AutoChem® III

Automated Catalyst Characterization System

micromeritics®

Operator Manual

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(Rev C)

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

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1. Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their respective brand names or trade names. We, therefore, make no express or implied warranty as to such products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree to in writing.
2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.
3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

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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.

PATENTS

For patent information, visit www.Micromeritics.com/patents.

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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

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

NOTE — Notes contain important information applicable to the topic.

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

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

GENERAL SAFETY

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

If closed-cell silicone foam pipe insulation is used in this instrument, it is REACH non-compliant.

Chemical Abstract Service and numbers:

- Octamethylcyclotetrasiloxane (D4) – CAS 556-67-2
- Decamethylcyclopentasiloxane (D5) – CAS 541-02-6
- Dodecamethylcyclohexasiloxane (D6) – CAS 540-97-6

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 AutoChem III weighs approximately 87 kg (130 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 AutoChem III sound level is 75 dBA. Hearing protection is optional.
- 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 SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are the responsibility of the operator.

INTENDED USE

The AutoChem III is a highly automated catalyst characterization system that can perform the following experiments individually or in various sequences:

- Temperature-Programmed Desorption Analysis (TPD)
- Temperature-Programmed Reduction Analysis (TPR)
- Temperature-Programmed Oxidation Analysis (TPO)
- Temperature-Programmed Surface Reaction (TPSR)
- Pulse Chemisorption
- BET Surface Area Analysis

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	o	o	o	o	o	o
Power Supplies	o	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 THE 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 kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

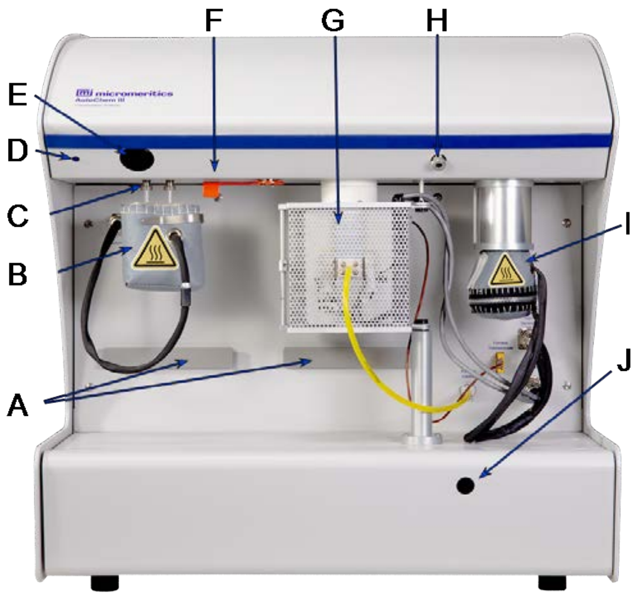
When working on an instrument where this symbol is displayed, refer to the corresponding Operator Manual for additional information.

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 Safety Data Sheet (SDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident.

Table of Contents

1 ANALYZER COMPONENTS



A. Bracket to hang Dewar shelf (2)
B. Heating Mantle
C. Ports for sorption trap, optional cold trap, and delay path
D. LED light
E. Mass Spectrometer port
F. Sample tube release lever
G. Furnace
H. Septum port
I. Vapor generator (optional)
J. Front cover release

Analyzer Components

Component	Description
Behind Front Cover (not shown)	Front cover is removed to expose the following: ■ Ethernet Port ■ Gas Inlet Ports (Carrier, Loop, and Preparation) ■ Exhaust Ports ■ Mass Spec connection ■ Air Source selector (Compressor or Tank) ■ Air In Valve ■ COM-1 Port ■ Air Filter
Furnace	Controls the temperature of the sample.
Heating mantle	For use with the sorption trap.
LED light	When lit, indicates the communication between the analyzer and the computer.

Analyzer Components (continued)

Component	**Description**
Mass spectrometer port	To attach an optional mass spectrometer.
Ports	For optional accessories (cold trap, sorption trap, delay path).
Sample tube release lever	Use to release the sample tube from the sample tube port above the furnace.
Septum port	To inject gas into an analysis.
Shelf bracket (not shown)	Bracket to support Dewar shelf
Vapor generator	Allows analysis using vapors from liquids carried by an inert gas.

EQUIPMENT OPTIONS AND UPGRADES

Parts and accessories are located on the Micromeritics web page.

Option	Description
B.E.T.	Includes delay line used to reduce the perturbation (disturbance) of the gas flow caused by injections of gas through the septum, a 10 mL syringe, additional needles for the syringe (pack of 3) and a plastic beaker for water at ambient temperature for B.E.T. analysis. (Requires purchase of the optional Dewar.)
CryoCooler	Analyses can be started at sub-ambient temperatures. The CryoCooler can be used to speed throughput by ramping the furnace temperature rapidly to ambient after an analysis. **CryoCooler II** - For use with all AutoChem instruments. **CryoCooler III** - For use on the AutoChem III only.
Mass Spectrometer	Separates isotopes, molecules, and molecular fragments according to mass. Connects directly to a dedicated port for detection and identification of low concentrations of condensable and/or reactive gases.
Sorption Trap	The Sorption Trap is used to trap water produced from Temperature Programmed Reduction (TPR) analyses or other temperature programmed experiments in which moisture needs to be removed from the flow path before reaching the detector.
Vapor Generator	Allows an inert carrier gas, such as helium, to be bubbled through the liquid at up to 100 °C. The vapor then passes through a cooler condensation zone maintained at an accurate temperature to ensure a stable vapor pressure. The vapor then passes to the loop valve zone (Fill/Inject valve) for precise injections onto the sample.

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 SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are 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.

Gas	Purity
Ar CO/He He He/Ar	99.999%

SPECIFICATIONS FOR THE AUTOCHEM III

Electrical

Voltage	100/120/230 VAC
Frequency	50-60 Hz
Power	1250 VA
Overvoltage category	II

Temperature System

Range	-100 °C to 1200 °C with CryoCooler option Ambient -5 °C to 1200 °C without CryoCooler option
Selection	Digitally set, 0.1 °C increments
Ramp Rates	1 °C/minute minimum, 100 °C/minute maximum Maintains scheduled rate within 5 °C or 1 minute, whichever is larger Heating rates: ≤ 25 °C/min at temperatures ≤ 1200 °C ≤ 50 °C/min at temperatures ≤ 1000 °C ≤ 100 °C/min at temperatures ≤ 800 °C Cooling rates: ≤ 2 °C/min at temperatures ≥ ambient + 25 °C ≤ 5 °C/min at temperatures ≥ 75 °C ≤10 °C/min at temperatures ≥ 110 °C ≤ 25 °C/min at temperatures ≥ 200 °C ≤ 50 °C/min at temperatures ≥ 300 °C ≤ 100 °C/min at temperatures ≥ 500 °C
Temperature Ramp Rate	± 1% of target

Wetted Materials

Standard System	Stainless steel, gold plated nickel/iron (filaments), Kalrez (most O-ring seals), quartz (sample tubes, thermocouple sheath), quartz wool (supports sample in tube), Inconel- sheathed sample thermocouple (can be placed outside tube or sheathed in quartz), PEEK (Poly Ether Ether Ketone - some ferrules), Teflon tubing (most exhaust lines; TCD exhaust is stainless steel for connection of external devices), copper (gas supply lines and BET delay path), nickel plated brass and steel (gas inlet valves), Polyaryletherketone/PTFE composite (rotary valves seals), Silicone/ Teflon sandwich (septum). Buna-N (gas inlet valve seals and O-rings).
System with Vapor Generator Option	As above, plus borosilicate glass vapor generator flask, Kalrez flask gasket.
System with Enhanced Chemical Resistant Option	As above, but stainless steel manifolds are protected from corrosion with vapor deposited Silcolloy by SilcoTek, stainless steel tubing, fittings, ferrules and rotary valves are replaced by Hastelloy-C versions, Teflon MFC seals, Kalrez is used for all O-ring seals and the seals inside the mass flow controllers, stainless steel grade 316 gas supply lines, stainless steel gas inlet valves with Kalrez seals.

Gases

Loop	H_2, CO, O_2, N2O, NH_2 vapors such as pyridine, water, etc.
Carrier	He, Ar, and other gases
Preparation	H_2, O_2, He, Ar, and others

Gas Flow Rate

All Mass Flow Controllers (MFCs)	
Manual Control	0 to 100 mL/minute[1]
Automatic Analysis	1 to 100 mL/minute

Gas Delivery

Inlet Ports	18 (6 each for preparation gas, carrier gas and loop gas)
Temperature Control	Internal gas lines and valves heated up to 150 °C

Sample Tube

Type	Fused quartz Flow-Thru sample tubes, for use up to 1200 °C, accepts powders and pellets up to 9 mm in diameter

Physical

Height	64 cm (25 in.)
Width	61 cm (24 in.)
Depth	64 cm (25 in.)
Weight	87 kg (192 lb.)

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[1] Rate for Nitrogen; other gases have a different range.

Environment

Temperature	15 °C to 35 °C (59 °F to 95 °F), operating 0 °C to 50 °C (32 °F to 122 °F), non-operating Maximum rate of change of 2 °C per hour
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
Location	Instrument should be located in a dust-free, vibration free environment, away from exposure to direct sunlight and direct air drafts.
Degree of Ingress Protection	IPX0

Computer Requirements

Operating System	Windows 10 or higher operating system 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. CAUTION 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.
Read/Write Permissions	All application users will need Read/Write permission to all directories and subdirectories where the application is installed.
Drives	USB port

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

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2 ABOUT THE SOFTWARE

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.

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.

BET Surface Area Analysis Tutorial on page 11 - 1
Pulse Chemisorption Analysis Tutorial on page 11 - 16
Temperature Programmed Desorption Analysis Tutorial on page 11 - 25
Temperature Programmed Oxidation Analysis tutorial on page 11 - 34
Temperature Programmed Reduction Analysis tutorial on page 11 - 43

The BET surface area analysis evaluates the total surface area of the catalyst before and after chemisorption. Pore-plugging phenomena, which might occur due to the irreversible adsorbed species during chemical reactions, and the occurrence of sintering1) , can be studied.

After outgassing the sample, a mixture of nitrogen and helium (typically 5 to 30% N_2) flows over the sample that is immersed in a liquid nitrogen (LN_2) bath. Both the adsorption and desorption of the N_2 are recorded. The amount of nitrogen desorbed at LN_2 temperatures and the sample weight is used to calculate the total specific surface area.

The entire BET analysis — or even repeat analyses — is performed in situ.

- **Langmuir Surface Area** Analysis. The Langmuir surface area analysis allows for evaluating the total surface area of the catalyst and is especially useful for adsorbate/adsorbent systems that adsorb only a monolayer. These materials typically exhibit a Type 1 isotherm and are often microporous. The Langmuir surface area analysis may be best applied to zeolites and microporous carbons. Typically the Langmuir surface area will exceed the BET surface area for these materials and provide a more accurate estimation of the total surface area. The Langmuir surface area report may be applied to a wide range of gas concentrations and is not limited to the typical BET range (5 to 30% N_2).

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1) Sintering is the fusing of small particles (or small features of a sample). Sintering tends to reduce the active surface area.

- **Total Pore Volume** Analysis. The total pore volume is a single-point estimate of the pore capacity of a material. The total pore volume analysis is usually conducted near the saturation pressure of the adsorbate (0.995 P/P_0). The total pore volume of a material can be determined on both fresh and used materials. The difference in pore volume may indicate pore plugging and directly relate to changes in the performance of catalysts and adsorbents.

  For a high surface area sample (> 100 m^2/g), a quantity less than 50 mg is recommended. The high sensitivity combined with a large amount of adsorbed gas allows smaller sample quantities to be used while maintaining high precision. The reduced sample quantity will also reduce the likelihood of saturating the high sensitivity detector.

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 on page 6 - 2

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.

MENU STRUCTURE

All program functions use standard Windows menu functionality.

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.
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 spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
File name	Selects a file name from the list shown or enter a file name. If the required file type is not shown, select the type of file from the list.
From / To	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, DESCRIPTION, AND LOCATION

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.
No Analysis	Sample files that have not been used to perform an analysis.

File Type and File Name Extension

File Type	File Name Extension
Analysis Conditions	.ANC
Methods	.MTH
Report Options	.RPO
Sample Information	.SMP
SPC Report	.SPC

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

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 + 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 + 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.
F6	Cascades open windows.
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.
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

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.
Always Open Edit View	Opens files with a *Complete* status in the tabbed file editor rather than in the Peak Editor view.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.

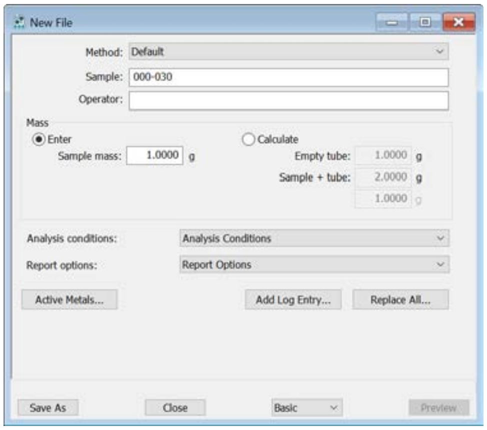
To change the view for the selected window, use the drop-down list at the bottom of the sample file editor.

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

Method: Default
Sample: 000-007
Operator:
Submitter:
Bar Code:
Mass
● Enter ○ Calculate
Sample mass: 1.0000 g
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Comments:

Advanced view



New File
Method: Default
Sample: 000-030
Operator:
Mass
● Enter ○ Calculate
Sample mass: 1.0000 g
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Analysis conditions: Analysis Conditions
Report options: Report Options

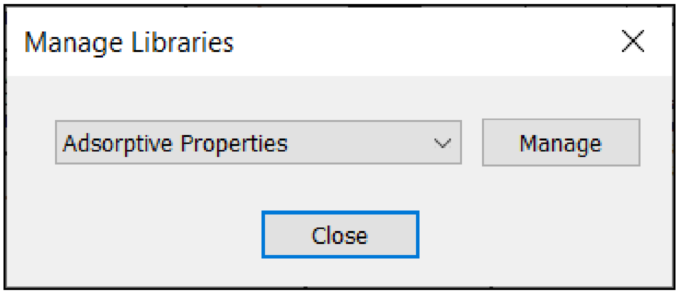
Basic or Restricted view

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.

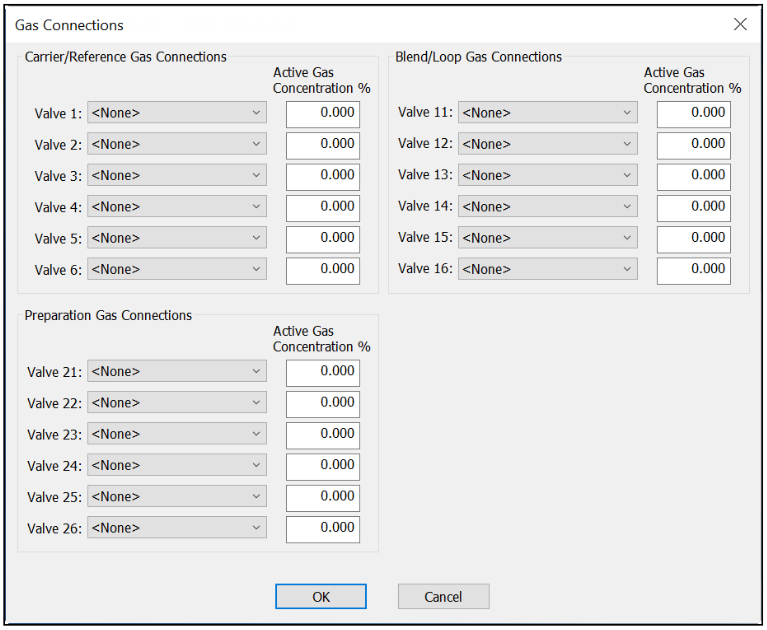
When *removing* a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when *deleting* files or folders from within a library, they are deleted from their original file storage location. Deleted files and folders can be recovered from the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

CONFIGURE THE ANALYZER

GAS CONNECTIONS

Unit [n] > Gas Connections

[Adsorptive Properties on page 4 - 2](#)



Gas Connections

Carrier/Reference Gas Connections

		Active Gas Concentration %
Valve 1:	<None>	0.000
Valve 2:	<None>	0.000
Valve 3:	<None>	0.000
Valve 4:	<None>	0.000
Valve 5:	<None>	0.000
Valve 6:	<None>	0.000

Blend/Loop Gas Connections

		Active Gas Concentration %
Valve 11:	<None>	0.000
Valve 12:	<None>	0.000
Valve 13:	<None>	0.000
Valve 14:	<None>	0.000
Valve 15:	<None>	0.000
Valve 16:	<None>	0.000

Preparation Gas Connections

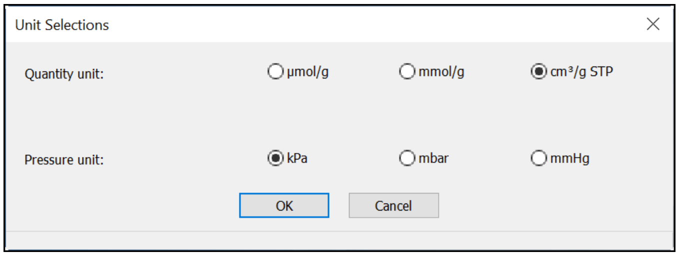
		Active Gas Concentration %
Valve 21:	<None>	0.000
Valve 22:	<None>	0.000
Valve 23:	<None>	0.000
Valve 24:	<None>	0.000
Valve 25:	<None>	0.000
Valve 26:	<None>	0.000

Use to configure the gas type connected to each analysis port. The gases available in the drop-down lists come from the Adsorptive Properties (.ADP) files. Click **OK** when done.

UNIT SELECTION

Options > Units

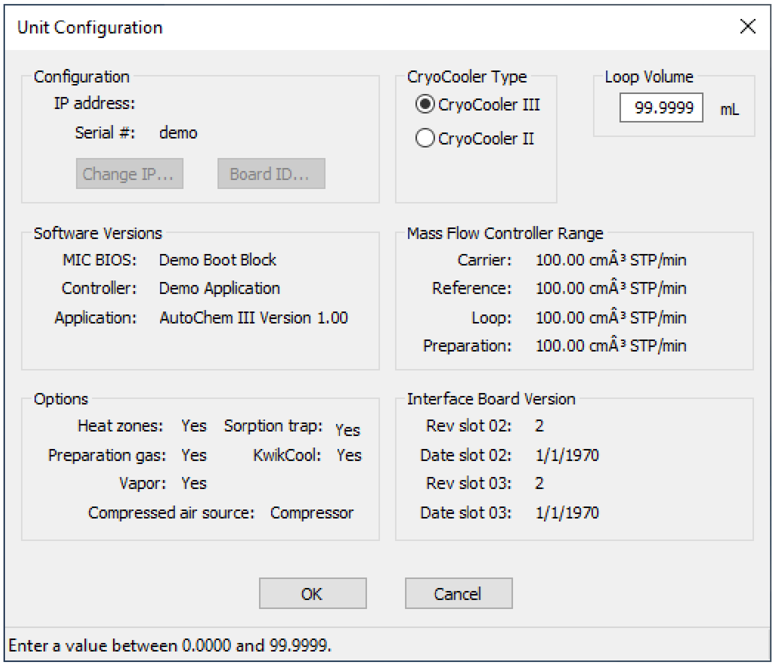
Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation in a standard installation environment.



UNIT CONFIGURATION

Unit [n] > Unit Configuration

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



Configuration
IP address:
Serial #: demo

CryoCooler Type
● CryoCooler III
○ CryoCooler II

Loop Volume
99.9999 mL

Software Versions
MIC BIOS: Demo Boot Block
Controller: Demo Application
Application: AutoChem III Version 1.00

Mass Flow Controller Range
Carrier: 100.00 cmÂ³ STP/min
Reference: 100.00 cmÂ³ STP/min
Loop: 100.00 cmÂ³ STP/min
Preparation: 100.00 cmÂ³ STP/min

Options
Heat zones: Yes Sorption trap: Yes
Preparation gas: Yes KwikCool: Yes
Vapor: Yes
Compressed air source: Compressor

Interface Board Version
Rev slot 02: 2
Date slot 02: 1/1/1970
Rev slot 03: 2
Date slot 03: 1/1/1970

Enter a value between 0.0000 and 99.9999.

Unit Configuration

Fields	Description
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. **Serial #**. Displays the analyzer serial number.

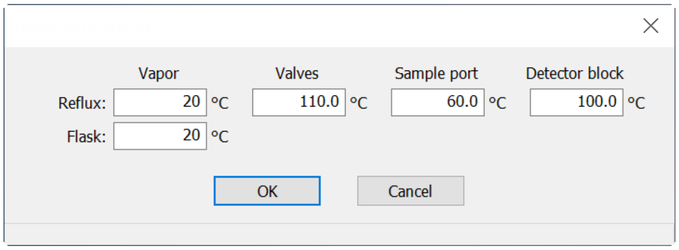
Unit Configuration (continued)

Fields	Description
CryoCooler [*group box*]	Select the CryoCooler type attached to the AutoChem. If no CryoCooler is attached to the instrument, this selection does nothing. **CryoCooler II** - For use with all AutoChem instruments. **CryoCooler III** - For use on the AutoChem III only.
Loop volume [*text box*]	Enter the volume of the gas injection loop. Three standard volume loops are provided with the analyzer. The loop volume is determined by performing a loop calibration. See ***Loop Calibration for TCD Analyzers Tutorial on page 11 - 13***.
Mass Flow Controller Range [*group box*]	Displays the range of each mass flow controller.
Options [*group box*]	Displays options installed on the analyzer.
Software Versions [*group box*]	Displays the software versions of the MIC BIOS, controller software, and analysis program.

SET THE INITIAL TEMPERATURE

Unit [n] > Initial Temperature

Use to set the temperature settings for heat zones when the instrument initializes.



	Vapor	Valves	Sample port	Detector block
Reflux:	20 °C	110.0 °C	60.0 °C	100.0 °C
Flask:	20 °C			

SIGNAL CALIBRATION FOR DYNAMIC ANALYSIS

Options > Signal Calibration

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.

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.

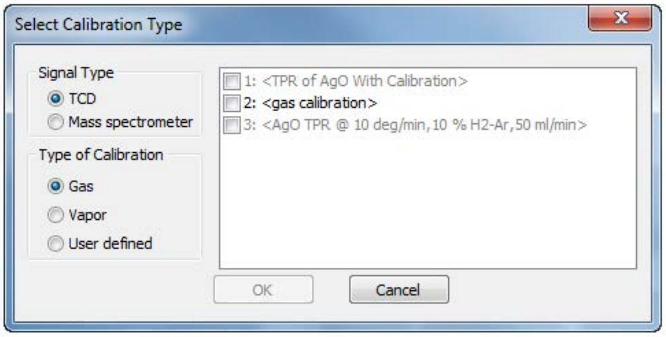
After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example, TPR yields peak area and the temperature 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 ***Options > 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.

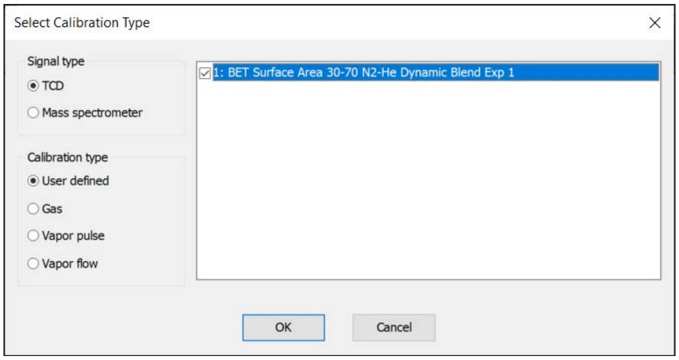
CREATE A NEW SIGNAL CALIBRATION FILE

Options > Signal Calibration > New



Signal Type: ● TCD
Type of Calibration: ● Gas

☐ 1: <TPR of AgO With Calibration>
☐ 2: <gas calibration>
☐ 3: <AgO TPR @ 10 deg/min,10 % H2-Ar,50 ml/min>

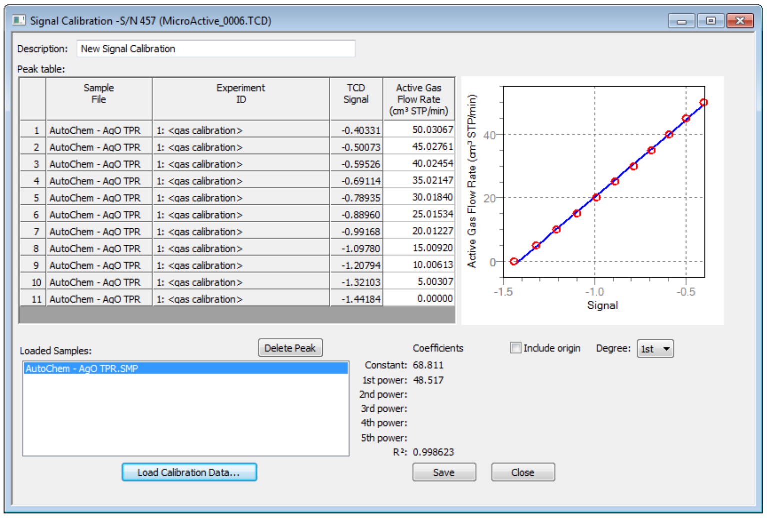


Signal type: ● TCD
Calibration type: ● User defined

☑ 1: BET Surface Area 30-70 N2-He Dynamic Blend Exp 1

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 -S/N 457 (MicroActive_0006.TCD)

Description: New Signal Calibration

Peak table:

	Sample File	Experiment ID	TCD Signal	Active Gas Flow Rate (cm³ STP/min)
1	AutoChem - AgO TPR	1: <gas calibration>	-0.40331	50.03067
2	AutoChem - AgO TPR	1: <gas calibration>	-0.50073	45.02761
3	AutoChem - AgO TPR	1: <gas calibration>	-0.59526	40.02454
4	AutoChem - AgO TPR	1: <gas calibration>	-0.69114	35.02147
5	AutoChem - AgO TPR	1: <gas calibration>	-0.78935	30.01840
6	AutoChem - AgO TPR	1: <gas calibration>	-0.88960	25.01534
7	AutoChem - AgO TPR	1: <gas calibration>	-0.99168	20.01227
8	AutoChem - AgO TPR	1: <gas calibration>	-1.09780	15.00920
9	AutoChem - AgO TPR	1: <gas calibration>	-1.20794	10.00613
10	AutoChem - AgO TPR	1: <gas calibration>	-1.32103	5.00307
11	AutoChem - AgO TPR	1: <gas calibration>	-1.44184	0.00000

Loaded Samples: AutoChem - AgO TPR.SMP

Coefficients

Constant: 68.811

1st power: 48.517

2nd power:

3rd power:

4th power:

5th power:

R²: 0.998623

☐ Include origin

Degree: 1st

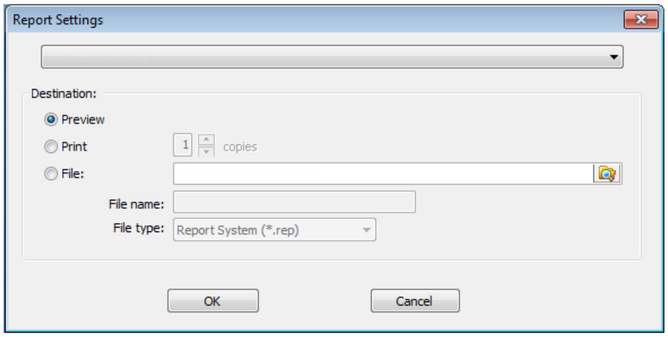
Signal Calibration

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 - 6*.

