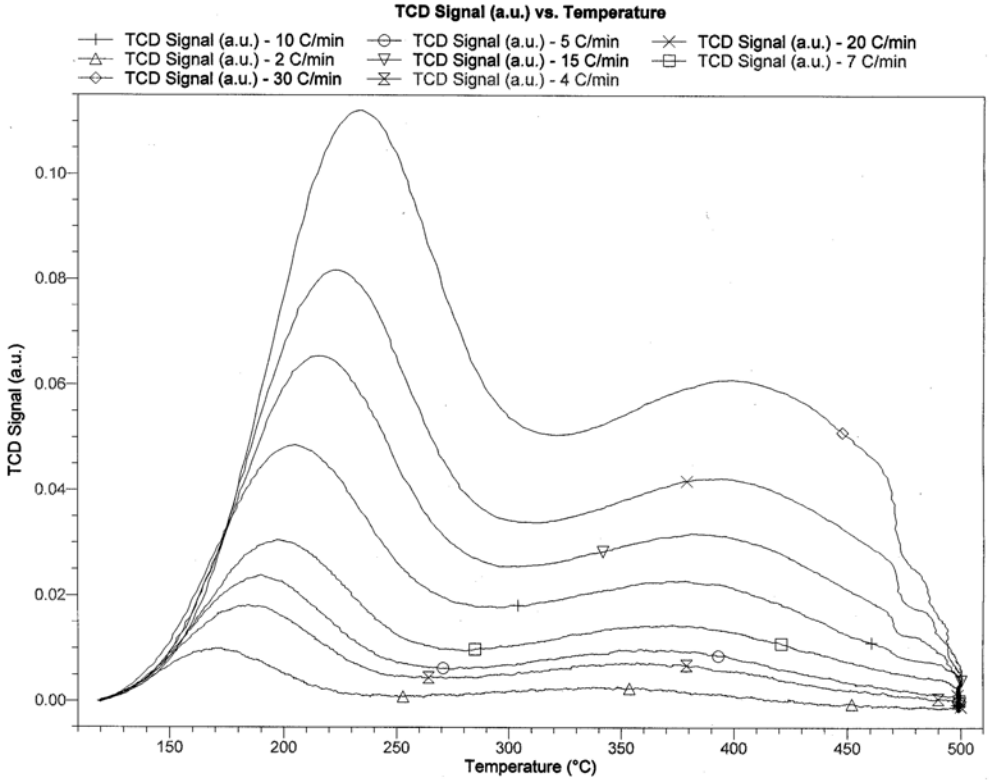
Unit 1 Serial # 256 Page 1

Sample: NH3 TPD -- ZSM-5
Operator:
Submitter:
File: C:\...\DATA\NH3_HOD.SMP

Started:
Completed:

Sample Mass: 0.1100 g
Report Time:

Comments: Heat of Desorption experiment using ZSM-5 and ammonia.



TCD SIGNAL VS TIME

micromeritics®

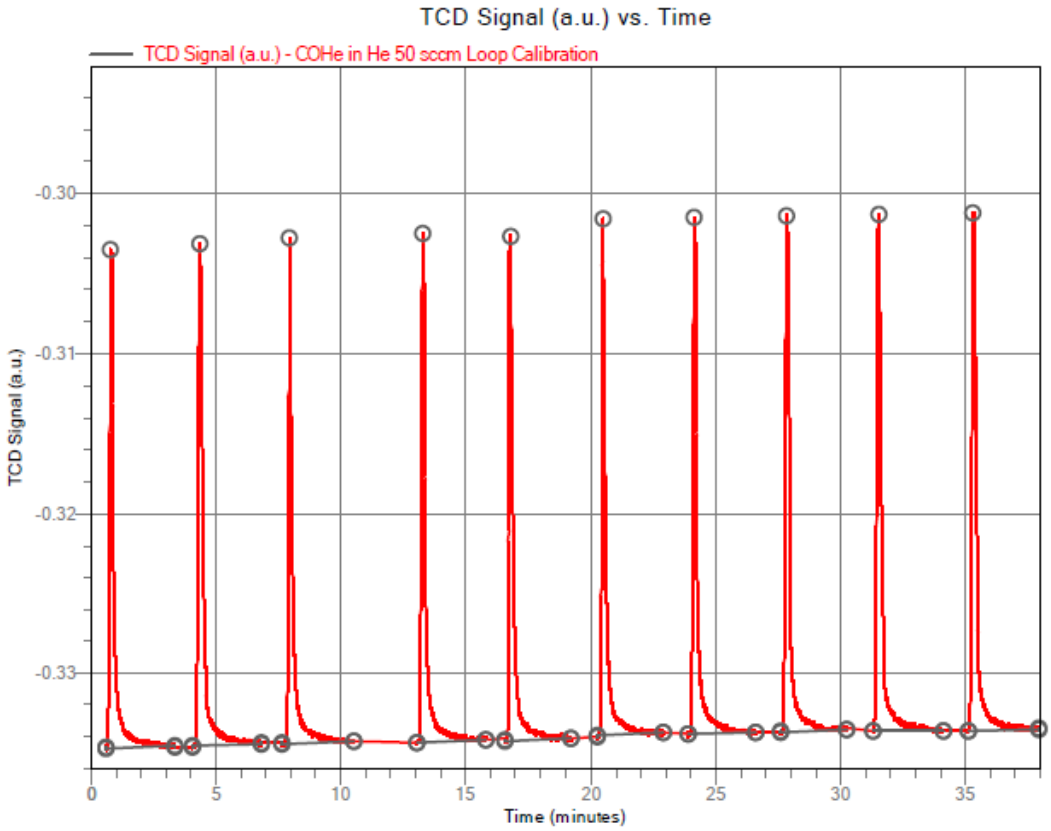
Micromeritics Instrument Corporation

3Flex Version
Serial # Unit 1 Serial # 315

Sample: 315-303 COHe in He 50 sccm Loop Calibration
Operator: wbc
Submitter:
File: C:\3Flex\data\examples\CD\315-303 COHe in He 50 sccm Loop Calibration.SMP

Started: 12/17/2015 6:54:48 AM
Completed: 12/17/2015 7:42:59 AM

Sample Mass: 1.0000 g
Report Time: 1/22/2016 9:08:05 AM



TCD SIGNAL VS TIME

micromeritics®

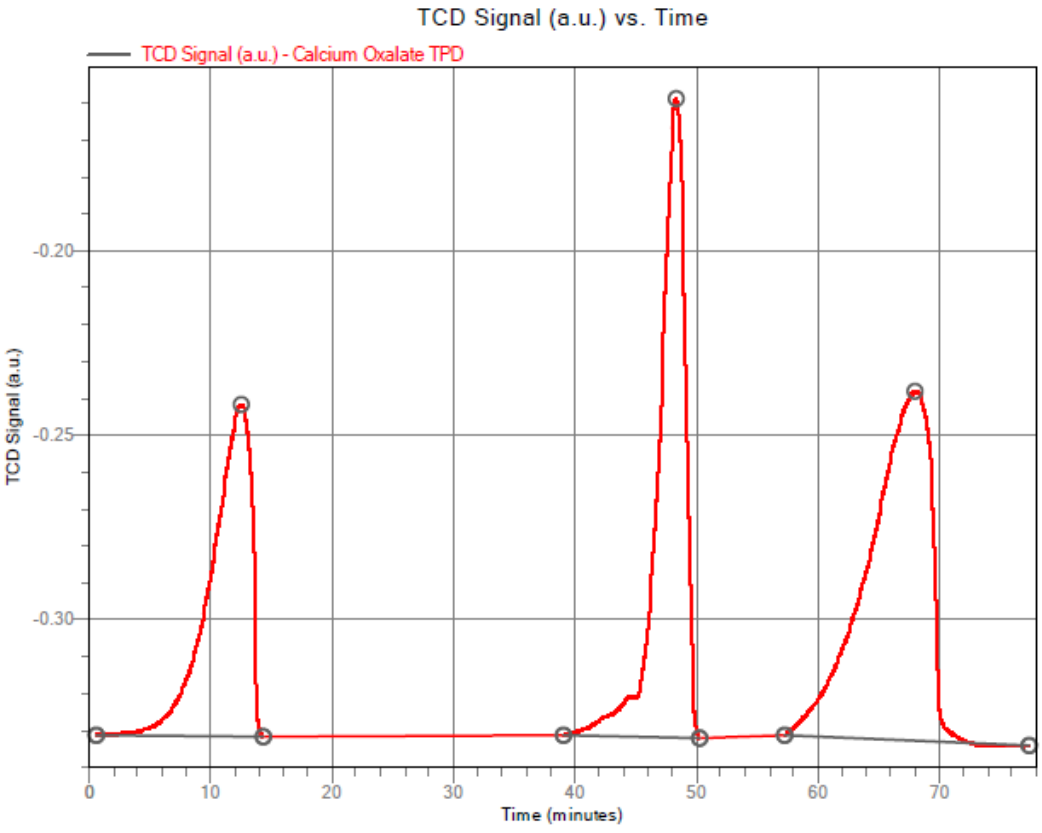
Micromeritics Instrument Corporation

3Flex Version ·
Serial # Unit 1 Serial # 315

Sample: 315-306 Calcium Oxalate TPD
Operator: wbc
Submitter:
File: C:\3Flex\data\examples\CD\315-306 Calcium Oxalate TPD.SMP

Started: 12/17/2015 2:45:31 PM
Completed: 12/17/2015 4:26:30 PM

Sample Mass: 0.0203 g
Report Time: 1/22/2016 9:08:04 AM



TCD SIGNAL VS TIME

micromeritics®

Micromeritics Instrument Corporation

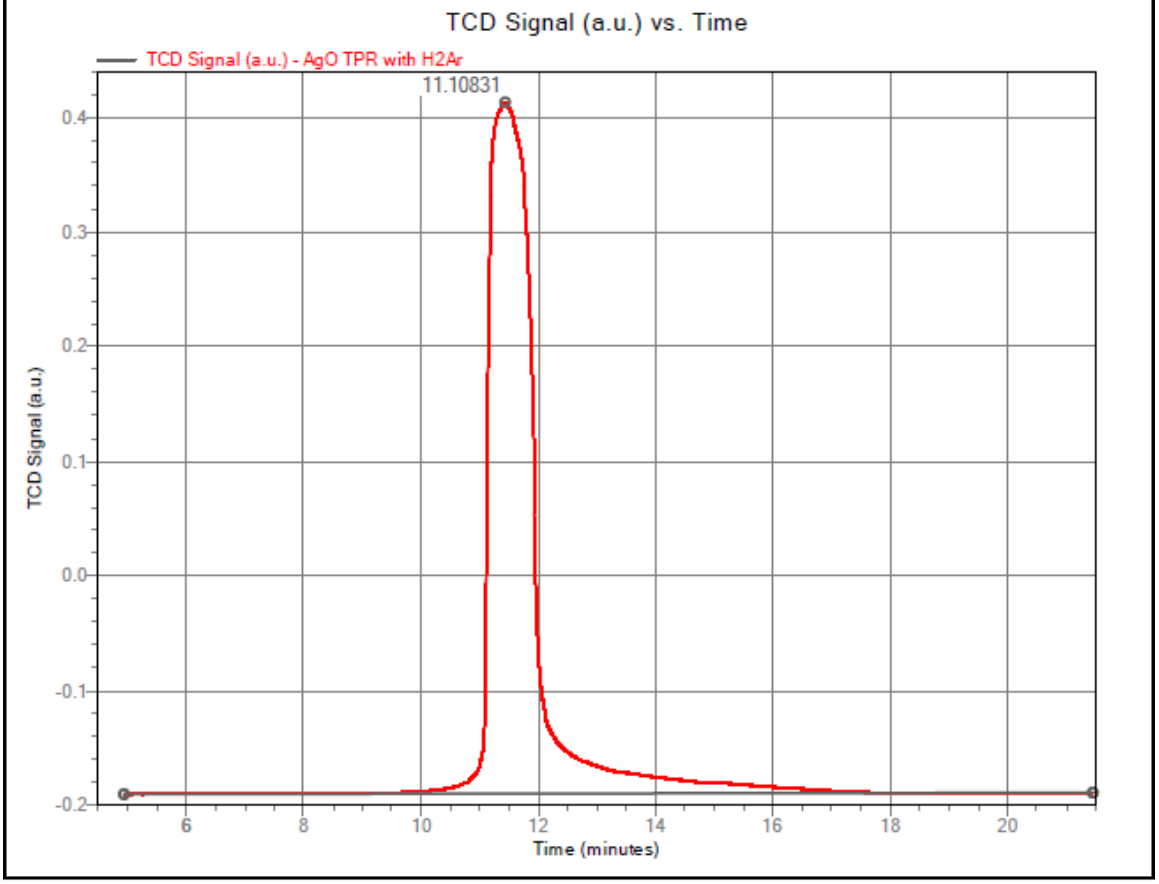
3Flex Version
Serial # Unit 1 Serial # 315

Sample: 315-058 AgO TPR with H2Ar
Operator: wbc
Submitter:
File: C:\3Flex\data\examples\CD\AgO TPR with H2Ar.SMP

Started: 9/10/2015 9:54:57 AM
Completed: 9/10/2015 10:37:47 AM

Sample Mass: 0.0498 g
Report Time: 1/22/2016 9:08:00 AM

Comments: LN2/IPA slurry



TCD SIGNAL VS TIME

micromeritics®

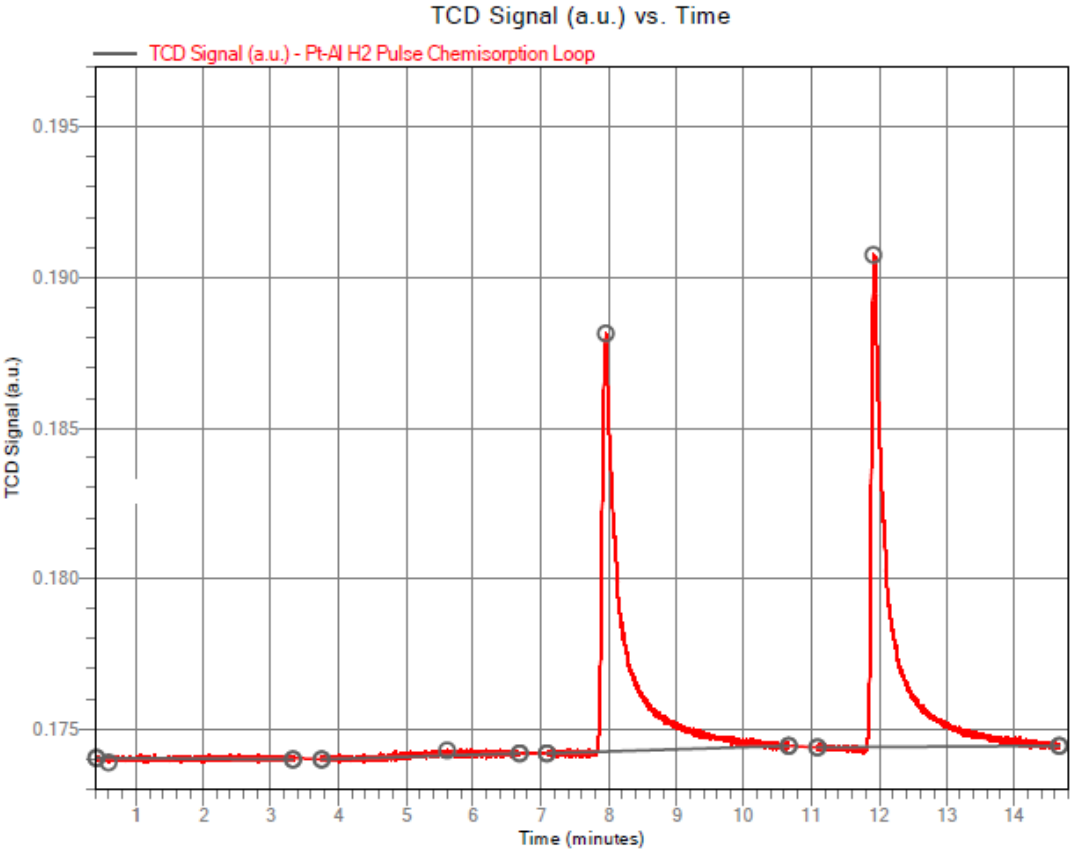
Micromeritics Instrument Corporation

3Flex Version
Serial # Unit 1 Serial # 315

Sample: 315-317 Pt-Al H2 Pulse Chemisorption Loop
Operator: wbc
Submitter:
File: C:\3Flex\data\examples\CD\315-317 Pt-Al H2 Pulse Chemisorption Loop.SMP

Started: 12/23/2015 7:24:36 AM
Completed: 12/23/2015 11:13:04 AM

Sample Mass: 0.8520 g
Report Time: 1/22/2016 9:08:01 AM



TCD SIGNAL VS TIME

micromeritics®

Micromeritics Instrument Corporation

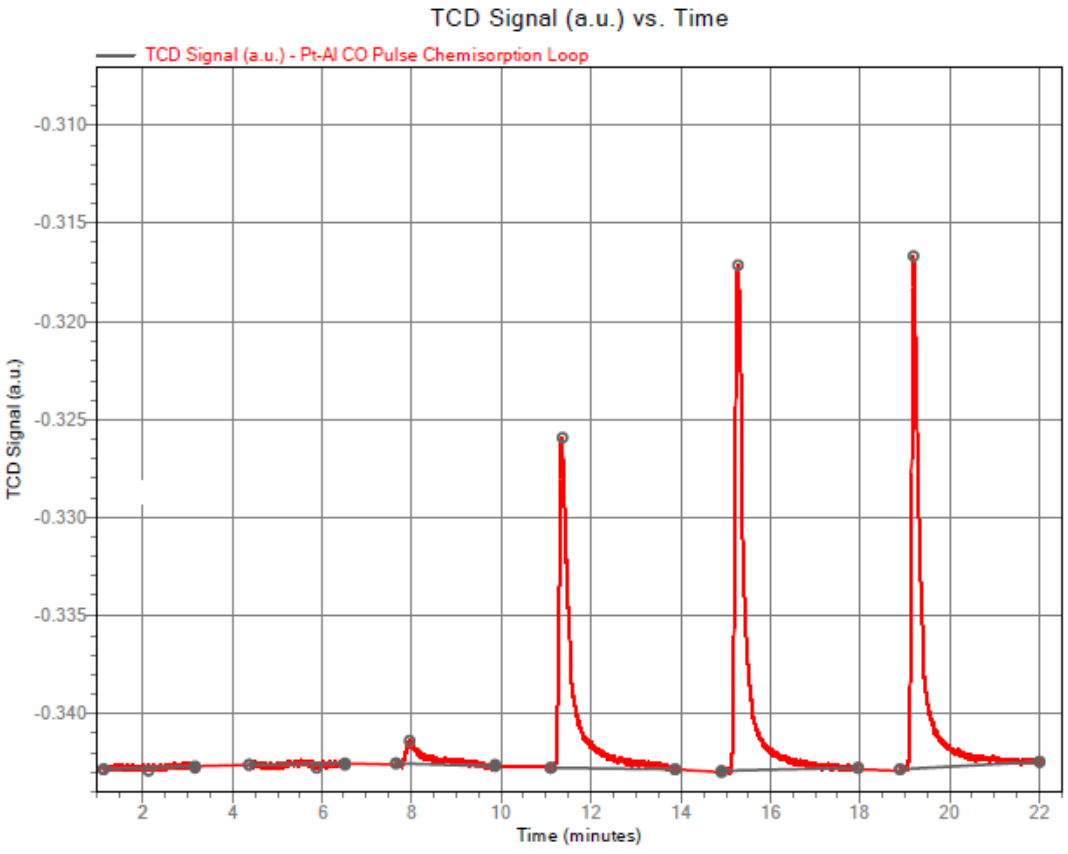
3Flex Version
Serial # Unit 1 Serial # 315

Sample: 315-332 Pt-Al CO Pulse Chemisorption Loop
Operator: wbc
Submitter:
File: C:\3Flex\data\examples\CD\Pt-Al CO Pulse Chemisorption Loop.SMP

Started: 1/5/2016 10:07:52 AM
Completed: 1/5/2016 1:07:54 PM

Sample Mass: 0.7648 g
Report Time: 1/22/2016 9:07:57 AM

Comments: Reweigh after analysis



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8 SELECTED REPORTS

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

CFR Note

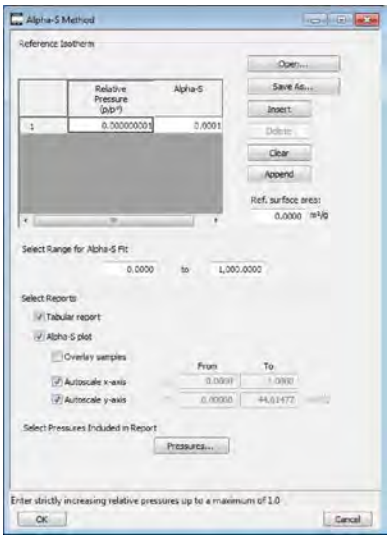
To edit reports, open the *Sample* file then select the *Report Options* tab. Highlight the report name in the *Selected Reports* list box and click **Edit**.

Report Name	P	C	DA
Advanced Reports - Python Module on page A - 1	✓	✓	✓
Alpha-S Method Report on page 8 - 3	✓		
BET Surface Area Report on page 8 - 5	✓		
BJH Adsorption/Desorption Report on page 8 - 7	✓		
DFT Pore Size Report on page 8 - 13	✓		
DFT Surface Energy Report on page 8 - 16	✓		
Difference Method Report on page 8 - 17		✓	
Differential Hysteresis on page 8 - 20	✓		
Dollimore-Heal Adsorption/Desorption Report on page 8 - 22	✓		
Dubinin Report on page 8 - 26	✓		
f-Ratio Method Report on page 8 - 31	✓		
First Order Kinetics Report on page 8 - 33			✓
Freundlich Report on page 8 - 34	✓	✓	
Graph Report Options on page 8 - 37 ■ TCD Concentration vs Time ■ TCD Signal and TCD Concentration vs Time ■ TCD Signal vs Temperature ■ TCD Signal vs Time ■ Temperature vs Time			✓
Horvath-Kawazoe Report on page 8 - 40	✓		
Isotherm Report on page 8 - 44	✓	✓	
Langmuir Report on page 8 - 46	✓	✓	

Report Name	P	C	DA
Loop Calibration Report on page 8 - 49			✓
MP-Method Report on page 8 - 50	✓		
NLDFT Advanced PSD Report on page 8 - 54	✓		
Options Report on page 8 - 58	✓	✓	✓
Pulse Chemisorption Report on page 8 - 59			✓
Sample Log Report on page 8 - 62	✓	✓	✓
Sinfelt and Difference Methods on page 8 - 63		✓	
Summary Report on page 8 - 64	✓		
Summary Report for Dynamic Analysis on page 8 - 65			✓
t-Plot Report on page 8 - 66	✓		
Temkin Report on page 8 - 69	✓	✓	
Validation Report on page 8 - 72	✓		

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.

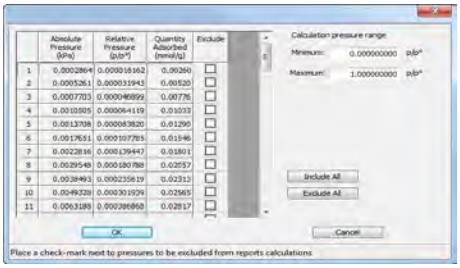


One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the Alpha-S values.

Alpha-S Method Report

Selections	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)

Selections	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 - 3***.**

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

Selections	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)

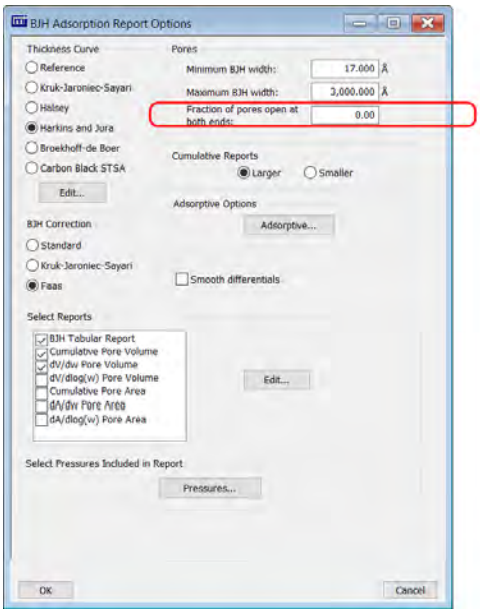
Selections	Description
	Include All. Select to include all pressure points in the table. **Insert Predefined.** Click to insert a predefined (default) set of points into the report. *Use Interpolation* must be selected to enable this button. This button displays for BET reports only. **Use Interpolation.** 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 - 3*.

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.

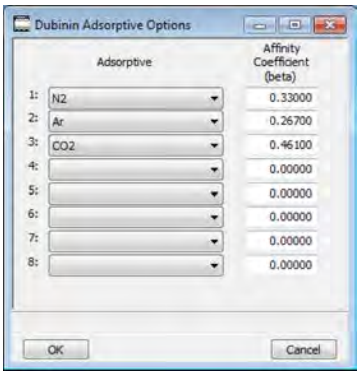


Circled selection is applicable to BJH Adsorption only

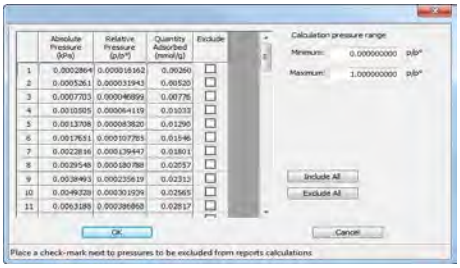
BJH Adsorption/Desorption Report

Selections	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)

Selections	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)

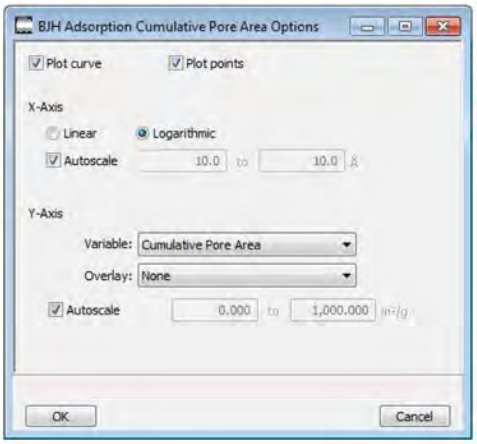
Selections	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate 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)

Selections	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 - 3*.

BJH Plot



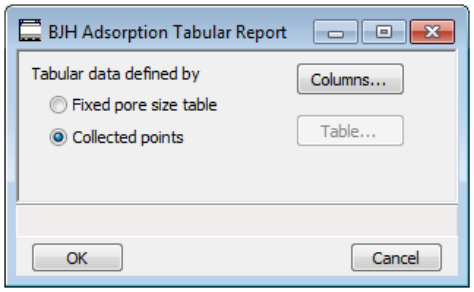
The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click **Edit**.

BJH Plot Report

Selections	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.

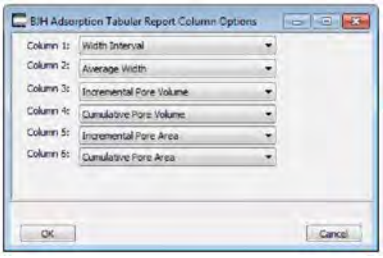
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

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.

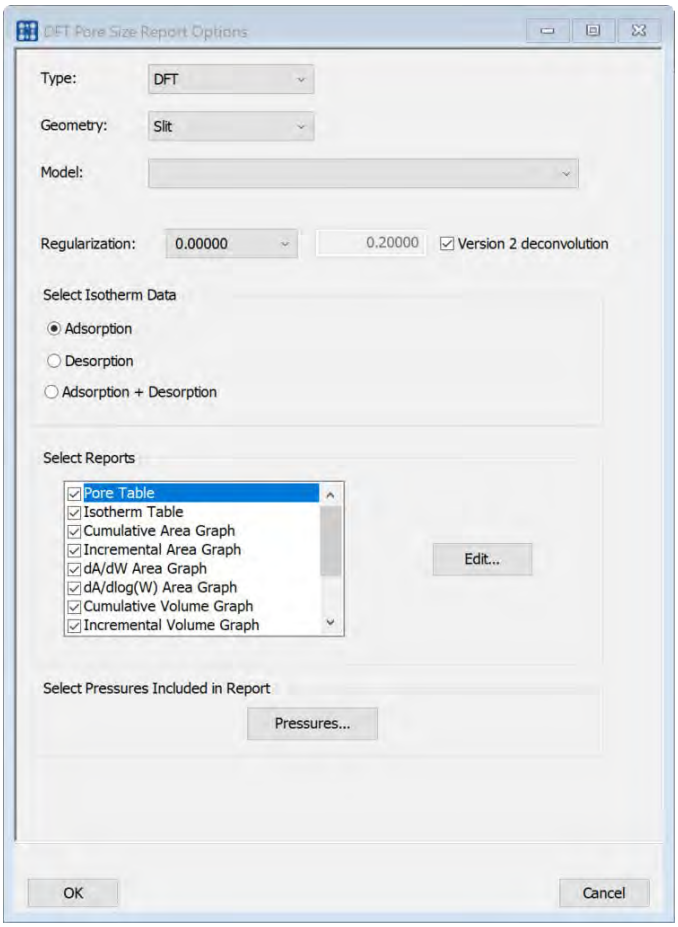
BJH Tabular Report

Selections	Description
Collected points [*selection*]	Use to include all relative pressure points collected. See 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. See the **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 - 3*.

DFT Pore Size Report

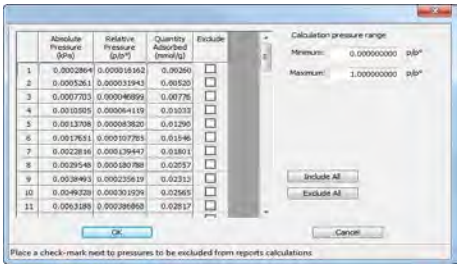
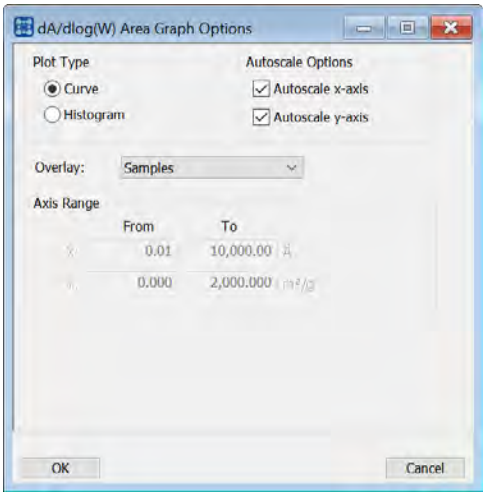
The *DFT Pore Size* report contains the results of pore size distribution analyses using a non-local DFT range of micro and mesopore ranges.



DFT Pore Size Report

Selections	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.

DFT Pore Size Report (continued)

Selections	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.
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 Isotherm Data [*group box*]	Select the isotherm data to be used in the report.
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.

DFT Pore Size Report (continued)

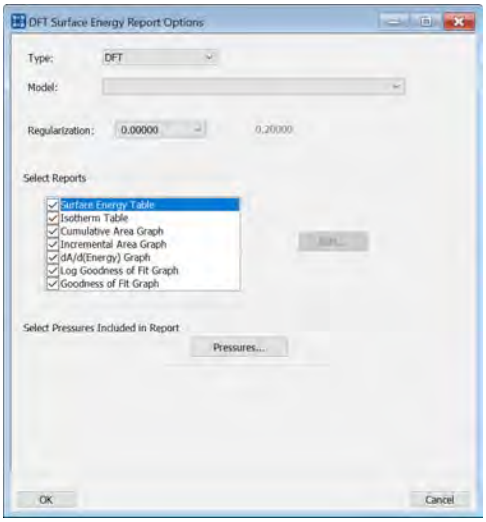
Selections	Description
	Axis Range. *From*/*To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes. ■ **x-axis.** Shows the pore size. ■ **y-axis.** Shows the area. **Overlay.** Select an overlay for the report. **Plot Type.** Select the method for data display.
Type [*drop-down box***]**	**Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See *DFT Models on page F - 1*. **DFT.** Model based on the density functional theory.
Version 2 deconvolution [*check box***]**	If enabled, adds improved performance for nonzero values of the DFT regularization. The DFT results will be slightly better than when the checkbox is not selected and when using a regularization parameter. When the checkbox is not selected, the legacy behavior of the DFT calculation used by earlier versions of the application is produced.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3***.**

DFT Surface Energy Report

DFT Pore Size Report on page 8 - 13

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*.

DIFFERENCE METHOD REPORT

The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified.

The y-intercept quantity adsorbed (Q_0) is used for several calculations in the *Difference* and *Sinfelt* reports. This value can be determined in two ways. If one point is selected, Q_0 is the quantity adsorbed for that point.

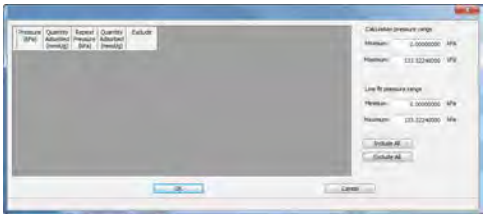
Difference Method. The repeat isotherm data are subtracted from the primary isotherm. Q_0 is the y-intercept of a straight line through the difference data.

Sinfelt Method. Both the primary and repeat isotherms are fitted to a straight line. Q_0 is the difference between the y-intercepts of the fit lines.

Difference and Sinfelt Report

Selections	Description
Analysis summary [*selection*]	**Analysis.** Generates a summary of the following for the first analysis: ■ Percent metal dispersion ■ Metallic surface area ■ Volume adsorbed ■ Slope ■ Correlation coefficient **Difference.** Generates a summary of the differences between the following information for the first and repeat analyses: ■ Percent metal dispersion ■ Metallic surface area ■ Average difference volume **Repeat.** (Sinfelt report only.) Generates a line fit plot for the secondary analysis.

Difference and Sinfelt Report (continued)

Selections	Description
Plot data [*selection***]**	**Analysis.** Includes a line fit plot for the primary analysis. **Autoscale x-axis / Autoscale y-axis.** Select to have the x- and/or y- axes automatically scaled. The application uses the highest values collected during analysis as the ending points for an axis range. X-axis shows the pressure. Y-axis shows the quantity of gas adsorbed. **Difference.** Plots the difference between the analysis and repeat analysis lines. **Overlay samples.** Overlays data from the current sample with that of other samples. Click **Overlays** on the *Report Options* window to choose other sample files. **Plot curve** and **Plot points**. Use to specify how to plot data. Plot data as a curve, points, or both. **Repeat.** Includes a line fit plot for the secondary analysis.
Pressures [*button***]**	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. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Line fit pressure range.** Enter the minimum and maximum pressures for line fit.

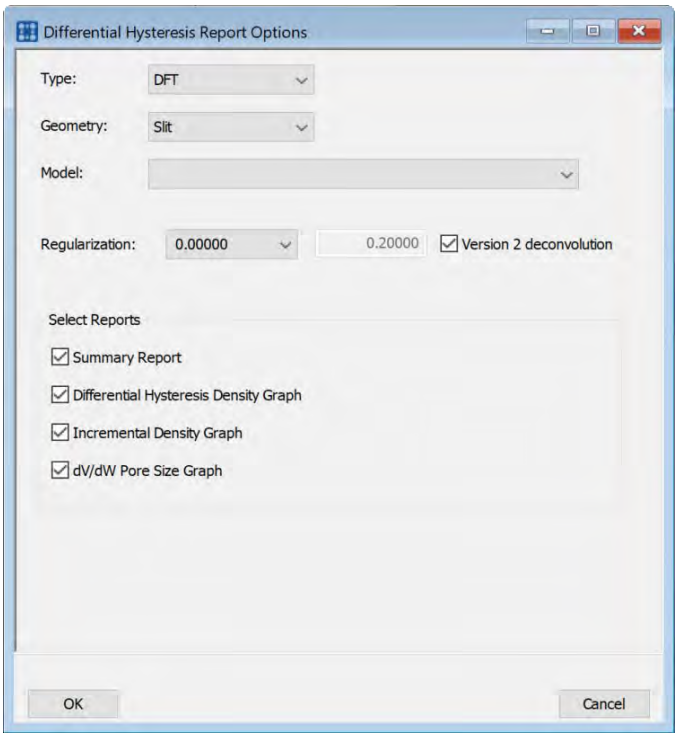
Difference and Sinfelt Report (continued)

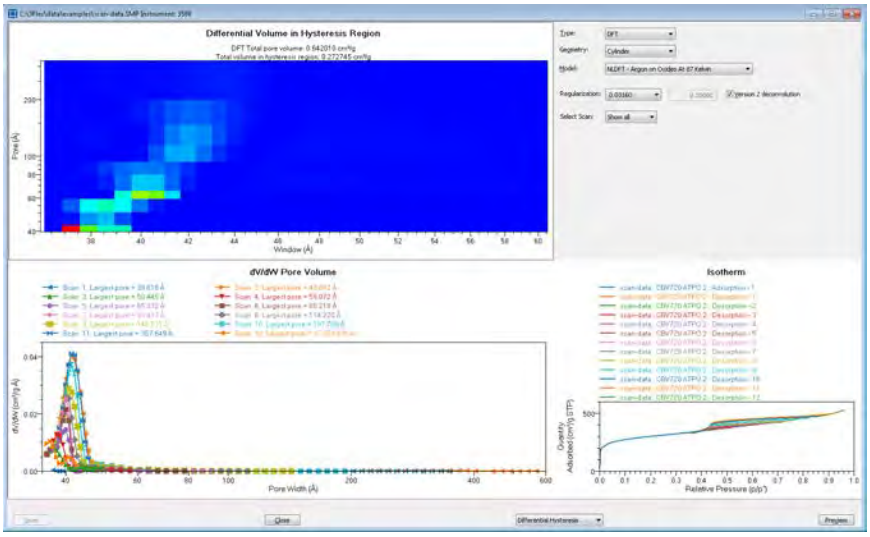
Selections	Description
Tabular report [*selection*]	Select to have a report of the pressure points generated.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

DIFFERENTIAL HYSTERESIS

This report seeks to determine a distribution of pore sizes for a sample material taking into account pores of varying geometry such as pyramidal, constricted, and occluded pore shapes. To extract this information, a sample material is subjected to multiple adsorption and desorption scans in order to cover the hysteresis region of the isotherm. If the scans are sampled to obtain a sufficiently dense set of data, a pore size distribution can be obtained by application of NLDFT calculations to each desorption scan and the results combined into a two dimensional distribution.





Differential Hysteresis Report

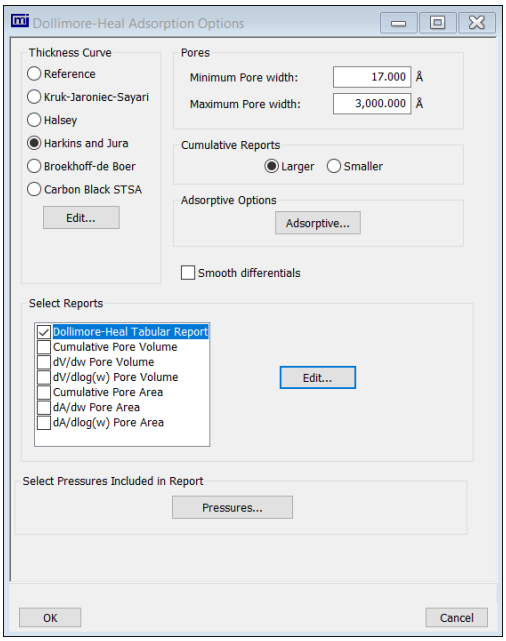
Selections	Description
Geometry [*drop-down box*]	Select the pore shape.
Model [*drop-down box*]	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
Regularization [*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 [*selection*]	Select the report names to include in the report.
Select Scan [*drop-down box*]	Select the scans to include in the report.
Type [*drop-down box*]	**Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See ***DFT Models on page F - 1***. **DFT.** Model based on the density functional theory.
Version 2 deconvolution [*check box*]	If enabled, adds improved performance for nonzero values of the DFT regularization. The DFT results will be slightly better than when the checkbox is not selected and when using a regularization parameter. When the checkbox is not selected, the legacy behavior of the DFT calculation used by earlier versions of the application is produced.

For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 3*****.**

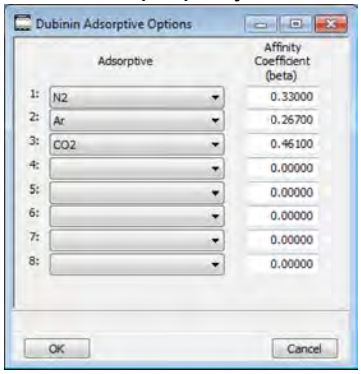
DOLLIMORE-HEAL ADSORPTION/DESORPTION REPORT

BJH Adsorption/Desorption Report on page 8 - 7

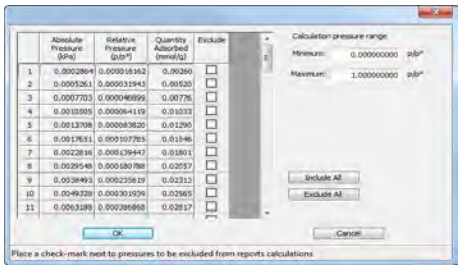
The *Dollimore-Heal Adsorption Report Option* and the *Dollimore-Heal Desorption Report Option* generate reports from both adsorption and desorption data.



Dollimore-Heal Adsorption/Desorption Report

Selections	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)

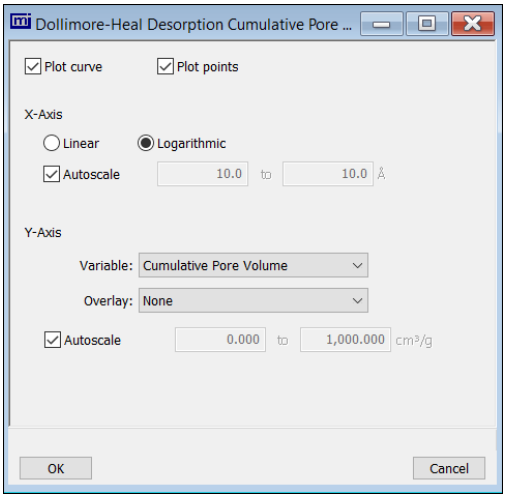
Selections	Description
Cumulative Reports [*group box*]	**Larger.** Use to report the total volume found in pores larger than the current pore size. **Smaller.** Use to report the total volume found in pores smaller than the current pore size.
Pores [*group box*]	Enter the minimum and maximum diameter (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)

Selections	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.

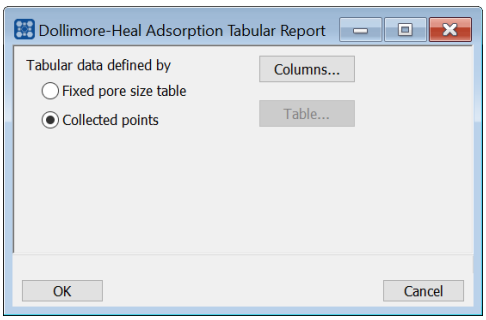
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

Dollimore-Heal Desorption Plot Options



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*.

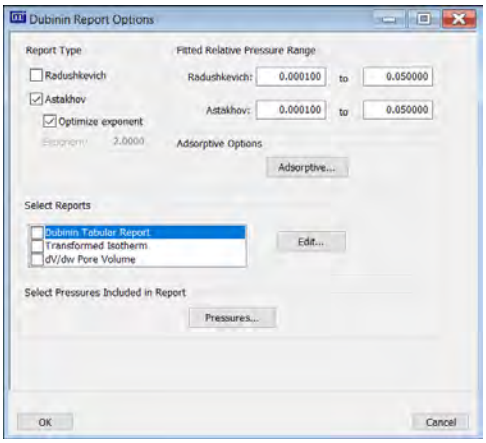
Dollimore-Heal Tabular Report Options



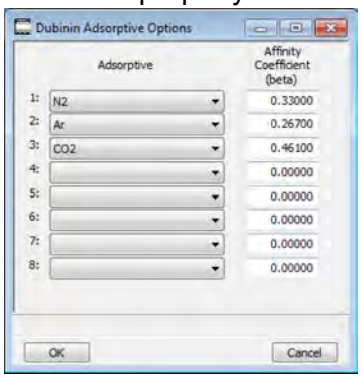
Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*.

DUBININ REPORT

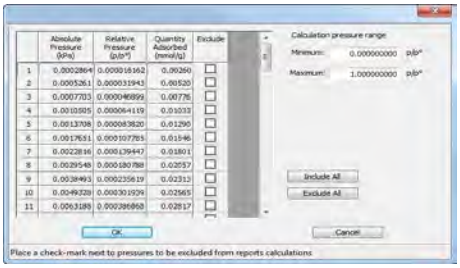
The *Dubinin* method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential.



Dubinin Report

Selections	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)

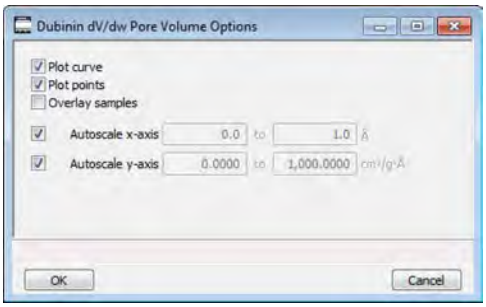
Selections	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate 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 - 3*.

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.



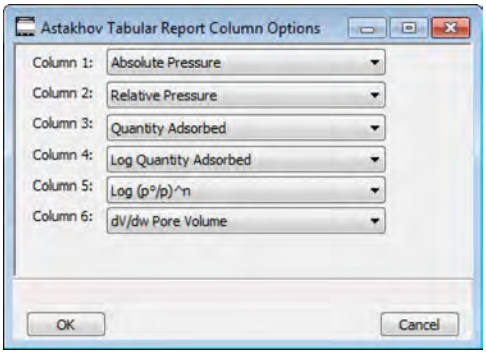
Dubinin Pore Volume Report

Selections	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 - 3*.

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.

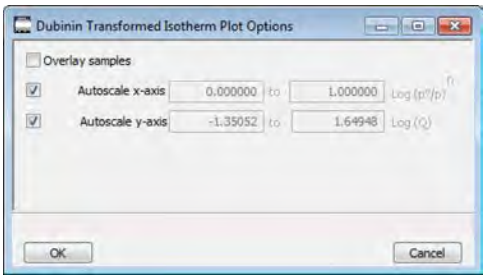


Log (p^o/p)^n. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

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.



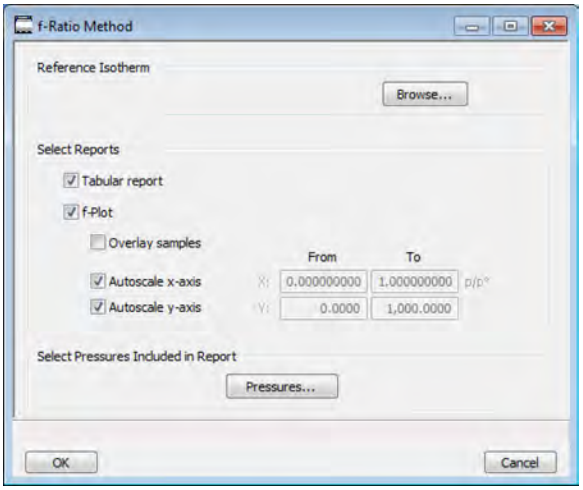
Dubinin Transformed Isotherm Plot Report

Selections	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.

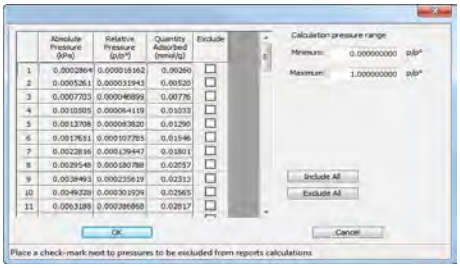
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

f-Ratio Method Report

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



f-Ratio Report

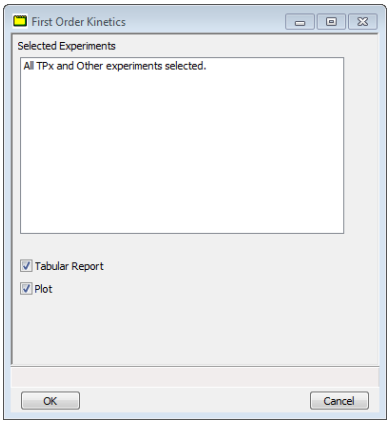
Selections	Description
Pressures [*button*]	Use to select a pressure range for report calculations and points for exclusion from calculations.  **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate 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.
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.

f-Ratio Report (continued)

Selections	Description
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 - 3*.

First Order Kinetics Report



First Order Kinetics Report

Selections	Description
Plot [*check box*]	Select to have the report formatted in a graph format.
Selected Experiments [*group box*]	Displays all related experiments in the current file. Only experiments with collected data are shown.
Tabular Report [*check box*]	Select to have report formatted in table format.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

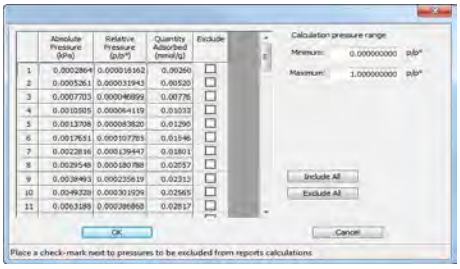
FREUNDLICH REPORT

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**.

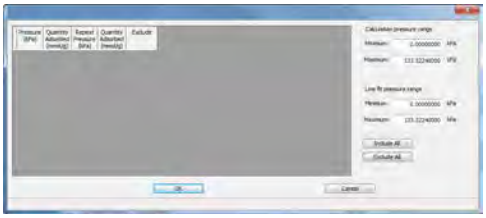
Physisorption

Chemisorption

Freundlich Report

Selections	Description
Calculations [*group box*] C	Select from the various calculation options. **Difference**. Plots the difference between the analysis and repeat analysis lines. **First Analysis**. Includes a line fit plot for the primary analysis. **Repeat Analysis.** Includes a line fit plot for the secondary analysis.
Pressures [*button*] P	Use to select a pressure range for report calculations and points for exclusion from calculations. 

Freundlich Report (continued)

Selections	Description
	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.
Pressures [*button***]**	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. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Line fit pressure range.** Enter the minimum and maximum pressures for line fit.
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.

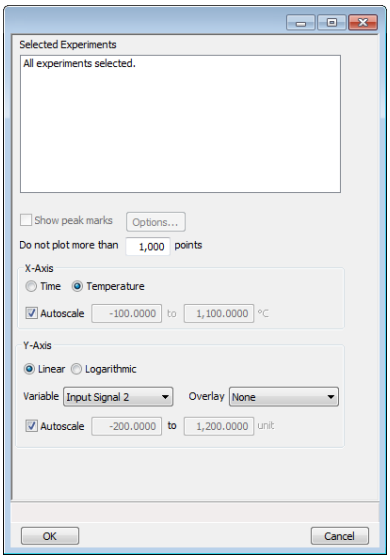
Freundlich Report (continued)

Selections	Description
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 - 3*.

GRAPH REPORT OPTIONS

- TCD Signal vs. Time
- Temperature vs. Time
- TCD Signal vs. Temperature
- TCD Concentration vs. Time
- TCD Concentration vs. Temperature
- TCD Signal and TCD Concentration vs. Time



Any signal (the detector signal or the 1 or 2 auxiliary signal) may be plotted against time or temperature. Any signal can be overlaid onto the primary signal.

For color output to a monitor or printer, signals are displayed in different colors. For black and white output, different symbols are used.

Graph Report Options

Selections	Description
Do not plot more than [*n*] points [*text box*]	Specifies a maximum number of points to plot. Plot speed may be affected when a higher resolution is entered.
Mass spec [*group box*]	**Autoscale.** 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.

Graph Report Options (continued)

Selections	Description
Options [*button*]	Enabled when the *Show peak marks* option is selected. Lists the peaks for the results of the highlighted experiment. Select any (or all) peaks to include in the graph. Select the peak labels to display on the graphs.
Overlay [*group box*]	Specify overlay options to include in the report.
Selected Experiments [*group box*]	Displays all related experiments in the current file. Only experiments with collected data are shown. This option is disabled for new files.
Show peak marks [*check box*]	■ Enables the **Options** button to specify the peaks to include in the experiment. ■ Displays the areas and baselines on the graph. ■ Draws a straight baseline between the selected peaks.
X-axis [*group box*]	Specify *Time* or *Temperature* for the x-axis variable. **Autoscale.** 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.

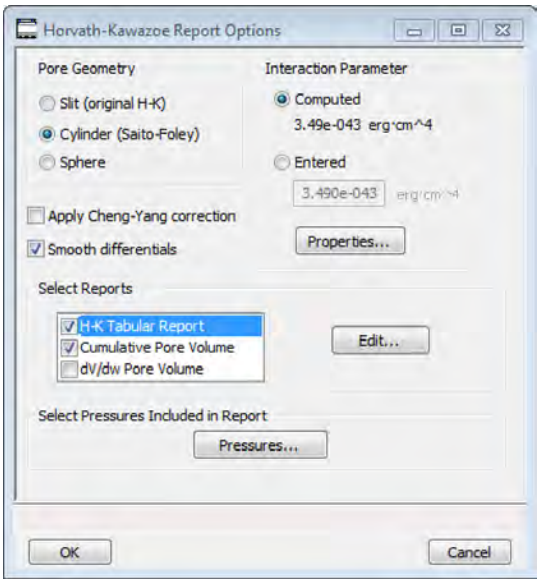
Graph Report Options (continued)

Selections	Description
Y-axis [*group box***]**	Specify *Time* or *Temperature* for the y-axis variable. **Autoscale.** 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. **Linear/Logarithmic**. Scales the graph as *Linear* or *Logarithmic*.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

HORVATH-KAWAZOE REPORT

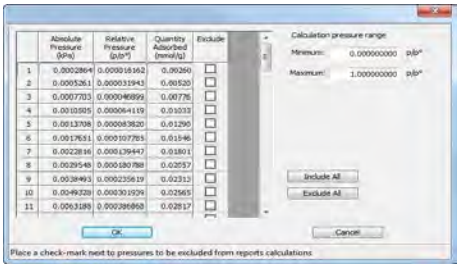
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.



Horvath-Kawazoe Report

Selections	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)

Selections	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. ■ **Diameter at zero energy.** Enter the diameter of an atom at zero

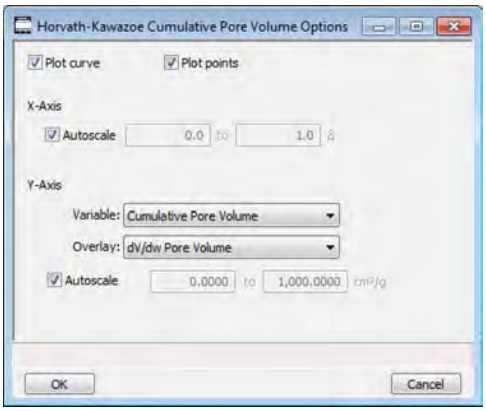
Horvath-Kawazoe Report (continued)

Selections	Description
	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 - 3*.

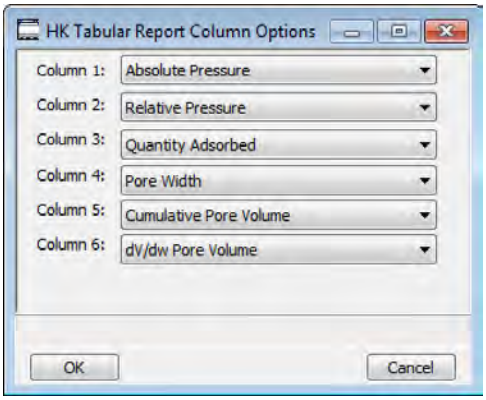
Horvath-Kawazoe Plot

See *BJH Adsorption/Desorption Report on page 8 - 7* 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.

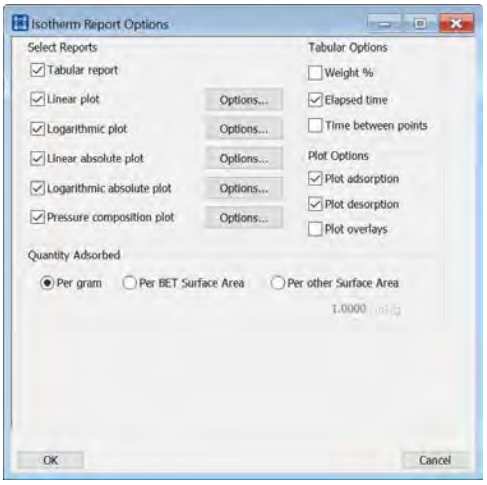
Horvath-Kawazoe Tabular Report



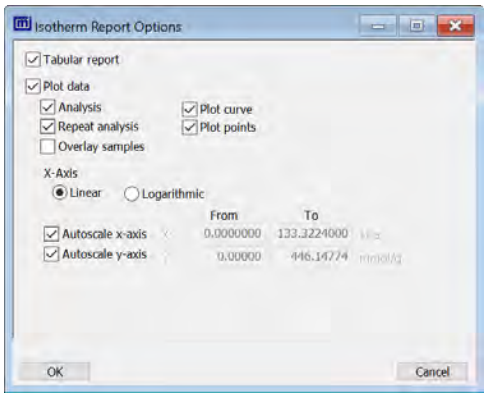
Highlight *H-K Tabular Report* in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit**. Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.

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.



Physisorption



Chemisorption

Isotherm Report

Selections	Description
Autoscale [*check box*] SC	**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.
Options [*button*] P	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 Data [*check box*] SC	Select each option to include in the final report.

Isotherm Report (continued)

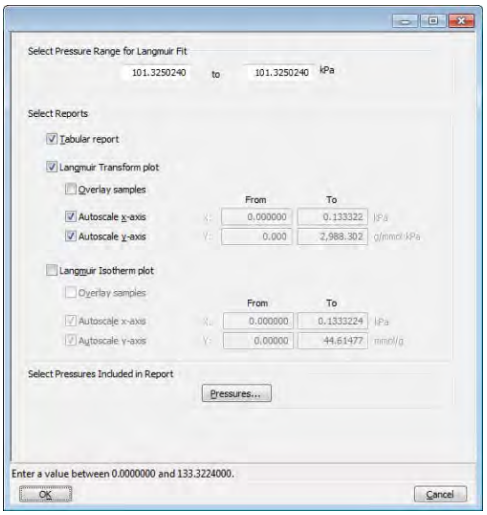
Selections	Description
Plot Options [*group box*] P	Select the types of isotherm to plot.
Quantity Adsorbed [*group box*] P	Select how to report the quantity adsorbed. ■ per gram (cm^3/g) STP ■ per BET Surface Area (cm^3/m^2) STP or mmol/g ■ per other Surface Area (cm^3/m^2) STP or $mmol/m^2$
Selected Reports [*group box*] P	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*] P	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.
Tabular Report [*group box*] SC	Select to include tabular data in the report.
X-axis [*group box*] SC	Indicate if the x-axis should be in linear or logarithmic format.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

LANGMUIR REPORT

This report is for physisorption and chemisorption analyses only.

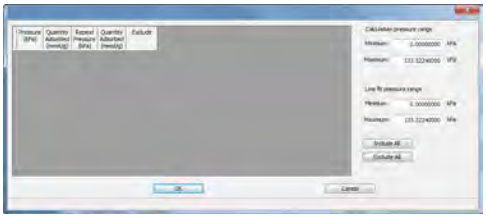
The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer. Langmuir uses a single layer model.



Physisorption

Chemisorption

Langmuir Report

Selections	Description
Calculations [*group box*] C	Select one or more of the calculation options to be used for analysis.
Pressures [*button*] C	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.

Langmuir Report (continued)

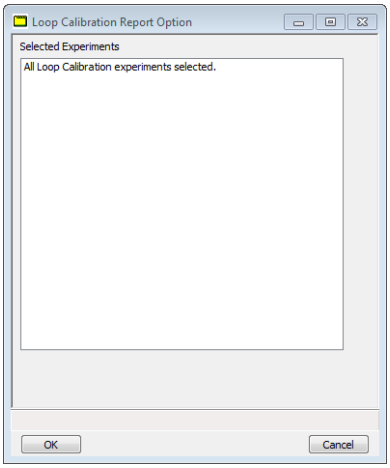
Selections	Description
	Exclude All. Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Line fit pressure range.** Enter the minimum and maximum pressures for line fit.
Pressures [*button*] P	This option is available when the sample 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*. **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*] P	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

Langmuir Report (continued)

Selections	Description
	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 - 3*.

LOOP CALIBRATION REPORT



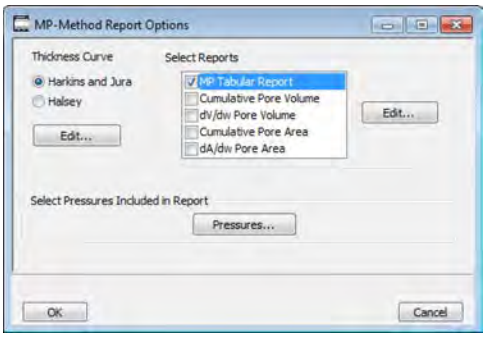
Loop Calibration Report

Selections	Description
Selected Experiments [*group box*]	Displays all related experiments in the current file. Only experiments with collected data are shown.

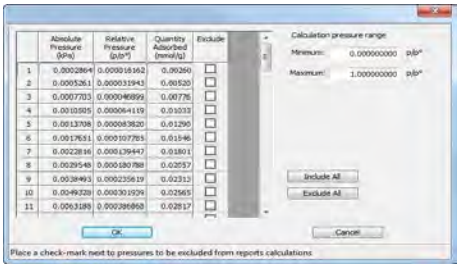
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

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

Selections	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)

Selections	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 - 3*.

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.

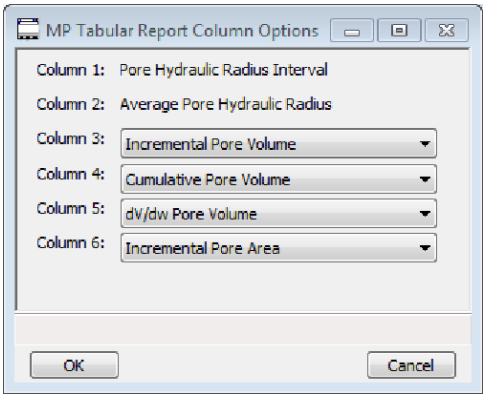
MP Method Plot Report

Selections	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 - 3*.

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.

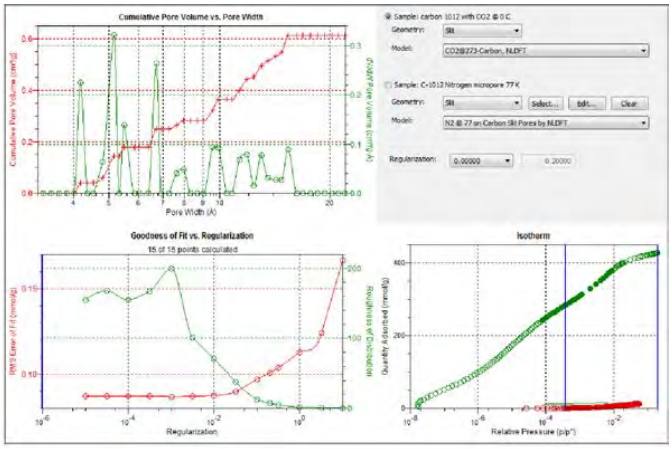


The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reported.

NLDFT Advanced PSD Report

The *NLDFT Advanced PSD* report allows for more advanced computation of the pore size distribution of a material using two separate analyses and two non-local DFT models.

The *NLDFT Advanced PSD* report option provides the same calculations as the DFT Pore Size report option and more. The NLDFT report compares two sample files. The models that can be selected are restricted to only those models which have the same analysis temperature and analysis gas as the sample file that is open. For instance, if the sample file was analyzed with N2 at 77 degrees Kelvin, then only the N2 DFT models at 77 degrees Kelvin will be available in the *Model* drop-down list.



The model curve fit is shown in the lower right quadrant along with the adsorption isotherm. This curve fit is updated each time the calculation parameters change (selection of isotherm data points, choice of model, choice of regularization parameter).

A second sample file and second model is used to compute a more accurate pore size distribution (PSD), which is shown in the upper left quadrant. Typically, the second sample file will have used the same sample material as the first sample file yet will have used a different analysis gas and temperature.

In general, the isotherm for this second sample will be different than the first sample. The advanced DFT calculation takes the data from both sample files and combines all this data into a more accurate calculation of the pore size distribution. More accurate means getting the pore distribution at smaller pore sizes (a few Angstroms) as well as larger pore sizes (one thousand Angstroms).

To make a successful *Advanced* calculation, a second sample file must be selected using the **Select** button. A second model must also be selected. Use the options next to the two sample file names to select the isotherm data points for each sample. After selecting an option, the blue bars in the isotherm graph will be toggled to select either the red points or the green points. Once these selections have been done, the results will appear in the left-hand plots and a second isotherm will appear in the isotherm plot (lower right) as well as a second curve-fit. As the selection of points is adjusted, the DFT editor will recalculate the PSD results and also recalculate the two model curve fits.

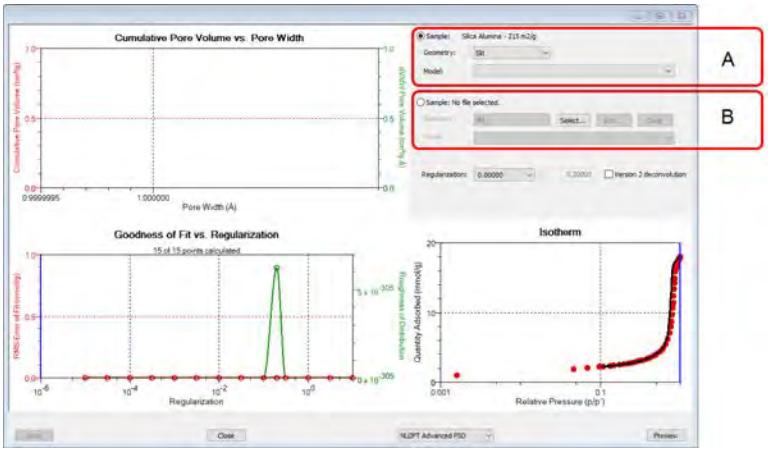
NLDFT Advanced PSD Report

Selections	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.
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*]	Use to select the second sample file.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

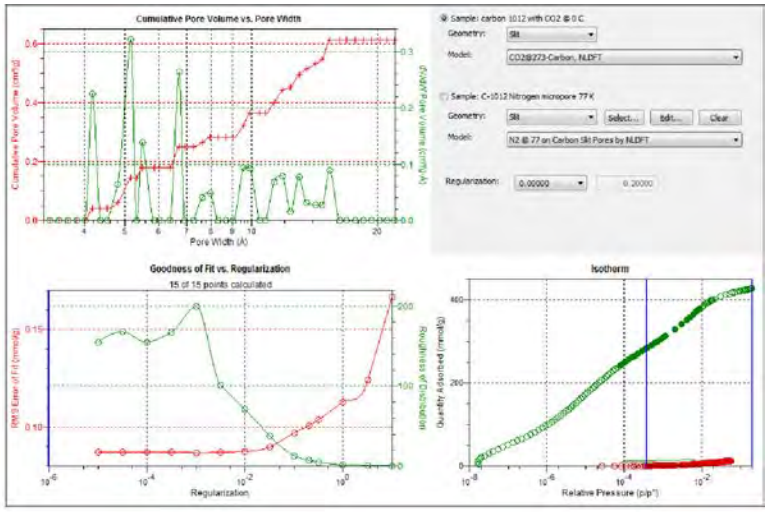
To run the NLDFT report:

1. Go to ***File > Open***. Select a sample file with a *Complete* status, then click **Open**.
2. In the view selector drop-down list at the bottom of the window, select *NLDFT Advanced PSD*. Graphs for the first sample file display and the sample description shows as the first group box title in the upper right corner of the window.



A. First selected sample file
B. Second selected sample file

   a. Select the *Geometry* and *Model* from the drop-down lists for the first sample file.
   b. To select isotherm data points for calculation for the first sample file, ensure the option to the left of the first sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Without a second sample selected, the report will perform a single model DFT calculation and show the results in the two left-hand result windows.
3. To calculate data from the second sample file, click **Select** to locate and open the second sample file with a *Complete* status. Graphs for the second sample file display and the sample description will display as the second group box title in the upper right corner of the window.
   a. Select the *Geometry* and *Model* from the drop-down lists for the second sample file.
   b. To select isotherm data points for calculation for the second sample file, ensure the option to the left of the second sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Data are automatically calculated for both sample files.
   c. Click **Edit** to make any necessary modifications to the second sample file.


Cumulative Pore Volume vs. Pore Width
Pore Width (A)
Sample: carbon 1012 with CO2 @ 0 C
Geometry:
Slit
Model:
CO2@273-Carbon, NLDFT
Sample: C-1512 Nitrogen micropore 77 K
Select...
Edit...
Clear
N2 @ 77 on Carbon Slit Pores by NLDFT
Regularization:
0.00000
0.20000
Goodness of Fit vs. Regularization
15 of 15 points calculated
Regularization
Isotherm
Relative Pressure (p/p°)

OPTIONS REPORT

Physisorption

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.

Chemisorption

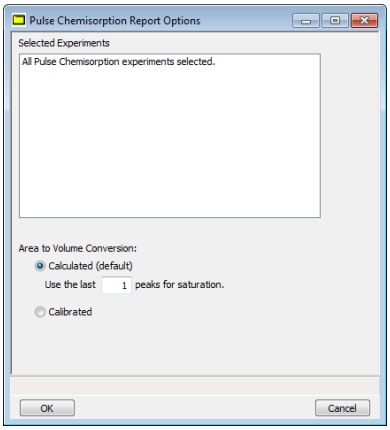
Produces a printed report of a predefined collection of sample file parameters. If *Automatically collected* is selected in the *Type of Data* group box on the *Sample Description* tab, the following information is reported:

- **Task Summary.** Lists conditions specified for each task selected.
- **Analysis Task Options.** Details conditions specified for the analysis task.
- **Experiment Log.** Identifies actual conditions under which each task transpired.
- **Leak Test Results.** Identifies outgas rates and the outcome for each leak test performed.

Dynamic Analyses

Produces a printed report of predefined collection of sample file parameters.

PULSE CHEMISORPTION REPORT



Pulse Chemisorption Report

Selections	Description
Area to Volume Conversion [*group box*]	**Calculated.** Uses the standard calculation and the raw signal. **Calibrated.** Uses selected peaks for the conversion. A calibration step must be included in the experiment to use this method.
Selected Experiments [*group box*]	Displays all related experiments in the current file. Only experiments with collected data are shown.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

Quantity Adsorbed Formulas

Situation 1	Pulse adsorption experiment, no calibration applied in peak editor, report option is *Calculated*. $Qads_x = \frac{L-(L\times\frac{A_x}{Avg_{lastN}})}{Mass_{sample}}$
Situation 2	Pulse adsorption experiment, no calibration applied in peak editor, report option is *Calibrated*. Report displays an error message that calibration is not found and uses the formula from Situation 1.
Situation 3	Pulse adsorption experiment, calibration is applied in peak editor, report option is *Calculated*. $Qads_x = \frac{Q_{end}-Q_x}{Mass_{sample}}$
Situation 4	Pulse adsorption experiment, calibration is applied in peak editor, report option is *Calibrated*. $Qads_x = \frac{L-Q_x}{Mass_{sample}}$
Situation 5	Pulse reaction experiment, no calibration applied in peak editor, report option is '*Calculated.* $Qads_x = \frac{(L\times\frac{A_x}{Avg_{firstN}})-(L\times\frac{A_{end}}{Avg_{firstN}})}{Mass_{sample}}$
Situation 6	Pulse reaction experiment, no calibration applied in peak editor, report option is *Calibrated*. Report displays an error message that calibration is not found and uses the formula from Situation 5.
Situation 7	Pulse reaction experiment, calibration is applied in peak editor, report option is *Calculated*. $Qads_x = \frac{Q_x-Q_{end}}{Mass_{sample}}$
Situation 8	Pulse reaction experiment, calibration is applied in peak editor, report option is *Calibrated.* $Qads_x = \frac{Q_x-0.0}{Mass_{sample}}$

where

A_{end}	=	Area of the last peak, shown as *Peak Area* in peak editor
Ax	=	Area of peak #X, shown as *Peak Area* in peak editor
Avg_{firstN}	=	Average of first N peak areas
Avg_{lasttN}	=	Average of last N peak areas
L	=	Loop quantity calculated from the injection volume, ambient temperature, ambient pressure, and active gas concentration. $$V_{inj} \times \frac{273.15}{T_{amb}+273.15} \times \frac{P_{amb}}{760mmHg} \times \frac{C_{active}}{100.0}$$
$Mass_{sample}$	=	Sample mass (grams)
N	=	*Use the last N peaks for saturation* in the Pulse Chemisorption Report Options. For pulse reaction experiments (where gas is emitted by the reaction and peak areas decrease over time) this is the first N peaks.
$Qads_x$	=	*Quantity Adsorbed per gram* for peak #X, shown in the Pulse Chemisorption Report's peak table. The report sets negative values to 0.0. To get total quantity adsorbed, multiply by $Mass_{sample}$ and sum across peaks.
Q_{end}	=	Quantity of the last peak, shown as *Peak Quantity* in peak editor.
Q_x	=	Quantity of peak #X, shown as *Peak Quantity* in peak editor

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

Not applicable to 21CFR11 environments. See ***Sample Audit Trail Report above***.