CREATE A SIGNAL CALIBRATION REPORT

Options > Signal Calibration > Report



Destination:

● Preview

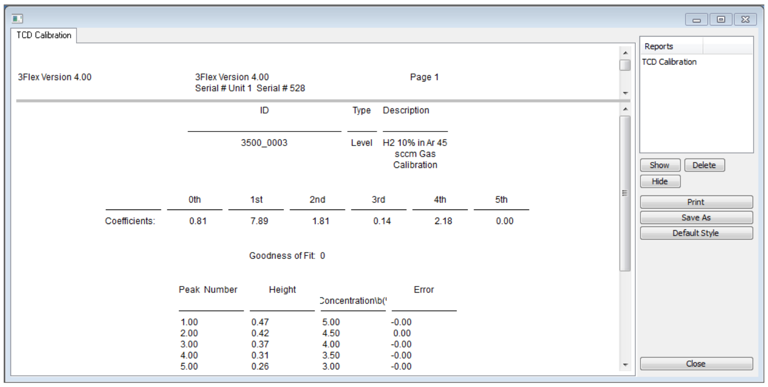
○ Print 1 copies

○ File:

File name:

File type: Report System (*.rep)

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



3Flex Version 4.00

3Flex Version 4.00
Serial # Unit 1 Serial # 528

Page 1

ID	Type	Description
3500_0003	Level	H2 10% in Ar 45 sccm Gas Calibration

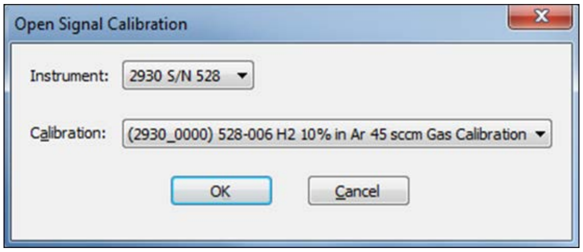
	0th	1st	2nd	3rd	4th	5th
Coefficients:	0.81	7.89	1.81	0.14	2.18	0.00

Goodness of Fit: 0

Peak Number	Height	Concentration\b('	Error
1.00	0.47	5.00	-0.00
2.00	0.42	4.50	0.00
3.00	0.37	4.00	-0.00
4.00	0.31	3.50	-0.00
5.00	0.26	3.00	-0.00

OPEN A SIGNAL CALIBRATION FILE

Options > Signal Calibration > Open



Instrument: 2930 S/N 528

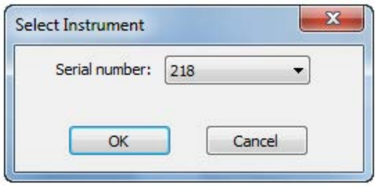
Calibration: (2930_0000) 528-006 H2 10% in Ar 45 sccm Gas Calibration

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.

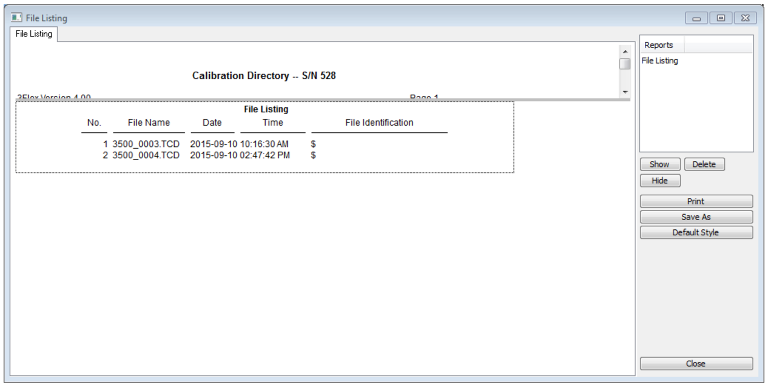
LIST SIGNAL CALIBRATION FILES

Options > Signal Calibration > List



Serial number: 218

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



Calibration Directory -- S/N 528

File Listing

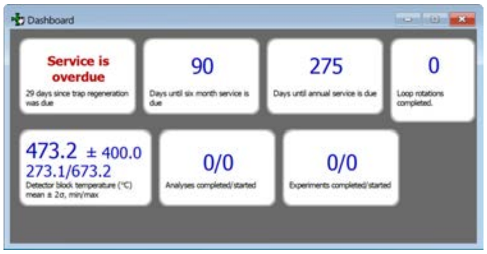
No.	File Name	Date	Time	File Identification
1	3500_0003.TCD	2015-09-10	10:16:30 AM	$
2	3500_0004.TCD	2015-09-10	02:47:42 PM	$

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.



Service is overdue
29 days since trap regeneration was due

90
Days until six month service is due

275
Days until annual service is due

0
Loop rotations completed.

473.2 ± 400.0
273.1/673.2
Detector block temperature (°C) mean ± 2σ, min/max

0/0
Analyses completed/started

0/0
Experiments completed/started

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**
Days since trap regeneration was due	Regular cleaning of the trap is recommended. The number of days until the anniversary of the last service is shown. The displayed value is updated at least once per day and when the service time is reset. When the displayed value is 30 or less, the value text is shown in red. Red negative numbers display if service is past due.
Days until six month service is due	Analyzer service is recommended every six months. The number of days until the anniversary of the last service is shown. The displayed value is updated at least once per day and when the service time is reset. When the displayed value is 30 or less, the value text is shown in red. Red negative numbers display if service is past due.
Days until annual service is due	Annual analyzer service is recommended. The number of days until the anniversary of the last service is shown. The displayed value is updated at least once per day and when the service time is reset. When the displayed value is 30 or less, the value text is shown in red. Red negative numbers display if service is past due.

Dashboard Gauges (continued)

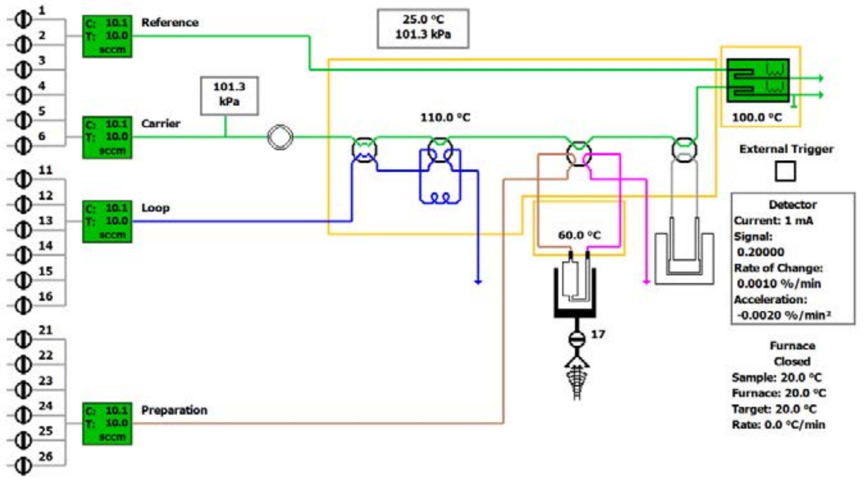
Selections	Description
Loop rotations completed	Displays the number of completed loop rotations.
Detector block temperature	Displays the statistics of the detector block temperature reading. The mean, the value at two standard deviations, the minimum and the maximum display.
Analyses completed / started	Displays *N/M* where *N* is the number of analyses that have finished data collection and *M* is the number of analyses started. Analyses canceled or terminated by errors before the termination stage starts are not counted as completed.
Experiments completed / started	Displays *N/M* where *N* is the number of experiments that have finished data collection and *M* is the number of experiments that have been started. Analyses canceled or terminated by errors before the termination stage starts are not counted as completed.

SHOW INSTRUMENT SCHEMATIC

Unit [n] > Enable Manual Control

Unit [n] > Show Instrument Schematic

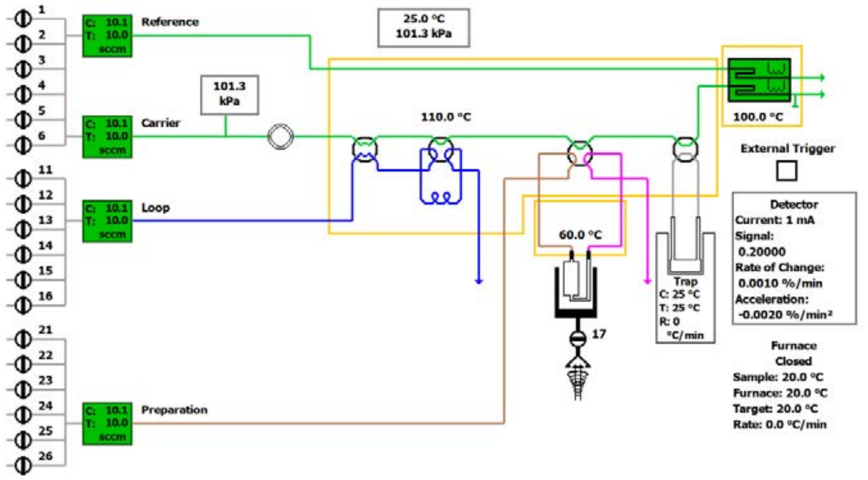
Enable Manual Control on page 10-4
Gas Pathways on page 2 - 35



Detector
Current: 1 mA
Signal:
0.20000
Rate of Change:
0.0010 %/min
Acceleration:
-0.0020 %/min²

Furnace
Closed
Sample: 20.0 °C
Furnace: 20.0 °C
Target: 20.0 °C
Rate: 0.0 °C/min

Standard Cooler

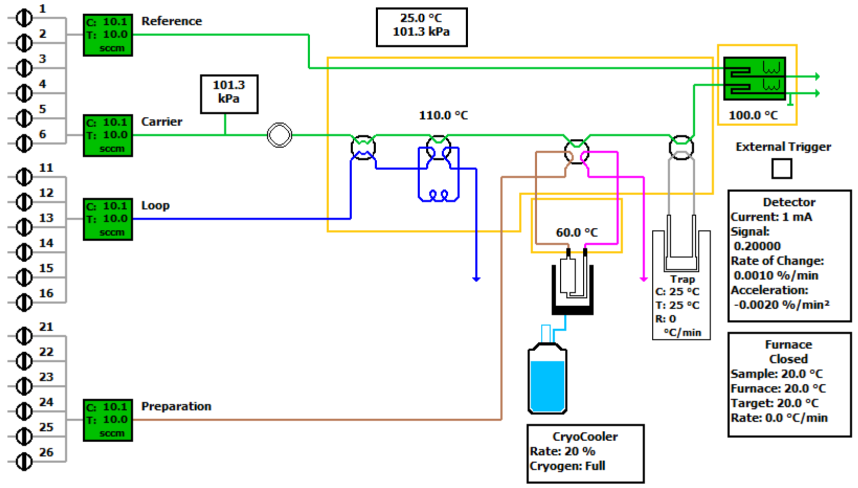


Trap
C: 25 °C
T: 25 °C
R: 0 °C/min

Detector
Current: 1 mA
Signal:
0.20000
Rate of Change:
0.0010 %/min
Acceleration:
-0.0020 %/min²

Furnace
Closed
Sample: 20.0 °C
Furnace: 20.0 °C
Target: 20.0 °C
Rate: 0.0 °C/min

Trap Heater



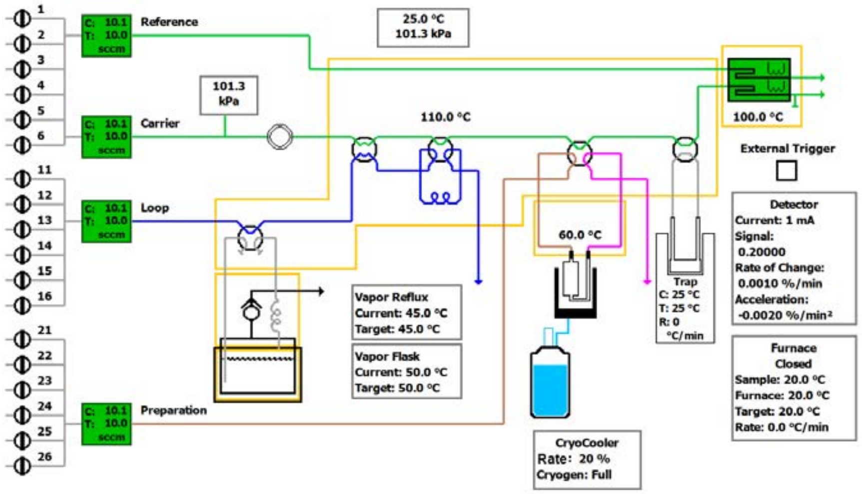
Trap
C: 25 °C
T: 25 °C
R: 0 °C/min

Detector
Current: 1 mA
Signal: 0.20000
Rate of Change: 0.0010 %/min
Acceleration: -0.0020 %/min²

Furnace
Closed
Sample: 20.0 °C
Furnace: 20.0 °C
Target: 20.0 °C
Rate: 0.0 °C/min

CryoCooler
Rate: 20 %
Cryogen: Full

CryoCooler



Vapor Reflux
Current: 45.0 °C
Target: 45.0 °C

Vapor Flask
Current: 50.0 °C
Target: 50.0 °C

Trap
C: 25 °C
T: 25 °C
R: 0 °C/min

Detector
Current: 1 mA
Signal: 0.20000
Rate of Change: 0.0010 %/min
Acceleration: -0.0020 %/min²

Furnace
Closed
Sample: 20.0 °C
Furnace: 20.0 °C
Target: 20.0 °C
Rate: 0.0 °C/min

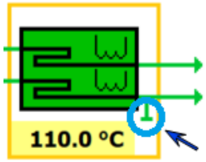
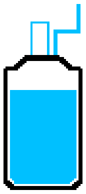
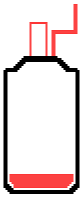
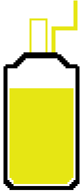
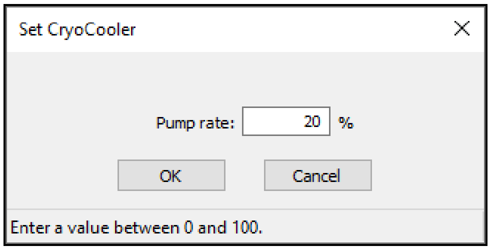
CryoCooler
Rate: 20 %
Cryogen: Full

Vapor Generator

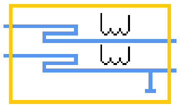
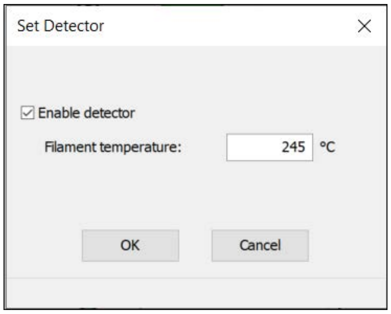
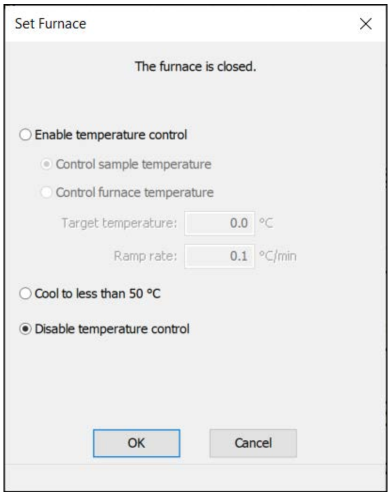
Analysis Valve Descriptions

Valve	Description
1 through 6	Reference and carrier gas inlet ports
11 through 16	Loop gas inlet ports
17 through 26	Preparation gas inlet ports

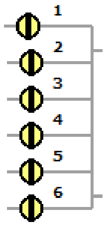
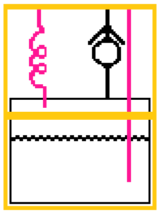
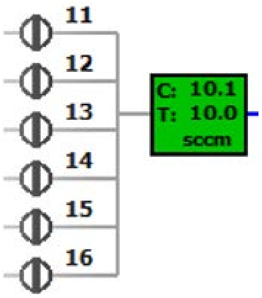
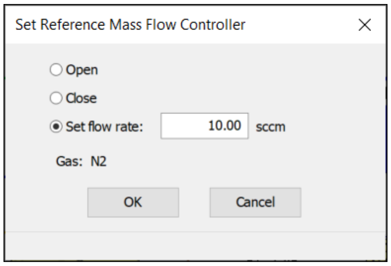
Analyzer Schematic Icons

Icon or Symbol	Description
	**AutoCool**. Displays only if a CyroCooler is not installed.
	**Auxiliary Port**. Connect an external detector (such as a mass spectrometer) to the analyzer. It cannot be manually controlled.
	**Closed Valve**. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
  	**CryoCooler**. Displays only if installed. The CryoCooler rate can be set from 0 to 100%.  Set CryoCooler Pump rate: 20 % Enter a value between 0 and 100. **Blue.** Indicates the Dewar is full **Red.** Indicates the Dewar is empty. When the rate speed is set to 100%, the Dewar displays as red and empty, a warning light illuminates, and the pump will beep continuously. **Yellow.** Indicates Dewar level is unknown and rate is set to zero (for CryoCooler III installations only).

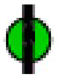
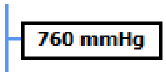
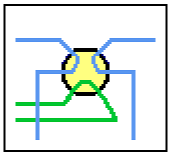
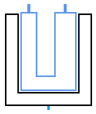
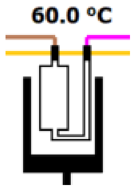
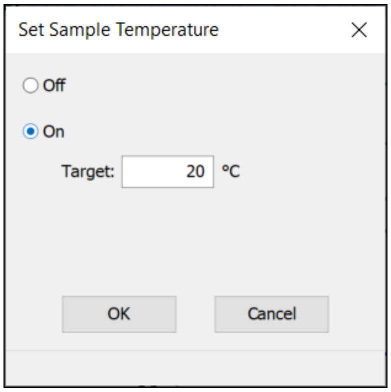
Analyzer Schematic Icons (continued)

Icon or Symbol	Description
	**Detector Heat Zone**. Set the detector heat zone by clicking anywhere in the orange box. Set the Filament Temperature by clicking the detector rectangle on the right side of the schematic.  ☑ Enable detector Filament temperature: 245 °C
External Trigger 🟨	**External Trigger**. Controls the contact closure used to trigger an external mass spectrometer.
Furnace **Closed** **Sample: 20.0 °C** **Furnace: 20.0 °C** **Target: 20.0 °C** **Rate: 0.0 °C/min**	**Furnace**. Controls the sample furnace temperatures and cooling level.  The furnace is closed. ○ Enable temperature control ● Control sample temperature ○ Control furnace temperature Target temperature: 0.0 °C Ramp rate: 0.1 °C/min ○ Cool to less than 50 °C ● Disable temperature control CAUTION Sample or Furnace temperatures significantly above 1100 °C will cause accelerated wear on furnace components and lead to premature failure of the furnace. Temperatures should be set at or below 1100 °C, or operator should minimize time operating above 1100 °C.

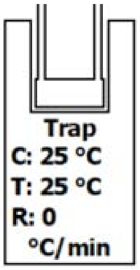
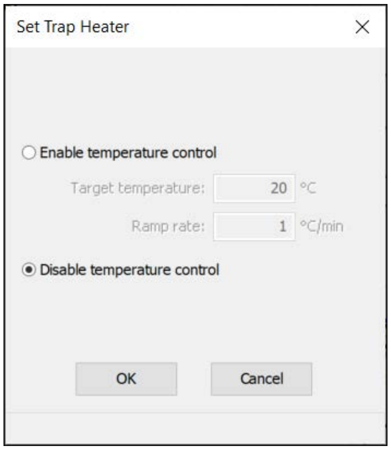
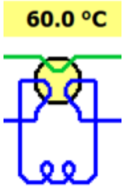
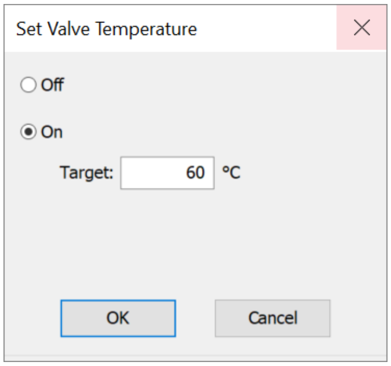
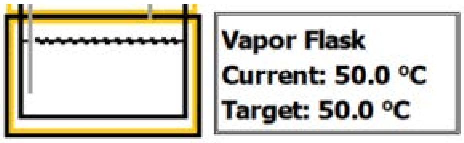
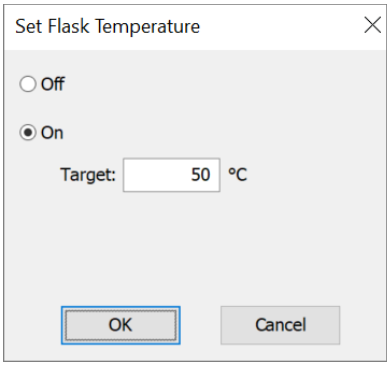
Analyzer Schematic Icons (continued)

Icon or Symbol	Description
	**Gas Inlet Valves**. The inlet displays the gas mnemonic if one is assigned. Right-click the inlet valve to open or close the valve.
	**Heat Zones**. Controls the temperature of various parts of the analyzer. Component heat zones are enclosed with an orange rectangle. Click a heat zone icon to display the current temperature and the target temperature. Right-click the icon, then select *Set Temperature* to change the heat zone's target temperature. Using the default temperature (110 °C) for all heat zones is recommended to ensure a cleaner system. The trap heat zone controls the temperature of the trap valve.
	**Mass Flow Controller**. Controls the flow of gas into the analyzer. The MFC target and flow rate are displayed. Right-click and select an option: ■ **Open**. Fully opens the MFC. ■ **Close**. Closes the MFC to stop gas flow. ■ **Set**. Displays the applicable Mass Flow Controller dialog box in which you can open the controller, close the controller, or set the flow rate. Set Reference Mass Flow Controller ○ Open ○ Close ● Set flow rate: 10.00 sccm Gas: N2 ■ **Zero**. Sets the MFC to read zero flow under current conditions.

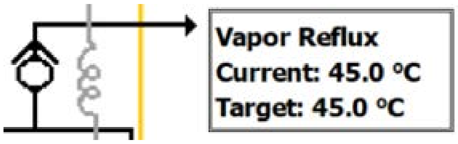
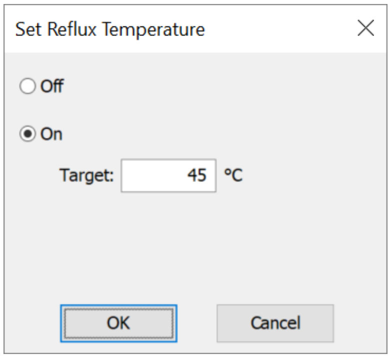
Analyzer Schematic Icons (continued)

Icon or Symbol	Description
	Zeroing a mass flow controller while gas is flowing will cause inaccurate readings. See Experiment Steps, Zero Mass Controller.
	**Open Valve**. Green indicates an open valve.
	**Pressure Transducer**. Displays the current pressure in the carrier gas path, upstream from the septum but downstream from the Mass Flow Controller. It cannot be manually controlled.
	**Rotary Valve**. Controls the gas flow through the trap, sample tube, loop, and optional vapor generator. Select the needed state. From left to right, the valves and their states are: ■ **Trap**. Bypass, Trap ■ **Analysis**. Analyze, Prepare ■ **Loop**. Fill, Inject ■ **Vapor**. Vapor, Bypass (optional)
	**Sample Furnace**. Controls the temperature of the sample. Click to display the sample temperature, the target temperature, the furnace temperature, and the ramp rate. Right-click the icon and select *Set Temperature* to change the *Set Point* and/or the *Ramp Rate*.
	**Sample Temperature**. Controls the sample *Target* temperature. Set Sample Temperature ○ Off ● On Target: 20 °C

Analyzer Schematic Icons (continued)

Icon or Symbol	Description
	**Trap Heater**. Controls the temperature and ramp rate.  Set Trap Heater ○ Enable temperature control Target temperature: 20 °C Ramp rate: 1 °C/min ● Disable temperature control
	**Valve Temperature**. Controls the *Target* temperature.  Set Valve Temperature ○ Off ● On Target: 60 °C
	**Vapor Flask**. Controls the *Target* temperature.  Set Flask Temperature ○ Off ● On Target: 50 °C

Analyzer Schematic Icons (continued)

Icon or Symbol	Description
 Vapor Reflux Current: 45.0 °C Target: 45.0 °C	**Vapor Reflux**. Controls the *Target* temperature.  Set Reflux Temperature ○ Off ● On Target: 45 °C

Instrument Schematic Shortcut Menus

Schematic Shortcuts

Icon or Symbol	Description
Valve options ☚	**Close.** Closes the selected valve. **Open.** Opens the selected valve.
Temperature control options 🌡	Select *Set Temperature* to modify furnace or heater settings.

Gas Pathways

The colored lines that lead from the gas inlets through the analyzer and to the exhaust represent the current flow path. A different color path represents each gas source:

Red	Exhaust gas
Green	Carrier gas
Brown	Preparation gas
Blue	Blended gas or loop gas
Gray	Inactive gas

> When a gas valve is opened, or the flow path is changed, it takes several minutes for the new gas to move through the entire system.

The behavior of the loop valve is one example. When an analysis includes a loop injection, the schematic shows the path available in *Fill* mode, then *Inject* mode.

- In *Fill* mode, the analysis gas enters the loop, and the excess gas vents out the exhaust.
- In *Inject* mode, the analysis gas bypasses the loop and exits the analyzer through the exhaust.

At that time, the carrier gas path is available from the inlet all the way through the loop, through the sample tube, and past the detector.

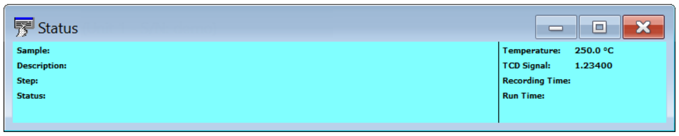
When the valve switches to *Inject* mode, the quantity of analysis gas contained in the loop — one exact unit — is pushed out of the loop and through the sample tube by the carrier gas. When the application is recording the TCD signal, the loop injection shows as a peak on the *Results* view of the *Analysis* window.

The analysis gas may be greatly dissipated as it travels out of the loop and through the sample tube, particularly if the carrier gas flow rate is low. Higher carrier gas flow rates minimize the dispersion of the loop gas.

SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



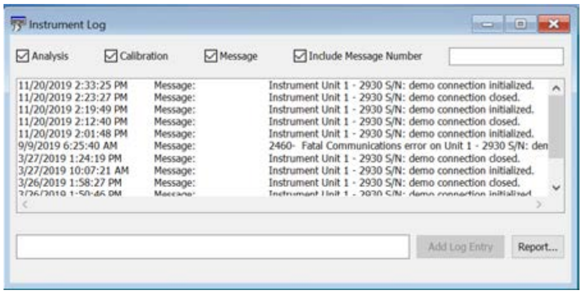
Sample:
Description:
Step:
Status:
Temperature: 250.0 °C
TCD Signal: 1.23400
Recording Time:
Run Time:

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

SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

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



```
11/20/2019 2:33:25 PM    Message:        Instrument Unit 1 - 2930 S/N: demo connection initialized.
11/20/2019 2:23:27 PM    Message:        Instrument Unit 1 - 2930 S/N: demo connection closed.
11/20/2019 2:19:49 PM    Message:        Instrument Unit 1 - 2930 S/N: demo connection initialized.
11/20/2019 2:12:40 PM    Message:        Instrument Unit 1 - 2930 S/N: demo connection closed.
11/20/2019 2:01:48 PM    Message:        Instrument Unit 1 - 2930 S/N: demo connection initialized.
9/9/2019 6:25:40 AM      Message:        2460-  Fatal Communications error on Unit 1 - 2930 S/N: den
3/27/2019 1:24:19 PM     Message:        Instrument Unit 1 - 2930 S/N: demo connection closed.
3/27/2019 10:07:21 AM    Message:        Instrument Unit 1 - 2930 S/N: demo connection initialized.
3/26/2019 1:58:27 PM     Message:        Instrument Unit 1 - 2930 S/N: demo connection closed.
3/26/2019 1:50:46 PM     Message:        Instrument Unit 1 - 2930 S/N: demo connection initialized.
```

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 - 6*.

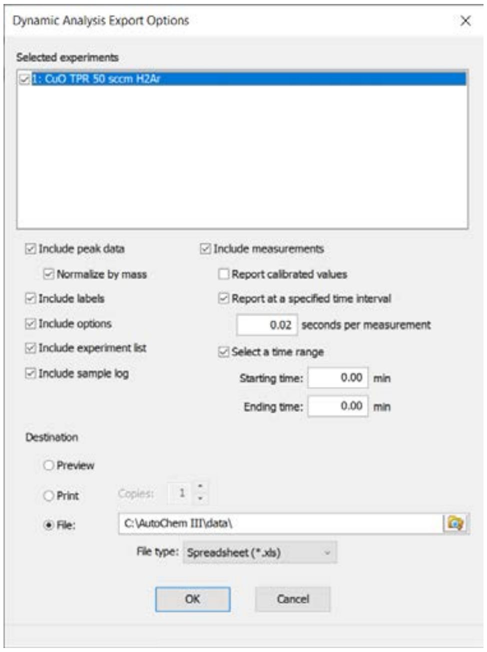
EXPORT FILES

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**.



Dynamic Analysis Export Options

Selected experiments

☑ 1: CuO TPR 50 sccm H2Ar

☑ Include peak data
  ☑ Normalize by mass
☑ Include labels
☑ Include options
☑ Include experiment list
☑ Include sample log

☑ Include measurements
  ☐ Report calibrated values
  ☑ Report at a specified time interval
    0.02 seconds per measurement
  ☑ Select a time range
    Starting time: 0.00 min
    Ending time: 0.00 min

Destination

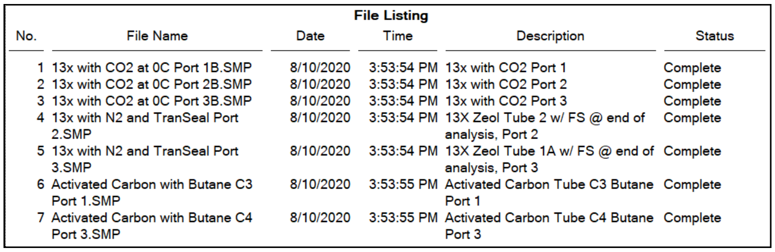
○ Preview
○ Print Copies: 1
● File: C:\AutoChem III\data\
File type: Spreadsheet (*.xls)

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

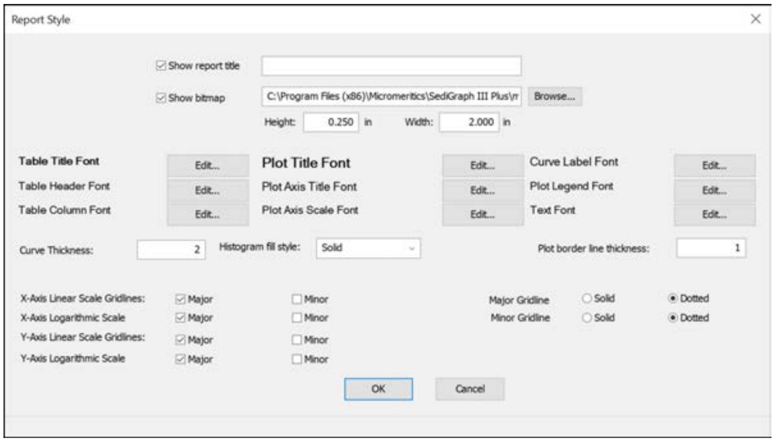
No.	File Name	Date	Time	Description	Status
1	13x with CO2 at 0C Port 1B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 1	Complete
2	13x with CO2 at 0C Port 2B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 2	Complete
3	13x with CO2 at 0C Port 3B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 3	Complete
4	13x with 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

REPORT STYLE

Options > Report Style

A report style can also be configured from an analysis with a *Completed* status.



☑ Show report title

☑ Show bitmap C:\Program Files (x86)\Micromeritics\SediGraph III Plus\r

Height: 0.250 in Width: 2.000 in

Curve Thickness: 2

Histogram fill style: Solid

Plot border line thickness: 1

X-Axis Linear Scale Gridlines: ☑ Major ☐ Minor

X-Axis Logarithmic Scale ☑ Major ☐ Minor

Y-Axis Linear Scale Gridlines: ☑ Major ☐ Minor

Y-Axis Logarithmic Scale ☑ Major ☐ Minor

Major Gridline ○ Solid ● Dotted

Minor Gridline ○ Solid ● Dotted

Report Style

Selections	**Description**
Curve Thickness [*text box*]	Enter a value to indicate the thickness of the curve on reports.
Font Options [*button*]	Click **Edit** to select font type, font style, and font size.
Histogram fill style [*drop-down box*]	Select how histograms are to appear on the report.
Linear Scale Logarithmic Scale [*check box*]	Select major and/or minor lines to display in reports for the logarithmic and linear scales. Deselect this option to remove the grid lines.
Major Gridline Minor Gridline [*button*]	Select if the major and/or minor grid lines should appear as solid or dotted lines.
Plot border line thickness [*text box*]	Enter a value to indicate the thickness of the plot borders on reports.
Show bitmap [*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.

Report Style (continued)

Selections	Description
Show report title **[***text 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 - 6*.	

SOFTWARE UNINSTALL

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.

3 SAMPLE FILES

Option Presentation on page 2 - 11

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.

SAMPLE FILES

File > New Sample > [.SMP File]

File > Open > [.SMP File]

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.

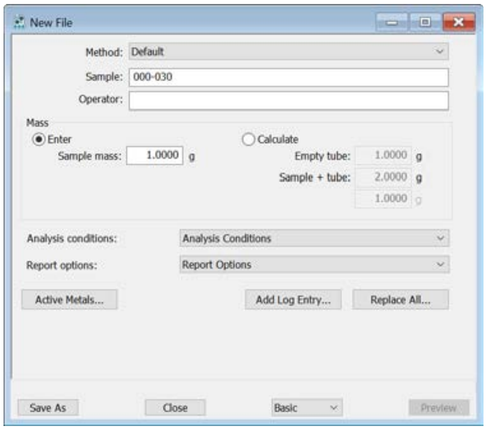
Each analysis must be linked with a sample file before the analysis can proceed. A sample file can consist of parameter files; however, parameter files can also stand alone.

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.

Method: Default
Sample: 000-007
Operator:
Submitter:
Bar Code:
Mass
● Enter
Sample mass: 1.0000 g
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Comments:

Advanced
option presentation



Method: Default
Sample: 000-030
Operator:
Mass
● Enter
Sample mass: 1.0000 g
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Analysis conditions: Analysis Conditions
Report options: Report Options

Basic or Restricted
option presentation

Sample Files

Selections	Description
Active Metals [*button*]	Displays a list of active metals. See ***Active Metals for Chemisorption Analyzers on page 3 - 6***.
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*]	Enter a value for sample mass. Mass can be changed any time before, during, or after analysis. **Enter.** Enables the *Sample mass* field. Enter a value for the sample mass. **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. The equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$
Method [*drop-down box*]	Select a method from the drop-down list.

Sample Files (continued)

Selections	Description
Operator [*text box*]	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods***.
Sample [*text box*]	Enter a sample description.
Submitter [*text box*]	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods***.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

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 Status	Displays
No Analysis	Tabbed file editor
Complete **Analyzing**	Peak Editor view

Method: Default

Sample: 000-007

Operator:

Submitter:

Bar Code:

Mass

● Enter

Sample mass: 1.0000 g

○ Calculate

Empty tube: 1.0000 g

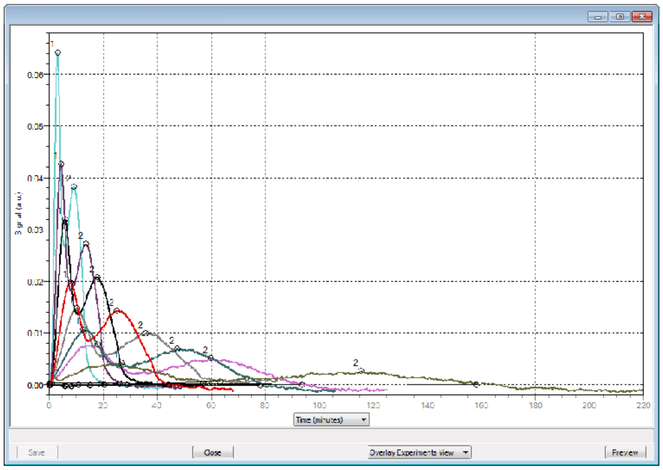
Sample + tube: 2.0000 g

1.0000 g

Comments:

Advanced

Tabbed file editor in Advanced view



Peak Editor View

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.

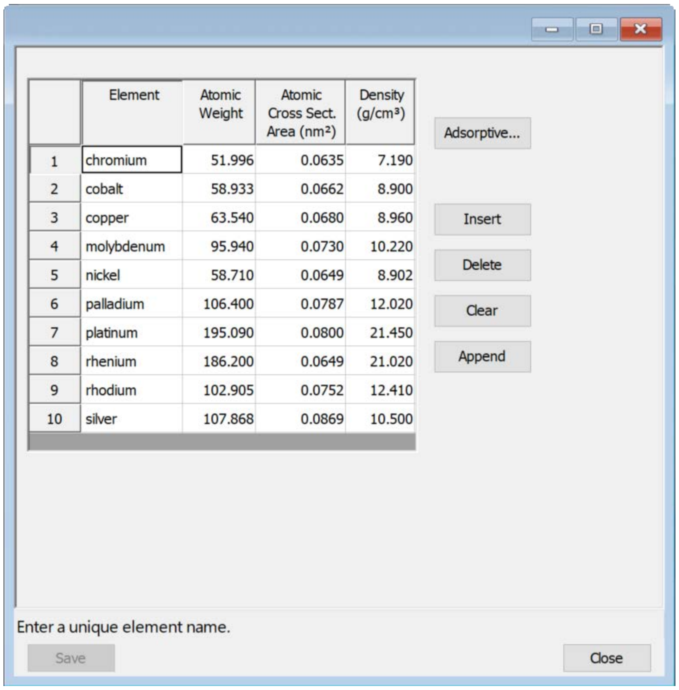
ACTIVE METALS FOR CHEMISORPTION ANALYZERS

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.



	Element	Atomic Weight	Atomic Cross Sect. Area (nm^2)	Density (g/cm^3)
1	chromium	51.996	0.0635	7.190
2	cobalt	58.933	0.0662	8.900
3	copper	63.540	0.0680	8.960
4	molybdenum	95.940	0.0730	10.220
5	nickel	58.710	0.0649	8.902
6	palladium	106.400	0.0787	12.020
7	platinum	195.090	0.0800	21.450
8	rhenium	186.200	0.0649	21.020
9	rhodium	102.905	0.0752	12.410
10	silver	107.868	0.0869	10.500

Enter a unique element name.

Active Metals

Selections	Description
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.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

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

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4 PARAMETER FILES

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

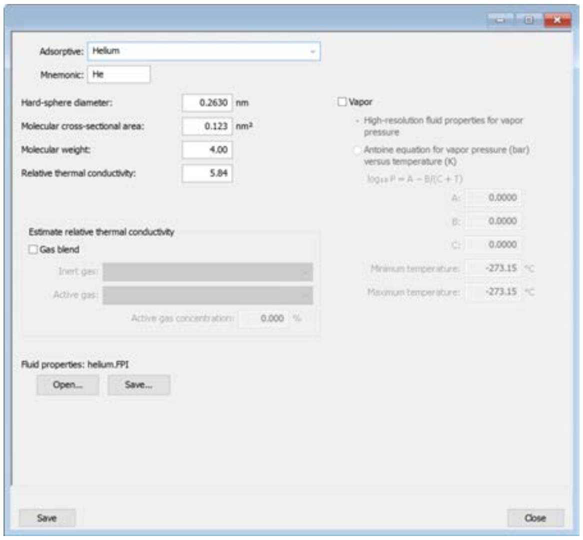
File Type	**File Extension**
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Method	.MTH
Report Options	.RPO

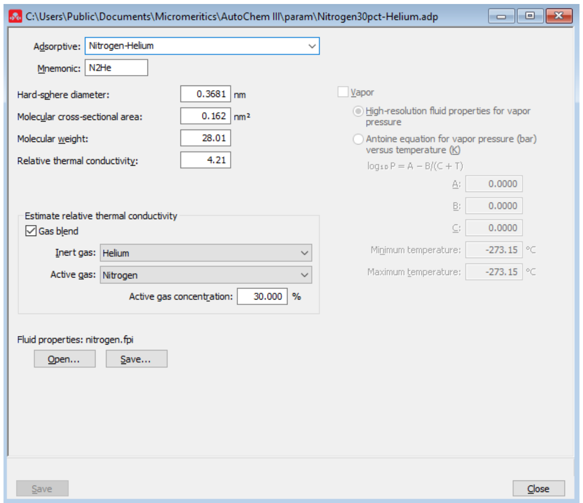
ADSORPTIVE PROPERTIES

File > Open > [.ADP File]

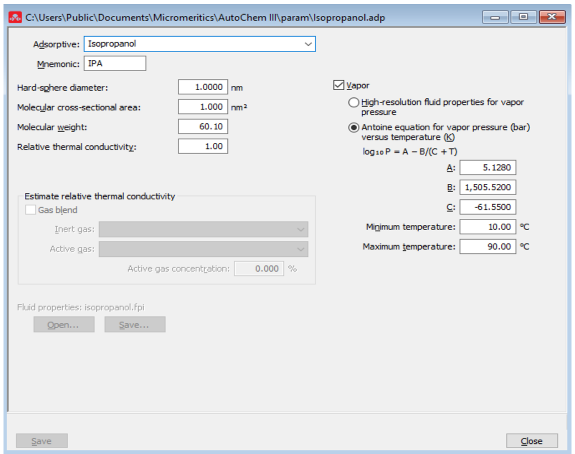
[*Gas Connections on page 2 - 14*]()
[*Gas Charts on page C - 1*]()

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


Adsorptive: Helium
Mnemonic: He
Hard-sphere diameter: 0.2630 nm
Molecular cross-sectional area: 0.123 nm²
Molecular weight: 4.00
Relative thermal conductivity: 5.84
☐ Vapor
○ High-resolution fluid properties for vapor pressure
○ Antoine equation for vapor pressure (bar) versus temperature (K)
$\log_{10} P = A - B/(C + T)$
A: 0.0000
B: 0.0000
C: 0.0000
Minimum temperature: -273.15 °C
Maximum temperature: -273.15 °C
Estimate relative thermal conductivity
☐ Gas blend
Inert gas:
Active gas:
Active gas concentration: 0.000 %
Fluid properties: helium.FPI


C:\Users\Public\Documents\Micromeritics\AutoChem III\param\Nitrogen30pct-Helium.adp
Adsorptive: Nitrogen-Helium
Mnemonic: N2He
Hard-sphere diameter: 0.3681 nm
Molecular cross-sectional area: 0.162 nm²
Molecular weight: 28.01
Relative thermal conductivity: 4.21
☐ Vapor
● High-resolution fluid properties for vapor pressure
○ Antoine equation for vapor pressure (bar) versus temperature (K)
$\log_{10} P = A - B/(C + T)$
A: 0.0000
B: 0.0000
C: 0.0000
Minimum temperature: -273.15 °C
Maximum temperature: -273.15 °C
Estimate relative thermal conductivity
☑ Gas blend
Inert gas: Helium
Active gas: Nitrogen
Active gas concentration: 30.000 %
Fluid properties: nitrogen.fpi

Gas Blend


C:\Users\Public\Documents\Micromeritics\AutoChem III\param\Isopropanol.adp
Adsorptive: Isopropanol
Mnemonic: IPA
Hard-sphere diameter: 1.0000 nm
Molecular cross-sectional area: 1.000 nm²
Molecular weight: 60.10
Relative thermal conductivity: 1.00
☑ Vapor
○ High-resolution fluid properties for vapor pressure
● Antoine equation for vapor pressure (bar) versus temperature (K)
$\log_{10} P = A - B/(C + T)$
A: 5.1280
B: 1,505.5200
C: -61.5500
Minimum temperature: 10.00 °C
Maximum temperature: 90.00 °C
Estimate relative thermal conductivity
☐ Gas blend
Inert gas:
Active gas:
Active gas concentration: 0.000 %
Fluid properties: isopropanol.fpi

Vapor

Adsorptive Properties

Selections	Description
Adsorptive [*drop-down box*]	Name of the adsorptive gas whose properties are being defined.
Thermal Conductivity [*group box*]	Select if a gas blend will be used for the estimated Relative Thermal Conductivity. Select the gases to be used in the blend. **Gas blend.** [*check box*] 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.
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.
Hard-sphere diameter [*text box*]	Estimate of the molecular size used in calculating the thermal transpiration correction.
Mnemonic [*text box*]	Enter the mnemonic name for the adsorptive.
Molecular cross-sectional area [*text box*]	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
Molecular weight [*text box*]	The molecular mass is used for the weight % column of the isotherm tabular report and the pressure composition isotherm plot.
Relative thermal conductivity (or Thermal conductivity) [*text box*]	Enter the thermal conductivity relative to Air.

Adsorptive Properties (continued)

Selections	Description
Vapor [*check box*]	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.

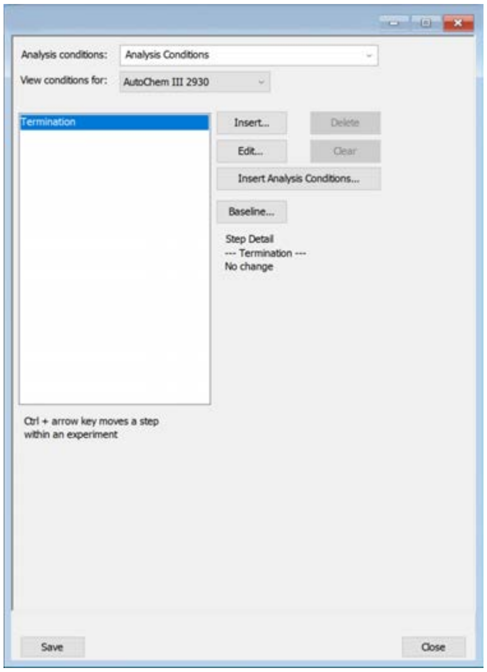
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

ANALYSIS CONDITIONS

File > Open > [.ANC File]

Analysis conditions specify the parameters used to guide an analysis.

When inserting experiments, select the instrument type in the *View conditions for* drop-down list.



Analysis conditions: Analysis Conditions

View conditions for: AutoChem III 2930

Termination

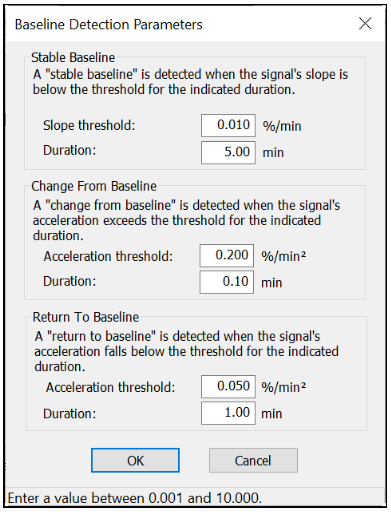
Step Detail
--- Termination ---
No change

Ctrl + arrow key moves a step within an experiment

Analysis Conditions

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.

Analysis Conditions (continued)

Selections	Description
Baseline [*button*]	Specifies baseline settings if a *Wait* step depends upon the *Baseline*.  Baseline Detection Parameters Stable Baseline A "stable baseline" is detected when the signal's slope is below the threshold for the indicated duration. Slope threshold: 0.010 %/min Duration: 5.00 min Change From Baseline A "change from baseline" is detected when the signal's acceleration exceeds the threshold for the indicated duration. Acceleration threshold: 0.200 %/min² Duration: 0.10 min Return To Baseline A "return to baseline" is detected when the signal's acceleration falls below the threshold for the indicated duration. Acceleration threshold: 0.050 %/min² Duration: 1.00 min Enter a value between 0.001 and 10.000. 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 (continued)

Selections	**Description**
Delete [*button*]	Deletes the currently selected step. If the step is an experiment, a prompt displays to confirm deletion of all steps for that experiment.
Edit [*button*]	Displays the applicable dialog box for the selected step.
Insert [*button*]	Inserts a new step into the task list. 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*]	Loads 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.
View conditions for [*drop-down box*]	Selects the instrument model to use, and the editor only shows the options available for that model. The selection is saved and reused when the file is reopened and disabled during and after analysis.

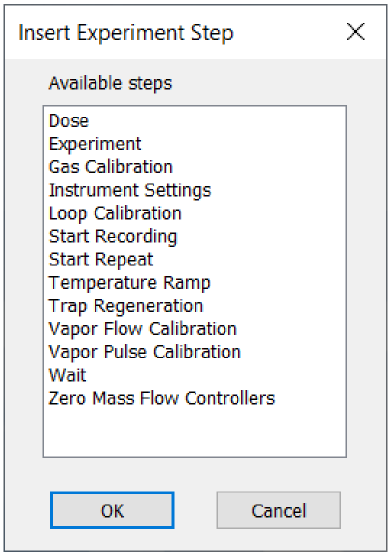
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

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.