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.

SINFELT AND DIFFERENCE METHODS

Difference Method Report on page 8 - 17

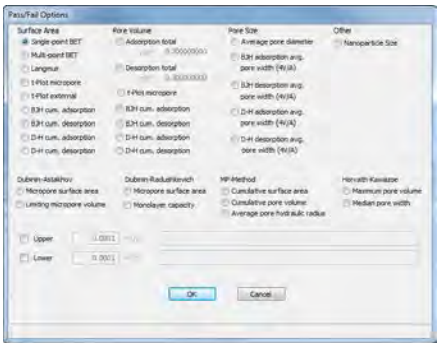
The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified.

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

Selections	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)

Selections	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 - 3*.

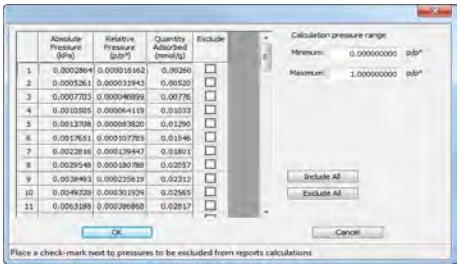
Summary Report for Dynamic Analysis

The *Summary Report* for dynamic analyses provides a condensed summary of the peaks and selected analysis parameters.

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

Selections	Description
Fitted thickness range [*text box*]	Enter the minimum and maximum thicknesses (in angstroms or nano-meters) 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)

Selections	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.

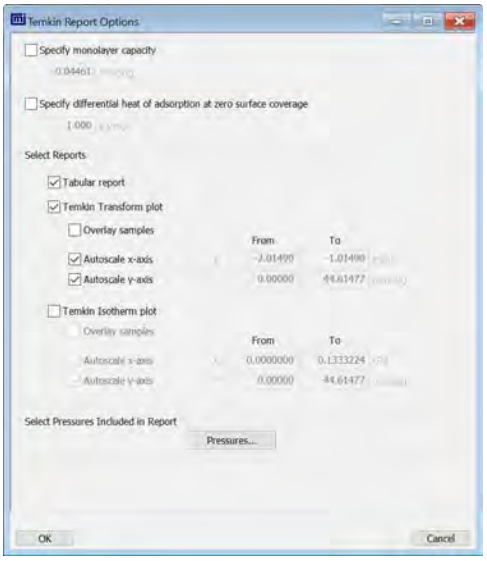
t-Plot Report (continued)

Selections	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 - 3*****.**

TEMKIN REPORT

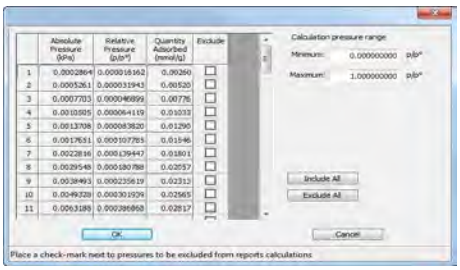
The *Temkin* isotherm is used to model adsorption data where the heat of adsorption drops linearly with increasing coverage.



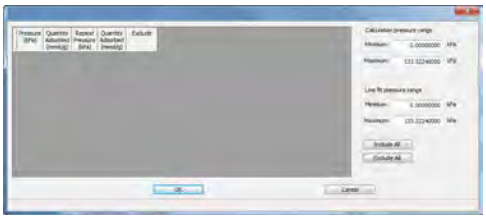
Physisorption

Chemisorption

Temkin Report

Selections	Description
Calculation [*group box*] C	Select one or more of the calculation options to be used for analysis.
Pressures [*button*] P	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.

Temkin Report (continued)

Selections	Description
	Include All. Select to include all pressure points in the table.
Pressures [*button*]	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. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Line fit pressure range.** Enter the minimum and maximum pressures for line fit.
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.

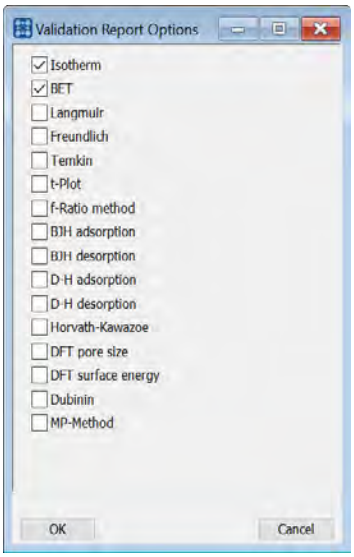
Temkin Report (continued)

Selections	Description
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 - 3*****.**

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.



9 DIAGNOSTICS

Unit [n] > Diagnostics

Use to display diagnostic readings, start and schedule 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.

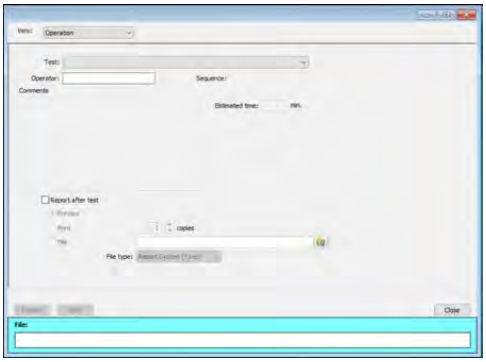
START DIAGNOSTIC TEST

Unit [n] > Diagnostics > Start Diagnostic Test

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.

It is recommended to schedule the *Analysis Manifold Leak Test* and the *P0 Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.

The *P0 Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.



Start Diagnostic Test

Selections	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.

Start Diagnostic Test (continued)

Selections	Description
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 - 3*.

SCHEDULE DIAGNOSTIC TESTS

Unit [n] > Diagnostics > Schedule Diagnostic Tests

Allows the specification of one-time or periodic running of a sequence of diagnostic tests. A separate list of tests is saved for each of the possible test frequencies. Tests are categorized and flagged as requiring intervention or not. If tests requiring intervention are scheduled, the operator has the option of omitting the tests if the operator does not respond within a specified time after an initial prompt is displayed and before the test is started. Events are logged in the analyzer log for all starting, ending, and omitted tests.

It is recommended to schedule the *Analysis Manifold Leak Test* and the *P0 Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.

The *P0 Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.

Schedule Diagnostics Test Frequency

Selections	Description
Available Tests [*drop-down box*]	Select one or more tests to run unattended.
Insert [*button*]	Inserts the selected test in the *Available Tests* drop-down list.

Schedule Diagnostics Test Frequency (continued)

Selections	Description
Skip these tests if the operator does not respond within [*n*] minutes [*check box***]**	Check this option if any test requiring operator intervention should be omitted if the operator does not respond within the specified time.
Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00 [*text box***]**	Enter a from and to time for an unattended test to begin if the instrument is idle at any time during the entered time frame.
Test Frequency [*selection***]**	Select how often the test is to run unattended.
Test Sequence [*group box***]**	Provides the test file identification and estimated run time. A checkmark in the *Intervention Required* column indicates that operator intervention is required. To remove a test from the sequence, select the test, then click **Delete**. To add a test to the test sequence, highlight a row in the *Test Sequence* box, select a test from the *Available Tests* list, then click **Insert**.

For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 3*****.**

DIAGNOSTIC TEST REPORT

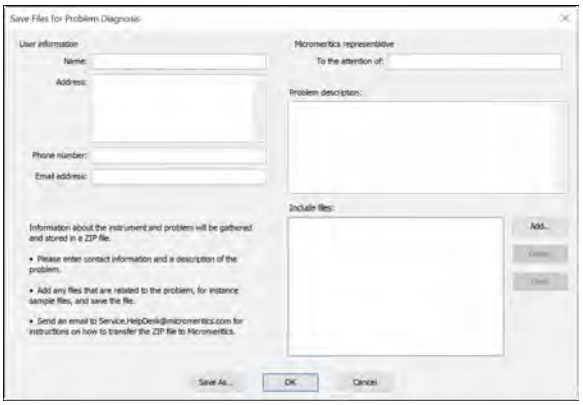
Unit [n] > Diagnostics > Diagnostic Test Report

Displays previously run diagnostic service tests. Separate directories store tests run once, daily, weekly, and monthly. Diagnostic test report files have a .SVT file extension and are stored in the ...\Service directory.

SAVE FILES FOR PROBLEM DIAGNOSIS

Unit [n] > Diagnostics > Save Files for Problem Diagnosis

Use to compress pertinent diagnostic information into a single zip file. This file can be sent to a Micromeritics Service Representative for problem resolution.



1. Complete the form. A default file named *Diagnostics-[date].zip* is created unless another file name is specified.
2. Add any files that are related to the problem — such as sample files — and save the file.
3. Send an email to Service.Helpdesk@Micromeritics.com for instructions on how to transfer the .ZIP files to Micromeritics.

Save Files for Problem Diagnostics

Selections	Description
Problem description [*text box*]	Enter information that would be helpful to the Micromeritics representative.
Include Files	**Add.** Click to select additional files to send with this problem diagnosis. **Delete.** Select the file in the *Include Files* box, then click **Delete** to remove the file from the list. **Clear.** Click to clear all files from the *Include Files* box.
Save As [*button*]	Click to specify the name and location of the compressed file. Make a note of the file name and location. This file will need to be sent to your Micromeritics representative for problem resolution.
Micromeritics representative [*text box*]	Enter the name of your Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).
User Information	Enter information for the person to be contacted by a Micromeritics

Save Files for Problem Diagnostics (continued)

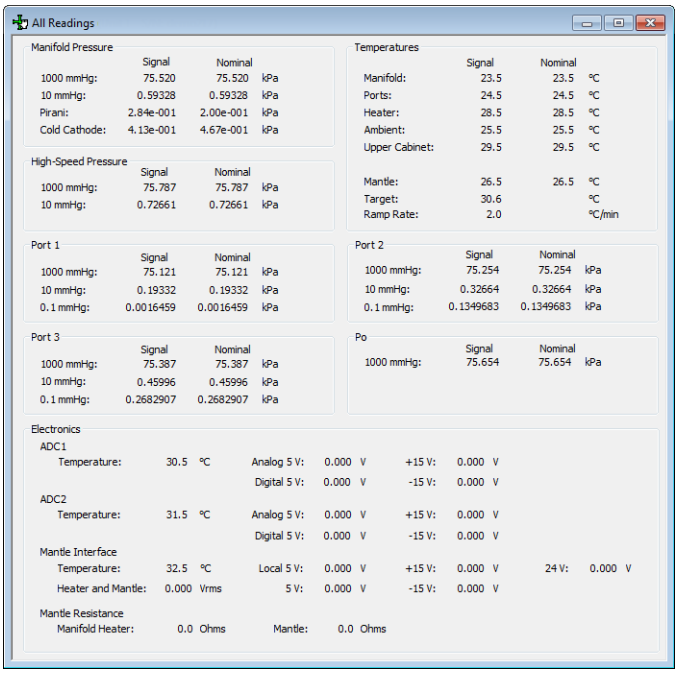
Selections	Description
[*text box*]	representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

SHOW ALL READINGS

Unit [n] > Diagnostics > Show All Readings

The *Show All Readings* window displays the calibrated and nominal readings of all sensors in the system.



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10 CALIBRATION

Unit [n] > Calibration

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

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.

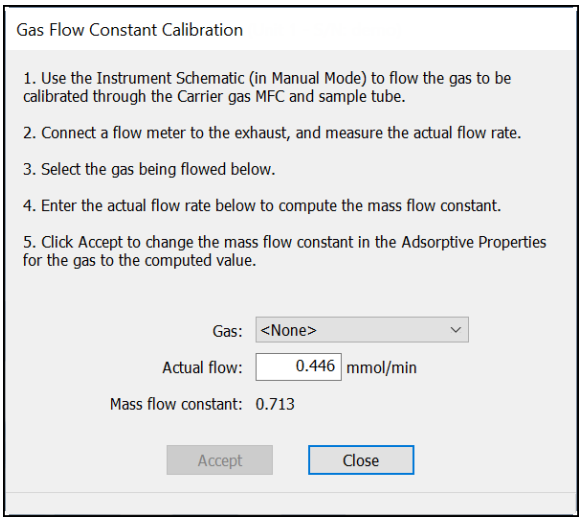
To review calibration details of the analyzer, go to ***Unit [n] > Unit Configuration***.

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.

GAS FLOW CALIBRATION

Unit [n] > Gas Flow Calibration

Gas Charts on page I - 1



Use the *Gas Flow Constant Calibration* option to determine a constant used by the analyzer to ensure accurate gas flows through each Mass Flow Controller (MFC). The Mass Flow Controllers were calibrated before shipping and calibration is typically not necessary.

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.*** Flow the gas to be calibrated through the Carrier gas MFC and sample tube.
2. Connect a flow meter to the exhaust and measure the actual flow rate.
3. In the *Gas* drop-down box, select the gas to be flowed. In the *Actual flow* field, enter the actual flow rate to compute the mass flow constant.
4. Click **Accept** to change the mass flow constant in the *Adsorptive Properties* for the gas to be the computed value.

MASS FLOW CONTROLLER CALIBRATION

Gas Charts on page I - 1

The analyzer uses a Mass Flow Controller (MFC) to control the flow of gases. The MFC requires a conversion constant for each gas or gas mixture to compensate for variations in gas flows resulting from variations in the gas properties.

In most cases, the default MFC conversion constant yields accurate data. A new conversion constant may be used if:

- A unique gas mixture is used.
- The gas to be used is not included in the table.
- A higher precision calibration of the MFC for a given gas is required.
- Unexpected analysis data lead you to believe that the default value is in error.

FLOW MEASUREMENT

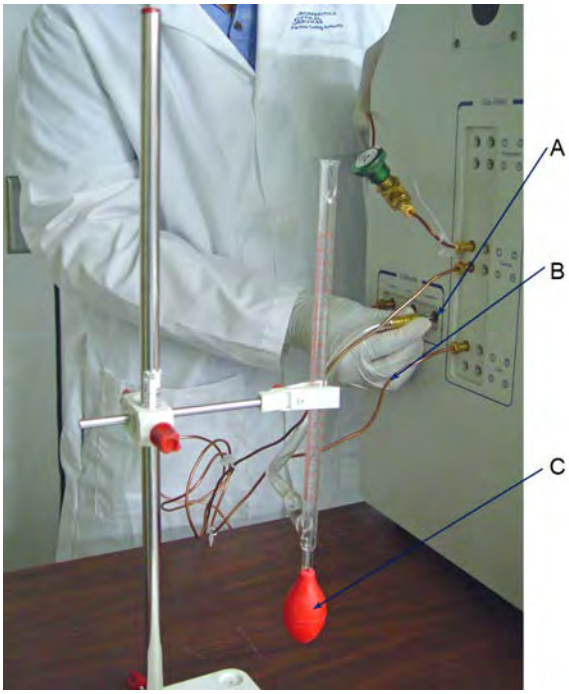
An external device that measures the flow of gas from the analyzer's exhaust is used during MFC calibration. A soap bubble burette is such a device.

For even more precise calibration, use another type of flow meter and follow these instructions for obtaining a new conversion constant, substituting your flow meter for the soap bubble burette.

INSTALL A SOAP BUBBLE BURETTE OR OTHER TYPE OF FLOW METER

These instructions are for assembling and using a burette obtained from Micromeritics. If using another type of flow meter, follow the meter manufacturer's assembly and operation instructions.

1. Carefully unwrap the glass tube and the rubber bulb.
2. Attach one end of the flexible tubing to the side arm of the glass tube.
3. Attach the metal tubing provided with the bubble burette to the other end of the flexible tubing.



A. Exhaust port
B. Flexible tubing
C. Bulb filled with leak detector

4. Locate the exhaust port that corresponds to the MFC to be used for this calibration.
5. Remove external plumbing from that port and attach the bubble burette using the connector provided with the burette. The soap bubble burette must be held in an upright position. For example, if using the Carrier Flow Controller, attach the bubble burette to the carrier gas exhaust port.
6. Attach the bulb to the bottom of the glass tube.

DETERMINE THE GAS FLOW CONSTANT FOR INDIVIDUAL GASES

Unit [n] > Gas Flow Calibration

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. Flow the gas to be calibrated through the Carrier gas MFC and sample tube.
3. Connect a flow meter to the exhaust and measure the actual flow rate. If using a bubble burette, observe a bubble as it rises through the glass tube. The lines on the glass tube indicate the beginning and ending points for measuring the progress of a bubble through the tube. Use a stopwatch to measure the amount of time that elapses from the moment the bubble passes the lower mark on the tube until the moment it passes the higher mark on the tube.
4. Go to ***Unit [n] > Gas Flow Calibration***.
5. Select the gas being flowed.
6. In *Actual flow* field, enter the actual flow rate to compute the mass flow constant.
7. Click **Accept** to change the *Mass flow constant* field in the *Adsorptive Properties* for the gas to the computed value.

MATCH TRANSDUCERS

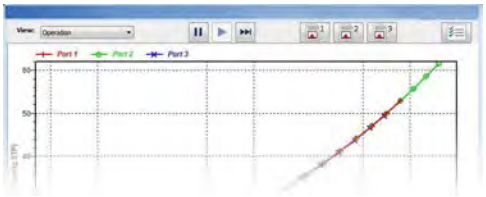
Unit [n] > Calibration > Match Transducers

CAUTION

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

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.



0.1 Torr transducer is not shown

1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. The window closes when the operation is complete. Click **OK** when the process is complete.

Match Transducers

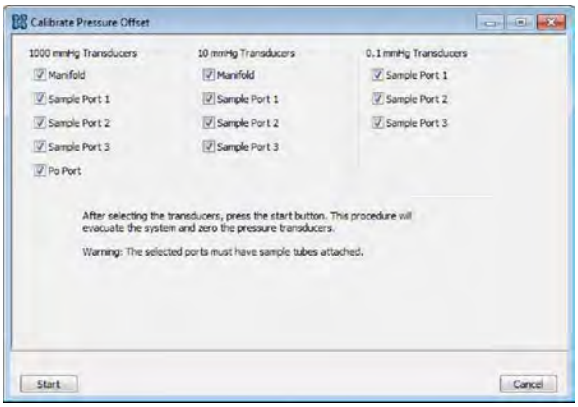
Selections	Description
1000 mmHg Transducers	Select the ports.
10 mmHg Transducers	Select the ports. Enabled only for ports with 10 mmHg transducers present.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

PRESSURE OFFSET

Unit [n] > Calibration > Pressure Offset

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.

Pressure Offset

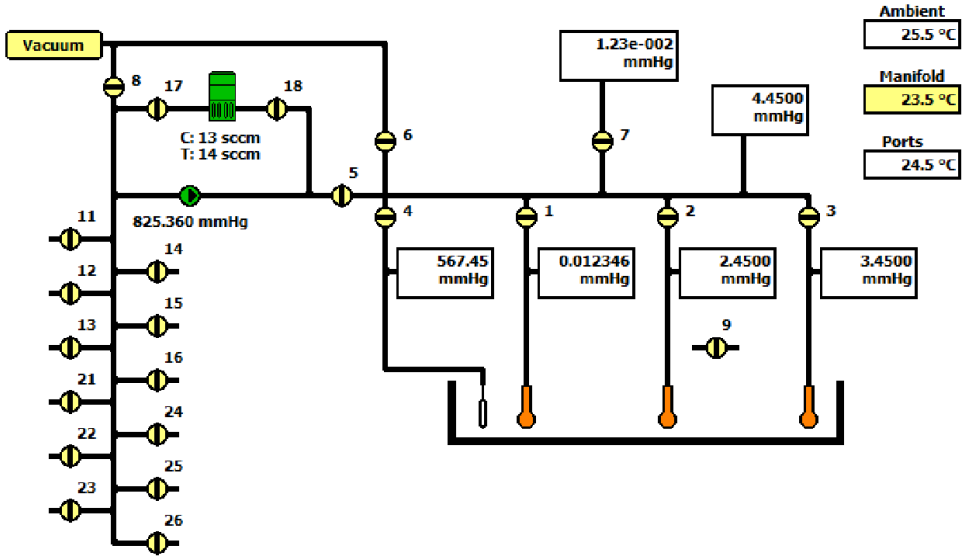
Selections	Description
1000 mmHg Transducers	Select the manifold and/or ports.
10 mmHg Transducers	Select the manifold and/or ports. Enabled only for the manifold and ports with 10 mmHg transducers present.
0.1 mmHg Transducers	Select the ports. Enabled only for ports with 0.1 mmHg transducers present.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

CHECK PRESSURE TRANSDUCER SCALE CALIBRATION

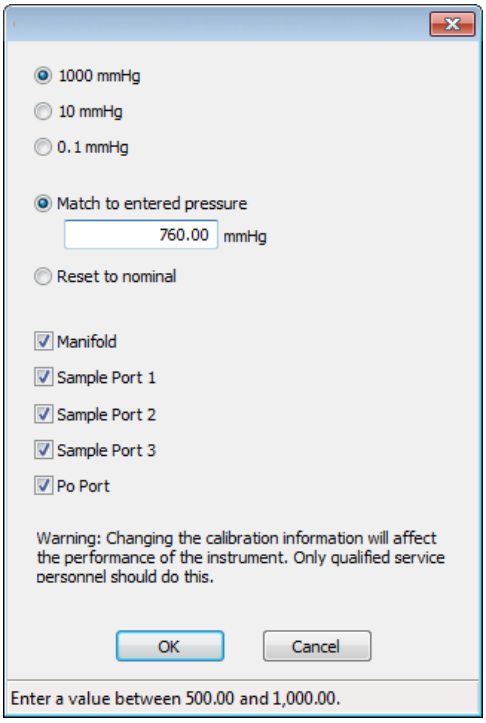
CAUTION

Do not change the pressure calibration unless absolutely necessary. Consult with Micromeritics Norcross, GA prior to changing calibration settings.



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. Ensure valves 6, 7, and 8 are closed.
3. Open valves 1 through 5 and the N_2 gas valve.
4. Set the servo to dose automatically to 760 mmHg by right-clicking the servo valve, select *Set* > *Dose* > *Automatic,* then enter *760 mmHg*.
5. Remove one of the port plugs. The pressure on all transducers will read atmospheric pressure.
6. Check all transducer readings against the external pressure gauge. If the readings are within 2 mmHg, do not change the transducer's calibration.
7. To calibrate, go to ***Unit [n] > Calibration > Pressure Scale***.
8. Use a pressure gauge to determine atmospheric pressure.

9. Select the appropriate transducer.



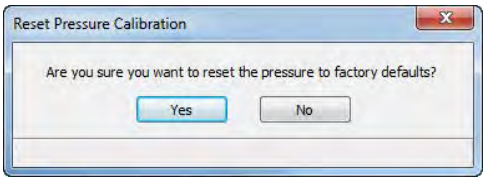
10. Enter the pressure from the pressure gauge in the *Match to entered pressure* field.
11. Click **OK** to close the window.
12. A confirmation message is displayed. Click **OK**.

The 10 mmHg and 0.1 mmHg transducers should not be calibrated at this time.

RESET PRESSURE CALIBRATION

Unit [n] > Calibration > Reset Pressure Calibration

This procedure resets the pressure calibration to the factory default settings.



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

SERVO VALVE

Unit [n] > Calibration > Servo Valve

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.

SIGNAL CALIBRATION FOR DYNAMIC ANALYSIS

Unit [n] > Signal Calibration > New

Analyses yield data on signal reading, peak area, temperature, and time. These data are sufficient for many applications; however, volume data may also be needed.

It is not necessary to perform a calibration if volume data are not needed. If volume data are needed, calibration may be performed either before or after the analysis.

This does **not** apply to loop calibration, ambient temperature, and atmospheric pressure. The correct values for these quantities must be entered under ***Unit [n] > Unit Configuration*** and ***Options > Environment Defaults*** before starting an analysis that uses them to determine the quantity of gas in an injection.

A group of automatic calibration routines is provided in the form of specialized experiment steps. A calibration run is an analysis using one of these experiment steps. The calibration run can be performed before or after the sample analysis. It can be included as a step within the analysis, or performed as a separate analysis.

After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example, TPR yields peak area and the temperature at which maximum reduction occurs. To obtain the volume of gas uptake, a calibration file must be associated with the analysis file. Then, reports are created in which the area data are converted to volume data.

A calibration file can be associated with a sample file by doing the following:

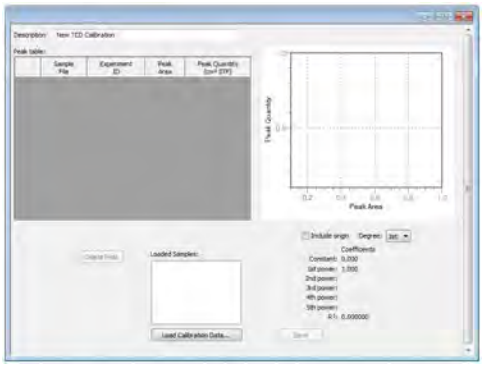
- Going to ***Unit [n] > Signal Calibration > Defaults*** and selecting a default calibration file for each experiment type.
- Using the default file or choose a different file in the *Unit [n] > Sample Analysis* window.
- Clicking **Set Calibration** on the *Peak Editor* window and select from a list of calibration files created after the sample file was used in an analysis.

CREATE A NEW SIGNAL CALIBRATION FILE

Unit [n] > Signal Calibration > New

When selecting a *Signal Type* and *Type of Calibration*, only those options used in the original analysis are enabled on the right side of the window.

1. Select the sample file to use for the new signal calibration file.
2. Select the *Signal Type* and *Type of Calibration* options to include in the new calibration file. As selections are made, the experiments from the selected analysis file display on the right side of the window. Only those experiments applicable to the selected signal type and calibration type are enabled. Click **OK**.
3. If prompted, enter the active gas concentration in the carrier gas and loop gas. Click **OK**.
4. Delete (or edit) data in the *Peak Table* as appropriate. Click **Save**.



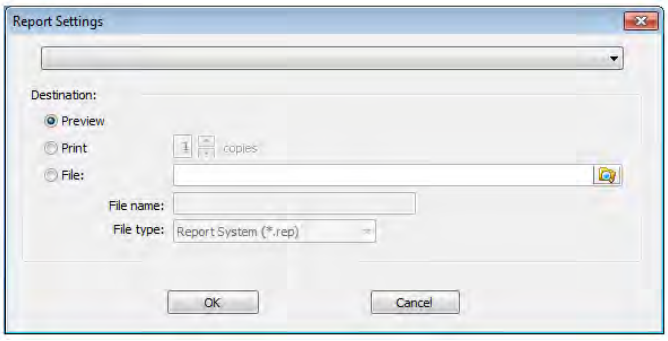
Signal Calibration

Selections	Description
Degree [*drop-down box*]	Select the power to display in the *Coefficients* list.
Delete Peak [*button*]	Removes a peak from the peak table. Select the peak and click **Delete Peak**.
Description [*text box*]	Enter a description of the file.
Include origin [*check box*]	Enables or disables the original in the graph.
Load Calibration Data [*button*]	Use to enter calibration data from a selected file.

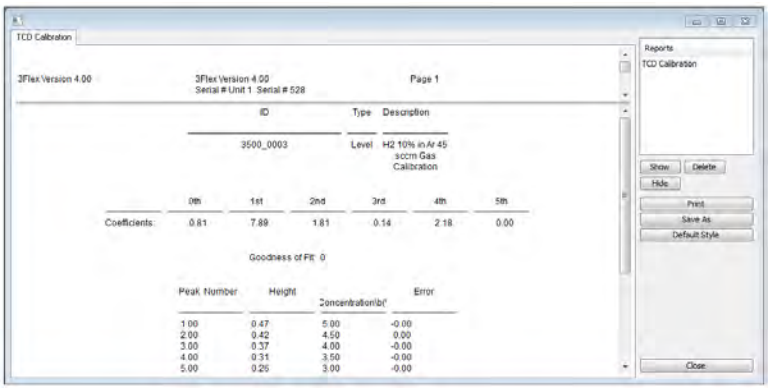
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

CREATE A SIGNAL CALIBRATION REPORT

Unit [n] > Signal Calibration > Report



Select a previously defined signal calibration file from the drop-down list, then select the print destination.



OPEN A SIGNAL CALIBRATION FILE

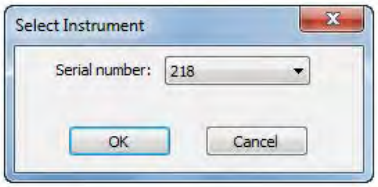
Unit [n] > Signal Calibration > Open

Select a previously defined signal calibration file from the drop-down list. If a calibration file already exists, select the serial number from the drop-down serial number list. When a new calibration file is saved with a new serial number, the new serial number displays in the *Serial Number* drop-down list.

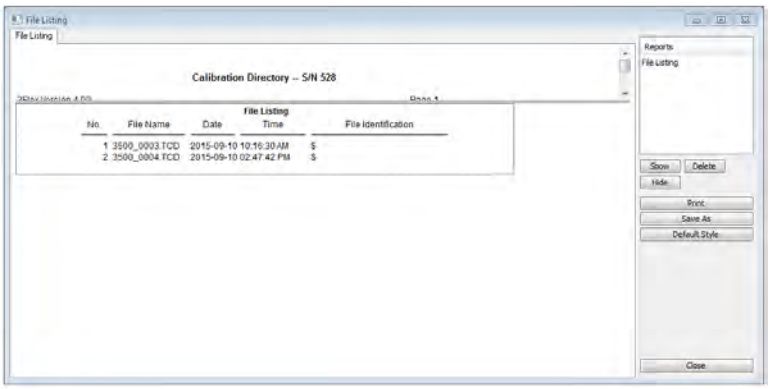
If this is the first calibration file to be saved, the *Serial Number* drop-down will not display until the first calibration file is saved.

LIST SIGNAL CALIBRATION FILES

Unit [n] > Signal Calibration > List



Displays a list view of previously defined signal calibrations for the selected serial number.



LOOP CALIBRATION FOR TCD ANALYZERS

Septum on page 11-12

Each calibration loop must be calibrated prior to its first use to determine its precise volume under local conditions. Calibration consists of:

- Determining the average area of a series of peaks generated by injections of a known volume of gas through the analyzer septum using a syringe.
- Determining the average area of a series of peaks generated by injections of the same gas using the analyzer's internal loop.
- Calculating the volume of the loop by comparing the average peak area generated by the loop injections with that generated by the syringe injections.
- Entering the calculated loop volume under ***Unit [n] > Unit Configuration***.

Options > Environmental Defaults

Environmental Defaults for TCD Analyzers on page 2 - 20

Ambient pressure and temperature at the time of the analysis affect the results of a loop calibration. In calculations, the analyzer uses the ambient pressure and temperature recorded in *Environmental Defaults*. Check and update the *Environmental Defaults* before beginning a loop calibration.

STEP 1: CREATE A SAMPLE FILE

Create the sample file and insert a *Loop Calibration* experiment on the *Analysis Conditions* tab.

A loop calibration must be created for each loop. For Loop Calibration, select a carrier gas, flow rate, and loop gas that will commonly be used for sample analyses.

STEP 2: PERFORM THE ANALYSIS

1. Install the correct injection loop.
2. Go to ***Unit > Sample Analysis*** and select the sample file for the loop calibration.
3. Click **Next** to accept the default values, then click **Start**.

Pay close attention to the instructions provided in each prompt and perform the steps in the order given. Most accurate data results from keeping injection size as consistent as possible. Injection errors may be evident in the data and may make it necessary to repeat the experiment.

After the last manual injection, the analyzer automatically makes the same number of injections using the loop.

STEP 3: GENERATE THE REPORT

Peak Editor for Dynamic Analysis on page 7 - 10

When properly performed, each manual and automatic injection results in a peak. When the *Results* view of the *Start Analysis* window is selected, each peak can be viewed as it is collected. The area under the peak corresponds to the amount of gas injected.

1. In the *Peak Editor*, open the sample file to ensure the peaks are properly marked.
2. Go to ***File > Open > [.SMP]*** and open the .SMP file containing the calibration experiment and verify (or correct) the defined peaks using the *Peak Editor*.
3. On the *Reports* tab, select only the *Loop Calibration* report option.
4. Go to ***Unit > Unit Configuration***. In the *Loop volume* field, enter the *Loop Volume*.

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

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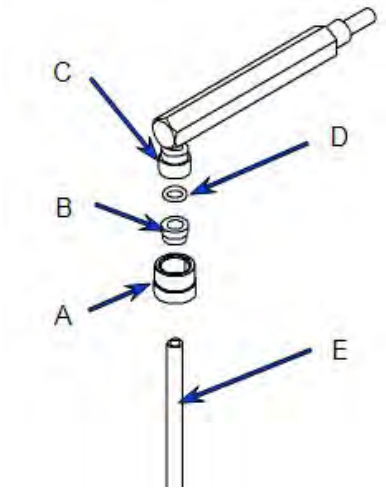
11 HARDWARE

COLD TRAP TUBE



A. Connector nut
B. Ferrule
C. Sample port
D. O-ring
E. Sample tube

To remove the cold trap tube

1. Loosen the connector nut on each stem of the cold trap tube.
2. Gently pull down on the cold trap tube to remove it from the ports. The connector nut and ferrule will remain on the cold trap tube stems. If the O-ring remains on the cold trap tube stem, examine the O-ring to ensure it is still in good condition prior to re-inserting it into the port. Replace the O-ring, if necessary. If the O-ring remains in the port, it may be gently pried out for replacement.

To Install the cold trap tube

1. Reassemble the cold trap tube. On each stem, assemble the connector nut, a ferrule (large side pointing toward the port), and an O-ring.
2. Slide each stem fully into the port, then tighten the connector nuts.

Prepare the Cold Trap for Idle Periods

If the analyzer is to be inoperative or unattended long enough for the liquid nitrogen in the cold trap Dewar to evaporate (or the dry ice to evaporate from the dry ice acetone slurry), remove the Dewar and U-tube so that impurities are not released into the system. Install a clean, dry U-tube. If additional U-tubes are unavailable, clean the current tube and allow to dry; then reinstall at the cold trap port.

If the analyzer is to be inoperative or unattended for a few days, reduce the gas flow to approximately one-quarter of its normal value and leave analyzer powered ON. The gas loss and power drain are very low and the analyzer will be immediately ready for use.

Regardless of how long the analyzer is to be idle, keep a sample tube installed at both sample ports. This ensures the integrity of the system for gas flow and prevents infusion of water and other vapors.

Enable Manual Control

Unit [n] > Enable Manual Control

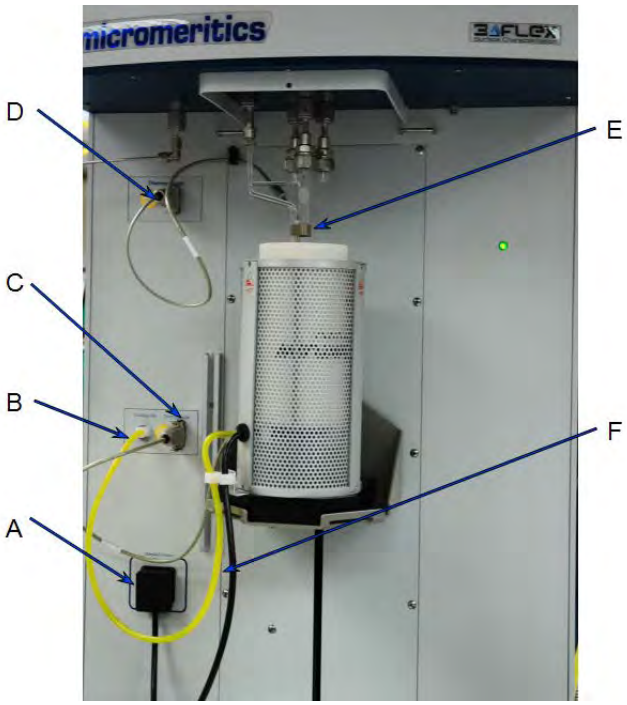
Show Instrument Schematic on page 2 - 24

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***.

FURNACE INSTALLATION FOR CHEMISORPTION

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

The furnace uses the same supply of compressed air as the instrument. There is an internal regulator that has been set at the factory for the correct flow rate of cooling air to the furnace.



A. Furnace power outlet
B. Cooling air connector
C. Furnace thermocouple connector
D. Sample thermocouple connector
E. Sample thermocouple clipped to sample tube
F. Tygon tubing

NOTE: This photo is shown without the safety shield. The safety shield should always be installed prior to running an analysis.

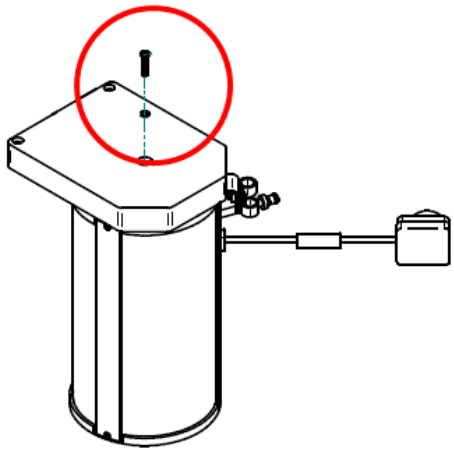
1. If the heating mantle is installed, it must first be removed and replaced with the furnace. To remove the heating mantle, unplug the heating mantle thermocouple connector and heating mantle power source from the front panel of the instrument.
2. Place and center the furnace on the elevator tray.
3. On the front of the analyzer, connect cables in the following order:
    a. Furnace thermocouple
    b. Furnace cooling air connector
    c. Furnace power connector
    d. Install the sample thermocouple.
    e. Insert the sample thermocouple connector into the thermocouple outlet
    f. Secure the thermocouple cable into the p-clip.

SECURE FURNACE TO ELEVATOR TRAY

This is an optional procedure for securing the furnace to the elevator tray in areas prone to earthquakes.

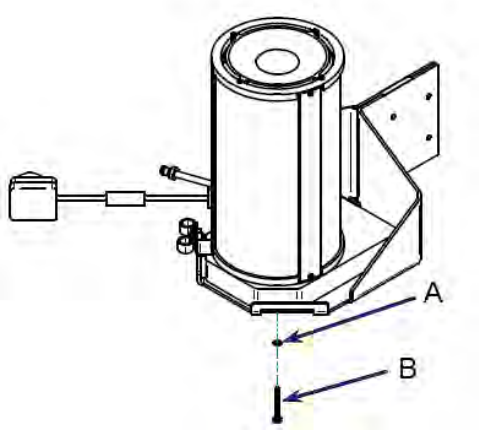
Replace the screw that holds the black plastic base to the furnace and replace it with a 8-32 × 1.5 in. screw [*part number 004-28622-00*].

1. Invert the furnace and remove the screw that holds the black plastic base to the bottom of the furnace. Save the lock washer and use it when inserting the longer screw.



Remove screw and lock washer. Save the lock washer for the next step.

2. Place the black plastic base onto the elevator aligning the hole in the base with the hole in the elevator.
3. Position the furnace on top of the black plastic base aligning the hole in the furnace bottom with the hole in the furnace base.



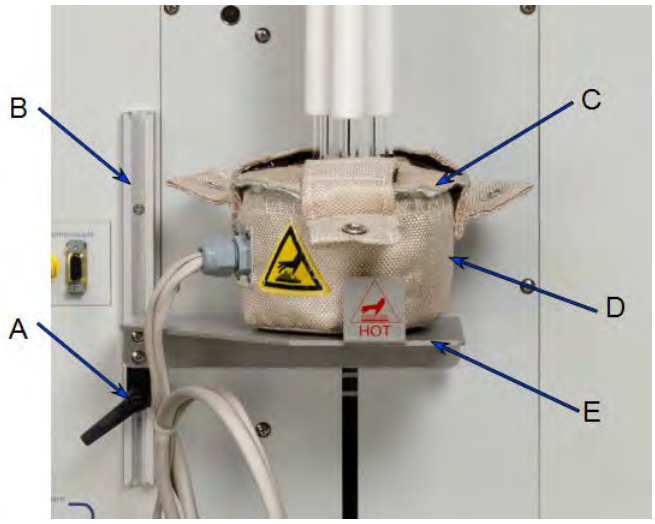
A. Lock washer
B. 8-32 × 1.5 in. screw

4. Raise the elevator. Position the lock washer in the hole in the elevator tray. Insert the 8-32 × 1.5 in. screw through the lock washer, the hole in the elevator, the black plastic base, and into the furnace.

HEATING MANTLE INSTALLATION

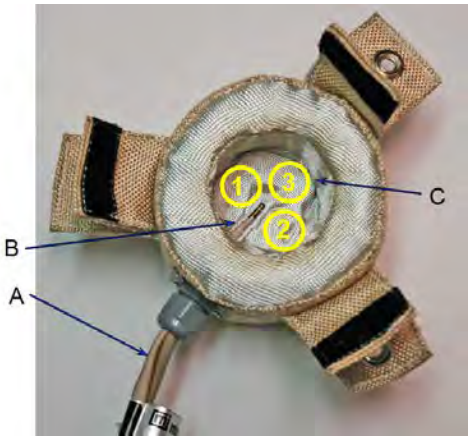
Pivot Shelf Installation on page 11-10

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



A. Shelf locking mechanism
B. Shelf track
C. Heating mantle lid
D. Heating mantle
E. Heating mantle shelf

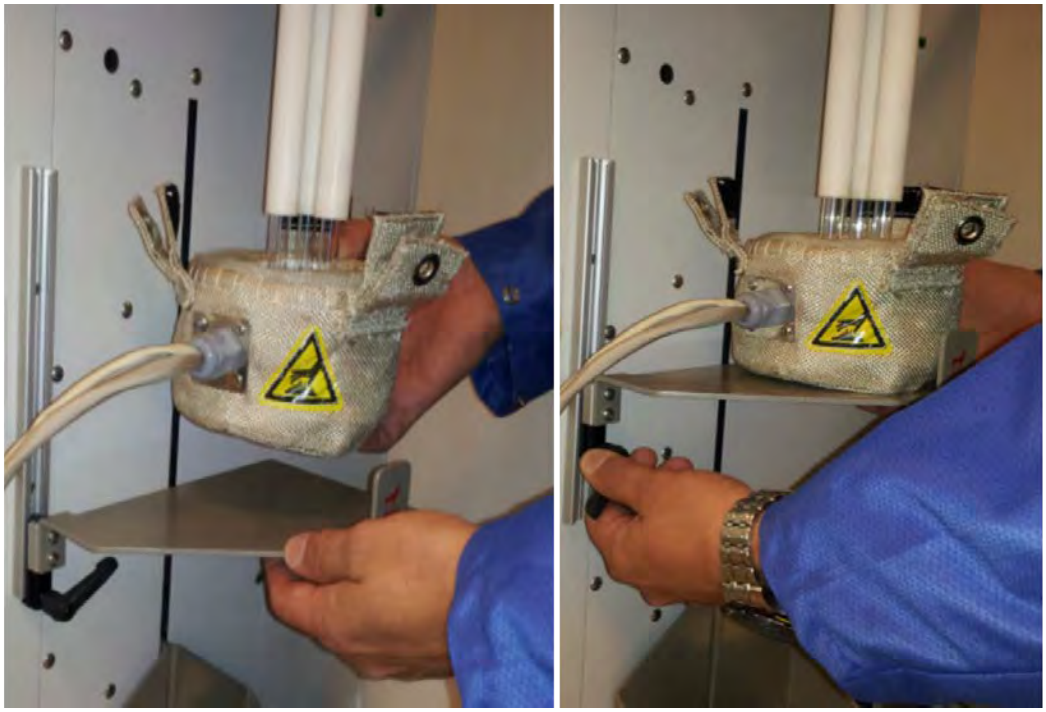
If using less than three sample tubes, position the heating mantle cable in the 8 o'clock position when resting on the shelf surface. Ensure that the bottom of all sample tubes are in full contact with the bottom of the heating mantle interior surface only and DO NOT touch the thermocouple located on the bottom surface of the mantle's interior. A single sample tube must be installed on port 2.



A. Place cable in 8 o'clock position
B. Thermocouple
C. Sample tube positions

1. Place the mantle around the sample tube bulbs. Ensure that the isothermal jackets are pushed up against the Dewar cover to avoid damage to the jackets.

2. While supporting the heating mantle with one hand, slide the shelf locking mechanism into the shelf track.
3. Raise the shelf on the track until the heating mantle rests securely on the shelf and the sample tubes touch the bottom of the inside of the heating mantle.
4. Turn the locking mechanism clockwise to secure the shelf.



5. Slide the heating mantle cover between the sample tube bulbs and the bottom of the isothermal jackets so that the sample tubes fit within the slots of the mantle cover.

Do not to apply force to the tubes while installing the lid.

6. Secure the heating mantle tabs onto the hook and loop fasteners of the heating mantle cover. Ensure there is at least a 1/2 in. (12 mm) gap between the top of the mantle cover and the bottom of the isothermal jackets. This will prevent damage to the jackets. Replace any damaged jackets.
7. Insert the mantle thermocouple and the mantle power plug into the analyzer's front panel.

A. Thermocouple connector
B. Mantle/furnace power connector

6. Acknowledge the prompt on the *Sample Analysis* window. The degas will proceed. When the degas is completed and the mantle has cooled below 45 °C, the *Sample Analysis* window will submit a prompt to remove the degas heating mantle and shelf, properly position the isothermal jackets and Dewar cover, and install the Dewar.

To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled.

7. To remove the heating mantle, take off the heating mantle cover, support the bottom of the heating mantle, then lower the shelf. The shelf must be removed prior to installing the Dewar.

PARTS AND ACCESSORIES

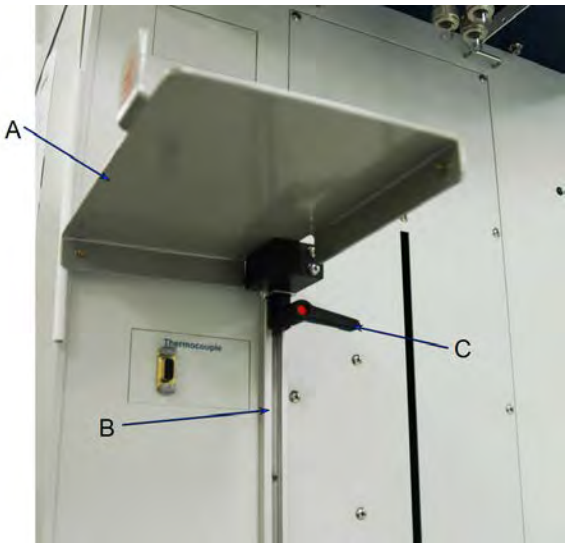
Parts and accessories are located on the Micromeritics web page.

PIVOT SHELF INSTALLATION

Heating Mantle Installation on page 11-6

A pivot shelf is used to support the vapor source container or the heating mantle. It can be pivoted for installation, removal, or height adjustment.

INSTALL THE PIVOT SHELF



A. Slide rail
B. Pivot tray
C. Lever

1. Hold the pivot shelf in your left hand and the pivot block and lever (underneath the shelf) with your right hand.
2. Pivot the shelf to the left to expose the block and lever.
3. Insert the slide block into the slide rail on the front of the analyzer.
4. To tighten the shelf into position, start with the lever to the right, push and turn the lever clockwise.
5. Release the lever, then move the lever back to the right position and again push and turn the lever clockwise.
6. Continue this process until the pivot shelf is snug in the slide rail.

REMOVE OR ADJUST THE PIVOT SHELF

1. Pivot the pivot shelf to the left to expose the pivot block and lever underneath the shelf.
2. Hold the pivot shelf in your left hand and the pivot block lever with your right hand.
3. To loosen the tension on the pivot shelf, start with the lever to the left, push and turn the lever counterclockwise.
4. Release the lever, then move the lever back to the left position and again push and turn the lever counterclockwise.
5. Continue this process until the pivot shelf can move on the slide rail.

SEPTUM

The septum is used to inject quantities of gas into the analyzer.

Septum port

CAUTION Do not use the septum at pressures above ambient. Gas may escape at elevated pressures.

Accuracy of data is diminished when poor techniques are used for injecting gas through the septum.

Injecting the gas through the septum causes a peak to appear, but it also causes a perturbation in the flow of gas through the analyzer. This perturbation is visible in the peak data. To minimize this perturbation, inject the gas more slowly into the septum. Prolonging the injection causes the peak to spread.

An injection method should be developed to balance the need to minimize the perturbation with the need for sharper peaks. See *Peak Editor for Dynamic Analysis on page 7 - 10* to adjust peak data to reduce the effects of perturbation.

- Always hold the syringe by its metal parts away from the needle. Holding the syringe by the glass allows body heat to affect the volume of gas in the syringe.
- After filling the syringe, allow the syringe to lie on a room temperature surface for about a minute. This ensures that the syringe and its contents are at room temperature.
- If the gas used is lighter than air, do not allow the filled syringe to remain in a vertical position (needle up) — the gas will diffuse out and the total volume will be reduced.

Fill the syringe

1. Empty the syringe completely.
2. Insert the syringe into a septum accessory installed on the gas regulator.
3. Draw the syringe plunger back until the syringe is completely filled with gas.
4. Remove the syringe from the septum and allow it to return to room temperature as described previously.
5. Press the plunger into the syringe until the correct amount of gas is contained in the syringe.

Alternate method to fill the syringe with nitrogen (evaporating from liquid nitrogen

1. Empty the syringe.
2. Hold the tip of the needle just above the surface the liquid nitrogen.
3. Draw the syringe plunger back until the syringe is filled with nitrogen gas.
4. Allow the syringe to stabilize at room temperature.
5. Push the plunger into the syringe until the correct amount of gas is contained in the syringe.

Inject gas into the analyzer

1. Insert the needle fully into the septum.
2. Press the plunger into the syringe completely to ensure that the entire quantity of gas is injected from the syringe.
3. Remove the syringe.

SEPTUM REPLACEMENT

Septum port

The septum usually requires replacing after approximately 100 injections when using the 1 mL syringe.

1. Turn the knurled nut counterclockwise and remove it from the injection port.
2. Tap the nut into the palm of your hand to remove the septum and discard the used septum.
3. If the washer came out when the septum was removed, place the washer back into the knurled nut first.
4. Place a new septum into the knurled nut.
5. Place the knurled nut back onto the injection port. Turn the nut clockwise to finger tighten.

Do not use a wrench to tighten the septum retaining nut. Do not overtighten the retaining nut. Doing so may damage the septum or the fitting.

TCD Assembly

The filaments in the thermal conductivity detector assembly are heated to high temperatures and may be exposed to corrosive gases, therefore they may eventually need to be replaced. Symptoms that the TCD filaments may need replacing:

- A stable TCD baseline cannot be maintained.
- Data collection results in very high or very low peaks that are inconsistent with previous experience.
- During a data collection, no TCD signal is displayed on the main display, or the application indicates that no TCD signal is present.

Clean the TCD Assembly

In some instances, contamination of the thermal conductivity detector filaments will produce the symptoms described above. To clean the filaments, set the filament zone to 150 °C and flow an inert gas through the analyzer for several hours. Contact your Micromeritics service representative if necessary.

VAPOR SOURCE CONTAINER INSTALLATION

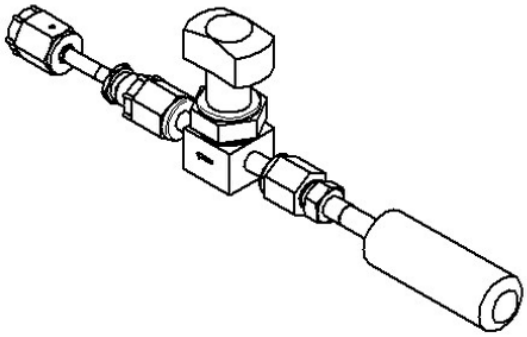
Vapor Purification on page M - 1

Your equipment may differ slightly. The photos in this section use View A; however, View B is installed in the same manner as View A. If using View B, the Vapor Source Mantle will be slightly shorter than shown in this section.

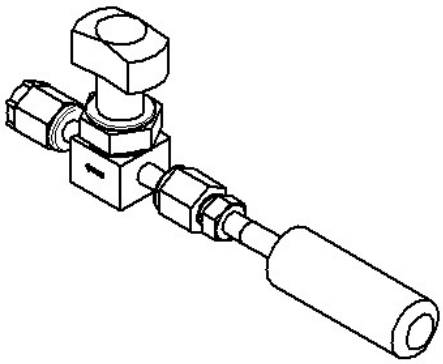
CAUTION

This device has been designed to be used for sample analysis via the analyzer control panel. Any other use may damage this device or the analyzer. Each time the Psat tube or vapor source container is replaced, a new gasket is required. Do not touch the sealing surfaces, the port fitting, or gasket with bare hands.

For chemisorption only the vapor mantle or the sample furnace can be connected at the time of analysis.

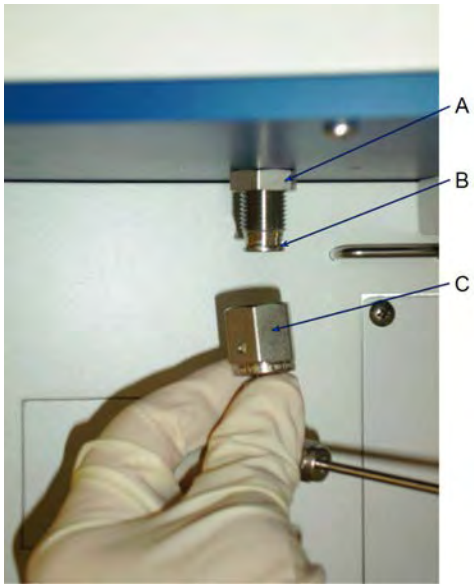


View A



View B

1. Use an appropriate wrench to loosen the connecting nut from the port fitting by turning the connecting nut counter-clockwise while using a second wrench to hold the port fitting stationary.



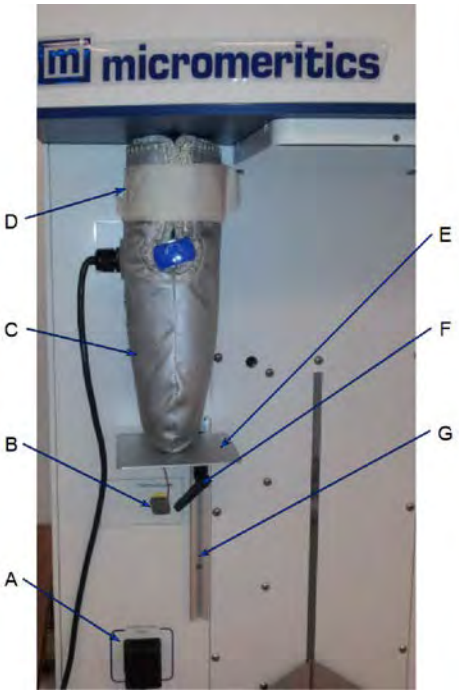
A. Port fitting
B. Gasket
C. Connecting nut

2. Remove the connecting nut and the attached assembly. After removal, the existing seal or a tight-fitting plastic cap can be used to protect the sealing surface assembly from scratches. Prior to reassembly, remove the existing seal or cap, then insert a new seal.
3. Install the vapor source container with a new seal by attaching the connecting nut to the port fitting.
4. Hand tighten the connecting nut by turning clockwise. Use an appropriate size wrench to tighten the assembly an additional 1/8 to 1/4 turn beyond finger tight, while using a second wrench to hold the port fitting stationary on the analyzer.

Turn on the vapor source isolation valve to adjust the vapor flow

5. Use the manual controls on the analyzer schematic, evacuate the space above the vapor source by opening valves 4 and 6 with all other valves closed. Then close valve 4 before

turning the vapor source isolation valve to the vertical (open) position. The P_0/vapor port pressure reading on the instrument schematic will show the vapor pressure.



A. Mantle power source
B. Thermocouple connector
C. Vapor source mantle
D. Hook and loop strap
E. Vapor source mantle shelf
F. Shelf locking mechanism
G. Shelf track

5. Slide the vapor source mantle over the vapor source container.
6. Extend the blue knob through the circular hole. Secure the hook and loop strap.
7. Insert the thermocouple plug into the connector labeled *Thermocouple*.
8. Insert the power plug into the outlet labeled *Mantle/Furnace Power*.
9. If using a support shelf, slide the shelf locking mechanism of the vapor source mantle shelf into the shelf track on the front of the analyzer.
10. Raise the shelf until the vapor mantle is pushed as close as possible to the underside of the upper cabinet.
11. To tighten the shelf, turn the locking mechanism clockwise.

12 MAINTENANCE

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)

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to 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 modify this instrument without the authorization of Micromeritics Service Personnel.

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 Flex weighs approximately 103 - 106 kg (227 - 234 lb) depending on configuration.

Use of a power cord or power supply not provided with the instrument could cause personal injury or damage to the equipment. If a replacement is needed, contact your Micromeritics Service Representative. Detachable power supply cords with an inadequate rating could cause significant instrument damage or physical harm.

Do not add anything between the power cord and the power source that would compromise the earth ground.

Do not remove or disable the grounding prong on the instrument power cord.

If the equipment needs to be relocated, check with your Micromeritics service representative. The equipment must be positioned such that the mains supply is not obstructed and is easily accessible to disconnect the equipment from the AC main power supply.

Prior to moving the instrument, disconnect and remove all glassware from the instrument. Ensure all gas shut-off valves on the gas cylinder have been closed and gas lines disconnected from the instrument. Contact your Micromeritics Service Representative.

SAFE SERVICING

Do not modify this instrument without the authorization of 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 6 - 2*.
- **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 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

Regulator Pressure Settings

Analyzer	Gauge should indicate
Flex	15 psig (103 kPag)

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

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 to 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.

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

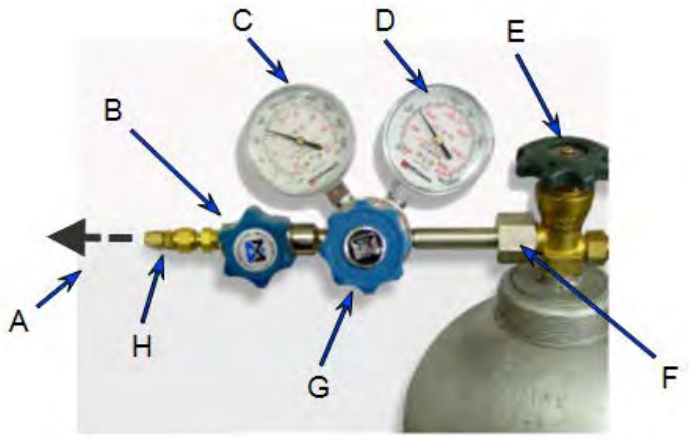
CLEAN AND VERIFY THE GAS LINE

SOFTWARE DIAGNOSTIC TEST TO CLEAN AND VERIFY GAS LINES

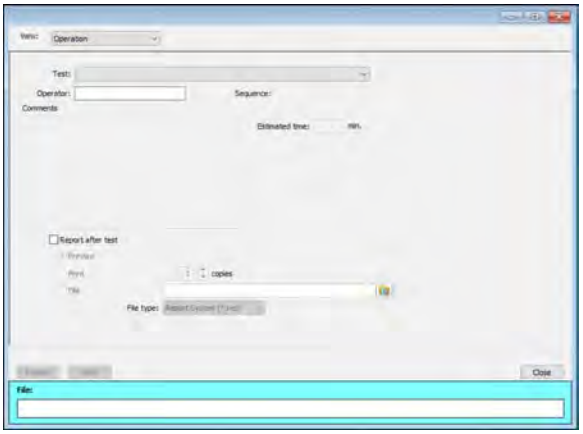
Unit [n] > Diagnostics > Start Diagnostic Test

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:

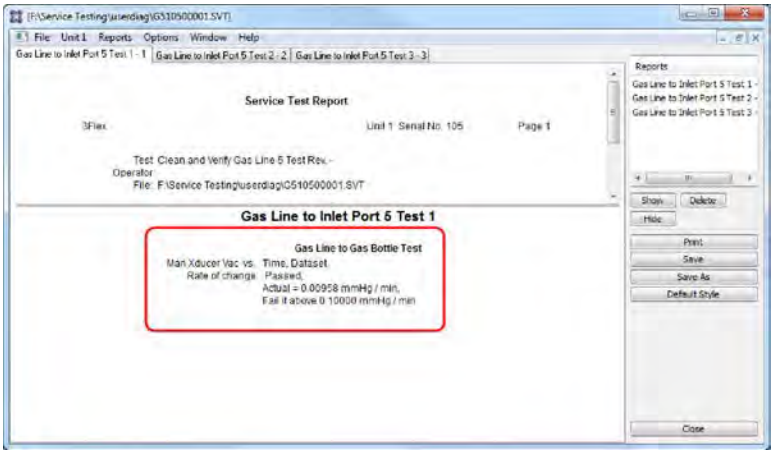


A. Gas tubing to instrument
B. Gas regulator shut-off valve - OPEN
C. Low pressure gauge - 14-15 psig
D. High pressure gauge
E. Gas cylinder shut-off valve - CLOSED
F. Regulator connecter nut
G. Regulator control knob - OPEN
H. Brass reducer fitting



1. Select *Clean and Verify Analysis Gas Line [n] Test Rev [n]* in the *Test* field. The length of time a test will run is also indicated on the window. The *Sequence* field indicates the file created as a result of this test.
2. In the *Operator* field, enter either the name or initials of the operator performing this test.

3. Resize the window (if necessary) to display the *Report after test* option, then select *Preview* as the destination. Click **Start**.
4. From the *View* drop-down list, select either *Operation*, *Instrument Log*, or *Instrument Schematic*.
5. The following series of prompts display on the window requiring operator response:
   a. This is the gas line clean and leak check test for inlet port [*n*]. Inlet ports being tested must be connected to a gas cylinder according to the user manual. A Nupro isolation valve should be installed on the line between the analyzer and the regulator.
   b. The test starts with a manual leak check (requires Snoop or equivalent, and IPA), then the line and regulator are evacuated for 20 minutes for cleaning. Next, the leak rate of the gas line is determined.
   c. With the regulator set to 15 psig, open the cylinder, regulator shut-off valve, and isolation valve. Check each joint for bubbles with Snoop or equivalent. If a joint is leaking, attempt tightening (without over-tightening) or replace ferrules.
   d. When there are no leaking joints, use IPA to remove water from each joint, then wipe dry.
   e. Close the gas cylinder valve. Leave the regulator shut-off and isolation valves open.
   f. User will be needed in 30 minutes to close the isolation valve. Click **OK** to begin automated testing.
6. A popup window indicates the test is complete. Click **OK**. The reports display.



7. Click each tab across the top of the window and look for a reading of *Passed*. A *Passed* reading indicates all valves are in a proper state for operation. If any test shows a *Failed* reading, refer to the following table to help determine the location of the gas leak.

Tab	Test	If Failed status, then...
Gas Line to Inlet Port [*n*] Test 1	Gas Line to Gas Bottle Test	This test will show a reading of *Failed* if any of the other tabs has a *Failed* reading. Correct the failed connection and rerun the test.
Gas Line to Inlet Port [*n*] Test 2	Gas Line to Isolation Valve Test	Check for a leak between the gas line and the isolation valve. Correct the problem and rerun the test.
Gas Line to Inlet Port [*n*] Test 3	Isolation Valve To Bottle Leak Rate	Check for a leak between the isolation valve and the gas cylinder. Correct the problem and rerun the test.

If the *Fail if above* field indicates *Failed*, one or more valves is not in the proper position. Set the valves, then ensure the appropriate pressure is displayed on the low pressure gauge.

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

1. Click **Close** to close the test report. Click **Close** again to close the test.
2. Repeat steps 1 through 8 for each gas line attached to the analyzer.

REPLACE A GAS CYLINDER

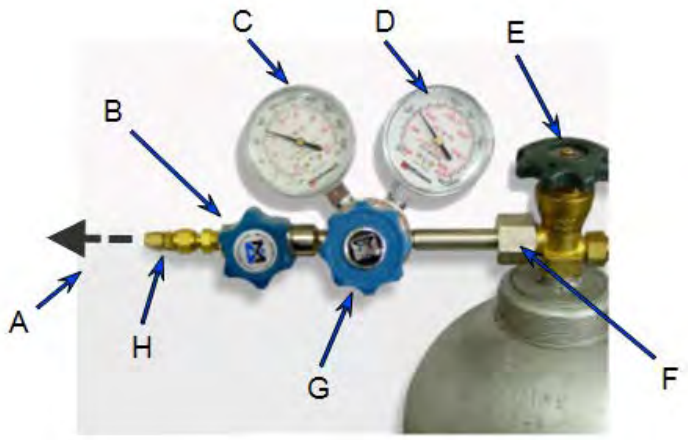
Regulator Pressure Settings

Analyzer	Gauge should indicate
Flex	15 psig (103 kPag)

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

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 counterclockwise. 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

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 starting 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*.
   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.

Clean the Power Supply Air Filter

Two power supply air filters are located on the lower rear panel of the analyzer and should be cleaned or replaced every 30 days (more often in environments with increased levels of dust).

1. Use a flat blade screwdriver to pry the air filter cover from the base. Do not remove the screws. Use caution when removing the cover to avoid breakage.

2. Use an air compressor to remove the dust, or rinse with tap water and dry thoroughly.
3. Replace the filter and cover by pressing the cover back into the base.

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
Dewar	Check and clean weekly.
Power supply air filters	Clean and replace every 30 days (more often in environments with increased levels of dust).
Analyzer exterior	Clean a needed or every 6 months.
Vacuum pump diaphragm(s)	Replace every 12 months.
Port gasket	Replace every 3 to 6 months (depending on the types of analyses that were run).
Sample tube O-ring	Replace as required or every 3 to 6 months.
Test analyzer for leaks	As required or every 12 months.
Septum	Inspect and replace every 6 months or after every 150 runs. Replace every 12 month or 300 runs.
TCD	Inspect and clean every 3 months or after every 75 runs. Replace every 12 month or 300 runs.
CryoStat	See *Preventive Maintenance for the CryoStat on page D - 12*.

CHECK AND CLEAN THE DEWAR

When handling Dewars, follow the precautions outlined in ***Dewar Precautions on page 6 - 2***.

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. (6 mm) in depth may jam between the bottom of the sample tube and the bottom of the Dewar, causing the Dewar not to raise fully.

Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tube. This causes the sample temperature to be slightly higher which, in turn, can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure.

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.

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 instrument. Doing so could result in damage to the unit.

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 TRIGEL grease *[Micromeritics part number 004-16166-00*] to the elevator screw, accessed from the back of the instrument, as needed.

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. External vacuum pump.
3. Analyzer.
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 dry forepump.
5. Smart VacPrep.

REPLACE THE PSAT FITTING GASKET

A gasket is attached to the Psat fitting. See ***Replace the Psat Tube Ferrules below***.

CAUTION

Each time the Psat tube or vapor source container is replaced, a new gasket is required. To avoid degassing problems, do not touch the sealing surfaces of the port fitting or gasket with bare hands.

REPLACE THE PSAT TUBE FERRULES

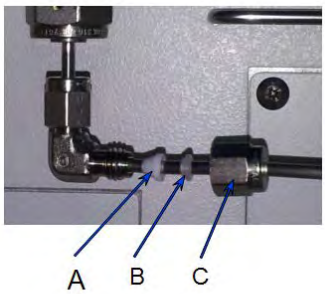
Over an extended period of time, pivoting the Psat tube may cause wear on the nylon and teflon ferrules housed in the Psat tube nuts. If the recommended weekly scheduled *P0 Port Leak Test* detects a leak by reporting *Failed* on the *Evacuated* or *Pressured* report, the first time a leak is detected, tighten the Psat nuts 1/2 turn and rerun the test. If the leak is still present, replace the nylon and teflon ferrules.

CAUTION

It is recommended that the VCR connector not be removed from the port fitting for this process.

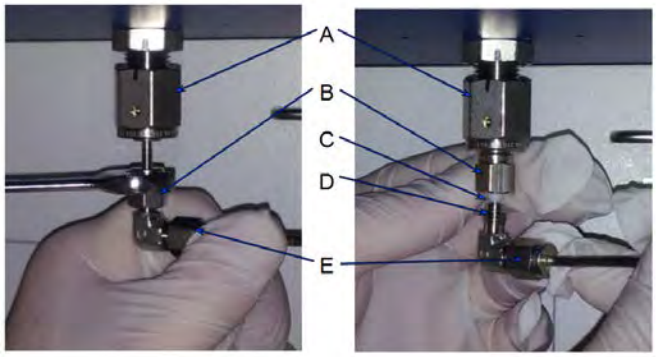
Two ferrule sets are located in the upper and lower Psat nuts. Both sets should be replaced. Additional ferrule sets were included in the analyzer's accessory kit.

1. Ensure the Psat tube is filled at atmospheric pressure with gas before loosening the Psat nut.
2. To remove the lower Psat nut, use a 7/16 in. (11 mm) wrench to loosen the Psat nut by turning the nut counter-clockwise.



A. Teflon ferrule
B. Nylon ferrule
C. Lower Psat nut

3. Remove the Psat tube from the Psat elbow.
4. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut. Position the ferrules as shown with the cone pointed out of the nut.
5. Insert the Psat nut onto the Psat tube, followed by a nylon ferrule, then a teflon ferrule.
6. Insert the Psat tube into the Psat elbow.
7. Hand tighten the Psat nut by turning the nut clockwise. Then use an appropriate size wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.
8. To remove the upper Psat nut, use a 7/16 in. (11 mm) wrench to loosen the Psat nut by turning the nut counter-clockwise.



A. VCR connector
B. Upper Psat nut
C. Nylon ferrule
D. Teflon ferrule
E. Lower Psat nut

9. Remove the Psat tube from the VCR connector.
10. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut.
11. Reinsert the Psat nut onto the VCR connector, followed by a nylon ferrule, then a teflon ferrule. Orient the ferrules as shown with the cone pointed out of the nut.
12. Insert the Psat elbow into the upper Psat nut.
13. Hand tighten the upper Psat nut by turning the nut clockwise. Then use a 7/16 in. (11 mm) wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.

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.

The chemisorption port frit is changed in the same manner as the sample port frit.

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.

CAUTION

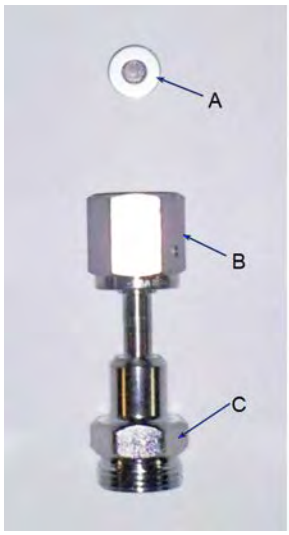
Use the proper size frit for the sample port. The analyzer will not operate properly if an incorrect size is used.

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 while using a second wrench to hold the port fitting stationary. Remove and discard the used frit.

CAUTION

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. Frit
B. Connector nut
C. Sample tube fitting

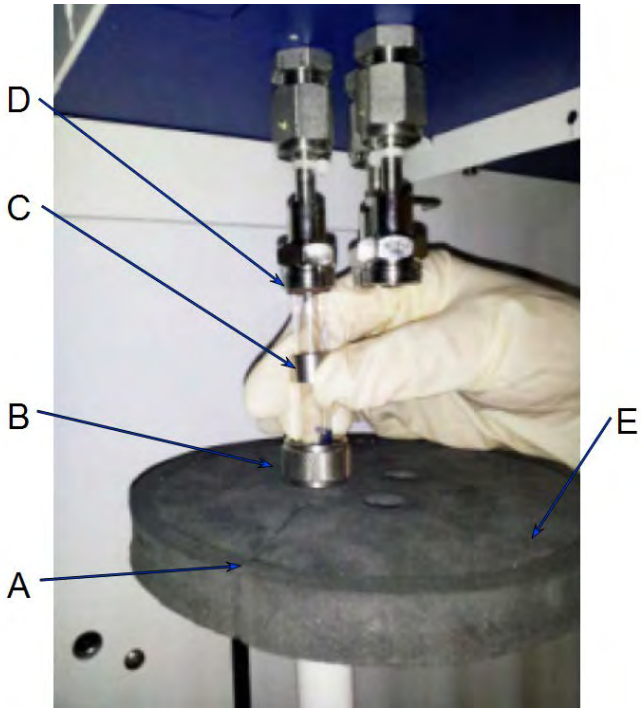
5. Attach the connecting nut to the sample port fitting and finger tighten. Use a wrench to tighten the nut 1/8 to 1/4 turn past finger tight while using a second wrench to hold the port fitting stationary.