To insert an experiment:

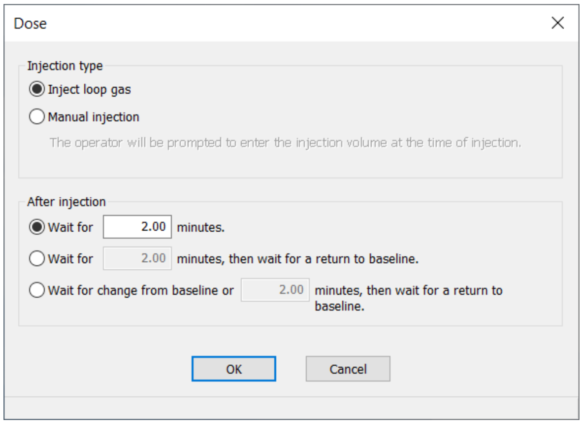
1. On the *Analysis Conditions* tab, click **Insert** to insert an experiment. A list of all available experiments is displayed.



2. Select the experiment step.
3. Click **OK**. The applicable step dialog box is displayed. See the *Experiment Fields* table for details on each step.

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

Experiment Fields

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Dose</td><td>


● Inject loop gas

○ Manual injection

The operator will be prompted to enter the injection volume at the time of injection.

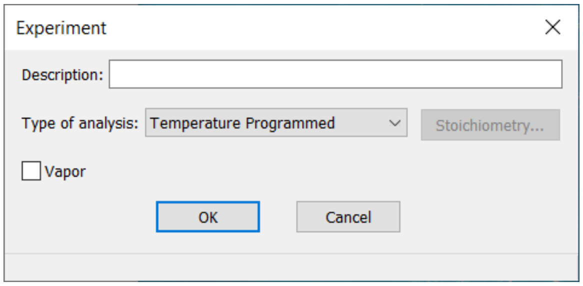
● Wait for 2.00 minutes.

○ Wait for 2.00 minutes, then wait for a return to baseline.

○ Wait for change from baseline or 2.00 minutes, then wait for a return to baseline.

Injection Type [group box]:

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. Specify the amount of time to wait after injection has occurred.
</td></tr>
<tr><td>Experiment</td><td>


Description:

Type of analysis: Temperature Programmed

☐ Vapor

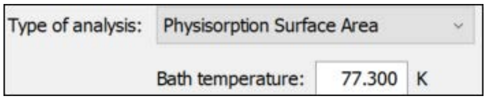
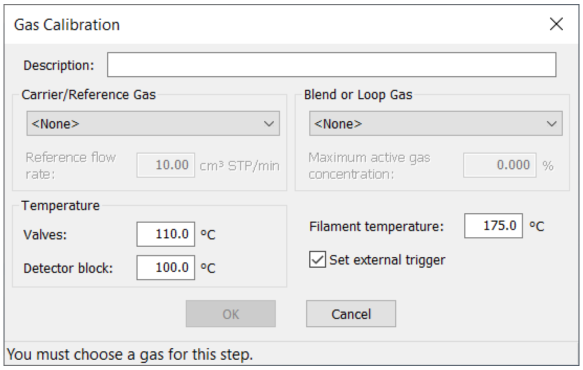
Description. [text box] Description of the experiment.

Type of analysis. [drop-down box]

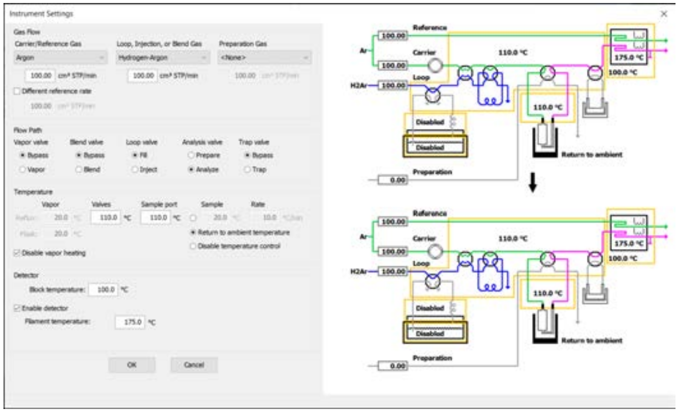
If Pulse Chemisorption is selected, the Stoichiometry button is enabled. When modifying the Active Metals table during the insertion of a Pulse Chemisorption experiment, changes to the Active Metals table on the Sample Description tab are not affected. This is useful when sequencing multiple experiments and Stoichiometry is different from one experiment to the next.

Bath temperature. [text box] Applies to Physisorption Surface Area
</td></tr>
</table>

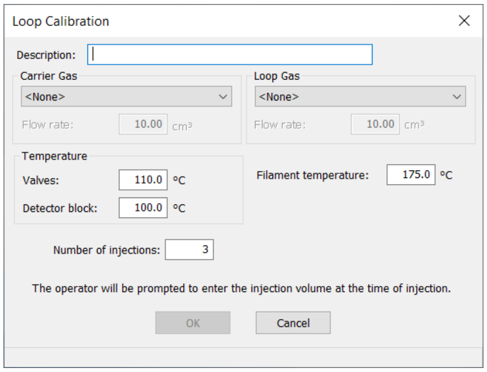
Experiment Fields (continued)

Selections	Description
	only. Calculates P_0 using the entered bath temperature. Type of analysis: Physisorption Surface Area Bath temperature: 77.300 K **Stoichiometry.** [*button*] Opens the *Active Metals* table. Specify the percent of sample weight of the active metals in the sample and the stoichiometry factor. See ***Active Metals for Chemisorption Analyzers on page 3 - 6***. **Vapor**. [*check box*] If *Temperature Programmed*, *Pulse* or *Other* is selected, the **Vapor** checkbox is enabled and a drop-down list is displayed. Select the Vapor to be used
Gas Calibration	 Description: Carrier/Reference Gas <None> Reference flow rate: 10.00 cm³ STP/min Blend or Loop Gas <None> Maximum active gas concentration: 0.000 % Temperature Valves: 110.0 °C Detector block: 100.0 °C Filament temperature: 175.0 °C ☑ Set external trigger You must choose a gas for this step. Use to calibrate the TCD so that peak areas can be converted to gas quantities. During a gas calibration, a series of known gas mixtures flows through the analyzer and the resultant signal readings are recorded. The analyzer can then use these data to calculate the concentrations of unknown mixtures flowing past the detector during subsequent analyses. **Calibration Gases.** [*drop-down box*] Select the *Carrier* and *Loop* gases to be used in this calibration. The gases available are those specified in an Adsorptive Properties [.ADP] file. ■ **Reference flow rate.** [*text box*] Enter a flow rate for the calibration gases. ■ **Maximum active gas concentration.** [*text box*] Enter the maximum percentage for the active gas concentration. **Description.** [*text box*] Description of the experiment. **Detector block.** [*text box*] Enter the temperature for the detector block.

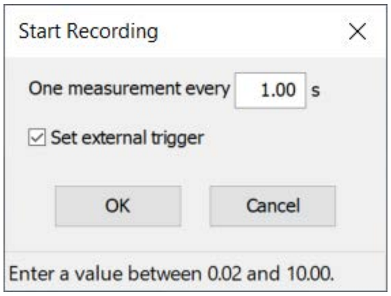
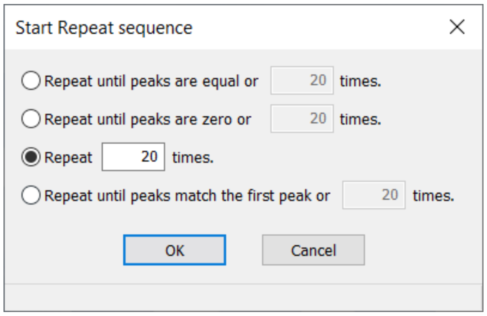
Experiment Fields (continued)

Selections	Description
	Filament temperature. [*selection*] Enter the temperature for the filament. **Set external trigger**. [*check box*] If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected. **Valves**. [*selection*] Enter the temperature for the valves.
Instrument Settings	As instrument settings are changed in the left window, changes are highlighted on the schematic at the bottom right window.  Instrument Settings Gas Flow Carrier/Reference Gas Argon 100.00 cm³ STP/min Loop, Injection, or Blend Gas Hydrogen-Argon 100.00 cm³ STP/min Preparation Gas <None> 100.00 cm³ STP/min ☐ Different reference rate 100.00 cm³ STP/min Flow Path Vapor valve ● Bypass ○ Vapor Blend valve ● Bypass ○ Blend Loop valve ● Fill ○ Inject Analysis valve ○ Prepare ● Analyze Trap valve ● Bypass ○ Trap Temperature Vapor Reflux: 20.0 °C Flask: 20.0 °C Valves 110.0 °C Sample port 110.0 °C ○ Sample 20.0 °C Rate 10.0 °C/min ● Return to ambient temperature ○ Disable temperature control ☑ Disable vapor heating Detector Block temperature: 100.0 °C ☑ Enable detector Filament temperature: 175.0 °C OK Cancel **Gas Flow**. [*group box*] Select the gases to be used in this analysis along with the flow rates. The gases available are those specified in an Adsorptive Properties [.ADP] file. See ***Adsorptive Properties on page 4 - 2*** and ***Gas Charts on page C - 1*** for gas combinations. Indicate if a **Different reference rate** should be used along with the applicable rate. **Flow Path**. [*group box*] Select the step to be performed at each valve. **Temperature**. [*group box*] Enter the sample and rate temperatures. ■ **Disable temperature control**. [*button*] Disables the temperature control. ■ **Disable vapor heating**. [*check box*] Disables the vapor flask heating mantle, the vapor reflux zone heater, and the vapor block heater. ■ **Return to ambient temperature**. [*button*] Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature between 14 °C and 50 °C.

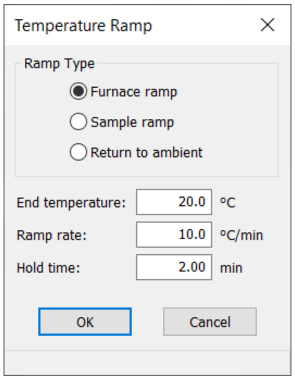
Experiment Fields (continued)

Selections	Description
	Detector [*group box*]: ■ **Block temperature**. [*text box*] Enter the block temperature. ■ **Enable detector**. [*check box*] Specify if the detector should be enabled and the applicable **Filament Temperature**.
Loop Calibration	 Loop Calibration Description: Carrier Gas <None> Flow rate: 10.00 cm^3 Loop Gas <None> Flow rate: 10.00 cm^3 Temperature Valves: 110.0 °C Detector block: 100.0 °C Filament temperature: 175.0 °C Number of injections: 3 The operator will be prompted to enter the injection volume at the time of injection. Use to verify the volume of the loop for use in calculations on analyses that use the loop. Sample analysis data yield signal vs. temperature data and peak areas. Associating the sample file with a loop calibration file makes it possible for the application to convert sample data to volume values. **Calibration Gases.** [*drop-down box*] Select the *Carrier* and *Loop* gases to be used in this calibration. The gases available are those specified in an Adsorptive Properties [.ADP] file. Enter a **Flow rate** for each gas. **Description.** [*text box*] Description of the experiment. **Filament temperature.** [*selection*] Enter the temperature for the filament. **Number of injections.** [*text box*] Enter the number of manual and automatic injections (3-10) to be done with the loop during calibration. **Temperature.** [*group box*] Enter the temperatures for the **Detector block** and **valves**.

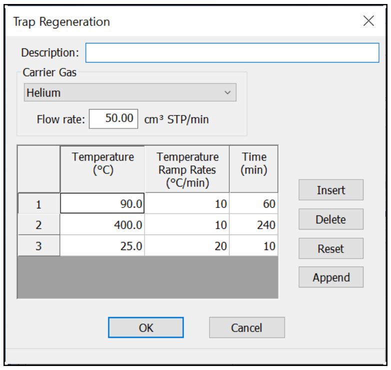
Experiment Fields (continued)

Selections	Description
Start Recording	 One measurement every 1.00 s ☑ Set external trigger Enter a value between 0.02 and 10.00. 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 the *Start Recording* and *Stop Recording* steps. **One measurement every [*n*] seconds.** [*text box*] Specify the frequency of measurements. **Set external trigger**. [*check box*] If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected. If this option is enabled, the external trigger signal will be set at the beginning of recording and turned off at the end of recording. 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 Sequence	 ○ Repeat until peaks are equal or 20 times. ○ Repeat until peaks are zero or 20 times. ● Repeat 20 times. ○ Repeat until peaks match the first peak or 20 times. 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.

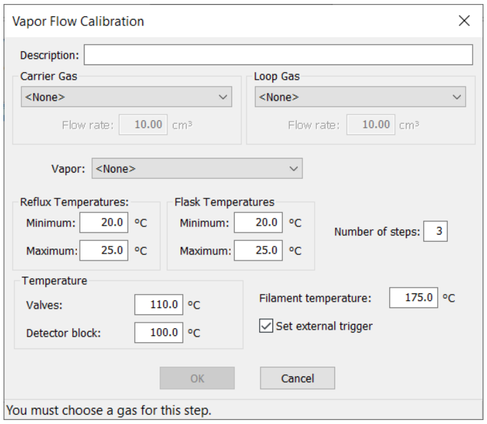
Experiment Fields (continued)

Selections	Description
	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*] 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	 Temperature Ramp Ramp Type ● Furnace ramp ○ Sample ramp ○ Return to ambient End temperature: 20.0 °C Ramp rate: 10.0 °C/min Hold time: 2.00 min Changes the sample temperature. CAUTION Temperatures above 850 °C can cause damage to a metal sample tube. Also, sample temperatures significantly above 1100 °C will cause accelerated wear on furnace components and lead to premature failure of the furnace. Experiments should be designed with sample temperatures at or below 1100 °C or to minimize operating time above 1100 °C.

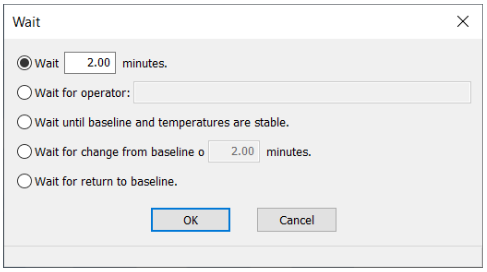
Experiment Fields (continued)

Selections	Description
	Ramp Type [*group box*]: ■ **Furnace ramp. [*button*]** Ramps the furnace temperature directly to the *End* temperature ignoring the sample temperature. ■ **Sample ramp.** [*button*] Ramps the sample temperature to the *End* temperature. The actual furnace temperature is adjusted to meet this target. ■ **Return to ambient. [*button*]** Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature to below 45 °C. **End temperature.** [*text box*] 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. **Hold time.** [*text box*] Temperature at which the sample is to be held while degassing. **Ramp rate. [*text box*]** The rate at which the temperature will change while advancing to the hold temperature.
Trap Regeneration	 Trap Regeneration Description: Carrier Gas: Helium Flow rate: 50.00 cm³ STP/min Temperature (°C) · Temperature Ramp Rates (°C/min) · Time (min) 1 · 90.0 · 10 · 60 2 · 400.0 · 10 · 240 3 · 25.0 · 20 · 10 **Description.** [*text box*] Enter a description for this step. **Carrier Gas.** [*group box*] Select the carrier gas and enter the **Flow rate**. **Insert**. [*button*] Inserts a new row at the top of the table. **Delete**. [*button*] Deletes the currently selected row. **Reset**. [*button*] Resets the table, returning it to the layout before the dialog box is closed. **Append**. [*button*] Adds a new row to the bottom of the table.

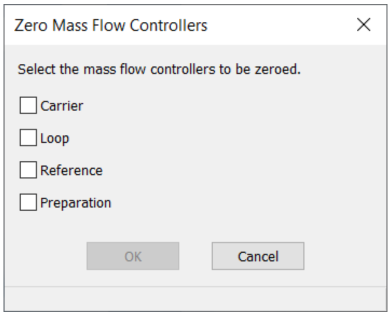
Experiment Fields (continued)

Selections	Description
Vapor Flow Calibration **Vapor Pulse Calibration**	 Vapor Flow Calibration Description: Carrier Gas: <None> Flow rate: 10.00 cm³ Loop Gas: <None> Flow rate: 10.00 cm³ Vapor: <None> Reflux Temperatures: Minimum: 20.0 °C Maximum: 25.0 °C Flask Temperatures: Minimum: 20.0 °C Maximum: 25.0 °C Number of steps: 3 Temperature: Valves: 110.0 °C Detector block: 100.0 °C Filament temperature: 175.0 °C ☑ Set external trigger OK Cancel You must choose a gas for this step. **Carrier Gas/Loop Gas.** [*drop-down box*] Select the gases to be used for this step and enter the **Flow rate**. **Description.** [*text box*] Enter a description for this step. **Flask Temperatures.** [*group box*] Enter a value between 20.0 and 120.0 degrees as the **Minimum** and **Maximum**. **Filament temperature.** [*selection*] Enter the temperature for the filament. **Number of steps.** [*text box*] Enter the number of steps (between 3 and 7) for this calibration. **Reflux Temperatures.** [*group box*] Enter a value between 20.0 and 100.0 degrees for the **Minimum** and **Maximum**. **Set external trigger.** [*check box*] If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected. **Temperature.** [*group box*] Enter a value between 20.0 and 150.0 degrees for the **Valves** and **Detector** block. **Vapor.** [*drop-down box*] Select the vapor to be used.

Experiment Fields (continued)

Selections	Description
Wait	 Wait ● Wait 2.00 minutes. ○ Wait for operator: ○ Wait until baseline and temperatures are stable. ○ Wait for change from baseline o 2.00 minutes. ○ Wait for return to baseline. OK Cancel Specify a waiting routine. **Wait [*n*] minutes.** [*button*] Specify the time to wait. **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 until baseline and temperature are stable.** [*button*] Specify if the analysis should wait until the baseline and temperature become stable, then click **Baseline** to specify the settings. **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 return to baseline.** [*button*] Waits for a return to baseline. If enabled, click **OK**, then click **Baseline** to specify the settings.

Experiment Fields (continued)

Selections	Description
Zero Mass Flow Controllers	 Select the mass flow controllers to be zeroed. When this step is executed, all gas inlet valves for each selected MFC will be closed and zeroed. The Zero MFC task is equivalent to performing the following manual procedure: 1. Close any open valves on the MFC's inlet manifold. 2. Open the MFC for at least 20 seconds. 3. Close the MFC and wait at least 5 seconds. 4. Select *Zero* from the context menu.

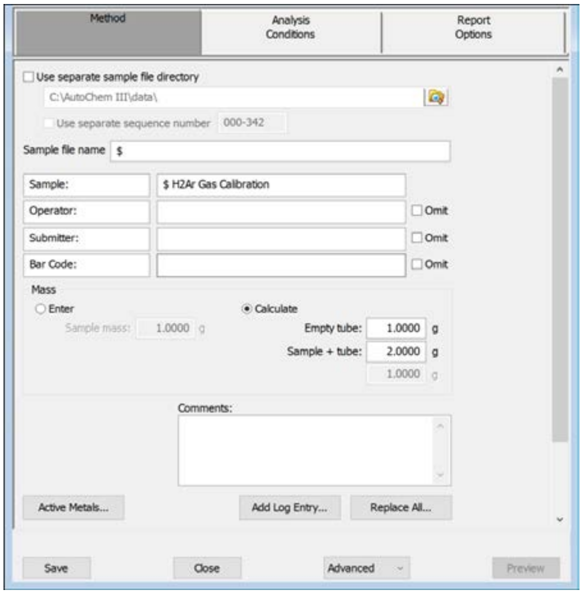
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

METHODS

File > Open > [.MTH File]

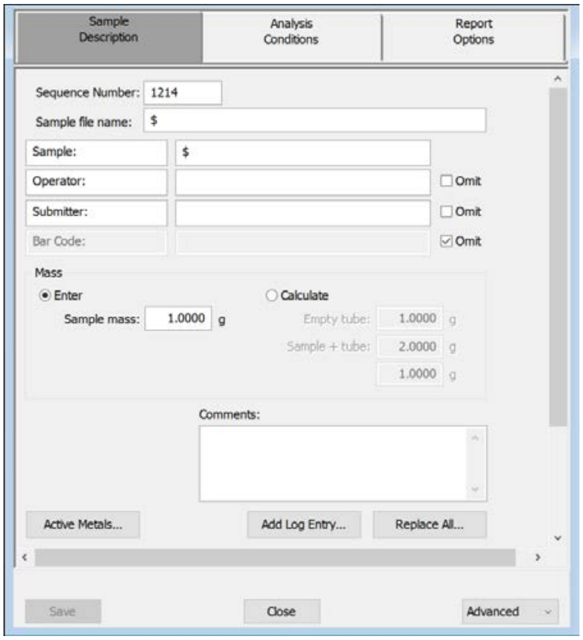
Options > Default Method

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.



☐ Use separate sample file directory
C:\AutoChem III\data\
☐ Use separate sequence number 000-342
Sample file name: $
Sample: $ H2Ar Gas Calibration
Operator: ☐ Omit
Submitter: ☐ Omit
Bar Code: ☐ Omit
Mass
○ Enter Sample mass: 1.0000 g
● Calculate Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Comments:

File > Open > [.MTH File]



Sequence Number: 1214
Sample file name: $
Sample: $
Operator: ☐ Omit
Submitter: ☐ Omit
Bar Code: ☑ Omit
Mass
● Enter Sample mass: 1.0000 g
○ Calculate Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Comments:

Options > Default Method

Methods

Selections	Description
Active Metals [*button*]	Opens the Active Metals dialog box that contains a list of active metals. See ***Active Metals for Chemisorption Analyzers on page 3 - 6***.
Comments [*text box*]	Enter comments to display in the report header about the sample or analysis.

Methods (continued)

Selections	Description
Mass [*group box*]	Specify the default data. **Enter.** Enables the *Sample mass* field. Enter a value for the sample mass. **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. The equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$
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.
Field names [*text box*]	Enter a name to use in the new sample file. A maximum of four fields can be configured. Default fields are Sample, Operator, Submitter and Bar Code. Check **Omit** to remove the field from the template. The first field cannot be omitted.
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. Select *Use separate sequence number* to specify a number other than the default.
Use separate sample file directory [*text box*]	Specify the directory to save the sample file other than the default shown. If the directory does not exist, the field is highlighted and an error message displays. Click the **Browse** button and select an existing directory.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

REPORT OPTIONS

File > Open > [.RPO File]

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

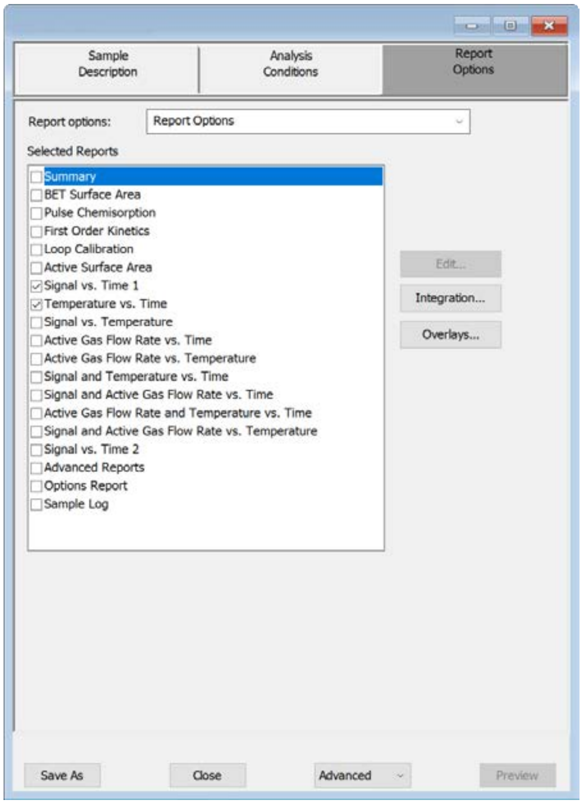
The *Calculations* document can be found on the Micromeritics web page (www.micromeritics.com).

Additional reports are available using the *Reports* menu.

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

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

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

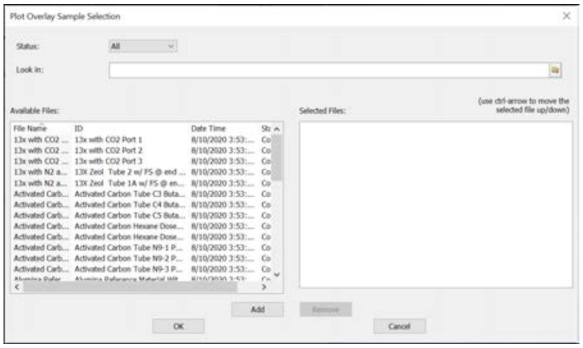


Report options: Report Options

Selected Reports

- ☐ Summary
- ☐ BET Surface Area
- ☐ Pulse Chemisorption
- ☐ First Order Kinetics
- ☐ Loop Calibration
- ☐ Active Surface Area
- ☑ Signal vs. Time 1
- ☑ Temperature vs. Time
- ☐ Signal vs. Temperature
- ☐ Active Gas Flow Rate vs. Time
- ☐ Active Gas Flow Rate vs. Temperature
- ☐ Signal and Temperature vs. Time
- ☐ Signal and Active Gas Flow Rate vs. Time
- ☐ Active Gas Flow Rate and Temperature vs. Time
- ☐ Signal and Active Gas Flow Rate vs. Temperature
- ☐ Signal vs. Time 2
- ☐ Advanced Reports
- ☐ Options Report
- ☐ Sample Log

Report Options

Selections	Description
Edit [*button*]	Select the report in the Selected Reports box. See ***Selected Reports on page 7 - 1***.
Integration [*button*]	See ***Peak Detection / Integration Options on page D - 1***
Overlays [*button*]	Select the files to overlay.  File Name ID Date Time St 13x with CO2 ... 13x with CO2 Port 1 8/10/2020 3:53:... Co 13x with CO2 ... 13x with CO2 Port 2 8/10/2020 3:53:... Co 13x with CO2 ... 13x with CO2 Port 3 8/10/2020 3:53:... Co 13x with N2 a... 13X Zeol Tube 2 w/ FS @ end ... 8/10/2020 3:53:... Co 13x with N2 a... 13X Zeol Tube 1A w/ FS @ en... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube C3 Buta... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube C4 Buta... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube C5 Buta... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Hexane Dose... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Hexane Dose... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube N9-1 P... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube N9-2 P... 8/10/2020 3:53:... Co Activated Carb... Activated Carbon Tube N9-3 P... 8/10/2020 3:53:... Co ***Example of Overlay window***
Report Options [*drop-down box*]	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports [*group box*]	Select the report names to include in the report.
Show graphic [*check box*]	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic'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 - 6*.