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.

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 counterclockwise.
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. P_0 slot
B. Connector nut
C. Ferrule
D. O-ring
E. Dewar cover

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*].

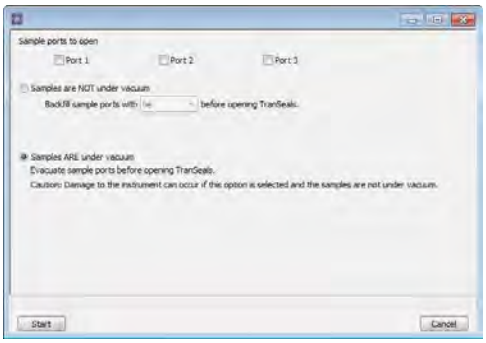
5. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.

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.

Open the TranSeal

Unit > Open TranSeal

Outlines the process of safely opening one or more TranSeals on sample ports.



Damage may occur to the analyzer if the samples are not under vacuum and the *Samples ARE under vacuum* option is selected.

Sample Tube O-ring

Selections	Description
Sample ports to open [*group box*]	Select the ports to open during analysis.
Samples are NOT under vacuum [*selection*]	Select if samples are NOT under vacuum and specify the amount of backfill and adsorptive to be used prior to opening TranSeals.
Samples ARE under vacuum [*selection*]	Select if the samples ARE under vacuum. This option evacuates sample ports prior to opening TranSeals. Do not select this option if the samples are not under vacuum as analyzer damage may occur.
Start [*button*]	Opens the TranSeals. The selected sample port will be either backfilled or evacuated as specified. The user will be prompted to open the TranSeals. For each selected port, an event is recorded in the Instrument Log file with the port pressure before and after opening the TranSeal.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

PURGE THE SYSTEM

PURGE AIR OR GAS

See *Guidelines for Connecting Gases on page 12 - 4* when connecting a new gas cylinder to one of the 12 primary gas inlets on the back of the analyzer for use as a carrier gas. After connecting a new gas cylinder to one of the four loop gas inlets on the side of the analyzer, open the gas inlet valve and allow gas to flow for a period of time to purge the gas line before using that gas in an analysis.

Changing the Gas Flow During an Analysis

When gases are changed during an analysis, allow the new gas to flow for a period of time before creating conditions that cause the experiment to begin (such as elevating the temperature). To avoid undesirable combinations of gases within the analyzer, purge one gas (by flowing an inert gas for a period of time) before starting to flow another gas.

To flow an inert gas between incompatible gases, insert a *Gas Flow* step (in which an inert gas is flowed for a period of time) and a *Wait* step (to wait for the inert gas to purge the analyzer) between other steps that involve incompatible gases.

When gas flows are changed while the analyzer is recording data, the gas flow is briefly disturbed. This may result in a brief period of noise or other visible disturbances on the peak data. Either disregard the disturbance or insert a Wait for stable baseline step immediately after changing gas flows.

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13 TROUBLESHOOTING

CFR Note	Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

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.

Sample is not within specifications.

Cause A: There may be a manifold leak.

Action A: See *Start Diagnostic Test on page 9 - 1*.

Cause B: Gas may be contaminated.

Action B:

- Perform a blank analysis. If results are good, perform a reference material analysis.
- Replace tank.
- Check for line leak, which could cause contamination.
- Flush the lines occasionally to help prevent contamination.

Cause C: Incorrect type of gas line.

Action C: Ensure the gas line is all metal. It is best to use the one shipped with the analyzer. Do not use polymer gas lines or flexible gas lines that may be coated internally with a polymer.

High vacuum pump indicator light does not come on.

Cause: No power to the high vacuum pump.

Action: Remove the lower panel on the front of the instrument. Check the power supply plug to the pump. Power off the high vacuum pump, then power it back on.

Vacuum gauge shows reading above 20 mmHg even after extended pumping through unrestricted valve with analysis or degas ports closed.

Cause A: Port filter is dirty.

Action A: Replace the port filter.

Cause B: No power to the vacuum pump.

Action B: Check the pump power plug, power switch, and line circuit breaker.

For oil-based pumps:

Cause A: Port filter is dirty.

Action A: Replace the port filter.

Cause B: Vacuum pump oil is low causing ineffective evacuation.

Action B: Add or change vacuum pump oil. Add oil to proper level according to the pump's indicator window.

Cause C: Alumina in the oil vapor trap is holding moisture because it was not sufficiently dried before being added to the trap.

Action C: Replace or dry the alumina.

For oil-free pumps:

Cause A: High vacuum pump may have timed out.

Action A: Remove the lower front panel from the analyzer. Power off the high vacuum pump, then power it back on.

Cause B: The pump diaphragm(s) is worn or damaged.

Action B: Contact your Micromeritics Service Representative.

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 Port Frit Replacement on page 12 - 19***.

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.

Vacuum pump system makes loud continuous noise.

Cause A: Sample tube connector nut is loose.

Action A: Turn the connector nut clockwise to tighten.

Cause B: Sample tube fitting is loose.

Action B: Tighten the fitting using a wrench.

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

Action C: Replace sample tube O-ring.

Cause D: Sample tube is cracked.

Action D: Replace the sample tube.

Cause E: No sample tube is loaded on selected port.

Action E: Ensure the port valve is closed. Install a plug or empty sample tube on the port.

Cause F: A gas inlet valve is open while the vacuum valve is open.

Action F: Enable manual control then use the analyzer schematic to close the gas inlet valve. See ***Show Instrument Schematic on page 2 - 24***.

Valves cannot be operated.

Cause A: Cable from computer to the instrument is loose.

Action A: Reconnect the cable.

Cause B: Circuit was opened by the circuit breaker.

Action B: Press the **Breaker** button located on the side of the analyzer near the power entrance. Contact your Micromeritics Service Representative if the button will not stay depressed.

A stable TCD baseline cannot be maintained.

Cause A: TCD filaments are contaminated or need to be replaced.

Action A: See ***TCD Assembly on page 11-15*** to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

Cause B: Possible leak in reference / carrier path.

Action B: Perform leak test in the reference / carrier path. See ***Perform a Leak Test on page 13 - 8***.

Data collection results in very high or very low peaks that are inconsistent with previous experience.

Cause A: TCD filaments are contaminated or need to be replaced.

Action A: See ***TCD Assembly on page 11-15*** to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

Cause B: A different (lower) TCD filament temperature is being used.

Action B: Repeat the analysis using a higher filament temperature.

During data collection, a TCD signal of zero is recorded, or no peaks are seen.

Cause A: TCD filaments are contaminated or need to be replaced.

Action A: See ***TCD Assembly on page 11-15*** to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

Cause B: TCD temperature was not reset after a run automatically suspended.

Action B: Re-enable the TCD after all run suspensions.

Smart VacPrep S/N [n] is already in use.

Cause: Cannot add Smart VacPrep with serial number [n] because a Smart VacPrep with the same serial number is already attached.

Action: Change the serial number if it was incorrect. Otherwise, remove the Smart VacPrep with the duplicate serial number.

POWER

The Flex is designed to operate with a universal input power supply (100/115/230 VAC) at 50/60 Hz. 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 115 VAC ±10% or 7.5 amps @ 230 VAC ±10%. These requirements can be checked by using a circuit analyzer (available at most hardware or electronic supply houses) or a multimeter. There should also be sufficient outlets for all peripheral devices.

The instrument should be connected to a switch which meets relevant requirements of IEC 60947-3 or a circuit breaker which meets the relevant requirements of IEC 60947-2.

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.

OIL-BASED VACUUM PUMP

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

O-RING COMPATIBILITY

O-ring selection for chemisorption measurements is based on temperature, time, and chemical compatibility. Chemical compatibility should be the first consideration when selecting an appropriate O-ring. The time at which the furnace, and subsequently the sample cell, is at elevated temperature can also affect the performance of the O-rings and should be a secondary consideration. Common O-ring materials include Buna-N (nitrile), Viton (fluoroelastomer), and Kalrez (perfluoroelastomer). Kalrez has historically been used extensively for chemisorption measurements due to compatibility with a wide range of chemicals and temperatures and should be suitable for all applications of the chemisorption option. 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 broader. 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.

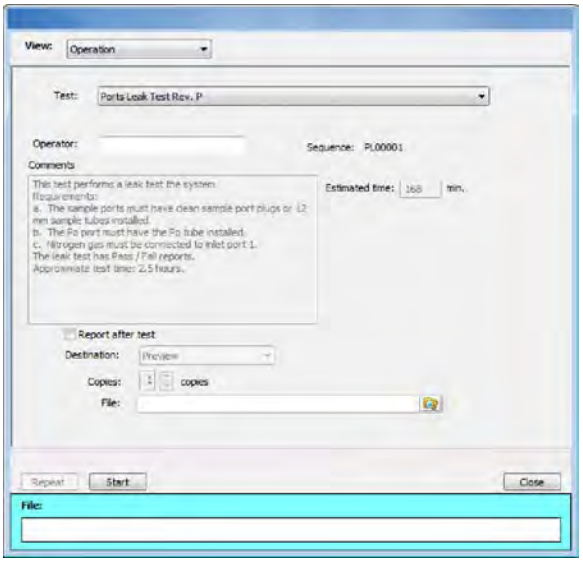
PERFORM A LEAK TEST

Unit [n] > Diagnostics > Start Diagnostic Test

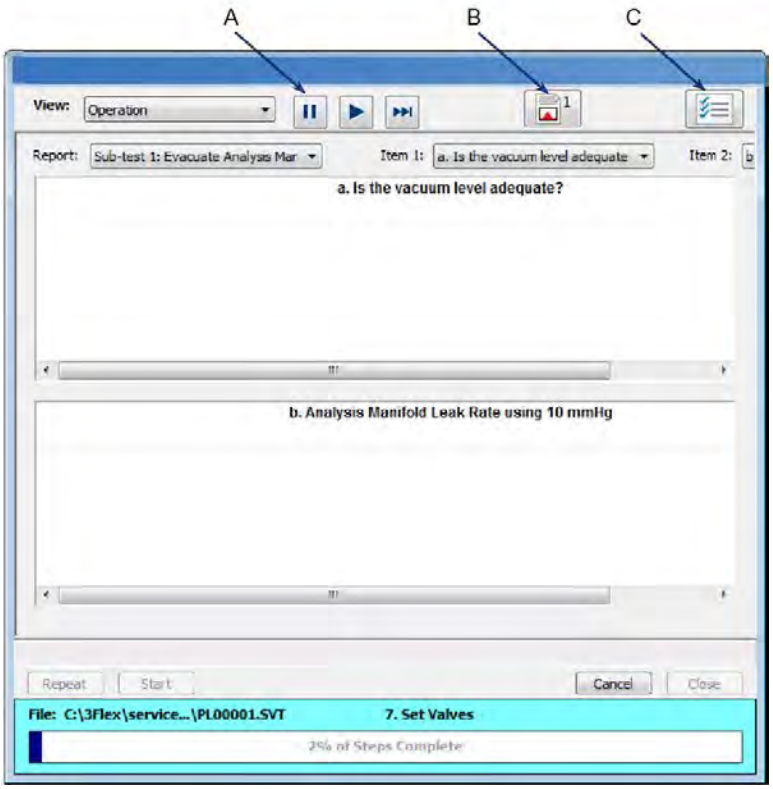
A Micromeritics Service Representative may request that a leak test be performed to determine if there is a system leak and may also require a copy of the report generated by this test.

The test provides:

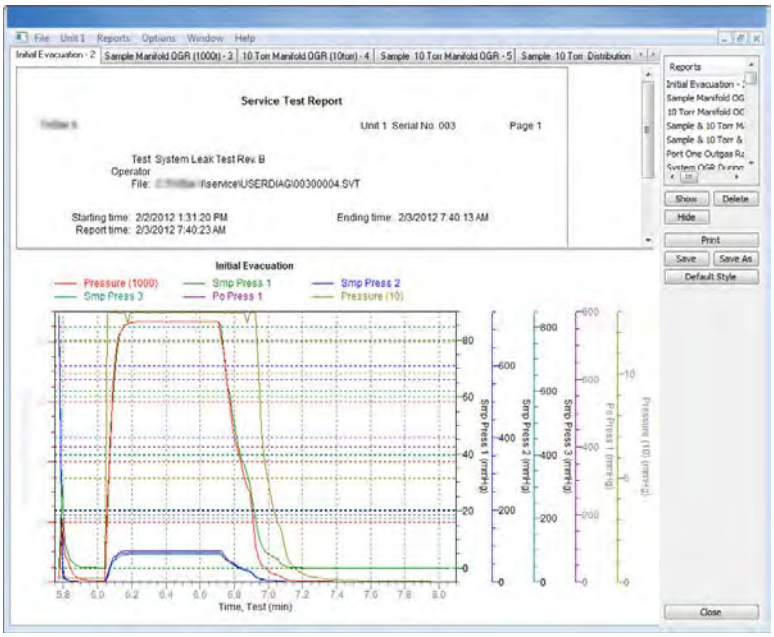
- Prompts on preparing the analyzer for the test.
- Approximate time period of the test.
- Prompts in which an operator response is required.



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



Pneumatic Pressure

The compressed air supply for the pneumatically actuated, hard seal valves and furnace cooling must be set to 100 psi ±20 psi for proper operation. Ensure that the regulator for the compressed air supply is correctly set. If a portable air compressor is used, perform maintenance as instructed in the air compressor manual.

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.

A ADVANCED REPORTS - PYTHON MODULE

CFR Note	In a 21CFR11 environment, the Advanced reports feature is applicable to members of the Developer group only.

The mic Python module is automatically imported when running a user supplied script. The module provides access to primary and overlay isotherm data and provides support for summary, tabular, and graphical reports.

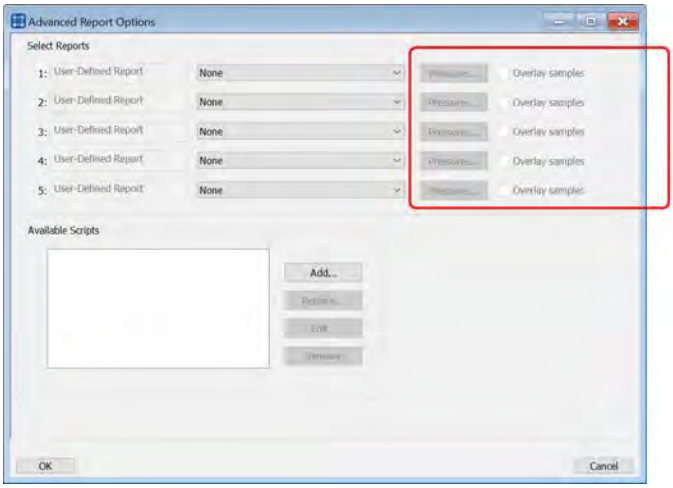
- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data. Tabular reports are created with the *mic.table* call.
- **Graphical reports**. Consist of a single graph with one or more curves on one or two y-axes. Graphical reports are created with the *mic.graph* call.

Calls for accessing the sample file data can be found in the *Mic Module Python Calls* section of this appendix. More advanced example python scripts are included in the analyzer software.

ADVANCED REPORT OPTIONS

Up to five Advanced reports, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports can be created. To use this feature, a file containing a Python script that imports a "mic" Python module must be created. See *MicModule Python Calls on page A - 22* for an example of a Python script and functions for the "mic" Python module.

1. Create the Python script and save it in the *Scripts* directory.
2. Open a sample file with a *Complete* status.
3. Select *Advanced* in the view selector drop-down list at the bottom of the window to return to the tabbed view.
4. On the *Report Options* tab, select *Advanced* in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add** in the *Available Scripts* group box to locate and select the Python script. Repeat for each script to be added.



These options are not available for dynamic analysis

6. In the *Selected Reports* group box, click the drop-down arrows to select up to five Python scripts previously added in the *Available Scripts* box.
7. On the *Report Options* tab, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.

Advanced Reports

Selections	Description
Advanced Report 1 through 5 [*drop-down box*]	Use the drop-down lists to select currently-defined functions used to define the report calculations and output.
Available Scripts [*group box*]	Lists the available reports and provides the option to add, replace, edit, or remove reports.
Overlay samples (if shown) [*check box*] PC	Use to overlay samples as defined by the function.

For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 3*.

SCRIPTS

Run a Script

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the view selector drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**.
6. Locate and select one or more python scripts then click **Select**. The selected scripts become a part of the drop-down list in the *Available Scripts* section of the *Advanced Report Options* window.
7. In the *Select Reports* section, select up to five *Advanced* reports in the drop-down lists.
8. Click **OK**.
9. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

Remove a Script

Select the script in the *Available Scripts* box then click **Remove**. The script is removed from the application however, the original .py text file is not affected.

Edit a Script

Selections	Description
Add [*button***]**	Adds one or more scripts to the *Available Scripts* box. The added scripts then become available as options in the *Selected Reports* section.
Edit [*button***]**	Edits the script stored within the application but does not affect the original .py text file.
Overlay samples [*check box***]**	Select to enable the overlay sample files process.
Pressures [*button***]**	Select to include or exclude pressures from the report. ■ **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. ■ **Cancel**. Discards any changes or cancels the current process. ■ **Exclude All.** Select to exclude all pressure points in the table. ■ **Include All.** Select to include all pressure points in the table. ■ **OK.** Saves and closes the active window.
Remove [*button***]**	Removes the script from the *Available Scripts* box but does not affect original .py text file.
Replace [*button***]**	Replaces the contents of the selected script however, the script name remains the same.

PYTHON REPORTS

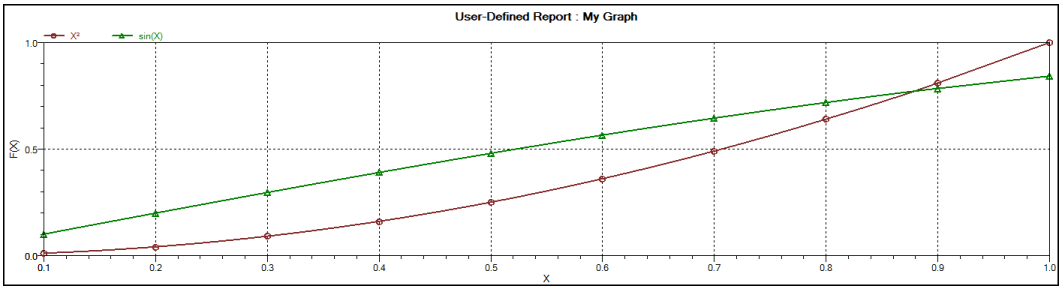
Graphic Report

This script is an example of the mic module producing a graph with two curves:

```
import mic
import numpy as np

mic.graph( 'My Graph', 'X', 'F(X)' )
myx = np.array( [0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0 ] )
mic.graph.add( 'X2', myx, myx*myx, marker='o' )
mic.graph.add( 'sin(X)', myx, np.sin(myx), marker='^' )
```

The results are:



Summary Report

This script produces a summary report with two summaries:

```
1  import mic
2  import numpy as np
3
4  mic.summary( "My Summaries" )
5  mic.summary.add( "Summary A",
6                   ["Label 1:", "Label 2:", "Label 3:"],
7                   ["val1", "val2", "val3"] )
8  mic.summary.add( "Summary B",
9                   ["Label 4:", "Label 5:", "Label 6:"],
10                  ["val4", "val5", "val6"] )
```

The result is:

Summary A
Label 1: val1
Label 2: val2
Label 3: val3

Summary B
Label 4: val4
Label 5: val5
Label 6: val6

Tabular Report

If more than one column is required, the call *mic.table* is employed. This script produces a tabular report consisting of two tables.

This script uses the Python package *numpy* and *c*-style formatting of the numerical values.

```
import mic
import numpy as np

mic.table( "My Tables" )
mic.table.addtable( "My Set A" )
mic.table.addcolumn( "X", ["1.0", "2.0", "3.0"] )
mic.table.addcolumn( "Y", ["0.5", "1.0", "1.5"] )
x1 = 0.2
x2 = 0.5
x3 = 3.14159/2
mic.table.addtable( "My Set B" )
mic.table.addcolumn( "X", ['{:8.3f}'.format(x1),
                           '{:8.3f}'.format(x2),
                           '{:8.3f}'.format(x3)] )
mic.table.addcolumn( "sin(X)", ['{:8.3f}'.format(np.sin(x1)),
                                '{:8.3f}'.format(np.sin(x2)),
                                '{:8.3f}'.format(np.sin(x3))] )
mic.table.addcolumn( "cos(X)", ['{:8.3f}'.format(np.cos(x1)),
                                '{:8.3f}'.format(np.cos(x2)),
                                '{:8.3f}'.format(np.cos(x3))] )
```

The result is:

My Set A

X	Y
1.0	0.5
2.0	1.0
3.0	1.5

My Set B

X	sin(X)	cos(X)
0.200	0.199	0.980
0.500	0.479	0.878
1.571	1.000	0.000

ACQUIRE BASIC INFORMATION

Physisorption

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic.isotherm*, *mic.sample_information*, and *mic.adsorptive_data* are applied.

This script produces a graph of the adsorption and desorption isotherms for both relative and absolute pressure, and prints summaries of the sample information and the adsorptive properties.

```
import mic

prel, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')
mic.graph( 'Graphical Report 1', 'Relative Pressure (P/Po)', 'Quantity Adsorbed (cm³/g STP)' )
mic.graph.add( 'Sample Isotherm', prel, qads )

pabs, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs')
mic.graph( 'Graphical Report 2', 'Absolute Pressure (mmHg)', 'Quantity Adsorbed (cm³/g STP)' )
mic.graph.add( 'Sample Isotherm', pabs, qads )

mass = mic.sample_information('sample mass')
Tanl = mic.sample_information('analysis temperature')
dens = mic.sample_information('sample density')

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information",
                 [ "Number of adsorption points:",
                   "Warm free space:",
                   "Cold free space:",
                   "Sample mass:",
                   "Description:",
                   "Analysis temperature:",
                   "Sample density:" ],
```

```
                  [ '{:d}'.format(n_ads),
                    '{:8.3f}'.format(warm_fs) + ' cm³',
                    '{:8.3f}'.format(cold_fs) + ' cm³',
                    '{:8.3f}'.format(mass) + ' g',
                    desc,
                    '{:8.3f}'.format(Tanl) + ' K',
                    '{:8.3f}'.format(dens) + ' g/cm³' ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
                  [ "Cross sectional area:",
                    "Hard sphere diameter:",
                    "Density conversion factor:",
                    "Molecular weight:",
                    "Analysis gas:" ],
                  [ '{:8.3f}'.format(csa) + ' nm²',
                    '{:8.3f}'.format(hsd) + ' Å',
                    '{:8.3f}'.format(dcf),
                    '{:8.3f}'.format(mol_weight),
                    analysis_gas ] )
```

Note the calls to *mic.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

Chemisorption

This script produces a graph of the primary, repeat, and difference isotherms, and prints summaries of the sample information and the adsorptive properties.

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic_chem.isotherm*, *mic.sample_information*, and *mic.adsorptive_data* are applied.

Note the calls to *mic_chem.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```
import mic

p_primary, q_primary = mic.chem_isotherm('primary')
p_repeat, q_repeat = mic.chem_isotherm('repeat')
p_difference, q_difference = mic.chem_isotherm('difference')
mic.graph('Graphical Report 1', 'Absolute Pressure (mmHg)', 'Quantity Adsorbed (cm³/g STP)')
mic.graph.add('Primary', p_primary, q_primary)
mic.graph.add('Repeat', p_repeat, q_repeat)
mic.graph.add('Difference', p_difference, q_difference)

mic.summary("Sample Information")
mic.summary.add( "Sample Information",
                 [ "Ambient free space:",
                   "Analysis free space:",
                   "Sample mass:",
                   "Description:",
                   "Analysis temperature:",
                   "Sample density:" ],
                 [ '{:8.3f}'.format(mic.sample_information('ambient freespace')) + ' cm³',
                   '{:8.3f}'.format(mic.sample_information('analysis freespace')) + ' cm³',
                   '{:8.3f}'.format(mic.sample_information('sample mass')) + ' g',
                   mic.sample_information('sample description'),
```

```
                    '{:8.3f}'.format(mic.sample_information('analysis tem-
perature')) + ' K',
                    '{:8.3f}'.format(mic.sample_information('sample density'))
+ ' g/cm³' ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
                 [ "Cross sectional area:",
                   "Hard sphere diameter:",
                   "Density conversion factor:",
                   "Molecular weight:",
                   "Analysis gas:" ],
                 [ '{:8.3f}'.format(csa) + ' nm²',
                   '{:8.3f}'.format(hsd) + ' Å',
                   '{:8.3f}'.format(dcf),
                   '{:8.3f}'.format(mol_weight),
                   analysis_gas ] )
```

Acquire Report Results for Physisorption

Sample file report results may be accessed using the *mic.report* call. This script prints a summary of the results of the *t*-plot and BET reports.

```
import mic

sa  = mic.report("bet", "surface area")
c   = mic.report("bet", "bet constant")
vm  = mic.report("bet", "monolayer capacity")
esa = mic.report("tplot", "external surface area")
vol = mic.report("tplot", "micropore volume")

mic.summary( "BET and T-plot Results" )

mic.summary.add( "Report Results",
                 [ "BET surface area:",
                   "BET constant:",
                   "BET monolayer capacity:",
                   "T-plot external surface area:",
                   "T-plot micropore volume:" ],
                 [ '{:10.5f}'.format(sa) + ' m²/g',
                   '{:10.5f}'.format(c),
                   '{:10.5f}'.format(vm) + ' cm³/g',
                   '{:10.5f}'.format(esa) + ' m²/g',
                   '{:10.5f}'.format(vol) + ' cm³/g' ] )
```

The result is:

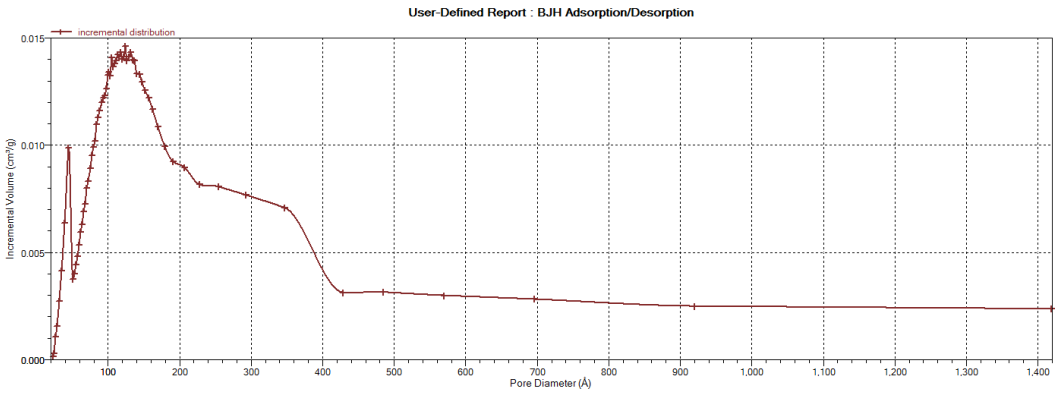
Report Results

BET surface area:	215.91368 m²/g
BET constant:	111.73509
BET monolayer capacity:	49.60593 cm³/g
T-plot external surface area:	210.36208 m²/g
T-plot micropore volume:	0.00121 cm³/g

Acquiring the results from a pore-distribution report such as the BJH method is done in a similar way as in the previous script except the return values from the *mic.report* call are slightly different since they involve lists of data. For example:

```
1  import mic
2  import numpy as np
3
4  xdat, ydat, desc = mic.report( 'bjhads', 'incremental distribution' )
5  if not (np.isnan(xdat[0]) or np.isnan(ydat[0])): # NaN indicates BJH cal-
   culation error.
6      mic.graph( 'BJH Adsorption/Desorption', 'Pore Diameter (Å)', 'Incremental
   Volume (cm³/g)' )
7      mic.graph.add( desc, xdat, ydat )
```

The result is:



ACQUIRE OVERLAY SAMPLE DATA FOR GAS ADSORPTION

Physisorption

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.overlay call*, and a summary of their usage is shown in the example in this topic.

```
import mic

p, q, n, fsw, fsc, mass, desc = mic.isotherm('rel')
p1, q1, n1, fsw1, fsc1, mass1, desc1 = mic.overlay( 1, 'rel')
p2, q2, n2, fsw2, fsc2, mass2, desc2 = mic.overlay( 2, 'rel')

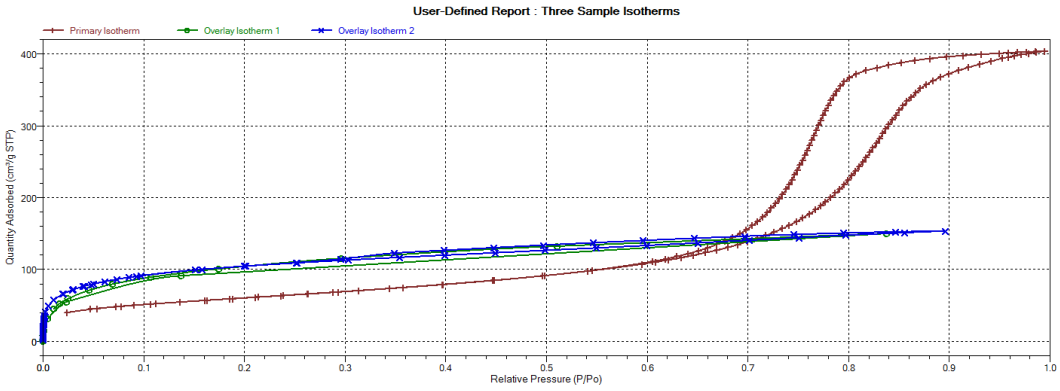
mic.graph( 'Three Sample Isotherms',
           'Relative Pressure (P/Po)',
           'Quantity Adsorbed (cm³/g STP)' )

mic.graph.add( 'Primary Isotherm', p, q )
mic.graph.add( 'Overlay Isotherm 1', p1, q1 )
mic.graph.add( 'Overlay Isotherm 2', p2, q2 )

mic.summary( "A Summary Report" )

mic.summary.add( "Two samples",
                 [ "Primary sample:",
                   "Overlay sample 1:",
                   "Overlay sample 2:" ],
                 [ desc,
                   desc1,
                   desc2 ] )
```

The results are:



Two samples

Primary sample: 12 mm Tube N1 Silica-Alumina ADS-DES with FS
Overlay sample 1: Activated Carbon Tube C4 Butane Port 3
Overlay sample 2: Activated Carbon Hexane Dosed from Port 3 - 2

To enable the use of overlay data in the Advanced reports, the following two actions must be taken prior to running the script:

- Sample files to overlay must be selected, and
- The *Overlay samples* checkbox on the *Advanced Report Options* window must be selected.

Chemisorption

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.chem.overlay call*, and a summary of their usage is shown in the example in this topic.

```
import mic

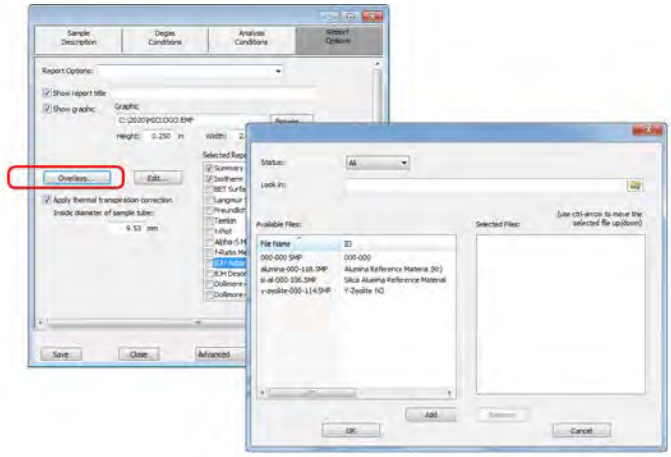
p0, q0 = mic.chem_isotherm('primary')
p0r, q0r = mic.chem_isotherm('repeat')
p1, q1 = mic.chem_overlay(1, 'primary')
p1r, q1r = mic.chem_overlay(1, 'repeat')
p2, q2 = mic.chem_overlay(2, 'primary')
p2r, q2r = mic.chem_overlay(2, 'repeat')
mic.graph('Graphical Report 1', 'Absolute Pressure (mmHg)', 'Quantity Adsorbed (cm³/g STP)')
mic.graph.add('Primary 0', p0, q0)
mic.graph.add('Repeat 0', p0r, q0r)
mic.graph.add('Primary 1', p1, q1)
mic.graph.add('Repeat 1', p1r, q1r)
mic.graph.add('Primary 2', p2, q2)
mic.graph.add('Repeat 2', p2r, q2r)

mic.summary("A summary report")

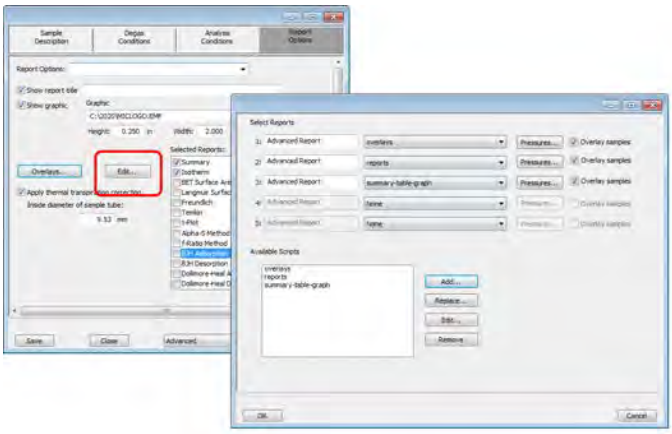
mic.summary.add("Sample and Two Overlays",
                ["Primary sample:",
                 "Overlay sample 1:",
                 "Overlay sample 2:"],
                [mic.sample_information('sample description'),
                 mic.sample_information('sample description', 1),
                 mic.sample_information('sample description', 2)])
```

ENABLE THE USE OF OVERLAY DATA

1. On the *Report Options* tab, click **Overlays**.
2. On the *Plot Overlay Sample Selection* window, to move a file from the *Available Files* list box to the *Selected Files* list box, either double-click a file name in the *Available Files* list box or click one or more files in the *Available Files* list box then click **Add**.



3. Click **OK**.
4. On the *Report Options* tab, highlight *Advanced* in the *Selected Reports* list box.
5. Click **Edit**.
6. Select the *Overlay samples* checkbox to the right of the selected report.
7. Click **OK**.
8. Run the script using the instructions found in ***Scripts on page A - 4***.



ACQUIRE METAL COMPOSITION DATA FOR CHEMISORPTION

The call to obtain information about active metals in a chemisorption sample is *mic.metal_composition*. Specifically, this call provides access to the data shown in the table of the *Active Metals* window. With no arguments specified, the call returns a list of all the active metals in the sample. When called with a metal specified, the method returns an associative array (python dictionary) of the metal's properties. With both the metal and property specified, the call returns the value for the specified metal property. The following example script illustrates these three usage patterns.