5 PERFORM AN ANALYSIS

When running an analysis and a CryoCooler is connected to the analyzer, a warning displays indicating that the Dewar level should be full before running an analysis.

ANALYSIS TEMPERATURE GUIDELINES FOR ALL ANALYSIS TYPES

- **Starting Temperature.** Experiments should be started at a temperature that is lower than that at which the reaction begins. If the reaction begins below ambient temperature, use the optional CryoCooler.
- **Maximum Temperature.** The experiment's maximum temperature is limited to the temperature at which the sample sinters.
- **Ramp Rate.** The recommended ramp rate is 5-10 °C/min, however a different ramp rate can be specified. For example, if using 500 °C as the starting temperature of the reaction, it is more expedient to ramp the temperature rapidly (for example, 50 °C/min.) to a value near 500 °C, then proceed with the experiment at 5-10 ºC/min.

COOLING OPTIONS

The furnace must cool during temperature programmed analyses if:

- a low temperature is required as part of the analysis, or
- to speed cooling of the furnace to increase sample throughput or reduce analysis time.

If cooling is required for the analysis, a CryoCooler may be necessary. Cooling options are:

- **Allow the temperature to return to ambient over time.** If time is not a consideration, allow the furnace to cool over time. It may take two hours or more for the furnace to cool from 1100 °C to near ambient. This may be acceptable when time is not limited.
- **Promote cooling by manually opening the furnace.** The furnace cools more rapidly when it is open. This option requires the availability of an operator to open and close the furnace at the appropriate times during analysis.

When it has been used recently, the furnace and/or the sample tube may be hot. Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

- **Connect a cooling gas or cryogen flow to the inlet on the front of the furnace.** A cooling flow of an appropriate gas or cryogen can be connected to the inlet on the furnace. Read the warning listed above. This option can provide rapid cooling of the sample but offers no control of the target temperature or the speed at which it is achieved. The gas or cryogen must be non-reactive — such as nitrogen, air, helium, argon, liquid nitrogen, or liquid argon.
- **Connect the optional CryoCooler to the inlet in the front of the furnace.** The CryoCooler provides control of the sample temperature to temperatures as low as -100 °C. The CryoCooler is required for accurate analyses that include signal recording below ambient. The CryoCooler Operator Manual can be found on the Micromeritics web page (www.micromeritics.com).

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 SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are the responsibility of the operator.

CAUTION	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.

PROTECT DETECTOR FILAMENTS BY FLOWING GAS

CAUTION

A carrier gas must be flowing through the detector whenever the detector filaments are turned on; otherwise, the filaments will deteriorate and lose sensitivity. If the gases are shut off, the filaments are automatically turned off after five minutes.

The flowing gas also continuously cleans the plumbing of any water vapor which can enter when the sample tube is removed. Ten cm^3/min is sufficient to perform this function.

Prepare for Analysis

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

Clean and Label Sample Tubes

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

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

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

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

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

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

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

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

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

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

PREPARE THE SAMPLE

To obtain the most repeatable, accurate results:

- Use approximately the same amount of sample for each analysis (especially if data are compared sample to sample).
- Ensure the sample is well dispersed in the tube, not against the side or walls of the tube.
- Ensure the sample particle size distribution remains fairly constant from sample to sample. (Large particles of a substance reduce at a different rate than small particles of the same substance.) For samples with a wide particle size distribution, care should be taken that each sample is representative of the entire lot's distribution.

Micromeritics recommends analyzing samples weighing between 20 mg and 2 g. When determining a sample amount, consider that the sample must be contained within the bottom 20 mm (3/4 in.) of the sample tube.

When determining sample size, consider the percentage of active metals in the sample. Materials with lower percentages of active metals may require larger sample amounts for analysis, while materials with high percentages of active metals can be analyzed using smaller samples.

> **CAUTION**
>
> Larger amounts of very fine powders may become packed in the sample tube and block gas flow. The resulting increase in gas pressure may force some of the sample out of the sample tube, causing contamination of, or damage to the analyzer. A possible solution is to expand the sample bed with quartz wool.

CREATE THE SAMPLE FILE

Sample Files on page 3 - 2

DETERMINE THE SAMPLE MASS FOR CHEMISORPTION

Use Quartz Filter Discs for Chemisorption on page 5 - 10
Sample Data Worksheet for Chemisorption on page F - 4

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

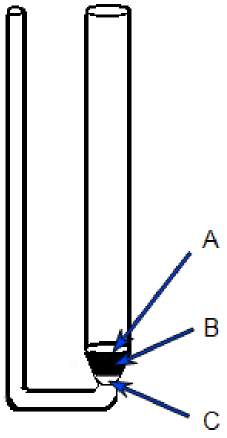
The equipment images in this topic may differ slightly from your equipment; however, the instructions are the same unless otherwise noted.

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.



A. Quartz disc
B. Sample
C. Quartz disc and 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

Sample Tube on page 8-23

CONNECT THE SORPTION TRAP

Sorption Trap on page 8-33

A sorption trap is necessary for TPR's or other temperature programmed experiments in which moisture needs to be removed from the flow path before reaching the detector. Make sure the sorption trap is connected and regenerated.

FILL AND INSTALL THE DEWAR

Dewar Precautions on page 5 - 3
Check and Clean the Dewar on page 9-20

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

If performing an analysis that requires use of the cold trap and cryogen, check the cryogen level in the Dewar. It should be approximately 25 mm (1 in.) from the top.

The cryogen must not be cold enough to trap the carrier gas or analysis gas. Do not use liquid nitrogen with argon carrier gas. For example, use an alcohol and liquid nitrogen slurry (-80 ºC).

PERFORM A PULSE CHEMISORPTION ANALYSIS

Pulse Chemisorption Analysis Tutorial on page 11 - 16

When running an analysis and a CryoCooler is connected to the analyzer, a warning displays indicating that the Dewar level should be full before running an 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

Loop Volume on page 11 - 15

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 Tutorial on page 11 - 13
Loop Volume on page 11 - 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.
Sorption or Cold Trap	The trap does not need to be cooled during the reference material analysis. This may vary depending on application or analysis method. The trap tube should remain on the instrument. Including the trap in the flow path is optional.
Pressure regulator	Gas cylinders should be set to a level between 14 and 16 psig (95 and 110 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 5 - 5***.

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.
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 on page 6 - 2***.

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 kit 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 on page 6 - 2

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.

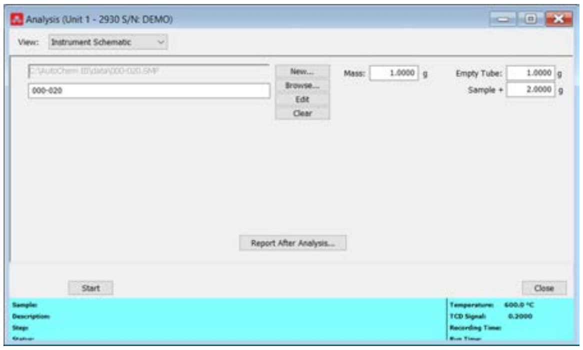
PERFORM A SAMPLE ANALYSIS

Unit [n] > Sample Analysis

[Analysis Tutorials on page 11 - 1](#)

> When running an analysis and a CryoCooler is connected to the analyzer, a warning displays indicating that the Dewar level should be full before running an analysis.

1. From the menu bar, select ***Unit [n]*** > ***Sample Analysis***.
2. Click **New** to create a new sample file, or click **Browse** to select an existing file.



Analysis (Unit 1 - 2930 S/N: DEMO)

View: Instrument Schematic

C:\AutoChem III\data\000-020.SMP

000-020

Mass: 1.0000 g

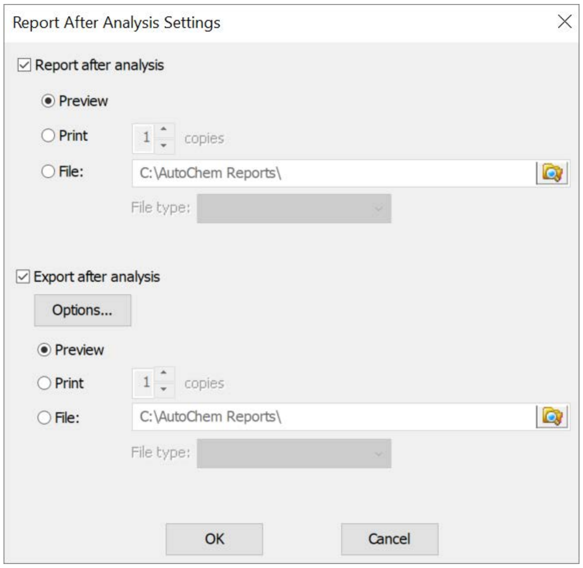
Empty Tube: 1.0000 g

Sample + 2.0000 g

Temperature: 600.0 °C

TCD Signal: 0.2000

3. Edit the *Mass*, *Empty Tube*, and *Sample +* fields, as needed.
4. Click **Report After Analysis** and specify the settings for the generated report.



Report After Analysis Settings

☑ Report after analysis

● Preview

○ Print 1 copies

○ File: C:\AutoChem Reports\

File type:

☑ Export after analysis

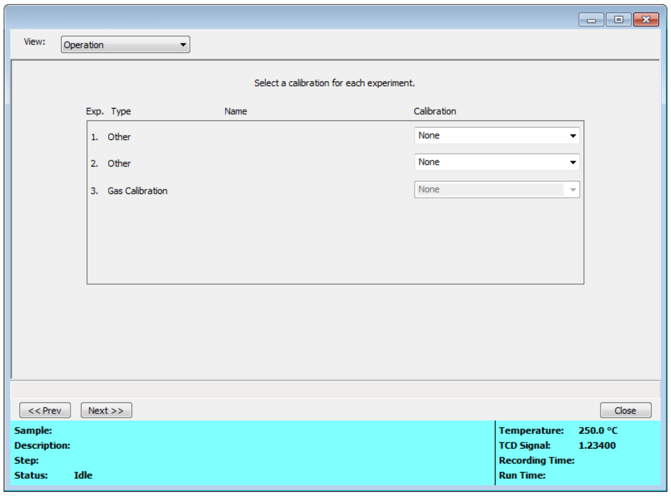
● Preview

○ Print 1 copies

○ File: C:\AutoChem Reports\

File type:

5. If applicable, click **Options** to select specific export options. See *Export Files on page 2 - 38*. Then click **OK**.
6. Click **Start** and select a calibration file for each experiment.



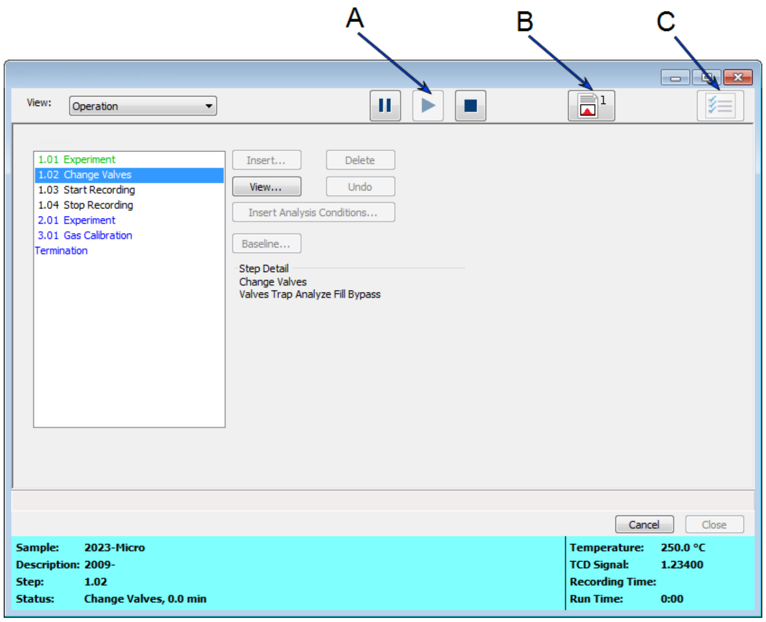
View: Operation

Select a calibration for each experiment.

Exp.	Type	Name	Calibration
1.	Other		None
2.	Other		None
3.	Gas Calibration		None

Sample:
Description:
Step:
Status: Idle
Temperature: 250.0 °C
TCD Signal: 1.23400
Recording Time:
Run Time:

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



View: Operation

1.01 Experiment
1.02 Change Valves
1.03 Start Recording
1.04 Stop Recording
2.01 Experiment
3.01 Gas Calibration
Termination

Step Detail
Change Valves
Valves Trap Analyze Fill Bypass

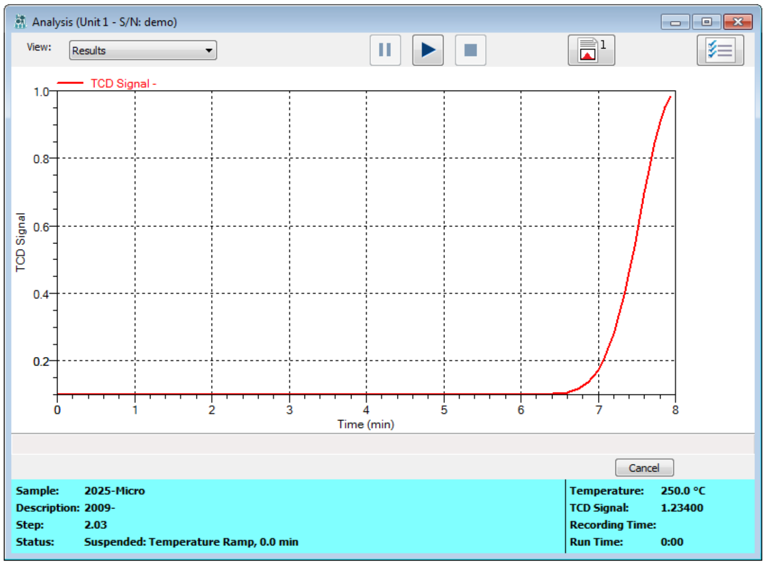
Sample: 2023-Micro
Description: 2009-
Step: 1.02
Status: Change Valves, 0.0 min
Temperature: 250.0 °C
TCD Signal: 1.23400
Recording Time:
Run Time: 0:00

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

The *Operation* view displays experiment steps and details as steps are performed. Text colors indicate the progress. Completed steps are shown in green. In progress steps are shown in light blue. Pending steps are shown in black, dark blue or dark green (depending on their nesting level).

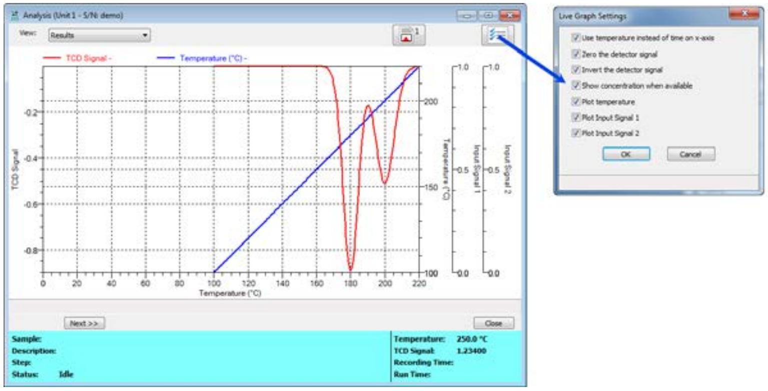
8. To change steps that have not been started:
   - Click **Suspend**, select the step, then click the appropriate button for modification.
   - Click **Resume** to continue the analysis.

9. Select the *Results* view to see the analysis in progress.



Sample: 2025-Micro
Description: 2009-
Step: 2.03
Status: Suspended: Temperature Ramp, 0.0 min
Temperature: 250.0 °C
TCD Signal: 1.23400
Recording Time:
Run Time: 0:00

10. Click the **Live Graph Settings** button to hide or show different settings.



Sample:
Description:
Step:
Status: Idle
Temperature: 250.0 °C
TCD Signal: 1.23400
Recording Time:
Run Time:

☑ Use temperature instead of time on x-axis
☑ Zero the detector signal
☑ Invert the detector signal
☑ Show concentration when available
☑ Plot temperature
☑ Plot Input Signal 1
☑ Plot Input Signal 2

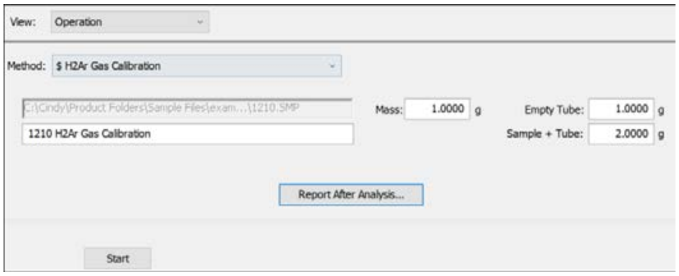
PERFORM A REFERENCE ANALYSIS

Unit [n] > Reference Analysis

[Analysis Tutorials on page 11 - 1](#)

When running an analysis and a CryoCooler is connected to the analyzer, a warning displays indicating that the Dewar level should be full before running an analysis.

1. Select the type of *View* from the drop-down list.
2. Select a *Method* from the drop-down list, or click **Browse** to select an existing file.
3. Edit the *Mass*, *Empty Tube*, and *Sample + Tube* fields, as needed.



View: Operation

Method: $ H2Ar Gas Calibration

C:\Cindy\Product Folders\Sample Files\exam...\1210.SMP

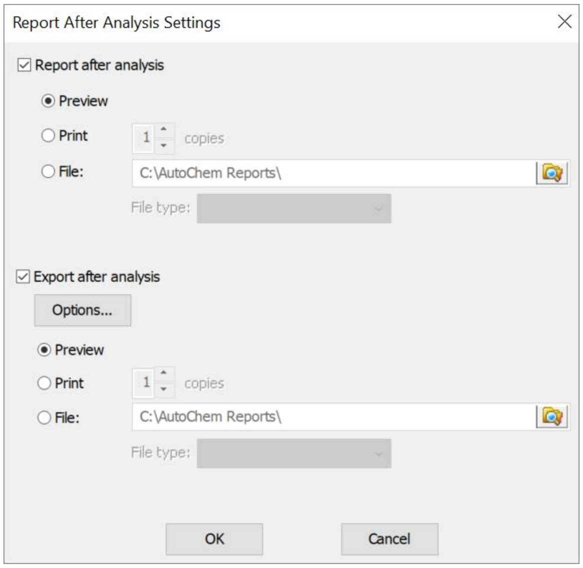
1210 H2Ar Gas Calibration

Mass: 1.0000 g

Empty Tube: 1.0000 g

Sample + Tube: 2.0000 g

4. Click **Report After Analysis** and specify the settings for the generated report.



Report After Analysis Settings

☑ Report after analysis

● Preview

○ Print 1 copies

○ File: C:\AutoChem Reports\

File type:

☑ Export after analysis

● Preview

○ Print 1 copies

○ File: C:\AutoChem Reports\

File type:

5. If applicable, click **Options** to select specific export options. See *[Export Files on page 2 - 38](#)*. Then click **OK**.
6. Click **Start**. The sample file is created and set up automatically.

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6 ABOUT REPORTS

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.

MICROACTIVE REPORTS

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* or *Analyzing* 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 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

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. 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.

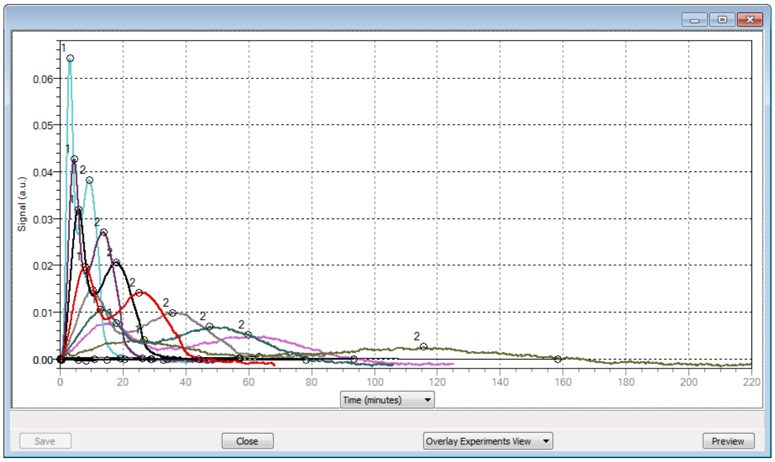
PEAK EDITOR VIEW

Open a Sample File on page 3 - 5

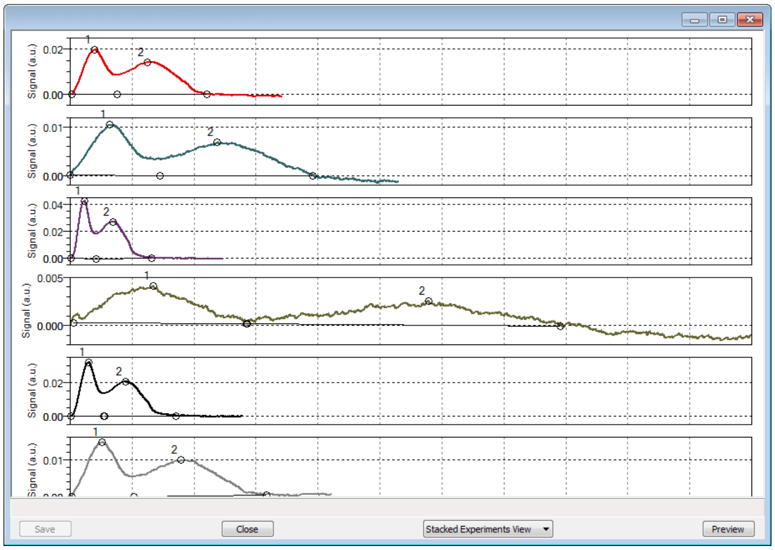
When a sample file with a *Complete* status is opened, three views are available:

- Overlay Experiments View
- Stacked Experiments View
- Peak Editor

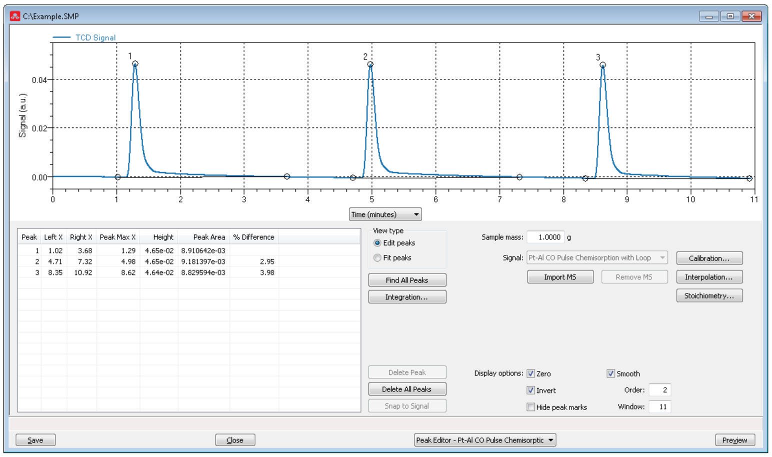
To change the view, select the view from the drop-down list at the bottom of the graph window. Only the *Peak Editor* view allows editing of the experiment.



Example of Overlay Experiments view



Example of Stacked Experiments view



Peak	Left X	Right X	Peak Max X	Height	Peak Area	% Difference
1	1.02	3.68	1.29	4.65e-02	8.910642e-03	
2	4.71	7.32	4.98	4.65e-02	9.181397e-03	2.95
3	8.35	10.92	8.62	4.64e-02	8.829594e-03	3.98

View type
● Edit peaks
○ Fit peaks

Sample mass: 1.0000 g

Signal: Pt-Al CO Pulse Chemisorption with Loop

Display options: ☑ Zero ☑ Invert ☐ Hide peak marks ☑ Smooth

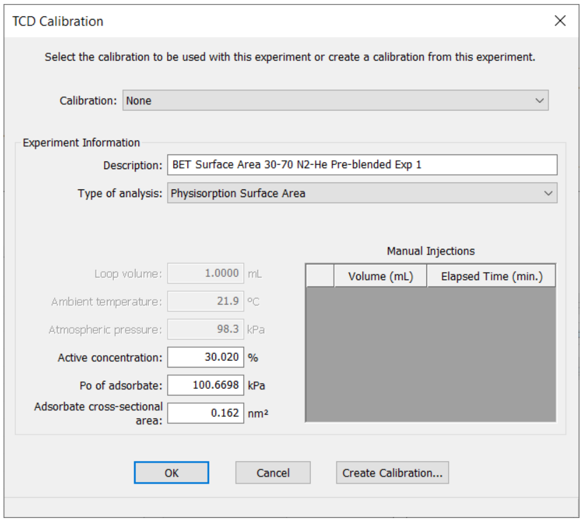
Order: 2

Window: 11

Peak Editor - Pt-Al CO Pulse Chemisorptic

Example of Peak Editor view

Peak Editor

Selections	Description
Calibration [*button*]	 TCD Calibration Select the calibration to be used with this experiment or create a calibration from this experiment. Calibration: None Experiment Information Description: BET Surface Area 30-70 N2-He Pre-blended Exp 1 Type of analysis: Physisorption Surface Area Loop volume: 1.0000 mL Ambient temperature: 21.9 °C Atmospheric pressure: 98.3 kPa Active concentration: 30.020 % Po of adsorbate: 100.6698 kPa Adsorbate cross-sectional area: 0.162 nm² Manual Injections Volume (mL) | Elapsed Time (min.) OK Cancel Create Calibration... **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.