```
import mic
import pprint as pp

# Without any arguments, the method returns a list of the active metals in
# the sample.
mnames = sorted(mic.metal_composition())
mic.summary("Metal Composition: " + pp.pformat(mnames))

# With only the 'metal' argument, the method returns a dictionary of the
# specified metals' properties.
mprops = sorted(mic.metal_composition(mnames[0]).items())
mkeys = []
mvals = []
for k, v in mprops:
    units = ''
    if 'atomic weight' in k:
        units = ' amu'
    elif 'cross sectional area' in k:
        units = ' nm²'
    elif 'density' in k:
        units = ' g/cm³'
    elif 'percent of sample mass' in k or 'percent reduced' in k:
        units = '%'
    mkeys.append(k + ':')
    mvals.append('{:8.3f}'.format(v) + units)
```

```
26  mic.summary.add("Properties for " + mnames[0], mkeys, mvals)
27
28  # With both first two arguments the method returns a single value for the
29  # specified property.
30  mweights = []
31  for mname in mnames:
32      mweights.append('{:8.3f}'.format(mic.metal_composition(mname, 'atomic
    weight')))
33  mic.summary.add("Active Metals and Atomic Weight (amu)", mnames, mweights)
```

MICMODULE PYTHON CALLS

TABLES

Available Mic Python calls for tables:

- Create a new tabular report
- Add a column
- Add a table

Add a Table

This script adds a table to the last created tabular report:

```
mic.table.addtable( name )

Keyword arguments:

  name --- the table name
```

Add a Column

This script adds a column to the last created table:

```
mic.table.addcolumn(header, values, align='r'):

Keyword arguments:

  header --- column header; must be a string (or convertible)
  values --- column values; must be a list of strings (or convertible)
  align  --- column alignment; 'r', 'l', 'c' for right, left, and center justified
```

Create a New Tabular Report

```
mic.table( title='User Table' )

Keyword arguments:

  title --- the tabular report title (default = 'User Table')
```

SUMMARY REPORTS

Add a Summary Section

This script adds a summary section to the last created summary report:

```
mic.summary.add(name, labels, values):

Keyword arguments:

  name   --- summary section name
  labels --- column of labels; must be a list of strings
             (or convertible) and the same length as values
  values --- column of values; must be a list of strings
             (or convertible) and the same length as labels
```

Create a New Summary Report

```
mic.summary( title='User Summary' )

Keyword arguments:

  title --- the summary title
```

GRAPHIC REPORTS

Add a Curve

This script adds a curve to the last created graphical report:

```
mic.graph.add(name, x, y, yyaxis=False, color=None, linestyle='-',
              marker='a', graphtype='both', interpolation='akima'):

Keyword arguments:

  name      --- the curve name
  x         --- list of x values; must be a list of floats
                (or convertible) and the same length as y
  y         --- list of y values; must be a list of floats
                (or convertible) and the same length as x
  yyaxis    --- place this curve on the yy-axis if True
                otherwise place on the y-axis (default = False)
  color     --- RGB color as an HTML hex string (e.g., '#4169e1')
                or a three-element list or tuple (e.g., [65,105,225]);
                if None, color is automatically selected (default = None)
  linestyle --- line style; (default = '-')
                   '-'        : solid
                   '--'       : dash
                   '.'        : dot
                   '-.'       : dash dot
                   '-..'      : dash dot dot
  marker    --- marker shape; (default = 'a')
                   '+'        : plus
                   'o' or '0' : circle
                   'x'        : cross
                   '^'        : up triangle
                   'v'        : down triangle
```

```
                    's'         : square
                    'd'         : diamond
                    '8'         : hourglass
                    '~'         : horizontal hourglass
                    '' or None  : no marker
                    'a'         : automatically selected
  graphtype --- graph type; (default = 'both')
                    'curve'  or 'c' : curve
                    'points' or 'p' : points
                    'both'   or 'b' : curve-and-points
                    'hist'   or 'h' : histogram
  interpolation -- linear or akima spline interpolation (default='akima')
                    'akima' use akima spline
                    'linear' use linear interpolation
```

Add a Curve Using the Second Y-Axis

This script adds a curve to the last created graphical report using the second y-axis:

```
mic.graph.addyy(name, xx, yy):

Add a curve to the last created graphical report using the second
y-axis. The arguments to this call are the same as to mic.graph.add.
```

Create a New Graphical Report

```
mic.graph(title='User Graph', xlabel='X axis', ylabel='Y axis',
          yylabel='YY axis',
          xlinear=True, ylinear=True, yylinear=True,
          xinvert=False, yinvert=False, yyinvert=False,
          xrange=None, yrange=None, yyrange=None, xbars_id=''):

Keyword arguments:

  title    --- the graphical report title (default = 'User Graph')
  xlabel   --- x-axis label (default = 'X axis')
  ylabel   --- y-axis label (default = 'Y axis')
  yylabel  --- yy-axis label (default = 'YY axis')
  xlinear  --- x-axis linear scale; if false, use log scale
               (default = True)
  ylinear  --- y-axis linear scale; if false, use log scale
               (default = True)
  yylinear --- yy-axis linear scale; if false, use log scale
               (default = True)
  xinvert  --- Invert x-axis if true (default = False)
  yinvert  --- Invert y-axis if true (default = False)
  yyinvert --- Invert yy-axis if true (default = False)
  xrange   --- None, or two values giving the min and max
               range of the axis.
  yrange   --- None, or two values giving the min and max
               range of the axis.
  yyrange  --- None, or two values giving the min and max
               range of the axis.
  xbars_id --- None, or the id of an xbar control created
               via the mic.control() object
```

GET EXPERIMENT INFORMATION

```
mic.dyn_chem_experiment(exp_num, sec_num=0, item=''):

  Get data associated with the indexed experiment

  Keyword arguments:

    exp_num    --- The index of the experiment to acquire
                   information for.  Indexing begins at 1

    sec_num    --- If zero, the primary experiment data is returned.
                   If greater than zero, returns associated secondary
                   experiment if any (typically these are mass specs).

    item       --- the specific experiment property to return
                   information on.  If '' or None, then return
                   the whole experiment dictionary

  Usage:

    exp = mic.dyn_chem_experiment(exp_num = 1)
    ms1 = mic.dyn_chem_experiment(exp_num = 1, sec_num = 1)
    expid = mic.dyn_chem_experiment(exp_num = 1, item = 'id')
    temp = mic.dyn_chem_experiment(exp_num = 1, item = 'temperature')

mic.dyn_chem_experiment_all():

  Return a dictionary of all experiments. The key is the experiment
  number, the value is a dictionary with keys from 0 (primary experiment
  data) through n where n is the number of secondary experiments (e.g.,
  mass spec data).
```

GET PRIMARY ISOTHERM DATA

Physisorption

```
mic.isotherm(press_type='rel', sample_number=0, item=''):

Get isotherm data.

Keyword arguments:

  press_type    --- the pressure basis; use 'rel' for relative pressure,
                    'abs' for absolute (default = 'rel'). This is ignored
                    if the 'item' argument is also used.

  sample_number --- Sample to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

  item          --- string identifying the item to be returned.
                    For example; 'absolute pressure', or 'quantity adsorbed'
                    The default is an empty string for which the return
                    value is the below list of quantities

Usage for specified item:
  prel = mic.isotherm(sample_number=0, item='relative pressure')

Usage with tuple returned:
  p, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')

  p       --- array of pressure (relative or absolute)
  qads    --- array of cumulative quantity adsorbed
  num_ads --- number of points in the adsorption curve
```

```
  warm_fs --- warm free-space
  cold_fs --- cold free-space
  mass    --- sample mass
  desc    --- sample description
```

Chemisorption

```
mic.chem_isotherm(branch='primary', item=None):

Get primary, repeat and difference isotherm data.

Keyword arguments:

  branch --- Specifies which analysis to get isotherm data;
             use 'primary' for the first analysis,
                 'repeat' for the repeat analysis
             and 'difference' for the difference of these two
  item   --- only get the specified item.  Allowed values are None
             or 'sample temperature'.  No argument means
             to return p,q

Usage:

  p, q = mic.chem_isotherm('primary')
  p, q = mic.chem_isotherm('repeat')
  p, q = mic.chem_isotherm('difference')

  temp = mic.chem_isotherm(branch='primary',item='sample temperature')

  p    --- array of absolute pressures
  q    --- array of cumulative quantity adsorbed
  temp --- array of sample temperatures
```

GET OVERLAY ISOTHERM DATA

Physisorption

```
mic.overlay(overlay_number=1, press_type='rel'):

Get overlay isotherm data.

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)
  press_type     --- the pressure basis; use 'rel' for relative pressure,
                     'abs' for absolute (default = 'rel')

Usage:

  p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')

  p       --- array of pressure (relative or absolute);
              empty-array if overlay is unavailable
  qads    --- array of cumulative quantity adsorbed;
              empty-array if overlay is unavailable
  num_ads --- number of points in the adsorption curve;
              0 if overlay is unavailable
  warm_fs --- warm (ambient) free-space; 0.0 if overlay is unavailable
  cold_fs --- cold (analysis) free-space; 0.0 if overlay is unavailable
  mass    --- sample mass; 0.0 if overlay is unavailable
  desc    --- sample description; empty-string if
              overlay is unavailable
```

Chemisorption

```
mic.chem_overlay(overlay_number=1, branch='primary', item=None):

Get overlay isotherm data.

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)

  branch --- Specifies which analysis to get isotherm data;
             use 'primary' for the first analysis,
                 'repeat' for the repeat analysis
             and 'difference' for the difference of these two
  item   --- only get the specified item.  Allowed values are None
             or 'sample temperature'.  No argument means
             to return p,q
Usage:

  p, q = mic.chem_overlay(1, 'primary')
  p, q = mic.chem_overlay(1, 'repeat')
  p, q = mic.chem_overlay(1, 'difference')

  temp = mic.chem_overlay(branch='primary',item='sample temperature')

  p    --- array of absolute pressures
  q    --- array of cumulative quantity adsorbed
  temp --- array of sample temperatures
```

GET ADSORPTIVE DATA FOR EACH SAMPLE

```
mic.adsorptive_data(sample_number=0):

Get adsorptive data for each sample

Keyword arguments:

  sample_number --- Identifier for the adsorptive data to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()
  csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data(0)

  csa          --- cross sectional area (nm^2)
  hsd          --- hard sphere diameter (angstroms)
  dcf          --- density conversion factor (dimensionless)
  mol_weight   --- molecular weight
  analysis_gas --- mnemonic for the analysis gas species
                       (e.g., 'CO', 'H2')
```

GET SAMPLE INFORMATION ITEM

```
mic.sample_information( item, sample_number = 0 ):

Keyword arguments:

  item          --- string identifying the item to be returned.
                    For example; 'sample mass', or 'sample description'
                    The default is an empty string for which the return
                    value is a list of all available keywords

  sample_number --- Sample to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  all_keywords = sample_information()
  mass         = sample_information('sample mass')
  mass         = sample_information('sample mass',0)
```

GET REPORT RESULTS FOR PHYSISORPTION

This script gets report results for the indicted report and sample:

```
mic.report(report_name='', result='', sample_number=0):

Get report results for the indicated report and sample

Keyword arguments:

  sample_number --- Identifier for the sample data to retrieve.
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  sa                  = mic.report('bet', 'surface area')

  porew, incvol, desc = mic.report('bjhads',
                                   'incremental distribution')

The available report keywords, result keywords and a corresponding
description of the result is listed in the table below

   Report        Result                    Description
-----------   --------------               -----------
    bet        surface area                Surface area (m^2/g)
    bet        bet constant                BET constant (dimensionless)
    bet        monolayer capacity          Monolayer capacity (cm^3/g)
    tplot      external surface area       External surface area (m^2/g)
    tplot      micropore volume            Micropore volume (cm^3/g)
    bjhads     incremental distribution    Incremental Distribution
    bjhdes     incremental distribution    Incremental Distribution
```

```
    dhads       incremental distribution    Incremental Distribution
    hk          incremental distribution    Incremental Distribution
    dft         incremental distribution    Incremental Distribution
    nldft       incremental distribution    Incremental Distribution


  where the incremental pore distribution result above (for those
  reports which return this) is a list with three components being,

  porew  --- array of pore dimension boundaries (angstroms);
             empty-array if unavailable.
  incvol --- array of incremental pore volumes (cm^3/g);
             empty-array if unavailable.
  desc   --- Name of data set; empty-string if unavailable.
```

GET IMPORTED PORE DATA

```
mic.imported_pore_data(import_number=1):

Get imported pore data.

Keyword arguments:

  import_number --- the import number (1 through 8)

Usage:

  porew, incvol, desc = mic.imported_pore_data(1)

  porew  --- array of pore dimension boundaries (angstroms);
             empty-array if unavailable.
  incvol --- array of incremental pore volumes (cm^3/g);
             empty-array if unavailable.
  desc   --- Name of data set; empty-string if unavailable.
```

GET METAL COMPOSITION FOR CHEMISORPTION

```
mic.metal_composition(metal='', metal_property='', sample_number=0):

Get information about the active metals in this sample

Keyword arguments:

  metal           --- the metal to return information about
                      if '' or None, then return a list of the
                      active metals

  metal_property  --- the specific property to return information on
                      if '' or None, then return all the properties
                      for the specified metal (requires metal to be
                      specified)

  sample_number --- Identifier for the metal data to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  metal_list  = mic.metal_composition()
  copper_prop = mic.metal_composition('copper')
  copper_perc = mic.metal_composition('copper',
                                      'percent of sample mass')

In the above first usage case, the list of active metals is returned.
In the above second usage case, a python dictionary type
is returned which includes all the properties of the metal
available and their corresponding values.  The last case returns
```

```
a single value (int, float, or string) for the specified property.

The metal_property keywords which one can use are

  atomic weight
  oxygen atoms
  density
  percent of sample mass
  metal atoms
  cross sectional area
  percent reduced
  stoichiometry GASNAME

where in the last keyword one substitutes the desired gas name to
obtain its stoichiometry factor.  One can also make the call
metal_composition(metalname) without any metal_property keyword
provided to see the whole dictionary of keywords and values.
```

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B ATOMIC WEIGHTS AND CROSS-SECTIONAL AREAS

CFR Note

Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

Atomic Weights and Cross-sectional Areas for Selected Metals

Metal	Symbol	Atomic Weight (g/mol)	Cross-sectional Area (nm^2)	Density (g/ml)
chromium	Cr	51.996	0.0635	7.19
cobalt	Co	58.933	0.0662	8.9
copper	Cu	63.54	0.0680	8.96
gold	Au	196.967	0.08696	18.9
hafnium	Hf	178.490	0.0862	13.3
iridium	Ir	192.220	0.0769	22.4
iron	Fe	55.847	0.0613	7.89
manganese	Mn	54.938	0.0714	7.43
molybdnum	Mo	95.940	0.0730	10.22
nickel	Ni	58.710	0.0649	8.9
niobium	Nb	92.906	0.0806	8.57
osmium	Os	190.220	0.0629	22.6
palladium	Pd	106.400	0.0787	12.02
platinum	Pt	195.090	0.0800	21.45
rhenium	Re	186.2	0.0649	21.02
rhodium	Rh	102.905	0.0752	12.1
tuthenium	Ru	101.070	0.0613	12.4
silver	Ag	107.868	0.0869	10.5
tantalum	Ta	180.947	0.0800	16.6
thorium	Th	232.038	0.1350	11.7
tin	Sn	118.710	0.1082	4.54
tungsten	W	183.850	0.0741	19.3
vanadium	V	50.942	0.0680	6.11
zirconium	Zr	91.220	0.0877	6.51

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C CALCULATE FREE SPACE VALUES FOR MICROPORE ANALYSES

Many microporous materials, such as zeolites and activated carbons, trap and hold helium in their complex pore structures for many hours after being exposed to helium. Helium trapped in micropores can interfere with the analysis at low pressures, causing an “S”-shaped curve at the lower end of the isotherm. For this reason, it is recommended to enter the ambient and analysis free space volumes when performing micropore analyses, therefore avoiding exposure of the sample to helium. Two techniques can be used for determining ambient and analysis free-space values.

The first method is to perform a short analysis on the sample after partial degassing (one pressure point with no incremental dosing), but prior to final sample preparation. Measure the free space during this analysis. The measured free space values will be printed on the report and may then be entered into the sample file after more thorough sample preparation.

The second method requires prior tests using empty tubes that will be employed later for the sample analyses. The measured free space data can be used thereafter on every analysis performed using these sample tubes. This small initial investment of time will save considerable time later. Perform an empty tube analysis on each sample tube to be used for micropore analysis. Measure the free space of each sample tube, taking only one pressure point

1. Create a sample tube file from each empty tube test.
2. Use the **Load from Sample File** button to read in the measured free space from the empty tube tests.
3. In the sample file for the sample analysis, pick the appropriate sample tube from the sample tube list.
4. Enter the mass and density of the sample.
5. *Select Calculate* in the free space options in the analysis conditions.

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D CRYOSTAT

The cryostat elevator is manually operated, however the analyzer elevator must first be disabled to prevent damage to the analyzer.

A CryoStat is an available option. Parts and accessories are located on the Micromeritics web page.

Unit [n] > Enable CryoStat

When selected:

- ***Unit [n] > Disable Elevator*** is automatically selected
- applicable cryostat fields within the application will be enabled

Unit [n] > Disable Elevator

When selected, the analyzer elevator will not move until this option is deselected.

CryoStat Operating Instructions

The cryostat provides automatic analysis temperature control over a wide range of sub-ambient temperatures.

- Read and fully understand this entire section prior to carrying out operations on the system.
- All system components must be powered ON in the sequence provided in this section.
- Pay attention to all local safety systems and requirements and consider the specifics of the experiment when operating the system.

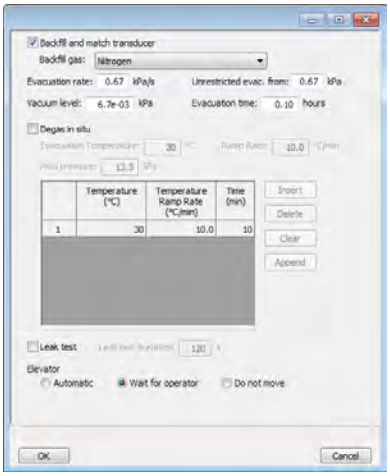
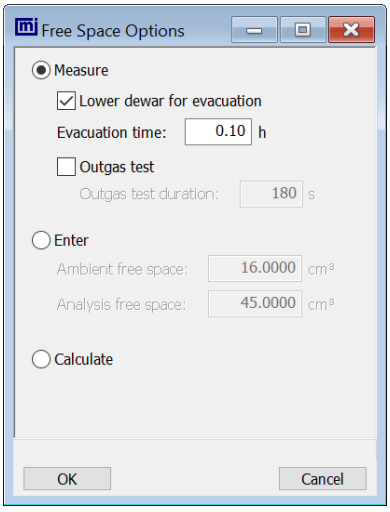
1. Prior to starting any analysis on the system, ensure the following:
   - Go to ***Unit [n] > Enable CryoStat*** and ensure a checkmark displays to the left of the menu option. A checkmark indicates the item has been selected and disables the elevator during an analysis when using the cryostat.
   - The cryostat is correctly mounted and held firmly in place by the metal retaining bands.
   - Release the brake on the cryostat cradle and move the system upwards to check that the sample well and the analyzer sample port is correctly aligned.
   - Check that the vacuum connection, helium connection, and wired connections to the compressor and temperature controller are in place and correctly fitted.
2. Prepare the samples.
   - Samples can be prepared in-situ using the analyzer heating mantle (if the heating mantle fits with the cryostat installed). See ***Degas in Situ on page 5 - 3***. This will depend on the type of heating mantle used.
   - The cryostat can be prepared to run analyses in the DOWN position while the samples are being prepared. Leave the insulating cover for the cryostat sample well in place while doing this.
3. Turn on the helium supply and adjust the rotameter to scale position 55. This corresponds to a flow rate of approximately 5 ml/min. See ***Rotameter Scale Readings on page D - 8***.
4. Allow the helium to purge the sample well for fifteen minutes. This purge procedure removes water vapor from the sample well and prevents water vapor from freezing when the system is in use at sub-ambient / cryogenic temperatures.
5. Power ON the compressor by flipping the first switch of the circuit breaker to the UP position. The compressor will now start to pump.
6. Press the **ALL OFF** button to power ON the temperature controller. Set the required temperature on the temperature controller or in the analysis application. If using the temperature controller, refer to the temperature controller manual supplied by the manufacturer.

7. Create a sample file (see ***Create Sample Files on page 3 - 2***). There are settings specific to physisorption analyses using a cryostat in ***Analysis Conditions for Physisorption and Chemisorption on page 4 - 5***.
8. With the sample tubes in place, preparation completed, and the tubes cooled to ambient temperature, slide the insulating cover up the sample tubes so that the cover fits snug against the mouth of the raised cryostat.
9. Remove the insulating cover from the top of the cryostat sample well. When prompted by the analyzer application, raise the cryostat.
10. Allow the samples to thermally equilibrate before starting the analysis.
11. When prompted by the analyzer application, lower the cryostat and place the insulating cover over the sample well of the cryostat.
12. If the sample tubes are to be removed, use ***Unit [n] > Backfill Ports*** or use the manual control option and pressurize the sample tubes to 760 mmHg at ambient temperature.
13. Weigh the samples after analysis and enter the sample weight into the sample file. The cryostat can now be used to analyze more samples at the same temperature or change the set point temperature.
14. Power OFF the compressor, vacuum pump, helium supply, and temperature controller. The cryostat should remain powered ON unless it will be unused for a long period of time. If it is necessary to power OFF the cryostat, set it to ambient temperature and allow it to come up to room temperature.

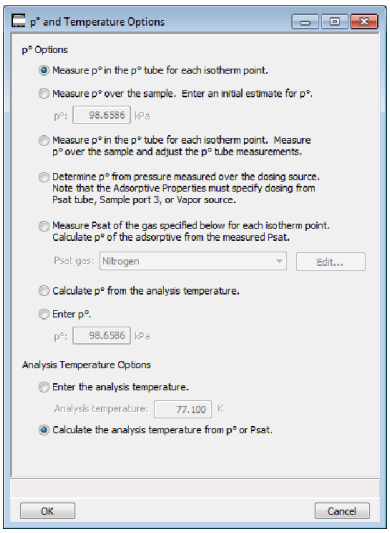
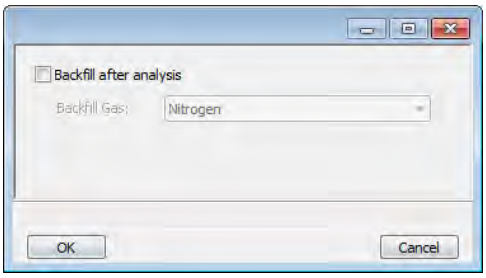
SAMPLE FILE CONFIGURATION FOR FLEX

1. Start the analysis program and open a sample file or create a new one.
2. Select the *Analysis Conditions* tab then use the *Analysis Conditions Buttons* table to complete the sample file.

Analysis Conditions Buttons

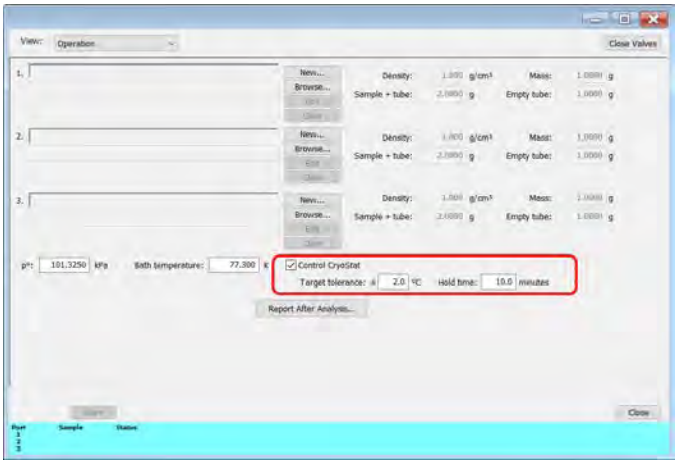
Button	Enter....
Preparation	 In the *Elevator* section, select *Wait for Operator*. Click **OK**.
Free Space	 Depending on the sample and proposed analysis, the free space can be *entered*, *measured*, *measured after analysis*, or *calculated*. If the free space is to be *measured*, then this can be done in the normal manner, but with the operator manually moving the cryostat assembly when prompted.

Analysis Conditions Buttons (continued)

Button	Enter....
p^0 and T	 In the *Analysis Temperature Options* section, select *Calculate the analysis temperature from p0 or Psat*. Click **OK**.
Termination	 Deselect *Backfill after analysis*.

3. Go to ***Unit [n] > Sample Analysis***.

4. Select *Control CryoStat*, enter a *Target Tolerance* and a *Hold Time*. The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.



INSTALL SAMPLE TUBE FOR THE CRYOSTAT

Sample Tube Installation for Physisorption on page 6 - 10

Sample tubes for the cryostat are installed in the same manner as the sample tubes for the analyzer.

REMOVE THE CRYOSTAT FROM THE ANALYZER

To run a normal analysis, the cryostat must be removed.

1. Move the cryostat to the DOWN position.
2. Place the brake in the bottom position.
3. Remove the 1/8 in. nylon helium tube.
4. Close vacuum valve.
5. Remove the 1/4 in. flexible stainless steel evacuation hose at the Swagelok valve.
6. Remove the large hose clamps and lift the entire cryostat off the cradle.
7. Lay the assembly on an adjacent bench so that other hoses do not need to be disconnected.
8. Close the helium purge supply, close the vacuum valve, power OFF the vacuum pump and compressor, turn OFF the temperature controller.

It is not necessary to remove the carriage assembly from the analyzer; however, the special safety shield (supplied with the cryostat kit) should be used. This shield has been modified to reach over the cryostat panel and hook onto the analyzer.

ROTAMETER SCALE READINGS

The rotameter scale corresponds approximately to flow rates:

A. Adjustment knob
B. Brake

Rotameter Scale to Flow Rate

Scale Reading	Flow Rate (ml/min)
65	5.8
55	5
45	4.3
35	3.3
25	2.5
15	1.8
5	1.3

TROUBLESHOOTING THE CRYOSTAT

Frost or ice accumulates around the outside of the cryostat in the area of the sample well

Cause: The internal vacuum is degrading and must be re-evacuated.

Action: Effective re-evacuation can be done only when the cryostat is warm, such as after an analysis. It is recommended that the vacuum pump always remain powered ON.

Elevator tries to rise

Cause: Analysis program is not set to enable the cryostat.

Action: In the analysis program, go to ***Unit [n] > Enable CryoStat*** and ensure that this option is selected. Failure to do so will cause the P_0 port to be evacuated and the elevator will try to rise.

Valve motor in cryostat cold head does not start when the compressor starts

Cause A: Cold head cable is not connected.

Action A: Stop the compressor. Connect the cable.

Cause B: Open circuit in the cold head cable.

Action B: Disconnect the cold head cable. Check each conductor for continuity. Replace the cable if necessary.

Cause C: Defective valve motor.

Action C: Contact your Micromeritics Service Representative.

Cause D: Blown fuse in the compressor's electrical box.

Action D: Refer to the manual supplied by the compressor manufacturer.

Valve motor in cryostat cold head hums but does not start

Cause A: Valve disk has stalled.

Action A: Check the operating pressures on the compressor. Contact your Micromeritics Service Representative.

Cause B: Defective valve motor.

Action B: Contact your Micromeritics Service Representative.

Cause C: Open circuit in the cold head cable.

Action C: Disconnect the cold head cable. Check each conductor for continuity. Replace the cable if necessary.

Valve motor on the cryostat cold head runs but there is no cooldown

Cause A: No insulating vacuum.

Action A: Check the vacuum system for operation and leaks.

Cause B: Gas line couplings are not fully engaged.

Action B: Ensure all couplings are fully engaged and torqued.

Cause B: Gas lines are connected incorrectly.

Action C: Reconnect the gas lines. Refer to the manual supplied by the compressor manufacturer.

Cause D: Compressor output is inadequate.

Action D: Troubleshoot the compressor. Refer to the manual supplied by the compressor manufacturer.

Shroud is sweating or abnormally cold

Cause: Loss of insulating vacuum.

Action: Check the vacuum system for operation and leaks.

Abnormally noisy operation after a sustained period of five to fifteen minutes

Cause A: Incorrect compressor pressures.

Action A: Troubleshoot the compressor. Refer to the manual supplied by the compressor manufacturer.

Cause B: Contaminants in the gas.

Action B: Perform *Gas Cleanup and Recharging* procedure on the cold head, compressor, and the gas lines. Refer to the manual supplied by the compressor manufacturer.Contact your Micromeritics Service Representative.

Intermittent operation

Cause: Compressor is cycling on and off.

Action: Troubleshoot the compressor. Refer to the manual supplied by the compressor manufacturer.

Temperature is cycling

Cause: Contaminated gas is causing a cold head freezing-thawing cycle.

Action: Perform *Gas Cleanup and Recharging* procedure on the cold head, compressor, and the gas lines. Refer to the manual supplied by the compressor manufacturer.Contact your Micromeritics Service Representative.

Sudden loss of refrigeration capacity

Cause A: Loss of insulating vacuum.

Action A: Check the vacuum system for operation and leaks.

Cause B: Compressor malfunction.

Action B: Troubleshoot the compressor. Refer to the manual supplied by the compressor manufacturer.

PREVENTIVE MAINTENANCE FOR THE CRYOSTAT

The preventive maintenance procedures can be located in the documents supplied by the cryostat manufacturer.

Maintenance Required	Frequency
Cold head	Every 13,000 hours
Compressor (absorber)	Every 30,000 hours

CRYOSTAT CALIBRATION

The cryostat temperature reading does not necessarily reflect the temperature at the sample. The discrepancy is large enough to cause significant differences between data taken with a cryogenic bath and data taken with the cryostat set to the temperature of the bath.

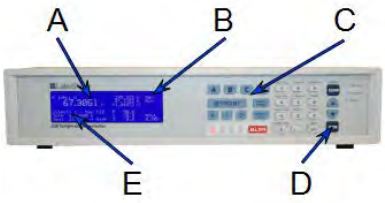
One solution is to modify the voltage-to-temperature curve stored in the temperature controller so that the reading accurately reflects the temperature of the sample. This option requires additional interaction with the temperature controller to download, upload, and select the voltage-to-temperature curves. However, this is straightforward with the Lakeshore controller and their Curve Handler software.

REQUIRED ITEMS

- A Flex analyzer equipped with an installed Micromeritics cryostat unit.
- A selection of adsorptives to cover the temperature range to be calibrated — such as nitrogen, argon, methane, ethane, and propane. Choose adsorptives whose saturation pressure is less than 800 mmHg at the measurement temperatures.
- The Lakeshore Curve Handler software (free download): http://www.lakeshore.-com/products/Pages/curvehandler.aspx.
- An empty sample tube for the Micromeritics gas adsorption analyzer being used.
- A spreadsheet application. Knowledge of how to load a .TXT file containing tabular data into a spreadsheet application is beneficial.

CALIBRATION PROCEDURE

1. In the analysis application, go to the *Unit* menu. Ensure there is a checkmark to the left of *Disable Elevator* and no checkmark to the left of *Enable CryoStat*. The cryostat will be adjusted manually.
2. Install a blank sample tube into the gas adsorption analyzer. Note that only one tube is required for the Flex.
3. Raise the cryostat and lock it in place following the general cryostat procedure.
4. Set the cryostat to the required temperature. On the LakeShore temperature controller, press **SETPOINT**, enter the temperature (in kelvin), then press **ENTER**.



A. Measured temperature
B. Probe signal voltage
C. Setpoint button
D. Enter button
E. Set temperature

5. Wait until the cryostat reaches the entered temperature. The time varies depending on how big the temperature jump is. For example, going from 200 K to 215 K may take 15 to 20 minutes; going from 298 K to 77 K might take 90 minutes. Once the cryostat has reached the operating temperature, record the set temperature, the measured temperature, and the cryostat signal probe voltage.

6. Create a sample file to measure saturation pressure.

   The analysis is to run in the absolute pressure dosing mode with an upper pressure limit of 950 mmHg. Increment volume dosing is to be employed using a dose volume of 20 cm^3/g STP with a 5 second equilibration time. In Analysis Conditions:

   a. Select the *Absolute pressure dosing* option.
   b. Select the adsorptive to be used.
   c. Enter the pressure settings shown in the *Pressure Settings for Flex* table below.

Pressure Settings for Flex

	Starting Pressure (mmHg)	Pressure Increment (mmHg)	Dose amount (cm^3/g STP)	Equilibration Interval(s)	Ending Pressure (mmHg)
Line 1	0.000000			5	100.000000
Line 2	100.000000		20.0000	5	900.000000

7. Once the analyzer is set up and the temperature for the first data point has reached equilibrium, start the analysis defined by the sample file. The analyzer will dose the sample tube with 20 cm^3/g STP of adsorptive . Once the tube pressure reaches the adsorptive saturation pressure, the generated isotherm will climb straight up. Take several points at this saturation pressure, then pause the analysis and set the cryostat to the next temperature.

   Once the cryostat has reached the set temperature and has equilibrated a few minutes, record the cryostat temperature and the probe signal, then resume the analysis. Perform this cycle for the number of temperature points required for the adsorptive being used. Typical isotherms are shown below. Note that the temperature labels are for clarity and are not a part of the normal report.

Additional adsorptives will allow the calibration to be extended. A set of local fits may be needed to cover a wide temperature change.

Depending on the selected adsorptive, a warning message may be given indicating that 950 mmHg is not the saturation pressure for the temperature being used. This is a normal message and will not interfere with the experiment. Click **OK** and continue with the analysis.

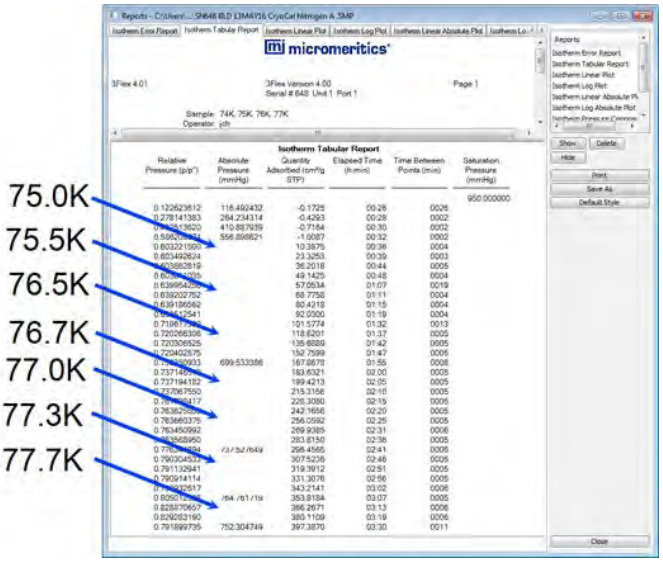
These analyses will not end and will have to be terminated manually by stopping the analysis window. To terminate the analysis, click **Cancel**, then wait a few seconds and click **Cancel** again (do not double-click the **Cancel** button).

The analysis will end abruptly with adsorptive in the sample tube. The sample tube must be manually evacuated before the cryostat is lowered.

Manually close all valves. Go to ***Unit > Evacuate Ports*** to evacuate the sample tube(s) used for the measurement.

The cryostat can be lowered once the sample tube has been completely evacuated.

This saturation pressure data can either be read from the graph or an isotherm report can be made and the saturation pressure read from the report:



8. Place the data in table format using a spreadsheet application.

Temperature and Saturation Pressure Values

	Set Temperature (K)	CryoStat Temperature (K)	Signal Probe Voltage	Measured Saturation Pressure (mmHg)	Sample Tube Temperature (K)
Nitrogen	65	64.999	1.04878	137.833	65.317
	67.5	67.498	1.04474	206.334	67.778
	70	69.999	1.04066	301.792	70.294
	73	72.996	1.03572	454.217	73.239
	73	72.996	1.03572	456.148	73.271
	73	72.998	1.03571	452.139	73.239
	75	75.002	1.03240	584.063	75.189
	75	75.002	1.03245	587.793	75.240
	77	76.993	1.02896	743.094	77.165
	77	76.995	1.02897	742.343	77.156
	78	78.002	1.02737	832.533	78.165
Ethylene	140	139.999	0.91437	111.175	142.470
	145	144.999	0.90456	168.125	147.470
	150	150.000	0.89477	245.617	152.350

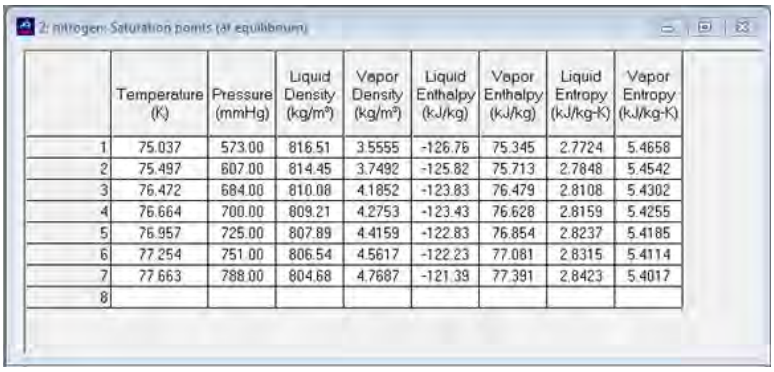
Set Temp — Cryostat temperature setting, in kelvin.

CryoStat Temperature — Temperature reported by the temperature controller.

Signal Probe Voltage — Signal probe voltage reading.

Measured Saturation Pressure — Equilibrium pressure measured by the instrument when the isotherm is vertical.

Sample Tube Temperature — Sample tube temperature calculated from the measure saturation pressure using NIST's REFPROP program (bundled with the Micromeritics applications).

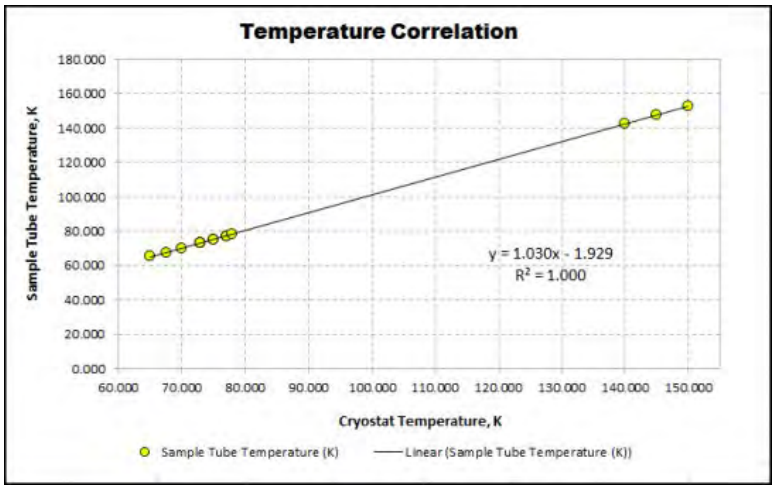


2: nitrogen Saturation points (at equilibrium)

	Temperature (K)	Pressure (mmHg)	Liquid Density (kg/m³)	Vapor Density (kg/m³)	Liquid Enthalpy (kJ/kg)	Vapor Enthalpy (kJ/kg)	Liquid Entropy (kJ/kg-K)	Vapor Entropy (kJ/kg-K)
1	75.037	573.00	816.51	3.5555	-126.76	75.345	2.7724	5.4658
2	75.497	607.00	814.45	3.7492	-125.82	75.713	2.7848	5.4542
3	76.472	684.00	810.08	4.1852	-123.83	76.479	2.8108	5.4302
4	76.664	700.00	809.21	4.2753	-123.43	76.628	2.8159	5.4255
5	76.957	725.00	807.89	4.4159	-122.83	76.854	2.8237	5.4185
6	77.254	751.00	806.54	4.5617	-122.23	77.081	2.8315	5.4114
7	77.663	788.00	804.68	4.7687	-121.39	77.391	2.8423	5.4017
8								

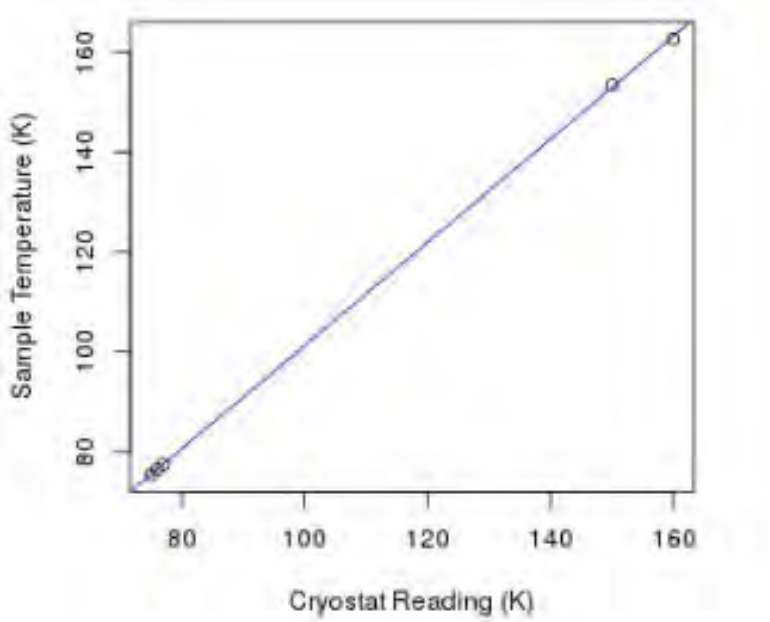
Screenshot from REFPROP software

The spreadsheet data can be graphed in the REFPROP spreadsheet program. Plot the REPROP to determine temperature versus the Cryostat reading. Fit the data points:



Temperature Correlation

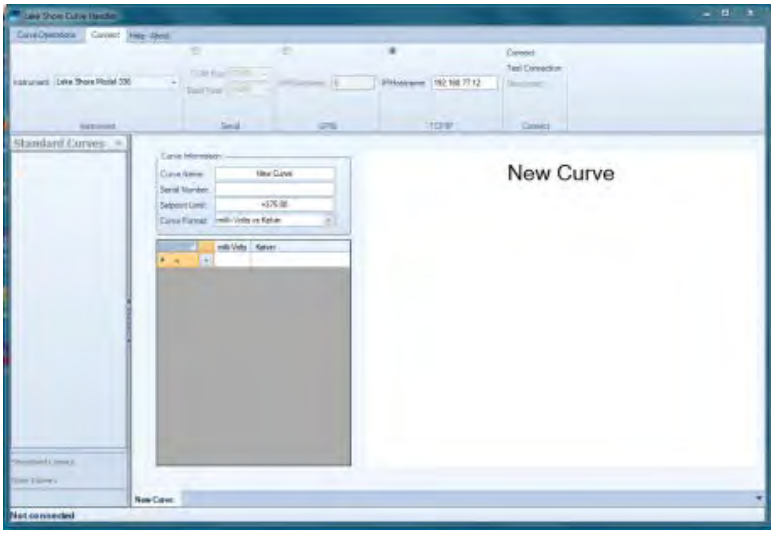
Additional adsorptives will allow the calibration to be extended. The following graph contains data for nitrogen combined with data from ethane:



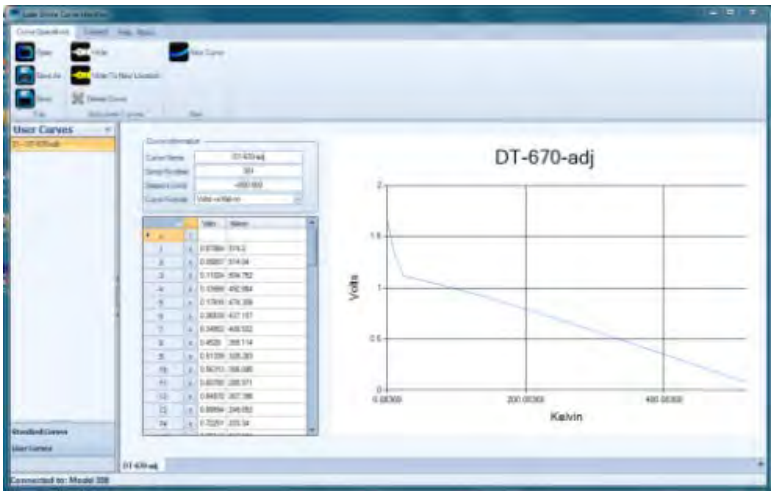
Temperature Correlation

9. The REPROP determined sample tube temperatures, combined with the signal probe voltages read from the LakeShore controller are then used to construct a cryostat calibration curve.

   The LakeShore Curve Handler program (shown below) is used for this operation. (Refer to the LakeShore Curve Handler manual for communications information.) Connect the cryostat to the computer via an Ethernet connection.



10. Click **Connect** to establish communication with the LakeShore Temperature Controller. Once communication is established, all the calibration curves in the LakeShore Temperature Controller will be listed.



11. The ColdEdge Cryostat/LakeShore Model 336 Temperature Controller uses the DT-670 silicon diode as the temperature sensor according to ColdEdge Micromeritics Interface Owner's Manual. Select the curve for the DT-670 temperature probe. Click **Save As** and save the DT-670 curve with a different name (i.e., *DT-670-xx*). Note that all calibration curves are stored as ASCII files in the Documents library with the file extension *.curve*. There will be a *DT-670.curve* file and a *DT-670-xx.curve* file.

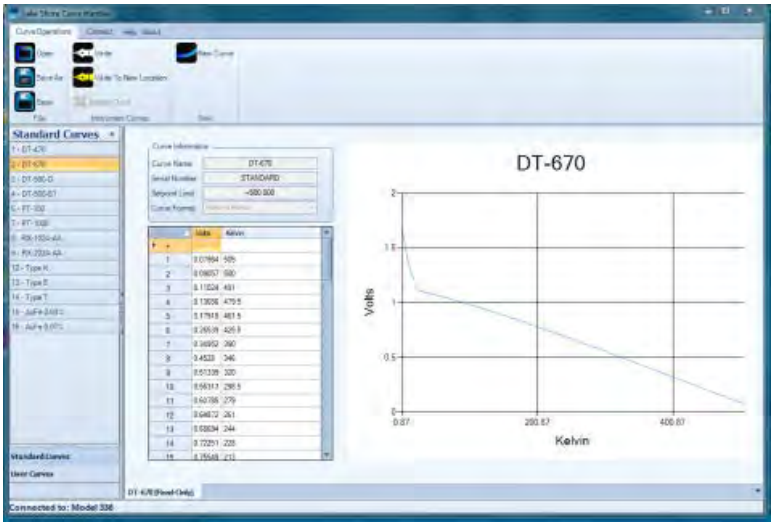
```
Sensor Model:   DT-670
Serial Number:  STANDARD
Data Format:    2       (Volts/Kelvin)
SetPoint Limit: +500.000

Measurement (Volts)     Temp (K)
7.964000E-02    5.050000E+02
9.057000E-02    5.000000E+02
1.102400E-01    4.910000E+02
1.365600E-01    4.795000E+02
1.791800E-01    4.615000E+02
2.653900E-01    4.255000E+02
3.495200E-01    3.900000E+02
4.528000E-01    3.460000E+02
5.133900E-01    3.200000E+02
5.631300E-01    2.985000E+02
6.078500E-01    2.790000E+02
6.487200E-01    2.610000E+02
6.869400E-01    2.440000E+02
7.225100E-01    2.280000E+02
7.554900E-01    2.130000E+02
7.869900E-01    1.985000E+02
8.170200E-01    1.845000E+02
8.445400E-01    1.715000E+02
8.695800E-01    1.595000E+02
8.932300E-01    1.480000E+02
9.144700E-01    1.375000E+02
9.343600E-01    1.275000E+02
9.529000E-01    1.180000E+02
9.701300E-01    1.090000E+02
9.860700E-01    1.005000E+02
9.989200E-01    9.350000E+01
1.010640E+00    8.700000E+01
1.021250E+00    8.100000E+01
1.031670E+00    7.500000E+01
1.041890E+00    6.900000E+01
1.051920E+00    6.300000E+01
1.062770E+00    5.640000E+01
1.074720E+00    4.900000E+01
1.091100E+00    3.870000E+01
1.096020E+00    3.570000E+01
1.100140E+00    3.330000E+01
1.103930E+00    3.120000E+01
```

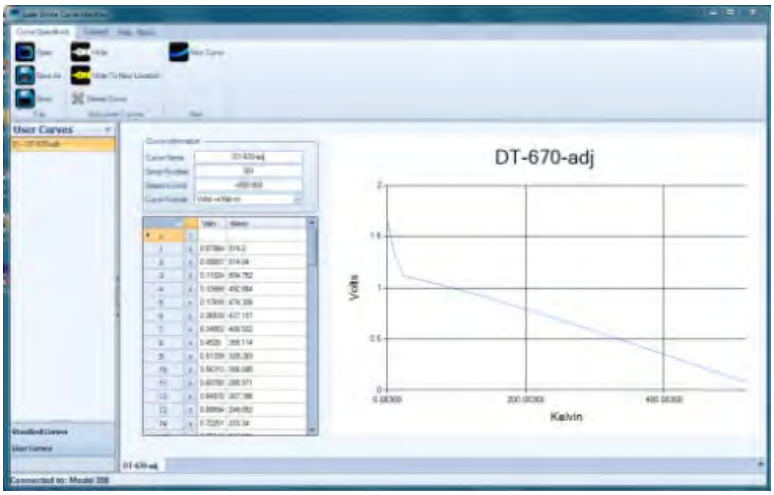
The LakeShore Curve Handler instruction manual provides several methods of entering user created calibration. The following process uses a file copy of a modified version of the DT-670 curve previously saved.

12. Produce a modified calibration curve by applying the fit from step 8 to the temperatures in DT-760 curve.

13. Start the LakeShore Curve Handler program and establish communication with the LakeShore Temperature Controller.



14. Click **Open** and select the modified *DT-670-xx.curve*. Place this curve in the first empty bin. User curve bins start at Number 21.
15. The new curve should now be able to be loaded into the LakeShore Curve Handler program. Click the *User's Curve* tab in the lower left side of the window.



16. Note in this example the modified curve *DT-670-xx.curve* is in bin 22 (left column) and the curve is displayed on the right. The values should be reviewed and any changes made. Click **Save** if changes have been made. Click **Write** to load the new curve into the LakeShore Temperature Controller. Exit the LakeShore program.
17. To select the new curve, on the temperature controller, press the *Input* command (key 7), select the *Input* channel (usually *A*), use the arrows to navigate to the curve menu, press **Enter**, then select the curve. Use curve 22 in this example. Press **Enter** once more, then press **Escape**. The new, modified curve is now in place. More details can be found in the LakeShore Model 336 Temperature Controller User's Manual.

The table below shows the effect of the new curve:

Temperature and Saturation Pressure Values

Set Temp (K)	Cryostat Temperature (K)	Measured Saturation Pressure (mmHg)	Uncalibrated Sample Tube Temperature (K)	Calibrated Sample Tube Temperature (K)	% Error (Calculated vs Uncalibrated)
73	72.997	473.450	73.553	73.250	-0.414
75	74.996	615.020	75.603	75.215	-0.517
77.3	77.002	786.618	77.648	77.029	-0.804

E DATA LOGGING

The Flex has the added capability to collect, access, and use real time transient data (pressures, temperatures, quantities adsorbed, etc.) during adsorption and desorption measurements. Data are collected every half second. Data logging can also be performed from a Smart VacPrep degasser attached to the analyzer. The *Smart VacPrep Operator Manual* can be found on the Micromeritics web page (www.Micromeritics.com).

This document provides:

- installation instructions for the PuTTY client and outlines how to access and use the data file, and
- installation instructions for Python components and outlines how the captured data can be handled using Python scripts.

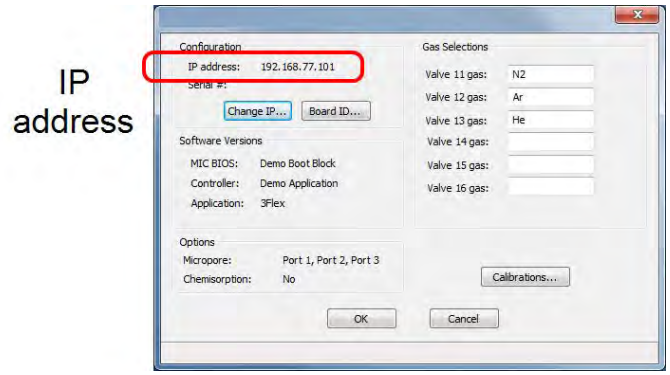
INSTALL THE PUTTY CLIENT

PuTTY is an open source SSH and telnet client that can be used to access the transient data output by the Flex analyzer. No additional Micromeritics application is required. The PuTTY.exe file runs directly from a desktop shortcut. Download the PuTTY application using the following URL (only save the *putty.exe* file):

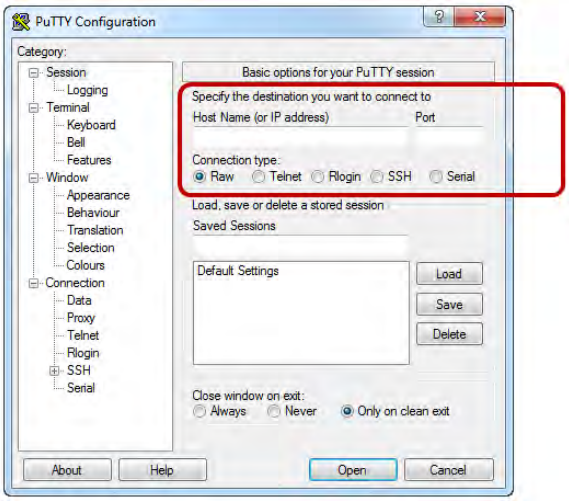
http://www.chiark.greenend.org.uk/~sgtatham/putty/download.html

To connect to the Flex analyzer:

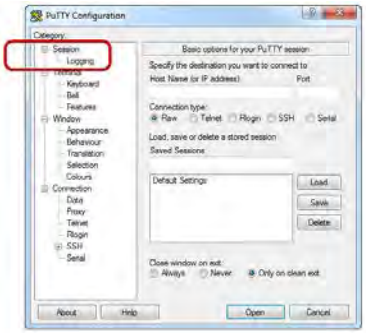
1. In the Flex application, go to ***Unit [n] > Unit Configuration*** and make a note of the analyzer IP address.

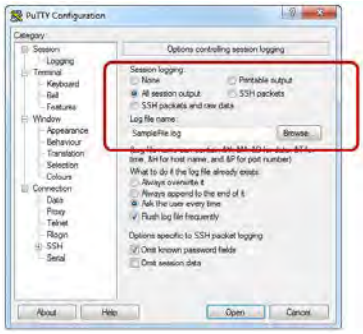


2. In the *PuTTY Configuration* window:
   a. Enter the analyzer IP address (from Step 1) into the PuTTY application.
   b. Enter *54000* 54000 in the *Port* field.
   c. Select *Raw* as the connection type.



3. To save the settings (optional):





   a. In the *Category* box, select ***Session > Logging***.
   b. In the *Session logging* group box, select *All session output*.
   c. In the *Log file name* group box, click **Browse** to select the destination and enter a .txt file name. It may be helpful to name this file the same as the sample file to be analyzed.
4. Click **Open** to start collecting the data.

The PuTTY application can be started before the analyzer starts collecting data — it will be *waiting* until data output from the analyzer begins.

ACCESS THE DATA LOG FILE

The text file created in the previous section (Step 3c) contains the transient data and can be accessed using any means that accepts tab separated value such as Notepad, Microsoft Excel (or other spreadsheet software), or programming languages that read .txt or .xls files such as MATLAB, Octave, and Python. MATLAB has the *xlsread()* function and Octave has the *textread()* function.

DATA FILE COLUMN DESCRIPTIONS

When the data file is viewed in a spreadsheet program, the following columns display:

Flex Data File Column Descriptions

A. 'manifold pressure (Torr)';
B. 'manifold volume (cc)';
C. 'manifold temperature (K)';
D. 'time (ms)';
E. 'port 1 pressure (Torr)';
F. 'port 1 volume (cc)';
G. 'port 1 temperature (K)';
H. 'port 1 ambient temperature (K)';
I. 'port 1 analysis temperature (K)';
J. 'port 1 warm freespace (cc STP)';
K. 'port 1 cold freespace (cc STP)';
L. 'port 1 quantity dosed (cc STP)';
M. 'port 1 quantity adsorbed (cc STP)';
N. 'port 1 has manifold ?';
O. 'port 1 valve open ?';
P. 'port 1 data points taken';
Q. 'port 1 current pressure table index';
R. 'port 1 last data point elapsed time (ms)';
S. 'port 1 last data point pressure Torr)';
T. 'port 1 last data point quantity dosed (cc STP)';
U. 'port 1 last data point quantity adsorbed (cc STP)';
V. 'port 1 last data point Po (Torr)';
W. 'port 2 pressure (Torr)';
X. 'port 2 volume (cc)';
Y. 'port 2 temperature (K)';
Z. 'port 2 ambient temperature (K)';
AA. 'port 2 analysis temperature (K)';
AB. 'port 2 warm freespace (cc STP)';
AC. 'port 2 cold freespace (cc STP)';
AD. 'port 2 quantity dosed (cc STP)';
AE. 'port 2 quantity adsorbed (cc STP)';
AF. 'port 2 has manifold ?';
AG. 'port 2 valve open ?';
AH. 'port 2 data points taken';
AI. 'port 2 current pressure table index';
AJ. 'port 2 last data point elapsed time (ms)';
AK. 'port 2 last data point pressure Torr)';
AL. 'port 2 last data point quantity dosed (cc STP)';
AM. 'port 2 last data point quantity adsorbed (cc STP)';
AN. 'port 2 last data point Po (Torr)';
AO. 'port 3 pressure (Torr)';
AP. 'port 3 volume (cc)';
AQ. 'port 3 temperature (K)';
AR. 'port 3 ambient temperature (K)';
AS. 'port 3 analysis temperature (K)';
AT. 'port 3 warm freespace (cc STP)';
AU. 'port 3 cold freespace (cc STP)';
AV. 'port 3 quantity dosed (cc STP)';
AW. 'port 3 quantity adsorbed (cc STP)';
AX. 'port 3 has manifold ?';
AY. 'port 3 valve open ?';
AZ. 'port 3 data points taken';
BA. 'port 3 current pressure table index';
BB. 'port 3 last data point elapsed time (ms)';
BC. 'port 3 last data point pressure (Torr)';
BD. 'port 3 last data point quantity dosed (cc STP)';
BE. 'port 3 last data point quantity adsorbed (cc STP)';
BF. 'port 3 last data point Po (Torr)';

INSTALL PYTHON COMPONENTS

To operate Python to view real time data, three components should be installed in the following order:

1. Install PYTHON
   - Download URL: https://www.python.org/downloads/
   - Download Python version 3.2 or later

   Depending on the computer operating system, download file versions may vary, however, the .msi file should be compatible with computers that operate the Flex analyzer.

2. Install NumPY (for numerical functions)
   - Download URL: http://sourceforge.net/projects/numpy/files/NumPy/1.6.2/
   - Download file: numpy-1.6.2-win32-superpack-python3.2.exe

   NumPY contains numerous numerical functions and libraries needed to properly handle the transient data extracted from the .txt file collected during an analysis. NumPY version 1.6.2 should be download to be compatible with the installed version of Python.

3. Install MatPlotLIB (for graphical display of the data)
   - Download URL: http://www.lfd.uci.edu/~gohlke/pythonlibs/#matplotlib
   - Download file: matplotlib-1.2.1rc1.win32-py3.2.exe

   MatPlotLIB version 1.2.1 is compatible with NumPY from Step 2.

SAMPLE PYTHON CODE

The following code was used to produce the data in this section. This is basic code for viewing transient data. More detailed scripts can be created to compare pressure or quantity adsorbed between ports, generate a separate figure for each port, etc. Contact Micromeritics' applications specialists to discuss and support creating custom scripts for individual applications.

```
import numpy as np
import matplotlib.pyplot as plt

myFile = 'carbon.txt'
data = np.genfromtxt(myFile, skip_header=2, skip_footer = 1)

# Time (minutes)
x=data[:,3]
x = x/1000/60

# Sample pressure PORT 1(Torr)
y=data[:,4]

plt.plot(x,y,'ko')
plt.ylabel('Pressure (Torr)')
plt.xlabel('time (minutes)')
plt.show()
```

SAMPLE CODE EXPLANATION

```
import numpy as np
import matplotlib.pyplot as plt
```

Import is needed to load the modules containing functions to be used — in this case, numerical and plotting functions. Coding is easier if abbreviations such as *np* and *plt* are used.

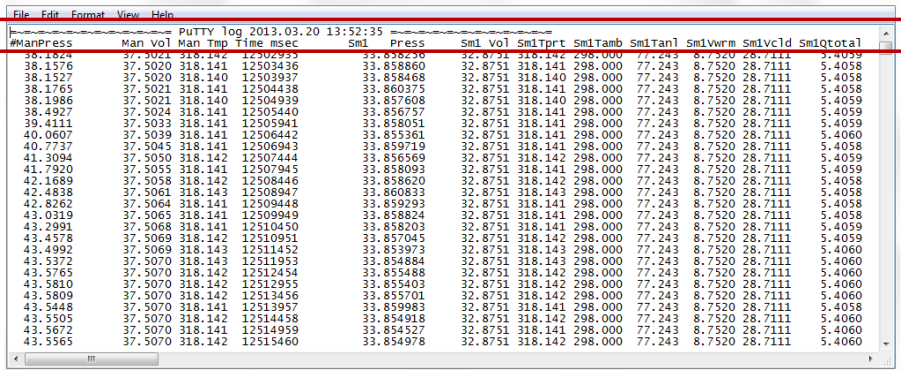
```
myFile = 'carbon.txt'
data = np.genfromtxt(myFile, skip_header=2, skip_footer = 1)
```

MyFile is a variable name for the .txt file to be read. As long as the .txt file and the script file are in the same directory, the script file can run automatically by double-clicking the script file after the correct file has been hard-coded.

The *genfromtxt()* function allows for the text file to be converted into a numerical matrix.

The *skip_header* setting allows for the first 2 rows of data to be excluded from the matrix. As shown in the following figure, the first row of data is ASCII *art* created by the PuTTY program, and the second row of data are the column labels — neither of which are numerical data.

The *skip_footer* setting is needed because, when the .txt file is read in real time, the entire last row of data may not be complete. This causes problems for creating the matrix and needs to be excluded. This means that the last half-second of data is not displayed when the data are viewed in real time.


File Edit Format View Help

~~~~~~~~~~~~~~~~ PuTTY log 2013.03.20 13:52:35 ~~~~~~~~~~~~~~~~

#ManPress	Man Vol	Man Tmp	Time msec	Sm1	Press	Sm1 Vol	Sm1Tprt	Sm1Tamb	Sm1Tanl	Sm1Vwrm	Sm1Vcld	Sm1Qtotal
38.1576	37.5020	318.141	12503436		33.858860	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
38.1527	37.5020	318.140	12503937		33.858468	32.8751	318.140	298.000	77.243	8.7520	28.7111	5.4058
38.1765	37.5021	318.141	12504438		33.860375	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
38.1986	37.5021	318.140	12504939		33.857608	32.8751	318.140	298.000	77.243	8.7520	28.7111	5.4059
38.4927	37.5024	318.141	12505440		33.856757	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4059
39.4111	37.5033	318.141	12505941		33.858051	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4059
40.0607	37.5039	318.141	12506442		33.855361	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4060
40.7737	37.5045	318.141	12506943		33.859719	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
41.3094	37.5050	318.142	12507444		33.856569	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4059
41.7920	37.5055	318.141	12507945		33.858093	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4059
42.1689	37.5058	318.142	12508446		33.858620	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4058
42.4838	37.5061	318.143	12508947		33.860833	32.8751	318.143	298.000	77.243	8.7520	28.7111	5.4058
42.8262	37.5064	318.141	12509448		33.859293	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
43.0319	37.5065	318.141	12509949		33.858824	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
43.2991	37.5068	318.141	12510450		33.858203	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4059
43.4578	37.5069	318.142	12510951		33.857045	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4059
43.4992	37.5069	318.143	12511452		33.853973	32.8751	318.143	298.000	77.243	8.7520	28.7111	5.4060
43.5372	37.5070	318.143	12511953		33.854884	32.8751	318.143	298.000	77.243	8.7520	28.7111	5.4060
43.5765	37.5070	318.142	12512454		33.855488	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4060
43.5810	37.5070	318.142	12512955		33.855403	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4060
43.5809	37.5070	318.142	12513456		33.855701	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4060
43.5448	37.5070	318.141	12513957		33.859983	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4058
43.5505	37.5070	318.142	12514458		33.854918	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4060
43.5672	37.5070	318.141	12514959		33.854527	32.8751	318.141	298.000	77.243	8.7520	28.7111	5.4060
43.5565	37.5070	318.142	12515460		33.854978	32.8751	318.142	298.000	77.243	8.7520	28.7111	5.4060

Exclude the first two rows of data

Sample of Transient Data

```
# Time (minutes)
x=data[:,3]
x = x/1000/60
```

Data File Column Descriptions on page E - 3 identifies the columns of data recorded. The fourth column of data contains time in milliseconds, however, the first index in Python is 0; therefore,
data[:,3] captures all of the rows *(:)* in the fourth column.

```
plt.plot(x,y,'ko')
plt.ylabel('Pressure (Torr)')
plt.xlabel('time (minutes)')
plt.show()
```

The above code shows the syntax for plotting. The label *'ko'* indicates filled black circles.
~~~~~~~~~~~~~~~~

DEGASSING AND CHEMISORPTION TEMPERATURE RAMP DATA

The same information provided above applies to capturing Flex data during degassing and during chemisorption temperature ramping, except:

DEGAS DATA

This data is available during programmed in situ degas sent once per second. Connect to port *54100* (rather than port *54000*) for in situ degas data (see ***Data Logging on page E - 1***). The data columns are:

A. time (ms)
B. manifold pressure (Torr)
C. manifold vacuum (Torr)
D. port 1 pressure (Torr)
E. port 1 temperature (C)
F. port 2 pressure (Torr)
G. port 2 temperature (C)
H. port 3 pressure (Torr)
I. port 3 temperature (C)

CHEMISORPTION TEMPERATURE RAMP DATA

Flex data can be captured during chemisorption analyses by connecting to port 54200 (see ***Data Logging on page E - 1***). Time and sample temperature are sent every half second when the external trigger is set to *ON*. This is typically done during a flowing preparation temperature ramp. The data columns are:

- Time (s)
- Sample temperature (°C)

The *External Trigger* in the analyzer application must be enabled for this data to be available.

- To manually set the trigger go to ***Unit [n] > Enable Manual Control***, right-click the *External Trigger* icon and select *ON*. The box is dark when enabled.
- Enable the *Set external trigger* box in a *Preparation Flow Task* in the Analysis Conditions to collect temperature data that is synchronized with an external mass spectrometer or other detector to be started by the external trigger signal.

F DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of 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 provided to 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 F - 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.

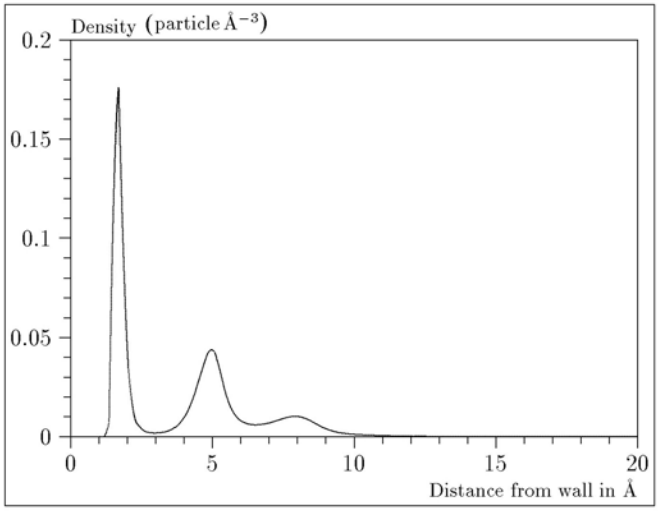


Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

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.

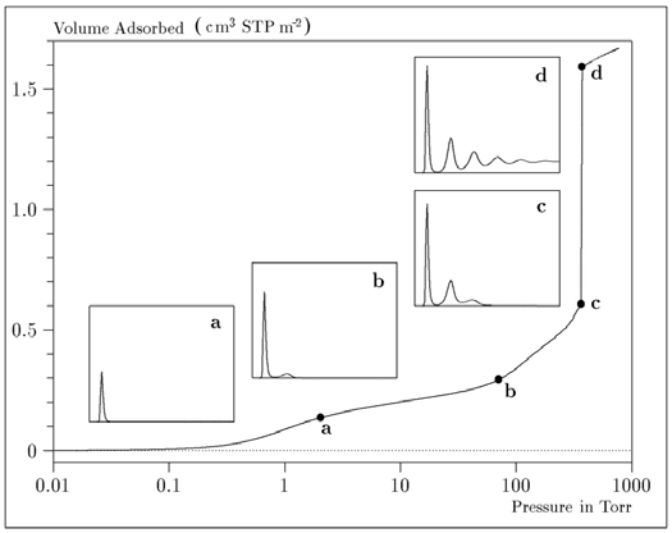


Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

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.

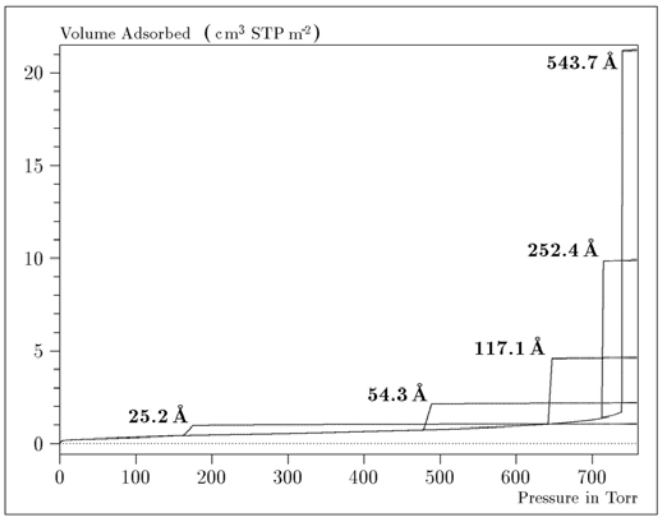


Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

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

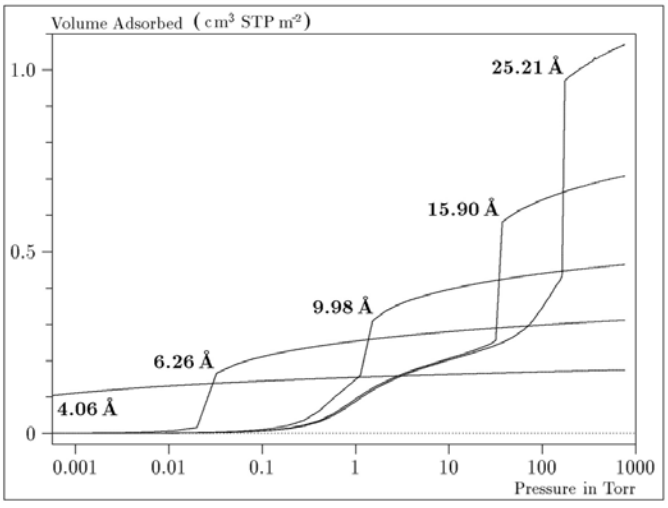


Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

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 F - 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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G EXPORTED DATA EXAMPLE

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Sample Information

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                  Operator:    jch
                 Submitter:
                 Mass type:    Calculated
                Empty tube:    34.6494 g
             Sample + tube:    35.3873 g
               Sample mass:    0.7379 g
                   Density:    1.000 g/cm³
              Type of data:    Automatically collected
           Instrument type:    3500 Chemi
  Original instrument type:    3500 Chemi
                  Comments:

                                        Active Metals Table

                                                                    Percent
                                                                 of  Percent
                                  Atomic Cross
Sect    Density     Sample      Reduced    MxOy    MxOy
   Element       Atomic Weight        Area (nm²)        (g/cm³)    Mass (%)
(%)XY
--------
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100.00       1       0
```

```
                    Adsorptive        Stoichiometry
                      Hydrogen                2.000
                        Oxygen                2.000
                Carbon Monoxide                1.500
                        Helium                1.000

  cobalt               58.9330               0.0662       8.900         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                      Hydrogen                2.000
                        Oxygen                2.000
                Carbon Monoxide                1.500
                        Helium                1.000

  copper               63.5400               0.0680       8.960         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                      Hydrogen                2.000
                        Oxygen                2.000
                Carbon Monoxide                1.500
                        Helium                1.000

  molybdenum           95.9400               0.0730      10.220         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                      Hydrogen                2.000
                        Oxygen                2.000
                Carbon Monoxide                1.500
                        Helium                1.000

  nickel               58.7100               0.0649       8.902         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
```

```
                    Hydrogen                2.000
                      Oxygen                2.000
                Carbon Monoxide             1.500
                    Ethylene                1.000

  palladium             106.4000              0.0787     12.020         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                    Hydrogen                2.000
                      Oxygen                2.000
                Carbon Monoxide             1.500
                      Helium                1.000

  platinum              195.0900              0.0800     21.450         0.50      -
100.00       1       0
                    Adsorptive        Stoichiometry
                    Hydrogen                2.000
                      Oxygen                2.000
                Carbon Monoxide             1.000
                      Helium                1.000

  rhenium               186.2000              0.0649     21.020         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                    Hydrogen                2.000
                      Oxygen                2.000
                Carbon Monoxide             1.500
                      Helium                1.000

  rhodium               102.9050              0.0752     12.410         0.00      -
100.00       1       0
                    Adsorptive        Stoichiometry
                    Hydrogen                2.000
```

```
                          Oxygen                  2.000
                  Carbon Monoxide                 1.500
                          Helium                  1.000

  silver                107.8680                0.0869     10.500         0.00      -
100.00       1       0
                       Adsorptive        Stoichiometry
                         Hydrogen                 2.000
                           Oxygen                 2.000
                  Carbon Monoxide                 1.500
                          Helium                  1.000


Analysis Conditions


                Analysis conditions:    Pt-Al Ref Matl with CO
     Pre-analysis evacuation time:      0.50 h
                    Repeat analysis:    Yes
  Repeat analysis evacuation time:      1.00 h
                 Equilibration time:    5


                           Adsorptive:    Carbon Monoxide (CO)
           Maximum manifold pressure:    123.323 kPa
  Therm. tran. hard-sphere diameter:    3.5900 Å
     Molecular cross-sectional area:    0.125 nm²
          Adsorbate molecular weight:    28.01
                   Mass flow constant:    1.000
                 Thermal conductivity:    1.00
```

Gas blend:	No
Fluid properties:	T:\fpi\version 3\carbon monoxide.fpi
Dosing method:	Normal

Pressure Table

Starting Pressure (kPa)	Pressure Increment (kPa)	Dose Amount (mmol/g)	Equilibration Interval (s)	Ending Pressure (kPa)
0.0000000			20	13.3322400
13.3322400	6.6661200		20	59.9950800

Preparation Options

Backfill and match transducer:	Yes
Backfill gas:	Nitrogen
Evacuation rate:	6.67 kPa/s
Unrestricted evac. from:	4.00 kPa
Vacuum Level:	6.7e-03 kPa
Evacuation time:	0.10 h

Analysis Task 1 - Evacuation

Evacuate for 30 min	below 2.7e-03 kPa.
Evacuation rate:	6.67 kPa/s
Unrestricted evacuation pressure:	4.00 kPa
Temperature:	110.0 °C
Temperature rate:	10.0 °C/min

Analysis Task 2 - Flow

```
                  Gas:    Hydrogen
            Flow rate:    50.0 cm³ STP/min
                 Time:    10 min
          Temperature:    100.0 °C
     Temperature rate:    10.0 °C/min
Set external trigger:    No

             Analysis Task 3 - Flow

                  Gas:    Hydrogen
            Flow rate:    50.0 cm³ STP/min
                 Time:    30 min
          Temperature:    400.0 °C
     Temperature rate:    10.0 °C/min
Set external trigger:    Yes

                Analysis Task 4 - Evacuation

               Evacuate for 30 min    below 2.7e-03 kPa.
                 Evacuation rate:    6.67 kPa/s
Unrestricted evacuation pressure:    4.00 kPa
                     Temperature:    400.0 °C
                Temperature rate:    10.0 °C/min

                Analysis Task 5 - Evacuation

               Evacuate for 10 min    below 2.7e-03 kPa.
                 Evacuation rate:    6.67 kPa/s
Unrestricted evacuation pressure:    4.00 kPa
                     Temperature:    35.0 °C
                Temperature rate:    100.0 °C/min
```

```
        Analysis Task 6 - Leak Test

 Outgas rate limit:    1.3e-03 kPa/min
       Temperature:    35.0 °C
  Temperature rate:    10.0 °C/min

                    Free Space Options

               Free space type:    Measure after analysis
               Evacuation time:    1.00 h
  Estimated ambient free space:    12.2664 cm³
Estimated analysis free space:    12.1432 cm³

         Temperature Options

  Temperature control:    Furnace
Analysis temperature:    35.0 °C
    Temperature rate:    10.0 °C/min

                 Dosing Options

    Absolute pressure tolerance:    0.6666 kPa
    Relative pressure tolerance:    5.0 %
Low pressure equilibration delay
                         Minimum:      0.00 h
                         Maximum:    999.00 h

         Termination Options

Backfill after analysis:    Yes
           Backfill gas:    Nitrogen
         Cool to < 50 °C:    Yes
```

```
Report Options


                          Report options:    Pt-Al Ref Matl with CO
                      Show report title:    No
                           Show graphic:    No
  Use thermal transpiration correction:    Yes
        Inside diameter of sample tube:    9.000 mm


        Isotherm: Yes


  Tabular report selected:    Yes
     Plot report selected:    Yes
              X-axis scale:    Linear
            Plot analysis:    Yes
     Plot repeat analysis:    Yes

   Isotherm Plot Options

        Plot curve:    Yes
       Plot points:    Yes
    Overlay samples:    No
  Autoscale X axis:    Yes
  Autoscale Y axis:    Yes


        Difference Method: Yes
```

H FREE-SPACE CORRECTION

Free space is that volume of the sample tube which is unoccupied by the sample. The quantity of gas dosed into the sample tube is calculated from the difference in pressures in the manifold before and after the dose is delivered. The quantity of gas adsorbed by the sample is calculated by subtracting the quantity of gas remaining in the free space of the sample tube after equilibrium is established from the quantity of gas originally dosed into the sample tube. Free space must be determined accurately to obtain a precise value for quantity adsorbed.