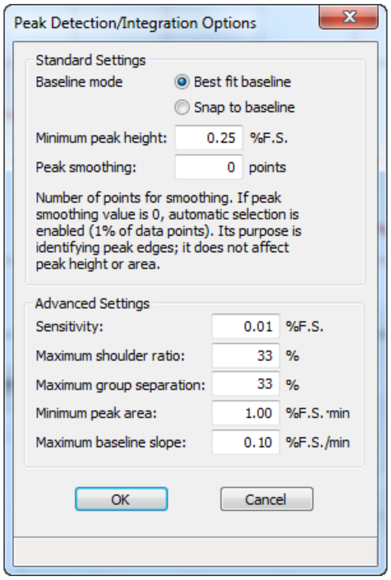
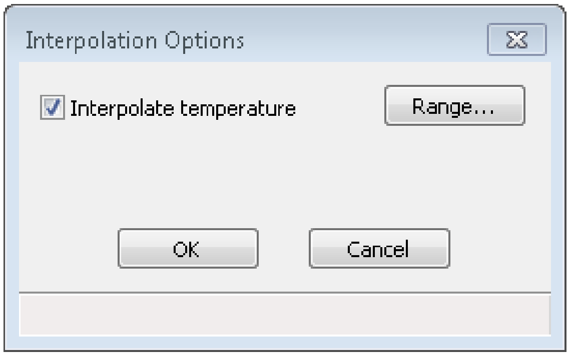
Peak Editor (continued)

Selections	Description
	This field is only visible for vapor experiments. - **Loop volume.** Displays the volume of the gas injection loop. - **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. - **P_0 of adsorbate.** Displays the saturation pressure of the adsorbate. - **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 [*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*]	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. 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*]	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*]	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*]	 Peak Detection/Integration Options Standard Settings Baseline mode ● Best fit baseline ○ Snap to baseline Minimum peak height: 0.25 %F.S. Peak smoothing: 0 points Number of points for smoothing. If peak smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges; it does not affect peak height or area. Advanced Settings Sensitivity: 0.01 %F.S. Maximum shoulder ratio: 33 % Maximum group separation: 33 % Minimum peak area: 1.00 %F.S.·min Maximum baseline slope: 0.10 %F.S./min See ***Peak Detection / Integration Options on page D - 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. **Suppress injection peaks**. Select to remove injection artifact spikes from the TCD signal during *Loop Calibrations* and *Dose* steps. The signal is linearly interpolated during a short time interval after the start and end of each injection.  Interpolation Options ☑ Interpolate temperature
Log Normal Skewed [*button*]	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*]	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).

Peak Editor (continued)

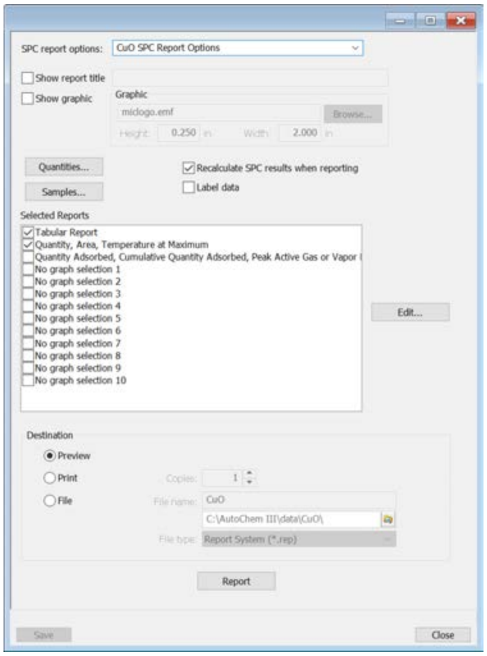
Selections	Description
Sample mass **[***text box***]**	Calculates the mass-specific quantities. Initial value is taken from the sample information at the start of the analysis.
Snap to Signal **[***button***]**	Places the selected baseline point on the signal curve.
Stoichiometry **[***button***]**	See ***Active Metals for Chemisorption Analyzers on page 3 - 6***.
View Type **[***group box***]**	**Edit Peak.** Locates peaks via signal integration over a baseline. **Fit Peaks.** Locates peaks via function fitting to minimize residual over a baseline.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

SPC REPORT

Reports > Open SPC 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.



SPC report options: CuO SPC Report Options

☐ Show report title

☐ Show graphic

Graphic: midogo.emf

Height: 0.250 in Width: 2.000 in

☑ Recalculate SPC results when reporting

☐ Label data

Selected Reports

- ☑ Tabular Report
- ☑ Quantity, Area, Temperature at Maximum
- ☐ Quantity Adsorbed, Cumulative Quantity Adsorbed, Peak Active Gas or Vapor
- ☐ No graph selection 1
- ☐ No graph selection 2
- ☐ No graph selection 3
- ☐ No graph selection 4
- ☐ No graph selection 5
- ☐ No graph selection 6
- ☐ No graph selection 7
- ☐ No graph selection 8
- ☐ No graph selection 9
- ☐ No graph selection 10

Destination

- ● Preview
- ○ Print
- ○ File

Copies: 1

File name: CuO

C:\AutoChem III\data\CuO\

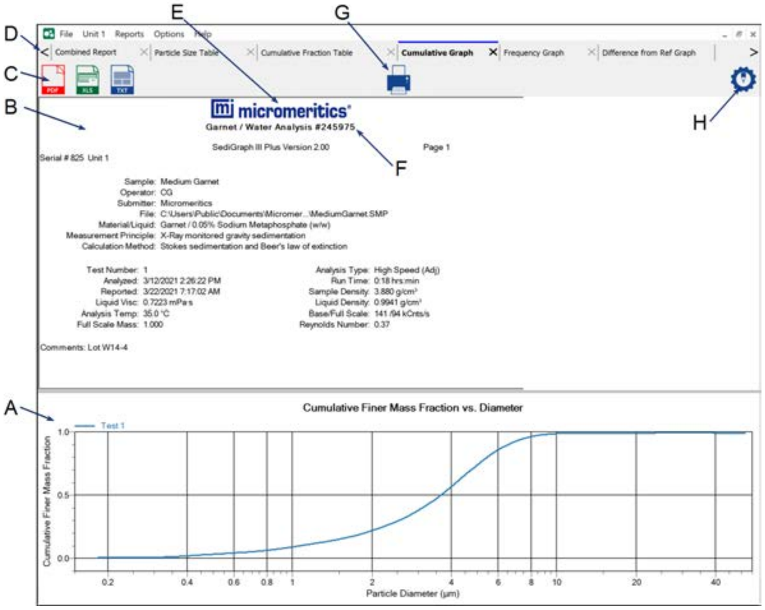
File type: Report System (*.rep)

REPORT FEATURES

Report Style on page 2 - 40

Click **Preview** on the MicroActive report window to display additional report information.

- 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.
- 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.
- Summary report headers contain the same information as tabular and graphical reports with the exception of notes.



Garnet / Water Analysis #245975

SediGraph III Plus Version 2.00

Page 1

Serial # 825 Unit 1

Sample: Medium Garnet
Operator: CG
Submitter: Micromeritics
File: C:\Users\Public\Documents\Micromer...\MediumGarnet.SMP
Material/Liquid: Garnet / 0.05% Sodium Metaphosphate (w/w)
Measurement Principle: X-Ray monitored gravity sedimentation
Calculation Method: Stokes sedimentation and Beer's law of extinction

Test Number: 1
Analyzed: 3/12/2021 2:26:22 PM
Reported: 3/22/2021 7:17:02 AM
Liquid Visc: 0.7223 mPa·s
Analysis Temp: 35.0 °C
Full Scale Mass: 1.000

Analysis Type: High Speed (Adj)
Run Time: 0:18 hrs:min
Sample Density: 3.880 g/cm³
Liquid Density: 0.9941 g/cm³
Base/Full Scale: 141 /94 kCnts/s
Reynolds Number: 0.37

Comments: Lot W14-4

A. Data display (graph or text)
B. Report header area
C. Report export options
D. Generated tabs
E. Report graphic
F. Title
G. Print icon
H. Style icon

Report Features and Shortcuts

Selections	Description
Data Display area	Displays either a graphic or text from the generated report. Click the tabs across the top to display the selected report.
Generated tabs	Tabs represent the reports selected on the *Report Options* window.
Print icon	Click to send the report to a designated printer.
Report export options	Select a file format option for the exported report details.

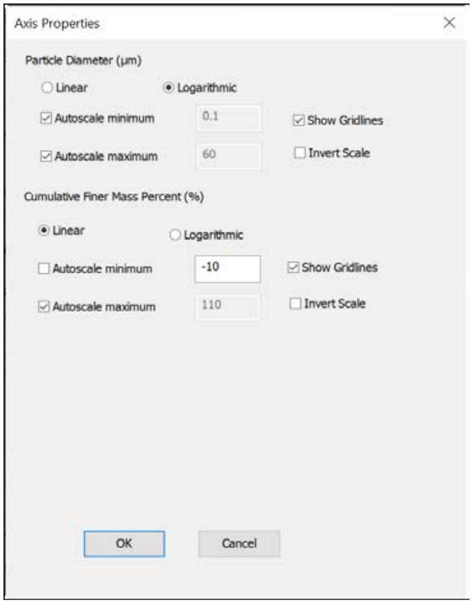
Report Features and Shortcuts (continued)

Selections	**Description**
Report graphic	A graphic can be displayed on the report by using the **Style** button.
Report header area	Displays information about the sample, instrument, and software.
Title	A report title can be displayed on the report by using the **Style** button.
Style icon	Click to customize report options, such as font size, graphics, report title, etc.

REPORT SHORTCUTS

Right-click in the graphic or tabular report area to display report shortcuts.

Report Shortcuts

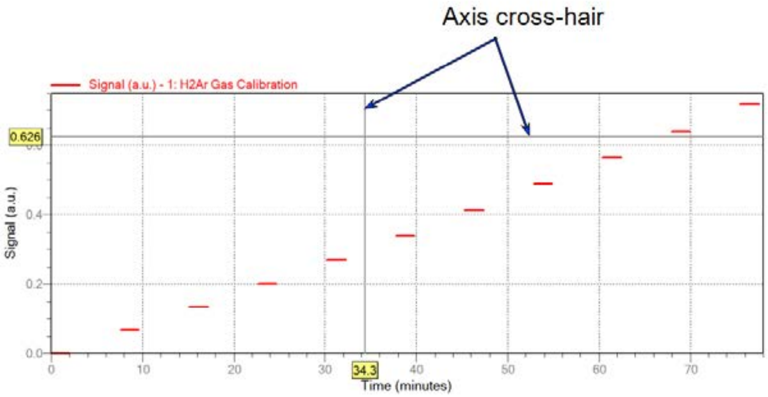
Selections	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy data	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns or copied as an overlay onto another graph.
Copy plot	Copies the graph to the clipboard. It can then be pasted into other software programs.
Copy table	Copies the table to the clipboard.
Edit curve	Edits available curves from the report.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Set scaling options	Edits the axis properties.  Axis Properties Particle Diameter (µm) ○ Linear ● Logarithmic ☑ Autoscale minimum 0.1 ☑ Show Gridlines ☑ Autoscale maximum 60 ☐ Invert Scale Cumulative Finer Mass Percent (%) ● Linear ○ Logarithmic ☐ Autoscale minimum -10 ☑ Show Gridlines ☑ Autoscale maximum 110 ☐ Invert Scale **Linear / Logarithmic.** [*button*] Select the option to scale the graph as linear or logarithmic. **Autoscale minimum / maximum.** [*check box*] To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box.

Report Shortcuts (continued)

Selections	Description
	Show Gridlines. [*check box*] Use to enable or disable report gridlines. **Invert scale.** [*check box*] Use to invert the scale.
Show curve	Displays a list of all curves. Select the curve(s) to display.
Show legend	Displays or hides the legend.

AXIS CROSS-HAIR

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



Example of
Axis cross-hair feature

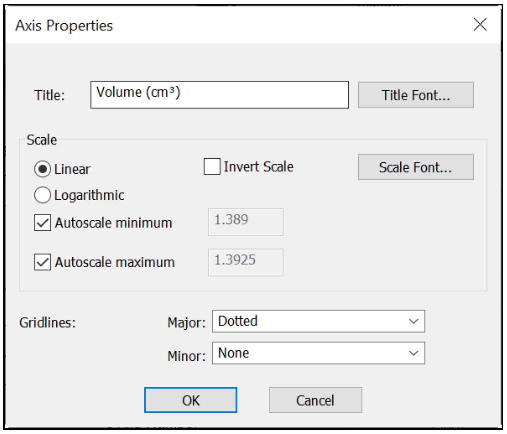
ZOOM FEATURE

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*.

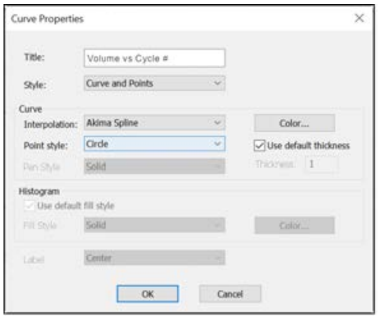
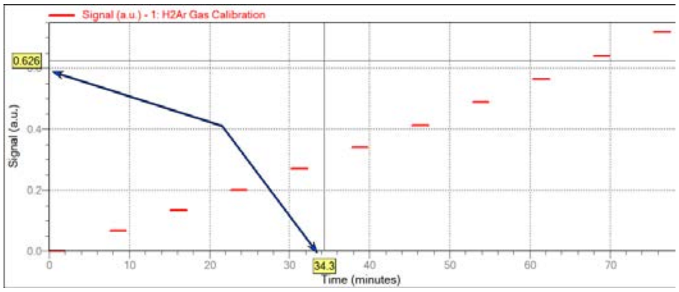
GRAPH FEATURES AND SHORTCUTS

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

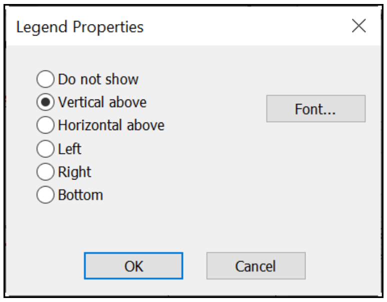
Graph Shortcut Options

Selections	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy graph	Copies the graph to the clipboard. It can then be pasted into other software programs.
Copy plot	Copies the plot onto the clipboard. It can then be pasted into other software programs.
Edit axis	Edits the selected axis properties.  Axis Properties Title: Volume (cm³) Scale ● Linear ○ Logarithmic ☐ Invert Scale ☑ Autoscale minimum 1.389 ☑ Autoscale maximum 1.3925 Gridlines: Major: Dotted Minor: None **Gridlines.** Changes how to display major / minor grid lines. **Scale.** - **Autoscale minimum/maximum.** To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. - **Invert scale.** Inverts the scale. - **Linear/Logarithmic.** Scales the graph as linear or logarithmic. - **Scale font.** Modifies the font for the scale label. Deselect *Use default font* to enable font options. **Title.** Edits the selected axis label. **Title font.** Modifies the font for the selected axis label. Deselect *Use default font*. Select new font attributes for report data. Enable *Use default font* to reset default fonts.

Graph Shortcut Options (continued)

Selections	Description
Edit curve	Edits selected curve properties.  Curve Properties Title: Volume vs Cycle # Style: Curve and Points Curve Interpolation: Akima Spline Point style: Circle ☑ Use default thickness Pen Style: Solid Thickness: 1 Histogram ☑ Use default fill style Fill Style: Solid Label: Center **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.

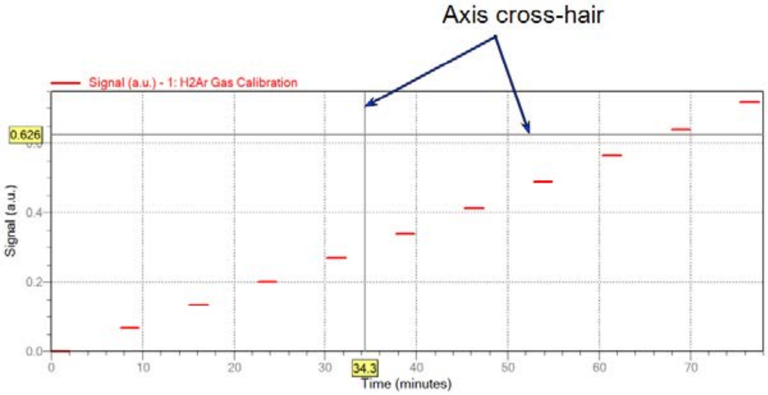
Graph Shortcut Options (continued)

Selections	Description
Edit legend	Changes the legend location and font.  Legend Properties ○ Do not show ● Vertical above ○ Horizontal above ○ Left ○ Right ○ Bottom
Edit title	Changes the report title.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
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 - 6*.

AXIS CROSS-HAIR

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



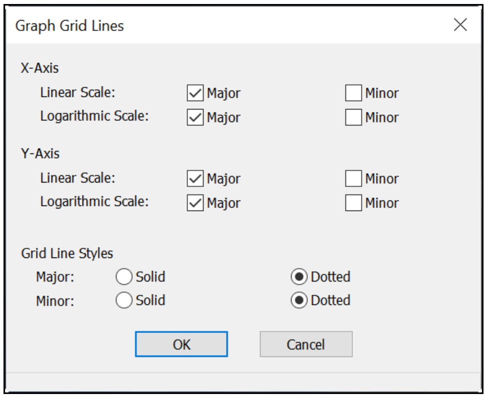
Example of
Axis cross-hair feature

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



Graph Grid Lines

X-Axis

Linear Scale: ☑ Major ☐ Minor

Logarithmic Scale: ☑ Major ☐ Minor

Y-Axis

Linear Scale: ☑ Major ☐ Minor

Logarithmic Scale: ☑ Major ☐ Minor

Grid Line Styles

Major: ○ Solid ● Dotted

Minor: ○ Solid ● Dotted

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

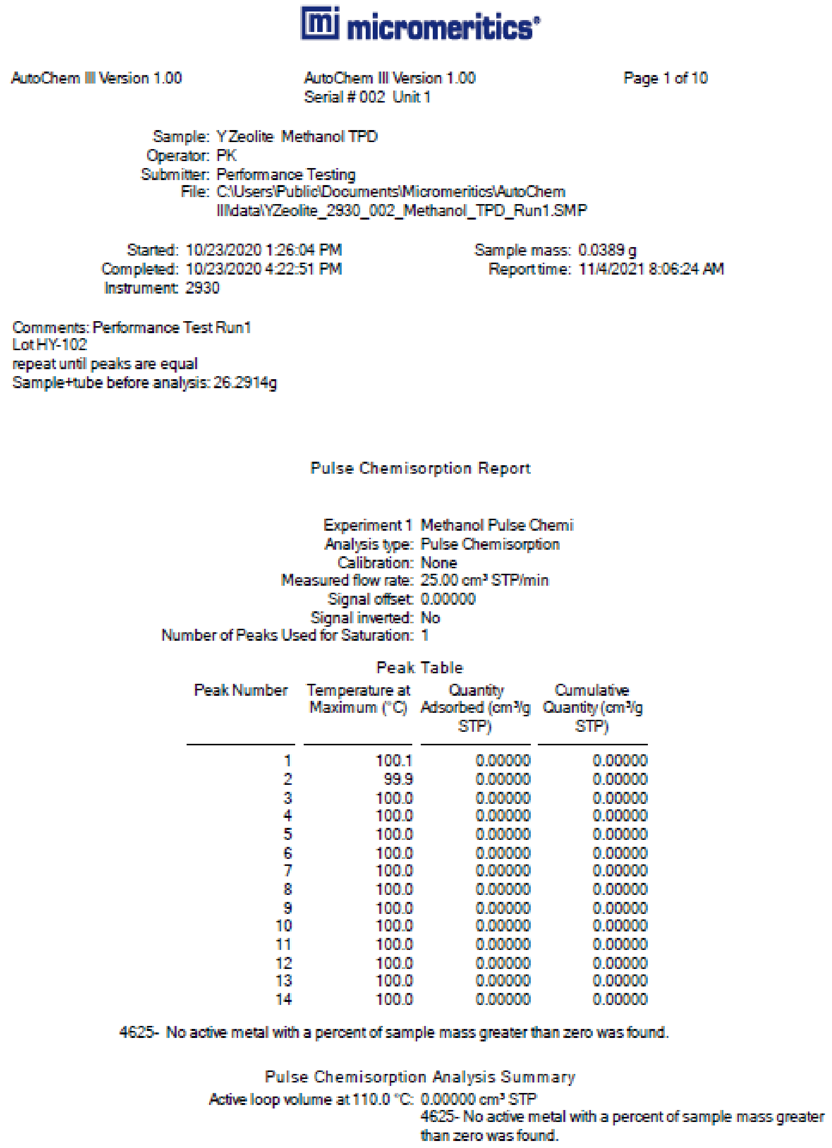
Graph Grid Lines

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 - 6*.

REPORT EXAMPLES

PULSE CHEMISORPTION REPORT



AutoChem III Version 1.00

AutoChem III Version 1.00
Serial # 002 Unit 1

Page 1 of 10

Sample: Y Zeolite Methanol TPD
Operator: PK
Submitter: Performance Testing
File: C:\Users\Public\Documents\Micromeritics\AutoChem III\data\YZeolite_2930_002_Methanol_TPD_Run1.SMP

Started: 10/23/2020 1:26:04 PM
Completed: 10/23/2020 4:22:51 PM
Instrument: 2930

Sample mass: 0.0389 g
Report time: 11/4/2021 8:06:24 AM

Comments: Performance Test Run1
Lot HY-102
repeat until peaks are equal
Sample+tube before analysis: 26.2914g

Pulse Chemisorption Report

Experiment 1 Methanol Pulse Chemi
Analysis type: Pulse Chemisorption
Calibration: None
Measured flow rate: 25.00 cm³ STP/min
Signal offset: 0.00000
Signal inverted: No
Number of Peaks Used for Saturation: 1

Peak Table

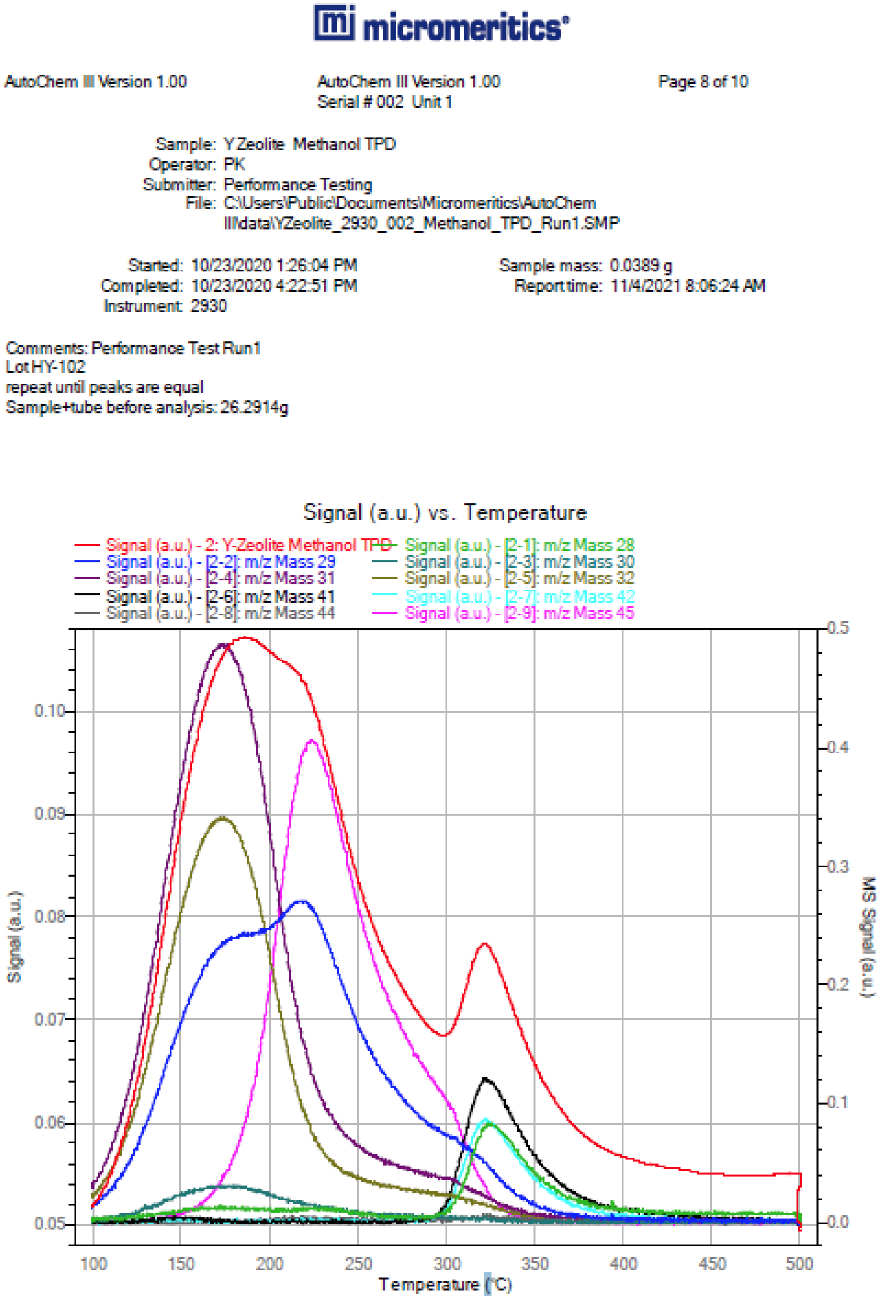
Peak Number	Temperature at Maximum (°C)	Quantity Adsorbed (cm³/g STP)	Cumulative Quantity (cm³/g STP)
1	100.1	0.00000	0.00000
2	99.9	0.00000	0.00000
3	100.0	0.00000	0.00000
4	100.0	0.00000	0.00000
5	100.0	0.00000	0.00000
6	100.0	0.00000	0.00000
7	100.0	0.00000	0.00000
8	100.0	0.00000	0.00000
9	100.0	0.00000	0.00000
10	100.0	0.00000	0.00000
11	100.0	0.00000	0.00000
12	100.0	0.00000	0.00000
13	100.0	0.00000	0.00000
14	100.0	0.00000	0.00000

4625- No active metal with a percent of sample mass greater than zero was found.

Pulse Chemisorption Analysis Summary

Active loop volume at 110.0 °C: 0.00000 cm³ STP
4625- No active metal with a percent of sample mass greater than zero was found.

SIGNAL VS TEMPERATURE REPORT



AutoChem III Version 1.00

AutoChem III Version 1.00
Serial # 002 Unit 1

Page 8 of 10

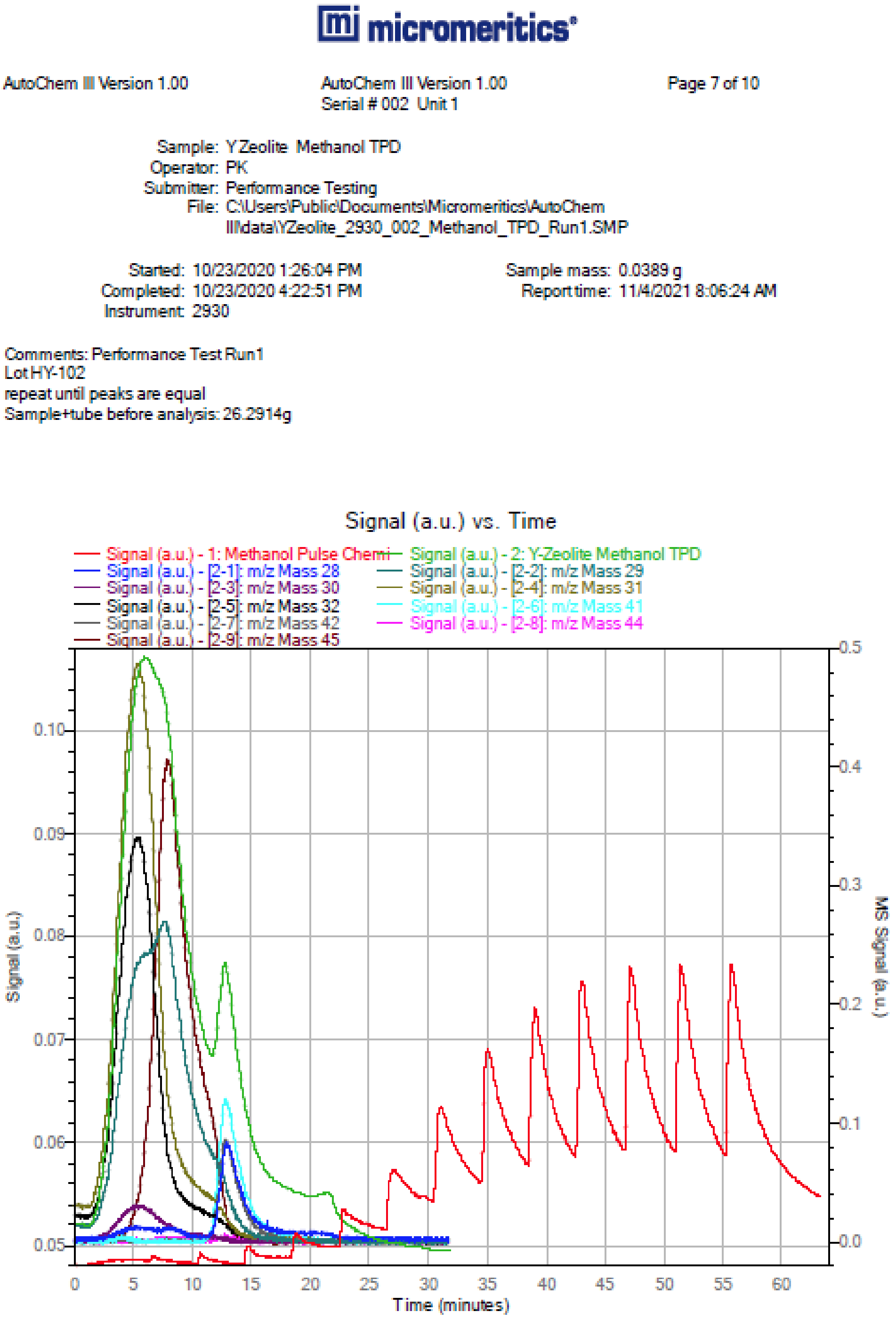
Sample: Y Zeolite Methanol TPD
Operator: PK
Submitter: Performance Testing
File: C:\Users\Public\Documents\Micromeritics\AutoChem III\data\YZeolite_2930_002_Methanol_TPD_Run1.SMP

Started: 10/23/2020 1:26:04 PM
Completed: 10/23/2020 4:22:51 PM
Instrument: 2930

Sample mass: 0.0389 g
Report time: 11/4/2021 8:06:24 AM

Comments: Performance Test Run1
Lot HY-102
repeat until peaks are equal
Sample+tube before analysis: 26.2914g

SIGNAL VSTIME REPORT



AutoChem III Version 1.00

AutoChem III Version 1.00
Serial # 002 Unit 1

Page 7 of 10

Sample: Y Zeolite Methanol TPD
Operator: PK
Submitter: Performance Testing
File: C:\Users\Public\Documents\Micromeritics\AutoChem III\data\YZeolite_2930_002_Methanol_TPD_Run1.SMP

Started: 10/23/2020 1:26:04 PM
Completed: 10/23/2020 4:22:51 PM
Instrument: 2930

Sample mass: 0.0389 g
Report time: 11/4/2021 8:06:24 AM

Comments: Performance Test Run1
Lot HY-102
repeat until peaks are equal
Sample+tube before analysis: 26.2914g

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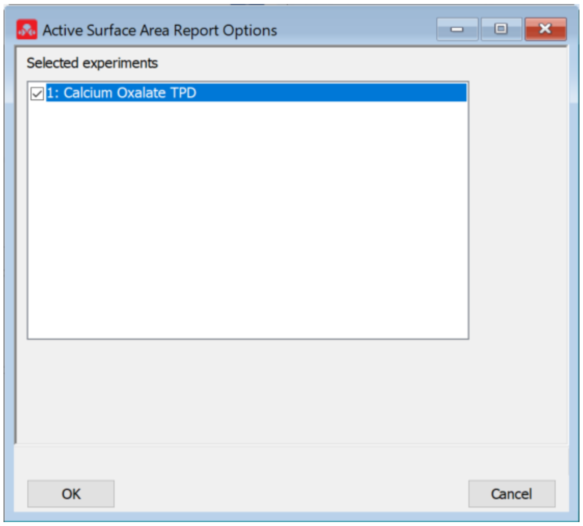
7 SELECTED REPORTS

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**.

ACTIVE SURFACE AREA REPORT

Use for temperature programmed or other experiment types where the gas quantity is determined from peak area by application of a calibration, from which active surface quantities can then be calculated. These quantities include:

- Metal Dispersion
- Metallic Surface Area
- Crystallite Size



☑ 1: Calcium Oxalate TPD

Active Surface Area 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 - 6*.

ADVANCED REPORTS - PYTHON MODULE

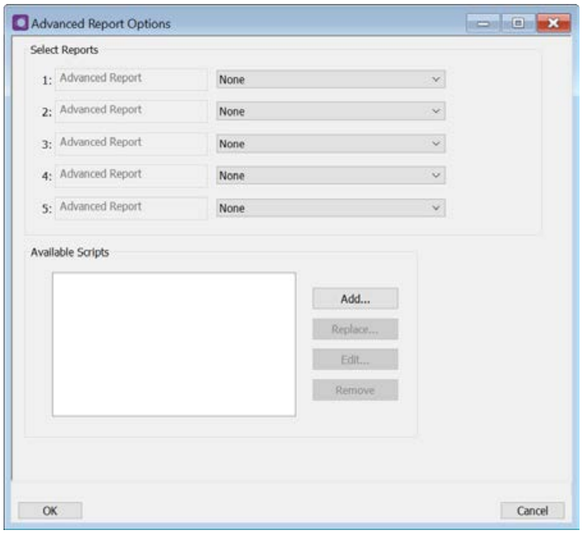
- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data. Tabular reports are created 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 REPORTS

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 - 11*** 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.



1: Advanced Report None
2: Advanced Report None
3: Advanced Report None
4: Advanced Report None
5: Advanced Report None

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.

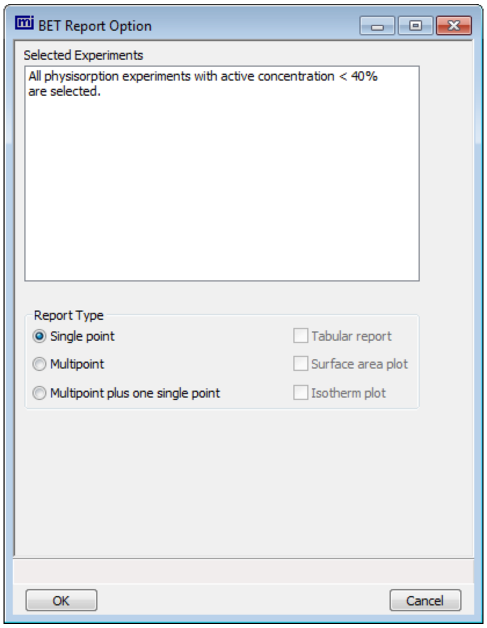
7. On the *Report Options* tab, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.

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.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

BET SURFACE AREA REPORT



All physisorption experiments with active concentration < 40% are selected.

● Single point
○ Multipoint
○ Multipoint plus one single point

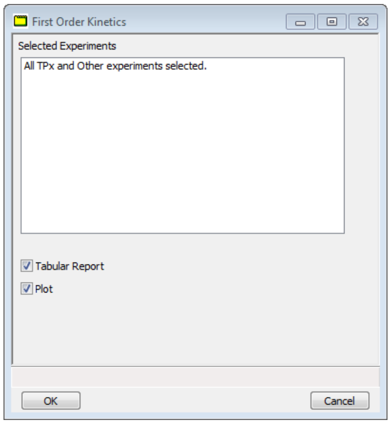
☐ Tabular report
☐ Surface area plot
☐ Isotherm plot

BET Report

Selections	**Description**
Selected Experiments [*group box*]	Displays all related experiments in the current file. Deselect report options to exclude from the experiment. If this is a new file, no experiments are listed. Experiments are not included in the list until BET data are collected.
Report Type [*group box*]	Select the report types to generate.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

FIRST ORDER KINETICS REPORT



All TPx and Other experiments selected.

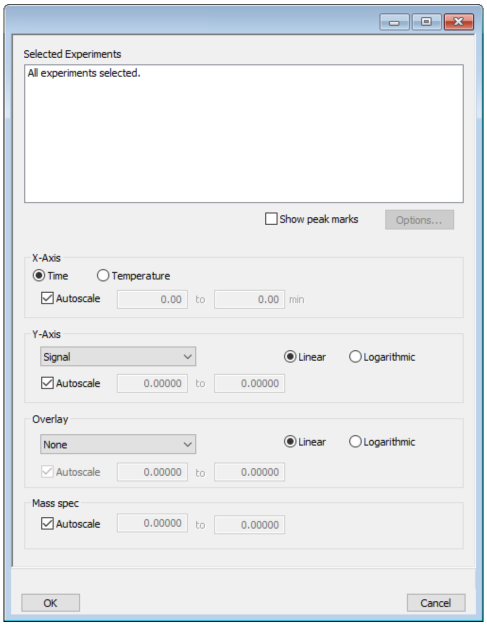
First Order Kinetics Report

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 - 6*.

GRAPH REPORT OPTIONS

- Active Gas Flow Rate and Temperature vs Time
- Active Gas Flow Rate vs Temperature
- Active Gas Flow Rate vs Time
- Signal and Active Gas Flow Rate vs Temperature
- Signal and Active Gas Flow Rate vs Time
- Signal and Temperature vs Time
- Signal vs Temperature
- Signal vs Time [*n*]
- Temperature vs Time



Selected Experiments

All experiments selected.

☐ Show peak marks

X-Axis

● Time ○ Temperature

☑ Autoscale 0.00 to 0.00 min

Y-Axis

Signal ● Linear ○ Logarithmic

☑ Autoscale 0.00000 to 0.00000

Overlay

None ● Linear ○ Logarithmic

☑ Autoscale 0.00000 to 0.00000

Mass spec

☑ Autoscale 0.00000 to 0.00000

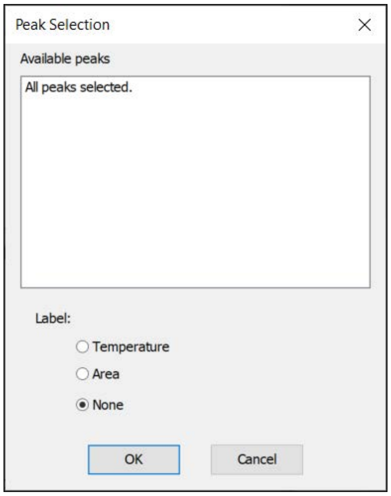
Any signal (the detector signal or the 1 or 2 auxiliary signal) may be plotted against time or temperature. Any signal can be overlaid onto the primary signal.

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
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)

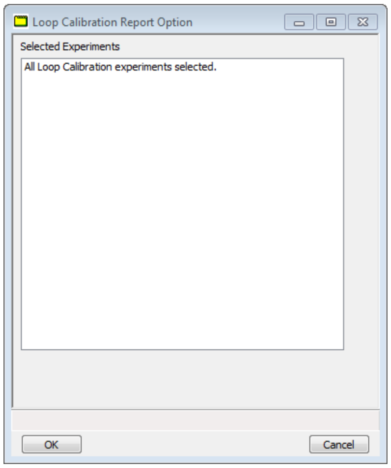
Selections	**Description**
Options [*button*]	Enabled when the *Show peak marks* option is selected. Lists the peaks for the results of the highlighted experiment. Select any (or all) peaks to include in the graph.  Peak Selection Available peaks All peaks selected. Label: ○ Temperature ○ Area ● None 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 - 6*.

LOOP CALIBRATION REPORT



All Loop Calibration experiments selected.

Loop Calibration Report

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

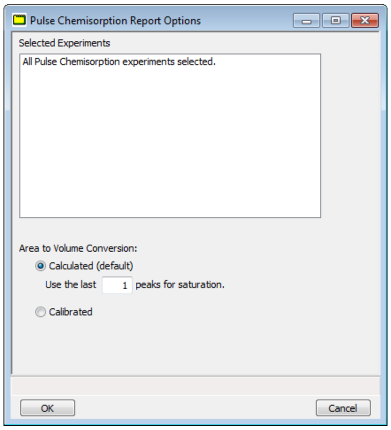
For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

OPTIONS REPORT

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

- Active metals
- Analysis conditions
- Report options
- Sample information

PULSE CHEMISORPTION REPORT



Selected Experiments

All Pulse Chemisorption experiments selected.

Area to Volume Conversion:

● Calculated (default)

Use the last 1 peaks for saturation.

○ Calibrated

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 - 6*.

Quantity Adsorbed Formulas

Situation 1	Pulse adsorption experiment, no calibration applied in peak editor, report option is *Calculated*. $Qads_{\mathrm{x}} = \frac{L-(L\times\frac{A_{\mathrm{x}}}{Avg_{\mathrm{lastN}}})}{Mass_{sample}}$
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 $\text{Mass}_{\text{sample}}$ 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 LOG REPORT

Sample Log reports cannot be edited.

Inserts a log of sample operations in the reports.

This report provides information on:

- Manual control operations performed during analysis.
- Information entered using *Add Log Entry* on the sample file editor.
- Warnings and/or errors which occurred during analysis.

SUMMARY REPORT

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

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8 HARDWARE

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).

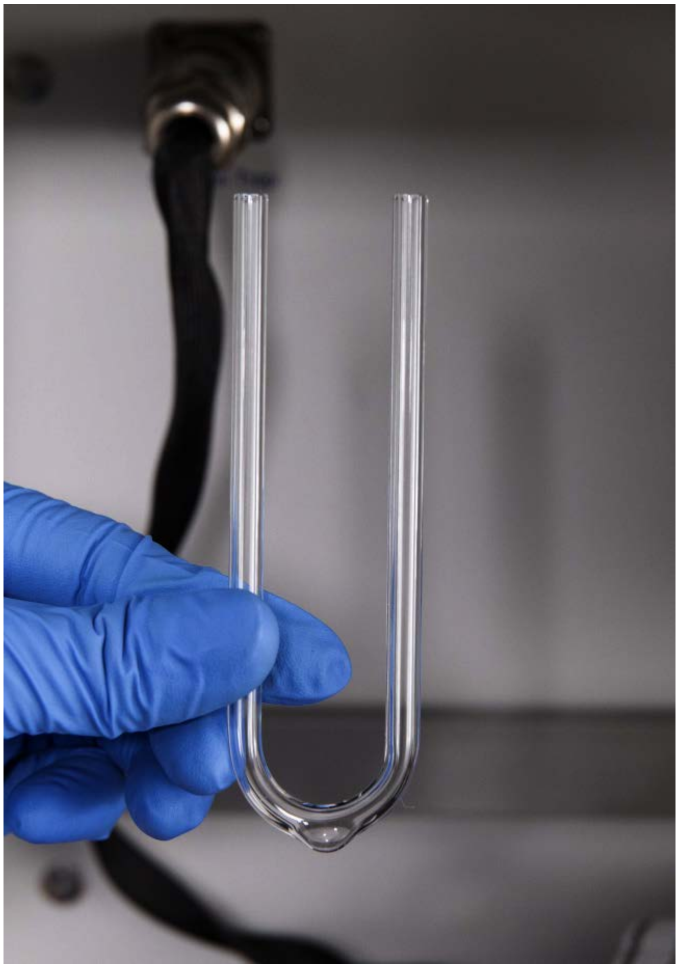
- CryoCooler II and CryoCooler III Operator Manual (PDF)
- Error Messages document (PDF)
- Parts and Accessories

COLD TRAP

Dewar Precautions on page 5 - 3

Parts and accessories are located on the Micromeritics web page.

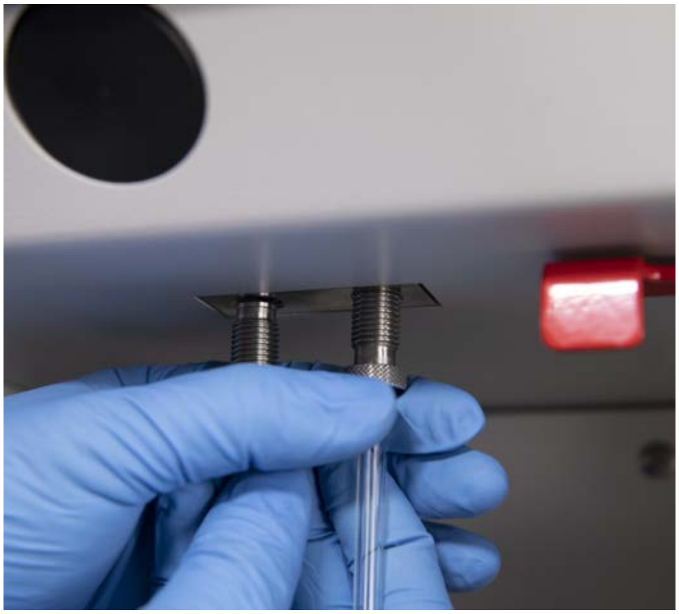
A *cold trap* is an available accessory used to condense liquids out of the gases flowing through the analyzer before reaching the detector. The use of a Dewar reduces the temperature of the cold trap.



Cold trap tube

1. On both ends of the tube, insert the retaining nut (knurled end first), the ferrule (small end first), and O-ring. Leave the O-rings close to the top of the tube.

2. Insert the open ends of the tube into the cold trap ports under the upper panel of the analyzer.



3. Press the tube up into the analyzer until it comes to a stop. Hold the tube in place and tighten the retaining nuts by hand. Tighten the nuts only enough to hold the tube securely in place and prevent leaks.

CAUTION Do not overtighten the retaining nuts. A sufficient seal is achieved when the fittings are finger tight.

4. Fill the Dewar with the appropriate coolant.
5. Carefully slide the Dewar over the cold trap tube.

6. Slide the shelf underneath the Dewar and hang the shelf on the bracket behind the Dewar.
7. Rest the Dewar on the shelf.

8. Place the two halves of the Dewar covers on the top of the Dewar and around the cold trap tube.

CHANGE OR CLEAN THE COLD TRAP TUBE

Occasional cleaning of the tubes is required.

1. If a Dewar is around the cold trap tube, raise the Dewar slightly and remove the Dewar shelf. Then remove the Dewar. Allow the tube to warm to room temperature.

> Depending upon the state of the analyzer, the tube fittings may be hot. Use caution. Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

2. To remove the tube, loosen the retaining nuts on the port fittings.
3. Remove the tube, retaining nuts, ferrules, and O-rings.
4. Clean the tube by flushing with isopropyl alcohol (IPA). Dry the tube in an oven or by blowing compressed air or nitrogen through it before replacing it.
5. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the tube. Otherwise, clean, dry, and re-install the O-rings.

> CAUTION
>
> Use only Kalrez O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.

6. On both ends of the tubes, insert the retaining nut (knurled end first), the ferrule (small end first), and O-ring. Leave the O-rings close to the top of the tube.

> CAUTION
>
> Do not over tighten the retaining nuts. Doing so may damage the O-rings. Finger tight is sufficient.

7. Hold the tube in place and tighten the retaining nuts. Tighten the nuts only enough to hold the tube securely in place and prevent leaks.

DELAY PATH

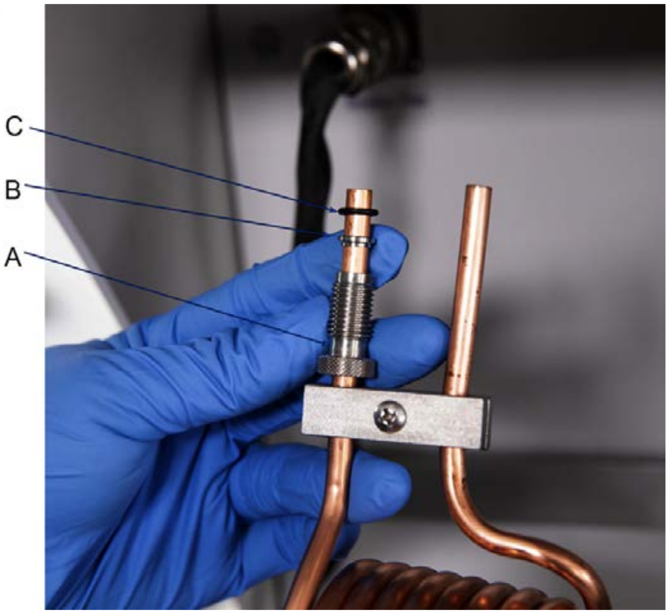
[Dewar Precautions on page 5 - 3](#)

Parts and accessories are located on the Micromeritics web page.

A *delay path* is used to reduce the perturbation (disturbance) of the gas flow caused by injections of gas through the septum.

Delay path tube

1. On both ends of the tube, insert the retaining nut (knurled end first), the ferrule (small end first), and O-ring. Leave the O-rings close to the top of the tube.



A. Retaining nut (insert knurled end first)
B. O-ring
C. Ferrule (insert small end first)

2. Insert the open ends of the tube into the delay path ports under the upper panel of the analyzer.
3. Press the tube up into the analyzer until it comes to a stop. Hold the tube in place and tighten the retaining nuts by hand. Tighten the nuts only enough to hold the tube securely in place and prevent leaks.

CAUTION Do not overtighten the retaining nuts. A sufficient seal is achieved when the fittings are finger tight.

4. Fill the Dewar with the appropriate coolant.

CHANGE OR CLEAN THE DELAY PATH TUBE

Occasional cleaning of the tubes is required.

1. If a Dewar is around the delay path tube, raise the Dewar slightly and remove the Dewar shelf. Then remove the Dewar. Allow the tube to warm to room temperature.

> Depending upon the state of the analyzer, the tube fittings may be hot. Use caution. Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

2. To remove the tube, loosen the retaining nuts on the port fittings.
3. Remove the tube, retaining nuts, ferrules, and O-rings.
4. Clean the tube by flushing with isopropyl alcohol (IPA). Dry the tube in an oven or by blowing compressed air or nitrogen through it before replacing it.
5. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the tube. Otherwise, clean, dry, and re-install the O-rings.

> CAUTION
>
> Use only Kalrez O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.

6. On both ends of the tubes, insert the retaining nut (knurled end first), the ferrule (small end first), and O-ring. Leave the O-rings close to the top of the tube.

> CAUTION
>
> Do not over tighten the retaining nuts. Doing so may damage the O-rings. Finger tight is sufficient.

7. Hold the tube in place and tighten the retaining nuts. Tighten the nuts only enough to hold the tube securely in place and prevent leaks.