Static-volumetric systems consist basically of a gas manifold joined to a sample tube by an isolation valve. The manifold section has connections for an absolute pressure transducer, a temperature gauge, and a vacuum system. It also has inlets for the adsorptive gas and helium. A Dewar flask containing a cryogenic liquid (usually LN_2 at approximately 77 K) is situated so that it can be raised to immerse most of the sample tube. Two temperature zones exist within the sample tube when immersed in the cryogenic bath: a warm zone (the volume above the liquid level and near ambient temperature) and a cold zone (the volume below the liquid level at cryogenic temperature). Not only must the total free space volume be determined, but it also is necessary to determine the quantity of gas residing within the “cold” zone since a nonideality correction must be applied to only that quantity of gas.

The total quantity of gas in the partly immersed sample holder cannot simply be determined using n = PV/RT because temperature is not constant over the total volume, but instead is distributed as two temperature zones with a steep temperature gradient between them. A convenient method for resolving this problem is to derive two factors which, for the existing temperature profile, can be multiplied by the prevailing pressure to reveal the molar volume of gas contained in the cold zone and the total quantity residing in the free volume of the immersed sample holder (the analysis free space).

The analyzer provides the following methods for free space determination:

- Measure
- Calculate
- Enter

MEASURE

Generally, this method, although requiring a little more time (approximately 10 minutes), is the most preferred one for determining free space. It is simple, automatic, requires very little information, and essentially is error-proof. With this method, the instrument first evacuates the manifold and sample tube (containing sample), then isolates the sample tube by closing the valve. Then the manifold is charged with helium, the pressure measured, and the valve opened allowing the helium to expand into the sample tube at ambient temperature. Again the pressure is measured.

The Dewar is raised and the sample tube is cooled to cryogenic temperature. Again pressure drops; when pressure has equilibrated, the value is recorded. Ambient and analysis free spaces are calculated from (1) system volume, (2) system, ambient, and bath temperatures, and (3) measured pressures. From these, the value of the portion of analysis free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable. Free space determination by the analyzer requires the use of helium.
- Analysis speed is a major factor. A helium free space measurement of 10 to 15 minutes is required.
- The sample tends to absorb and retain helium for a prolonged period of time or if it adsorbs helium.

CALCULATE

This method is the most rapid and efficient way of compensating for free space. Ensure the following is accomplished:

- Perform a blank analysis on the sample tube.
- Load the blank analysis file data into the sample tube file.
- Enter the analysis bath temperature (found on the *p° and Temperature* window).
- Enter the sample mass and density (found on the *Sample Description* tab).

ENTER

This method allows for entering predetermined values for the ambient and analysis free spaces. The values to enter may be obtained in one of two ways:

- A pre-analysis free space calibration of the sample tube containing sample.
- The total free space of an empty sample tube is measured and the displacement of the sample calculated from its mass and density and subtracted from the total free space.

In either procedure, ensure that the level (or, in cases where the Isothermal Jacket is used, the effective level) of the cryogen bath on the sample tube is the same when the analysis is performed as it was when gathering data for free space calculations.

I GAS CHARTS

RELATIVE THERMAL CONDUCTIVITY OF GASES

Name	Chemical Formula	Conductivity (Relative to Air)
Air		1.00
Ammonia	NH_3	0.92
Argon	Ar	0.68
Butane	C_4H_{10}	0.60
Carbon Dioxide	CO_2	0.62
Carbon Monoxide	CO	0.97
Ethane	C_2H_6	0.79
Helium	He	5.84
Hydrogen	H_2	7.07
Krypton	Kr	0.37
Methane	CH_4	1.29
Neon	Ne	1.87
Nitric Oxide	NO	0.99
Nitrogen	N_2	1.00
Nitrogen Dioxide	NO_2 or N_2O_4	1.51
Nitrous Oxide	N_2O	0.65
Oxygen	O_2	1.02
Sulfur Dioxide	SO_2	0.38
Water Vapor	H_2O	0.67

TYPICAL GASES USED

Flow Rate of 50 cm^3/min				
Test	**Preparation Gas**	**Carrier Gas**	**Loop Gas**	**Other**
TPR Experiment	Argon	10% H_2 in Argon	N/A	
Calibration	N/A		Argon	TCD Level Calibration
TPD Ammonia	Helium or 15% NH_3 in Helium	Helium	N/A	
Calibration	N/A		NH_3 in Helium	TCD Level Calibration
TPD Hydrogen	10% H_2 in Argon	Argon	N/A	Calibration
Calibration	N/A		10% H_2 in Argon	TCD Level Calibration
TPD Oxygen	10% O_2 in Helium	Helium	N/A	
Calibration	N/A		10% O_2 in Helium	TCD Level Calibration
TPO Experiment	Helium	10% O_2 in Helium	N/A	
Calibration	N/A		Helium	TCD Level Calibration
H_2 Pulse Chemisorption	10% H_2 in Argon	Argon	10% H_2 in Argon	
CO Pulse Chemisorption		Helium	10% CO in Helium	
Calibration	Not Required			
BET Surface Area	Helium	30% N2 in Helium	N/A	
Calibration	N/A			User-defined manual injections of N_2 (0.5, 1.0, 1.5, and 2.0 cm^3)

GAS CONVERSION CONSTANTS

Dynamic analysis uses a Mass Flow Controller (MFC) to control the flow of gases. The MFC requires a conversion constant for each gas or gas mixture to compensate for variations in gas flows resulting from variations in the properties of gases. A default gas table containing MFC conversion constants is included on the *Options* menu. The following table provides a more complete list of gases and their conversion constants.

Gas Conversion Constants for the MFC

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Acetylene	C_2H_2	0.6535
Air (mixture)		0.9901
Allene	C_3H_4	0.4752
Ammonia	NH3	0.7822
Argon	Ar	1.3861
Arsine	A_5H_3	0.7525
Boron Trichloride	BCl_3	0.4356
Boron Trifluoride	BF_3	0.5743
Bromine Pentafluoride	BrF_5	0.2871
Bromine Trifluoride	BrF_3	0.4356
Butane	C_4H_{10}	0.2871
Butene	C_4H_8	0.3267
Carbon Dioxide	CO_2	0.7723
Carbon Monoxide	CO	0.9802
Carbon Tetracloride	CCl_4	0.3465
Carbon Tetrafluoride	CF_4	0.4752
Carbonyl Fluoride	COF_2	0.2673
Carbonyl Sulfide	COS	0.6733
Chlorine	Cl_2	0.8218
Chloroform	$CHCl_3$	0.4356
Chlorine Trifluoride	ClF_3	0.4257
Cyanogen	C_2N_2	0.4950

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Cyclopropane	C_3H_6	0.5050
Deuterium	D_2	0.9901
Diborane	B_2H_6	0.5446
Dichlorosilane	SiH_2Cl_2	0.4356
Dimethylamine	$(CH_3)_2NH$	0.6634
Dimethylether	$(CH_3)_2O$	0.5842
Ethane	C_2H_6	0.5446
Ethyl Chloride	C_2H_5Cl	0.2871
Ethylene	C_2H_4	0.6139
Ethylene Oxide	C_2H_4O	0.5842
Fluorine	F_2	0.9208
Fluroform	CHF_3	0.5644
Freon 11	CCl_3F	0.3762
Freon 12	CCl_3F_2	0.3861
Freon 13	$CClF_3$	0.4257
Freon 13 B1	$CBrF_3$	0.4059
Freon 14	CF_4	0.4703
Freon 21	$CHCl_2F$	0.4554
Freon 22	$CHClF_2$	0.5050
Freon 23	CHF_3	0.5644
Freon 113	CCl_2F-$CClF_2$	0.2277
Freon 114	CCl_2F_4-$CClF_2$	0.2554
Freon 115	$CClF_2$-CF_3	0.2713
Freon 116	CF_3-CF_3	0.2277
Germane	GeH_4	0.6436

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Helium	He	1.3762
Hexamethyldisizane	HMDS	0.1386
Hydrogen	H_2	1.0000
Hydrogen Bromide	HBr	0.9703
Hydrogen Chloride (Dry)	HCl	0.9802
Hydrogen Fluoride	HF	0.9901
Hydrogen Iodide	HI	0.9505
Hydrogen Selenide	H_2Se	0.8317
Hydrogen Sulfide	H_2S	0.8416
Isobutane	C_4H_{10}	0.3069
Isobutylene	C_4H_8	0.3366
Krypton	Kr	1.3762
Methane	CH_4	0.8020
Methylamine	CH_3NH_2	0.5644
Methyl Bromide	CH_3Br	0.6436
Methyl Chloride	CH_3Cl	0.6832
Methyl Fluoride	CH_3F	0.7525
Methyl Mercaptan	CH_4S	0.5842
Neon	Ne	1.3861
Nitric Oxide	NO	0.9901
Nitrogen	N_2	0.9950
Nitrogen Dioxide	NO_2	0.7525
Nitrogen Trioxide	N_2O3	0.4356
Nitrogen Trifluoride	NF_3	0.5446
Nitrous Oxide	N_2O	0.7426

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Oxygen	O_2	0.9802
Ozone	O_3	0.7327
Pentaborane	B_5Hg	0.2871
n Pentane	C_5H_{12}	0.2376
Perchloryl Fluoride	ClO_3F	0.4455
Phosgene	$COCl_2$	0.5050
Phosphine	PH_3	0.7822
Phosphorous Pentafluoride	PF_5	0.3465
Propane	C_3H_8	0.3861
Propylene (Propene)	C_3H_6	0.4653
Silane	SiH_4	0.6733
Silicon Tetrachloride	$SiCl_4$	0.3168
Silicon Tetrafluoride	SiF_4	0.3960
Sulfur Dioxide	SO_2	0.7228
Sulfur Hexafluoride	SF_6	0.2970
Trichlorosilane	Cl_3HSi	0.3267
Trimethylamine	$(CH_3)_3N$	0.3168
Tungsten Hexafluoride	WF_6	0.2871
Uranium Hexafluoride	UF_6	0.2178
Vinyl Bromide	C_2H_3Br	0.5248
Vinyl Chloride	C_2H_3Cl	0.5347
Vinyl Fluoride	C_2H_3F	0.5743
Xenon	Xe	1.3762

J MAINTAIN HIGH-PURITY GASES

The analysis system was designed to accurately measure the surface area of all types of materials. It is important that the gases (especially krypton) used for these measurements be of highest purity, especially when analyzing low surface area samples. Three ways to ensure high-purity gases are to always maintain:

- Thoroughly purged gas pressure regulators.
- Non-permeable gas lines.
- Leak-free connections.

Impure gas is strongly indicated, for example, if a series of measurements on a low surface area material yields decreasing specific surface areas with decreasing quantities of sample. The analyzer uses very small amounts of helium; therefore any residual air in the regulator can distort results of subsequent analyses for quite some time.

Micromeritics offers the following suggestions to assist in maintaining high-purity gases (particularly helium):

- Use metal gas lines only.
- Remove trapped air from the regulator and gas lines.

USE METAL GAS LINES

Always use metal gas lines which have been carefully cleaned of any oils and greases used in the manufacturing process. Do not use plastic or rubber gas lines. When these types of permeable, nonmetallic gas lines are used with helium, contaminants accumulate at a much faster rate. This causes errors in analysis results and can also contaminate a clean sample.

REMOVE TRAPPED AIR

When connecting the regulator to the gas cylinder, air is unavoidably trapped on the high and low pressure sides of the regulator, as well as in the gas lines. Remove as much of this air as is possible ***before*** opening the gas cylinder valve. If this air is allowed to remain in the regulator, it will mix with the helium and cause inaccurate results in subsequent analyses. Or if the valve is open for any length of time, the air trapped on the high pressure side may diffuse back into the gas cylinder and contaminate its entire contents.

There are two methods for removing trapped air from the regulator lines: the Purge Method and the Evacuation Method.

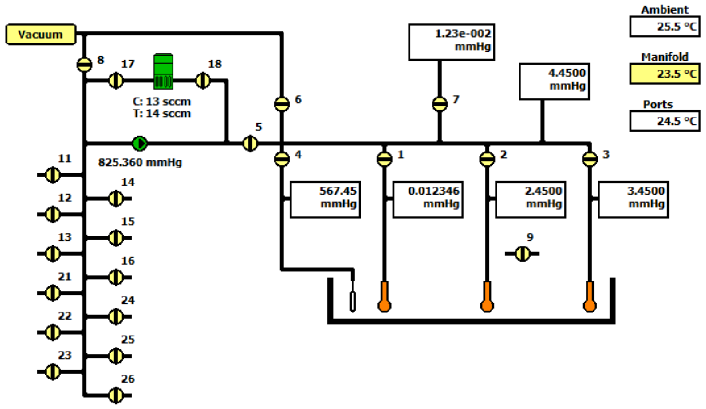
PURGE METHOD

This is the preferred method for removing trapped air.

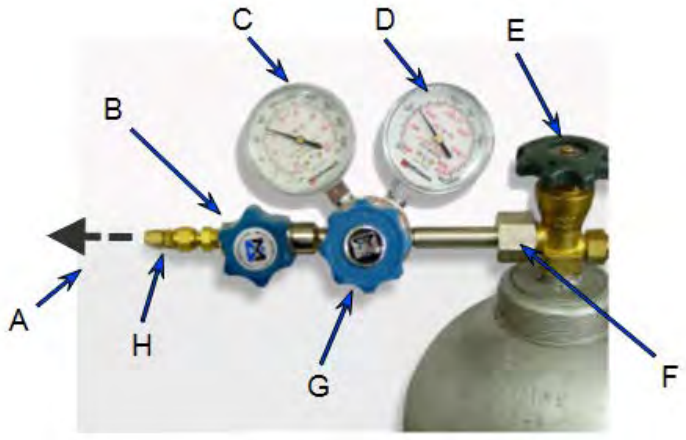
1. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. If the analyzer schematic does not display, go to ***Unit [n] > Show Instrument Schematic.***

If multiple instruments are installed, choose the correct *Unit* menu.

2. Close all valves by right-clicking each valve, then click **Close**.



3. Open the regulator shut-off valve.
4. Open the gas cylinder valve ***briefly*** and allow the regulator to be charged with gas until the high pressure gauge reads just over half the tank pressure, then quickly close the valve.



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

5. Use the Pressure Control knob to set the output pressure (gas cylinder pressure gauge) to 15 psig.

6. Loosen the fitting at the instrument helium inlet until the low pressure side drops to approximately 3 psig (0.02 MPa), then tighten the fitting.
7. Repeat steps 4, 5, and 6 three times.
8. Briefly open the gas cylinder valve, then use the Pressure Control knob to reset the regulator output pressure to 15 psig.
9. After the pressure has stabilized (indicating there are no leaks), open the gas cylinder valve.

EVACUATION METHOD

To use this method, the gas tank must be within 10 feet of the instrument.

1. Do one of the following:

If...	Then...
The regulator has not been filled with gas and the gas line is attached to the instrument:	Close the gas cylinder valve.
	Open the regulator shut-off valve.
The regulator is filled with gas:	Close the gas cylinder valve.
	Open the regulator shut-off valve.
	Loosen the helium inlet fitting (or nut) on the instrument.
	Allow all of the gas in the regulator to expel from the line (pressure reading will be zero).
	Retighten the helium inlet fitting (or nut).

2. Go to ***Unit > Enable manual control*** (if the instrument schematic is not displayed, go to ***Unit > Show instrument schematic***).

If multiple instruments are installed, ensure the correct *Unit* menu is selected.

3. Close all valves, then open valves 6, 7, and 10.
4. Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas cylinder. The manifold pressure transducer should fall close to zero.

Allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

5. Close valves 6, 7, and 10.

K PEAK DETECTION / INTEGRATION OPTIONS

CFR Note

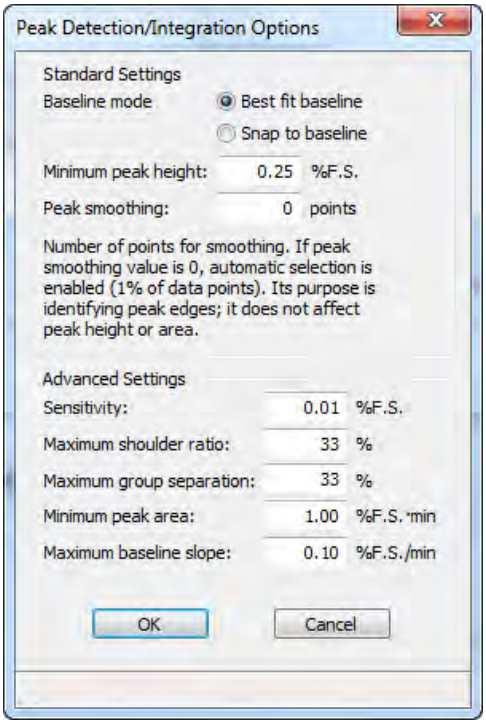
Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

This section applies only to dynamic analysis samples.

File Open > [.RPO file] > [Integration button***]***

Or, select the **Integration** button while in the Peak Editor.

Peak detection parameters can be customized using the **Integration** button on the *Report Options* window or the **Integration** button on the Peak Editor window. Peak detection options can be customized while creating the sample file or after analysis. For Pulse experiments and Loop calibrations, peaks are found using events that occurred during the analysis — such as the time an injection started — instead of peak detection options.

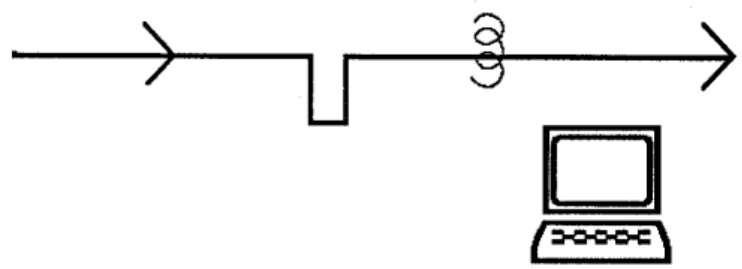


L TEMPERATURE PROGRAMMED ANALYSES

CFR Note	Dynamic Analysis and Chemisorption options are not applicable when the software is installed in 21CFR11 environments.

Most temperature-programmed experiments are based on the following highly simplified steps:

1. Gas flows into the analyzer.
2. The gas interacts with the sample as the temperature changes.
3. Gas flows past the detector.
4. The detector collects data.
5. The application plots and calculates results.



How the Detector Works

The detector contains heated filaments that measure the difference in gas thermal conductivity sensed between the gases flowing over the sample and reference filaments.

The gases flowing past the detector cool the filament by extracting heat. How quickly any type of gas removes heat from the detector is determined by its thermal conductivity[1)] . A gas with a high thermal conductivity cools the filament rapidly, and more power is required to maintain its temperature. A gas with a lower thermal conductivity removes heat from the filament more slowly.

When the sample reacts with the gas, it causes changes in the composition of the gas and, consequently, changes the thermal conductivity of the gas. These changes are sensed by the detector as an increase or decrease in the amount of power required to maintain the filament at a constant temperature.

1) The thermal conductivity of a gas is its ability to conduct heat. Each gas has a distinct thermal conductivity.

Data that are Collected

The detector reports the amount of electricity (in volts) required to keep its temperature constant during the analysis.

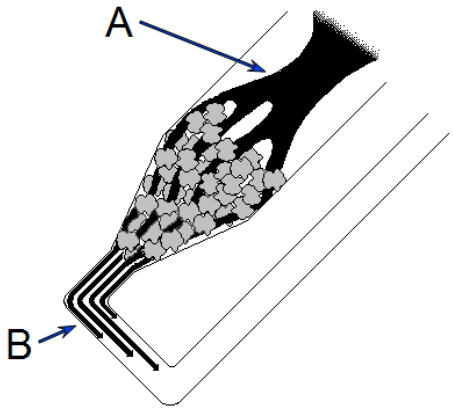
The Role of the Sample Temperature

Because the sample's temperature determines how rapidly it interacts with the analysis gas (or if it reacts at all), data are collected over the range of temperatures specified.

In some experiments, you may prefer to start collecting data at a very low temperature to establish a baseline where the gas is completely unaffected by the sample. In other cases, you may prefer to collect data after a reaction has begun. In still other experiments, your primary interest may be determining the temperature at which the maximum reaction occurs.

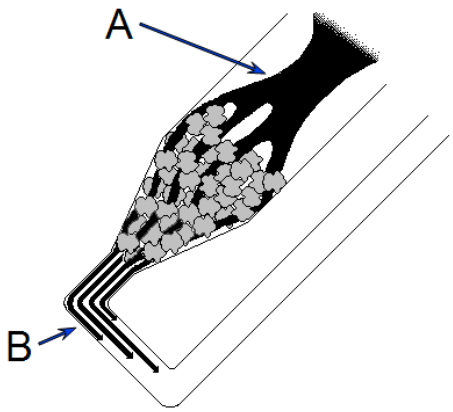
For example, consider the example of a Temperature-Programmed Reduction (TPR). During the TPR, a metal oxide is reacted with hydrogen to form a pure metal. This reaction is referred to as "reducing" the metal; for example, TPR of a catalyst containing Platinum. Argon, which has a very low thermal conductivity, is used as a carrier gas. It is blended in a fixed proportion with hydrogen, an analysis gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector.

When the gas blend begins flowing over the sample, a baseline reading is established by the detector. This baseline is established at a low enough temperature that no reduction of the sample is occurring, so the baseline level recorded by the detector is that of the thermal conductivity of the two gases in their fixed proportion. In other words, the proportion of gases flowing over the detector is the same as the proportion of gases entering the analyzer, because at the low temperature, there is no interaction with the sample.



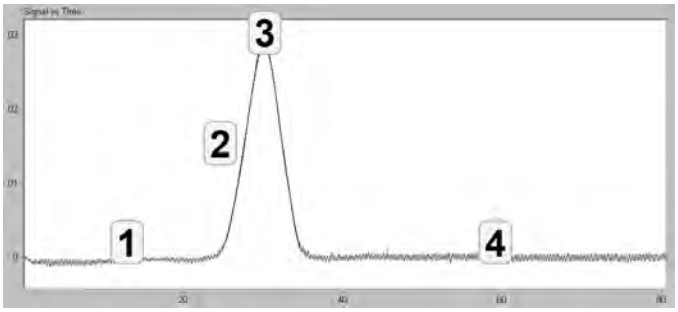
A. Gas flows into the sample tube and through the sample.
B. Before the reaction begins, the concentration of gases exiting the sample tube is unchanged after flowing through the sample.

The temperature is then changed, and when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming H_2O molecules. The H_2O molecules are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon / hydrogen gas blend inside the analyzer decreases, and the proportion between the two gases shifts in the direction of argon, as does the mixture's thermal conductivity.



A. As the temperature changes, the sample begins to react with the gas(es) flowing through it.
B. The concentration of gases exiting the sample tube is altered after passing through the sample. This change in the gas concentration is recorded by the Thermal Conductivity Detector downstream.

Since argon has a lower thermal conductivity than hydrogen, the mixture's thermal conductivity consequently decreases. The flowing gas removes heat from the filament more slowly, requiring less electricity to maintain a constant filament temperature. The analyzer records the electrical demand as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. When these readings are graphed, the data form one or more peaks. Peaks can be positive or negative; negative peaks are show in this example.



Negative Peaks Legend

Item	Description
1	Baseline readings. The gas(es) is (are) not reacting with the sample, so there is no change in the signal from reading to reading.
2	As the temperature changes, the sample begins to react with one of the gases. Therefore the gas mix is then made up of a larger proportion of the other gas. This causes a shift in the mixture's thermal conductivity. The detector measures this change by recording the change in the amount of electricity required to maintain constant filament temperature.
3	As temperature continues to increase, the interaction reaches a maximum, then begins to diminish.
4	As fewer and fewer sample atoms are available to bond with the analysis gas, there is less and less change in the mix of gases flowing into the analyzer and past the detector, so the thermal conductivity shifts back toward the baseline value.

This example illustrates the fundamental concept upon which the analyzer operates. Of course, the various types of analyses the analyzer can perform result in different types of traces. For example, a pulse chemisorption analysis results in a series of peaks that gradually increases in size as the sample is dosed with separate increments of gas. Initially, the gas uptake by the sample results in smaller peaks. But when all the active sites are saturated, no more gas can be taken up and the peaks become equal.

Peak Area

The area beneath each peak is calculated to provide information about the volume of gas reacted during the analysis. The *Calculations* document can be found on the Micromeritics web page (www.Micromeritics.com).

Automatic Operation

The analyzer application provides a simple format to specify all the analysis conditions for the experiment; create a sample file which contains sample information and a list of specific steps the analyzer will follow to perform the experiment(s). Then, the analyzer automatically performs the analysis, from controlling the gas mixture and flow rate to monitoring the temperature and pressure. After analysis, use the Peak Editor to adjust the peaks to create reports that contain the data needed, without baseline noise or other undesirable effects.

Because up to 99 experiments can be specified and each experiment can contain up to 99 steps, the analyzer can perform a wide variety of preparation and analysis functions automatically.

SORPTION TRAP

In some cases, it is preferred to trap substances resulting from the reaction. In the previous example, H_2O is produced during the analysis. If the gas flow is passed through the sorption trap at an appropriate temperature, the water can be removed before the gas flows past the detector.

INJECTION LOOP

Injection loops are provided for injecting carefully measured doses of gases for analyses such as Pulse chemisorption. The analyzer is shipped with a 0.5 cm^3 loop installed. A 1 ml loop is also available. If the sample file is set so that a loop is used for introducing gas into the analyzer, the analyzer will automatically dose the sample as specified in the sample file.

SAMPLE PREPARATION AND CALIBRATION

Depending on the type of experiment(s) to be performed, sample preparation and/or calibration may be required. Specific instructions are contained in the appropriate sections of this manual.

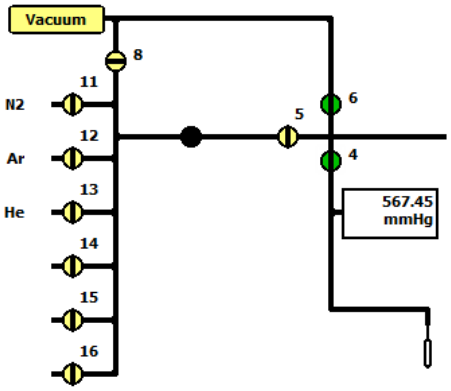
A sample is prepared for analysis by removing unwanted adsorbates from the surface of the sample. This is usually accomplished by flowing gas over the sample and may include heating the sample. The flowing gas may be inert or chemically active gases may be used to activate the surface.

Calibration routines provide the analyzer and application with the appropriate information to convert electrical signals to physically meaningful data such as volume adsorbed, loop volume, and gas concentration.

M VAPOR PURIFICATION

Physical adsorption of gases is generally used to calculate pore size and surface area of solid materials. Using vapor adsorptives allows the sorption capacity and isosteric heat of adsorption, the energy released as molecules adsorb on the sample surface, to be calculated by using isotherms collected at different temperatures with the same vapor and adsorbent. Heats of adsorption data are very useful in research applications. In the case of competitive adsorption, the vapor with the highest heat of adsorption will adsorb first and have the strongest interaction with the surface. However, if the heat of adsorption is too high, the molecule will be so strongly adsorbed that desorption or regeneration of the material may be difficult. In order to properly collect vapor isotherms, a purified vapor must be used. A liquid-filled reservoir is used as the source of the vapor.

This procedure describes a freeze-thaw method to remove dissolved gases and air within the reservoir so the vapor purity is suitable for analysis on the Flex. The general principles of this method could also apply to vapor purification on other gas adsorption instruments. The basic principle is to evacuate non-condensed species while the vapor reservoir is immersed in a cryogenic bath. At the pressures achieved during the purification process, nitrogen and oxygen are not condensed. The use of liquid nitrogen is limited to vapors that will not sublimate at cryogenic temperatures.



Valve 4 Open

A. Vapor shut-off valve

Vapor Source Container

1. Fill a clean vapor reservoir with liquid. Fill halfway (10 ml) if using water. Fill with approximately 20 ml for other liquids.
2. Attach vapor reservoir to the Flex. See ***Vapor Source Container Installation on page 11-16*** in the Flex operator manual.
3. Firmly close the vapor shut-off valve above the liquid-filled reservoir.
4. On the schematic, open valve 4 to evacuate the space above the vapor shut-off valve.
5. Close valve 4 on the instrument schematic, then open the vapor shut-off valve on the reservoir. Allow the pressure to equilibrate in the vapor container.

6. Submerge the vapor reservoir in a cryogen bath. The use of liquid nitrogen as the cryogen should be limited to vapors with no sublimation pressure at cryogenic temperatures. Wait for the pressure in the vapor container to drop as low as possible. This may only be 150 Torr for the first cycle but should be near zero after two or three cycles. A lab jack is useful to hold and adjust the Dewar filled with liquid nitrogen.
7. Once the vapor has condensed and has frozen, open valve 4 on the instrument schematic with the cryogen bath still in place and evacuate the vapor container (the vapor shut-off valve should still be open). Pressures in the range of 10^{-4}-10^{-5} Torr should be achievable.
8. Close valve 4 on the instrument schematic and remove the cryogen bath. Let the vapor container thaw. The vapor shut-off valve should remain open during this step. To expedite the process, a warm water bath may be used temporarily to raise the temperature of the vapor container closer to the ambient temperature. However, a layer of ice formed on the reservoir could also create a barrier for heat transfer, so it is best to not introduce the water bath immediately. If pressure stabilizes near the calculated saturation pressure at room temperature, the vapor is free of impurities.
9. If the pressure does not stabilize around the calculated saturation pressure, repeat steps 3 to 8 until the pressure at the last step stabilizes near the calculated saturation pressure of the vapor. When the vapor is pure, the pressure should stabilize around the same value after each thawing cycle. Typically, three total purification cycles (two repeats) is sufficient.

N WETTED MATERIALS

Contact Micromeritics for assistance before running a gas such as ammonia or pyridine that is incompatible with some of the system components.

Wetted Materials

Material	Location
304 stainless steel , 403 stainless steel , Ceramic (Al2O3), Silicon, SiO2, Si3N4, Gold, Viton, Low out gassing epoxy resin	Vacuum gauge
316 Stainless Steel, Hastelloy C-22, 17-7 PH, 430SS, Nickel, Kalrez (FFKM)	Mass Flow Controller (MFC) (for chemisorption only)
Aluminum	NW/KF ring in vacuum line
Aluminum alloys, stainless steels, fluoroelastomer and nitrile O-rings, hydrocarbon lubricant, felt, rare earth magnets, silicon nitride, phenolic resin, carbon-fiber reinforced epoxy resin, fire retardant polypropylene, polyamide and PVC.	Turbo pump
Borosilicate Glass	Physisorption sample tubes, filler rods
Buna-N	■ Gas inlet manifold, valve plungers, and O-rings ■ NW/KF ring in vacuum line
Buna-N, stainless steel, Platinum	TranSeal (optional)
Copper	Gas inlet lines
FFKM (Kalrez)	■ Optional sample port O-rings ■ Servo valve
Gold-plated copper	Turbo vacuum pump gasket and vacuum gauge
Gold-plated nickel / iron	TCD filaments
Inconel	Transducers
Kel-F (PCTFE)	■ Valve seats analysis manifold ■ Valve seat exhaust valve (chemisorption only)

Wetted Materials (continued)

Material	Location
PCTFE (Kel-f), Buna-N, FKM (Viton), Stainless steel, Borosilicate Glass	Check seal (optional)
PTFE, FPM (fluroelastomer), Aluminum	Diaphragm roughing pump
Quartz	Chemisorption sample tubes, filler rods, wool filter discs
Stainless steel	■ Manifolds, valve bodies, plumbing, VCR gaskets, port transducers, filler rods. ■ Gas inlet lines (optional) ■ Ferrules in exhaust line (chemisorption only) ■ TCD block
Teflon (PTFE)	Ferrules in gas inlet manifold, P^0 tube
Viton type A (FKM)	Sample port O-rings

O WORKSHEETS

Worksheets in this section may be copied as needed.

Sample Data Worksheet for Chemisorption on page O - 3
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:

SAMPLE DATA WORKSHEET FOR CHEMISORPTION

Sample tube identification: _____

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

Notes:

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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:

FLEX™ SERIES Surface and Catalyst Characterization Analyzer
Models: 3Flex (3500)

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 61010-2-010:2014 - *Particular requirements for laboratory equipment for the heating of materials*
EN 61010-2-081:2015 - *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 61000-3-3:2013 - *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:

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