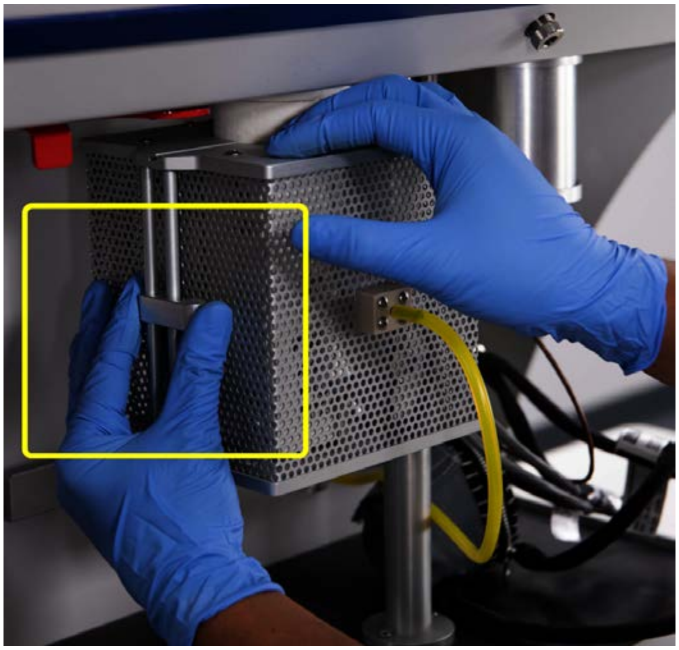
FURNACE

When performing BET analyses, the furnace must be removed to allow placement of a cold bath Dewar around the sample.

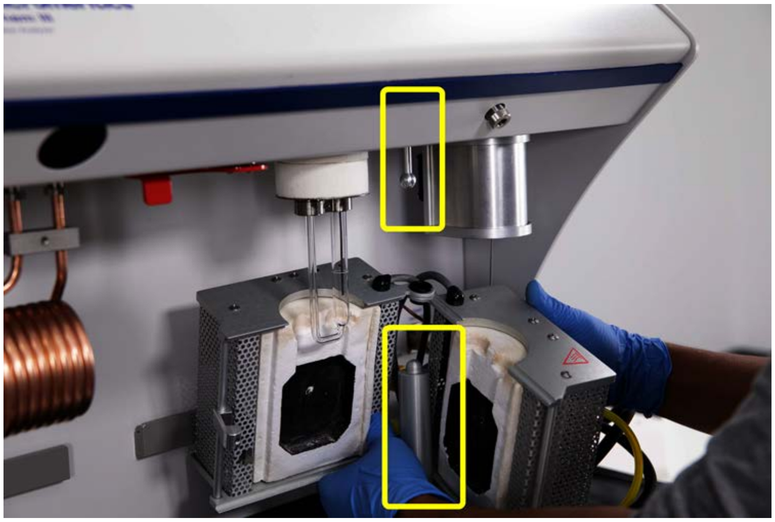
FURNACE REMOVAL

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

1. Unplug the furnace cooling plug, the furnace thermocouple plug, and the furnace power plug from the analyzer.
2. Release the latch on the left side of the furnace, then open the furnace.



3. Gently lift the right side of the furnace until it is free from the support post.

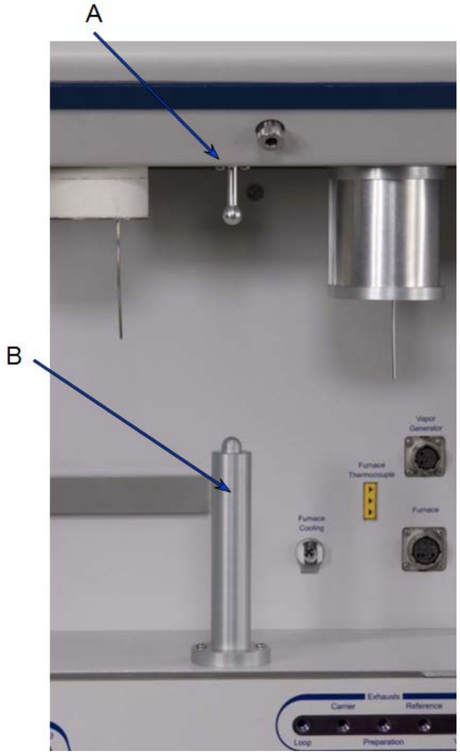


Support post and retaining knob

4. Lower the furnace, moving it away from the support post until it is free of the retaining knob.
5. Close the furnace and secure the furnace latch. Place the furnace in a location where it will be protected from damage.

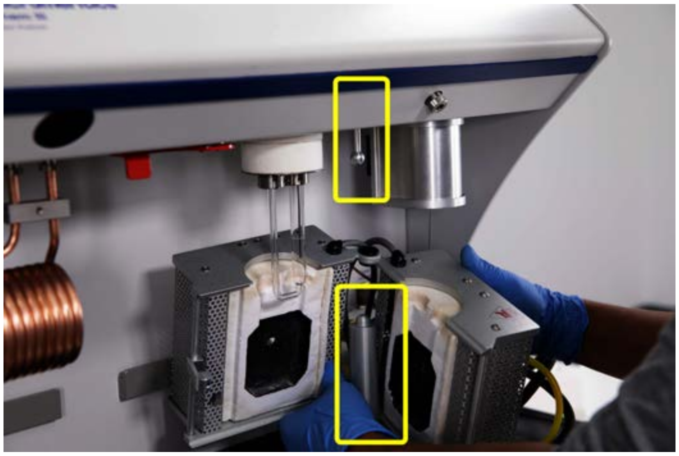
FURNACE INSTALLATION

1. Hold the open furnace next to the furnace support post. Lift the furnace, moving it above the support post until the retaining knob is inserted in the hole in the top of the furnace.



A. Retaining knob
B. Support post

2. When the retaining socket on the bottom of the furnace is located above the support post, gently lower the furnace until it rests securely on the post.



Support post and retaining knob

3. When ready to begin the experiment, close the furnace around the sample tube and secure the latch.
4. Plug the furnace cooling plug, the furnace thermocouple plug, and the furnace power plug into the appropriate connectors on the analyzer. These connectors are located on the recessed front panel.

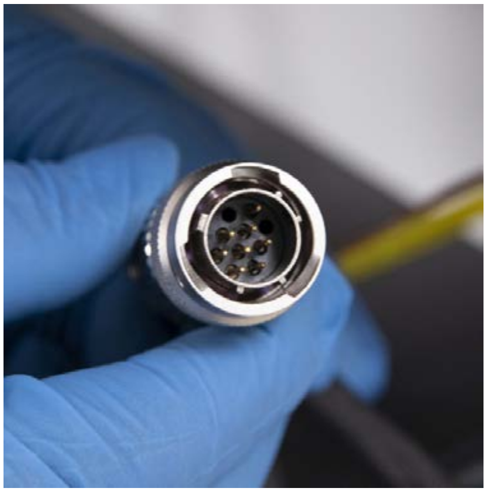
HEATING MANTLE

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

Heating mantle shown installed

The heating mantle plug installs behind the diversion ports on the front panel of the analyzer. If installing the heating mantle with a tube already installed, take extra precaution to ensure that the tube does not break while installing the heating mantle. These instructions install the heating mantle first.

1. Locate the wide tab on the heating mantle plug. This wide tab will be installed in the upper-most position when inserting it into the heating mantle port on the front panel of the analyzer.



Heating Mantle Plug

Sorption Trap Port

2. Push the heating mantle plug firmly into the heating mantle port on the analyzer. When the plug is firmly seated, twist the outer collar of the plug firmly to the right until you feel and hear a snap. This ensures the cable is properly seated in the mantle port.
3. Lay the heating mantle aside until after the sorption trap, cold trap, or delay path is installed.
4. Place the heating mantle around the tube and slide the mantle up as far as it will go.
5. Secure the mantle in place with a mantle clip.

To remove the heating mantle connector, push the twist lock connector in, then turn left.

SAMPLE THERMOCOUPLE

THERMOCOUPLE POSITION

Use the Thermocouple Clamp on page 8-20

The thermocouple must be attached to the analyzer prior to running an analysis. There are three possible configurations for the thermocouple:

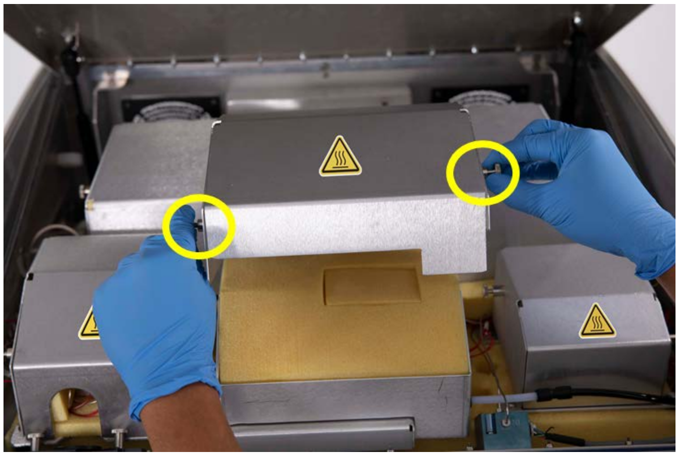
- inside the sample tube
- inside the sample tube surrounded by a quartz sheath
- outside the sample tube

Best accuracy is obtained when the bare thermocouple is placed inside the sample tube. In some cases, one or more of the gases used during the experiment may react with the thermocouple's Inconel material. A quartz sheath is available to protect the thermocouple when the gases or sample used in the experiment may damage it. In this case, the sheath is placed around the thermocouple and the thermocouple is positioned inside the sample tube. When placed inside the tube, the thermocouple can be adjusted so that the end is in the sample or just above it. Always try to position the end of the thermocouple as close to the furnace thermocouple as possible.

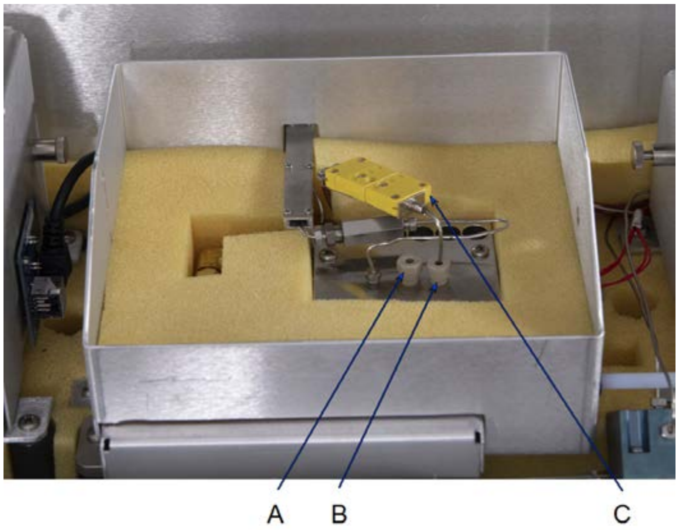
Positioning the thermocouple along the exterior of the sample tube is less accurate than the other configurations. If the thermocouple is placed outside the sample tube, ensure it is clamped against the sample tube, and that the end does not extend below the point where the sample tube begins to taper.

1. Disconnect the analyzer from the power source.
2. Lift the top cover of the analyzer.

3. The valve cover is held in place by two knobs or plungers. Remove the valve cover by pulling the plungers outward until the valve cover is released. Pull the cover up and out.



4. Remove the insulation that surrounds the valve. The thermocouple ports are located toward the front of the block.



A. Left port - use to place the thermocouple outside the tube
B. Right port - use to place the thermocouple inside the tube
C. Thermocouple connector - disconnect to ensure the thermocouple tube does not get bent

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

If the zone is not near room temperature, loosening or tightening the fittings may damage the analyzer components.

5. Disconnect the thermocouple connector.

6. Use an open-ended wrench to loosen the sample thermocouple fitting.

> The sheath is very fragile and can be easily broken. Because the sheath is made of quartz, cotton gloves must be worn when handling the sheath. Oils from fingers may lower the quartz melting point.

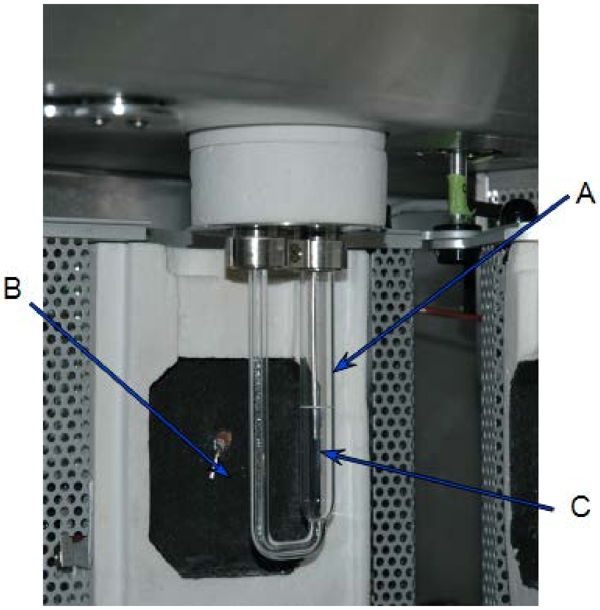
7. Adjust the thermocouple:

To change the position:	a. Loosen the fitting for the plug in the alternate thermocouple opening and remove the plug. b. Remove the fitting that retains the sample thermocouple. c. Move the sample thermocouple from its current position to the new position. d. Place the plug in the unused thermocouple opening and tighten the fitting. Reinstall the sample thermocouple retaining screw. If using the quartz sheath, perform the following before proceeding. The sheath can only be used when the thermocouple is being placed inside the sample tube. i. Remove the ferrule from the thermocouple, straighten the thermocouple as much as possible. ii. Insert the thermocouple into the sheath. iii. Place a larger ferrule around the sheath.
To adjust the length:	See ***Determine the length of the thermocouple on the facing page***. Adjust the thermocouple wire up or down as needed.

8. Tighten the thermocouple fitting.
9. Place the insulation in its original position. Reinstall the valve cover.
10. Return the upper front panel and the top panel to their original closed positions.
11. Power ON the analyzer.

DETERMINE THE LENGTH OF THE THERMOCOUPLE

When adjusting the length of the exposed portion, consider the following:

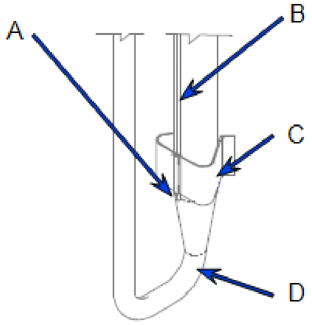


A. Thermocouple
B. Recessed area inside furnace
C. End of thermocouple should be below the line marked on the sample tube

- The end of the thermocouple should be aligned as closely as possible with the furnace thermocouple.
- The end of the thermocouple should never be placed outside the painted (black), recessed area inside the furnace.
- If the thermocouple is positioned inside the tube, its end should generally be located approximately 2 to 3 mm (1/8 in.) below the line marked on the sample tube.
- If the thermocouple is positioned outside the tube, its end should not extend below the point where the sample tube begins to taper.

USE THE THERMOCOUPLE CLAMP

If the thermocouple is placed outside the sample tube, a clamp must be used to secure the thermocouple to the sample tube.



A. Do not allow the thermocouple to extend below the point where the sample begins to taper
B. Thermocouple
C. Clamp
D. Sample tube

RECALIBRATE THE SAMPLE THERMOCOUPLE

Calibration on page 13 - 1

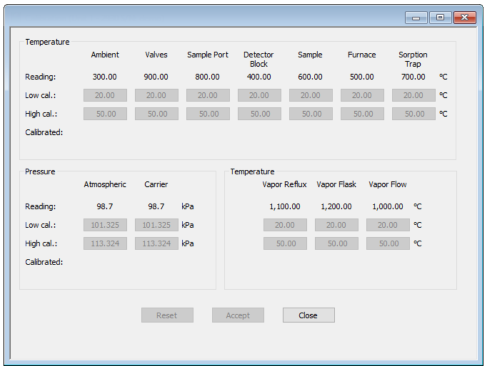
A calibration file is created specifically for each analyzer before the analyzer is shipped. This calibration file is installed during installation. If the original calibration file is corrupted or missing, or if the sample thermocouple is replaced, recalibration will be needed.

> **CAUTION** Do not attempt to calibrate anything other than the sample thermocouple without prior approval from a qualified Micromeritics Service Representative.

Prior to recalibration, ensure:

- The analyzer is powered ON and the application is running.
- All heat zones to be calibrated are stabilized at ambient temperature.
- Familiarity with manually controlling the analyzer.
- All gas flows through the analyzer are stopped.
- The furnace is open, and the sample tube is removed (the thermocouple is bare and exposed).
- A traceable thermocouple meter and a water bath (ambient temperature) to immerse the sample thermocouple are available.

1. Go to ***Unit [n] > Calibration > Calibrate***.



Temperature

	Ambient	Valves	Sample Port	Detector Block	Sample	Furnace	Sorption Trap	
Reading:	300.00	900.00	800.00	400.00	600.00	500.00	700.00	°C
Low cal.:	20.00	20.00	20.00	20.00	20.00	20.00	20.00	°C
High cal.:	50.00	50.00	50.00	50.00	50.00	50.00	50.00	°C
Calibrated:								

Pressure

	Atmospheric	Carrier	
Reading:	98.7	98.7	kPa
Low cal.:	101.325	101.325	kPa
High cal.:	113.324	113.324	kPa
Calibrated:			

Temperature

Vapor Reflux	Vapor Flask	Vapor Flow	
1,100.00	1,200.00	1,000.00	°C
20.00	20.00	20.00	°C
50.00	50.00	50.00	°C

2. Place the water bath around the thermocouple so that the lowest 25 mm (1 in.) is immersed in the water. Place the thermocouple meter (reference thermocouple) parallel to the sample thermocouple so that both thermocouples are at the same temperature.
3. After the reference thermocouple meter reading stabilizes, enter the ambient temperature reading in the *Low Cal.* field on the *Instrument Calibration* window.
4. Remove the water bath. Move the tip of the reference thermocouple so that it touches the sample thermocouple tip yet allows the furnace to be closed.

5. 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.*** Set the furnace target temperature to 1000 °C. Use a ramp rate of 50 °C/min. Observe the schematic to ensure that both the furnace temperature and sample temperature are increasing.
6. After the furnace and sample temperature have stabilized, enter the reading from the reference thermocouple in the *High Cal.* field on the *Instrument Calibration* window.
7. Use manual control of the furnace to return the temperature to ambient.
8. On the *Instrument Calibration* window, click **Accept** to save the new values. Make a backup copy of the settings when prompted.

SAMPLE TUBE

SAMPLE TUBE INSTALLATION

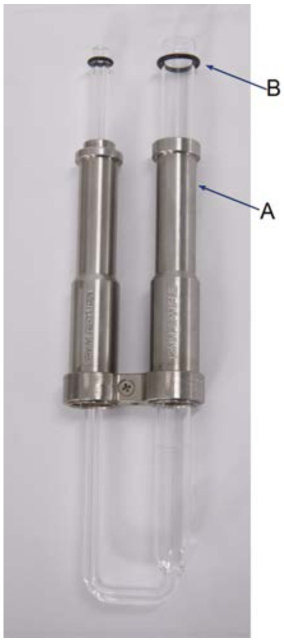
Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

CAUTION

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.

CAUTION

Use only Kalrez (or equivalent) O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.



A. Sample tube collar
B. O-rings

1. Open the furnace.
2. Remove any sample tube stoppers immediately before connecting the sample tube to the analyzer.
3. Carefully slide the sample tube collar over the open ends of the sample tube. The ferrules should be placed (narrow end down) on the smaller side of the sample tube.

CAUTION For quartz tubes, do not allow the collar to slide down rapidly to the curved portion of the tube. This could damage the tube.

4. Place an O-ring around each opening of the sample tube.
5. Insert the open ends of the sample tube into the openings under the upper panel, aligning the thermocouple in the proper position.
6. Push the sample tube up into the sample port until it comes to a stop. Push the lever to the left of the sample tube forward until it stops.

7. If the thermocouple is placed on the outside of the sample tube, clamp it to the tube.
8. Close and latch the furnace. The furnace will not heat unless closed.

SAMPLE TUBE REMOVAL

The furnace and sample tube may remain very hot for some time after analysis has ended. The sample tube retaining nuts are extremely hot, approximately 110 ºC. Use extreme caution!

It is recommended to allow the furnace and sample tube to return to ambient temperature before touching them. If the sample tube has cooled, use rubber gloves when removing it. If it is still hot, use the cotton gloves provided.

1. Unlatch and open the furnace.
2. Support the sample tube with one hand and push the sample tube lever to the left to release the sample tube.

CAUTION

Support the sample tube retaining nuts so that they do not fall rapidly out of the analyzer. This may damage the sample tube.

3. Loosen the sample tube retaining nuts, be very careful and support them. As the nuts are loosened, support the sample tube and pull it gently down out of the analyzer. When the nuts are sufficiently loose, the sample tube, retaining nuts, ferrules, and O-rings should slide out of the analyzer. If necessary, use the extractor tool to pull the O-rings out of the ports.
4. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the sample tube. Additional O-rings are shipped with the analyzer. Otherwise, clean, dry, and re-install the O-rings.

SAMPLE TUBE FILTER OR O-RING REPLACEMENT

A frit filter is used to protect the analyzer internal components from contaminants that may be forced out of the sample tube during an analysis. As the analyzer is used, the filter may gradually become clogged with trapped particles. If the filter does become blocked, it must be removed and cleaned or replaced. One indication of a clogged filter is a sample pressure greatly exceeding atmospheric pressure. The nominal sample pressure reading should be a few mmHg above atmospheric pressure

1. Loosen the analysis port fittings while holding the sample tube.

> The sample tube and nearby components may be hot. Allow the sample tube to cool before removing it. Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

2. Remove the sample tube by holding the tube with your right hand while pushing the sample tube lever back toward the analyzer with your left hand.
3. Remove the left (smaller) sample tube fitting (it has a tube within a tube).
4. Use a small diameter probe to remove the O-ring and frit filter disk from the sample tube fitting.

5. Install a new (or cleaned) analysis port filter in the sample tube fitting.
6. Inspect the O-ring for cracks or cuts. If the O-ring is damaged, replace it with a new O-ring. Otherwise, clean the O-ring, dry it, and place it in the sample tube fitting. Ensure the O-ring is securely in place.
7. Insert the open ends of the sample tube into the openings under the upper panel, aligning the thermocouple in the proper position.
8. Push the sample tube up into the sample port until it comes to a stop. Pull the lever to the left of the sample tube forward until it stops.

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 on page 6 - 2*** 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 loop exhaust port or 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

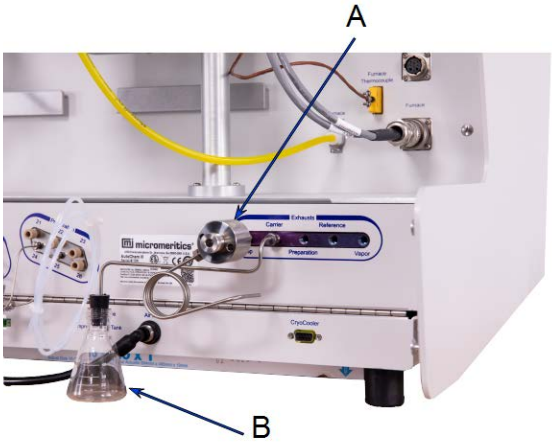
1. Empty the syringe.
2. Hold the tip of the needle just above the surface the liquid nitrogen.
3. Draw the syringe plunger back until the syringe is filled with nitrogen gas.
4. Allow the syringe to stabilize at room temperature.
5. Push the plunger into the syringe until the correct amount of gas is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

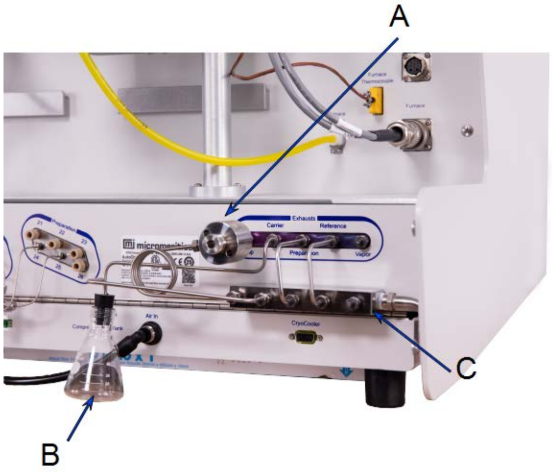
Use an External Sampling Septum

An external sampling septum and a condensate trap can be attached to the exhaust manifold. The external sampling septum is attached to the Loop port and the condensate trap is attached to the Carrier port.



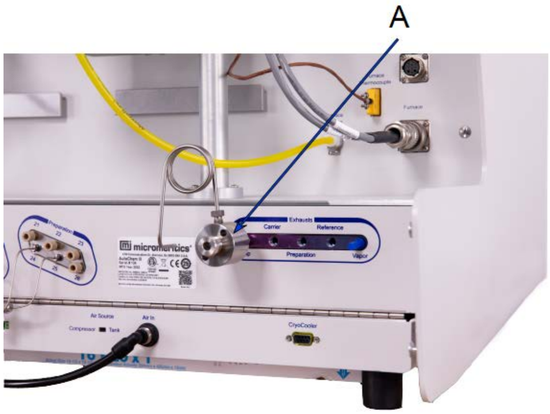
Without Exhaust Manifold Assembly

A. External sampling septum
B. Condensate trap
C. Exhaust manifold assembly



With Exhaust Manifold Assembly

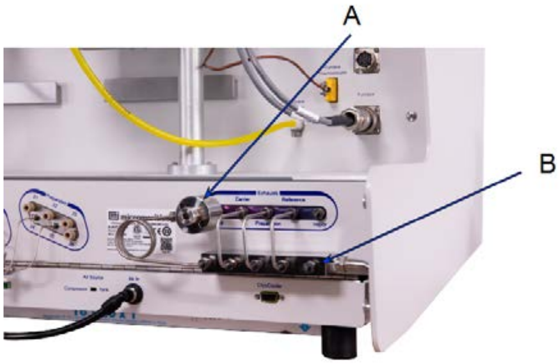
External Sampling Septum without Exhaust Manifold Assembly



A. External sampling septum

1. Insert the tubing of the external sampling septum into the Loop port of the exhaust manifold.

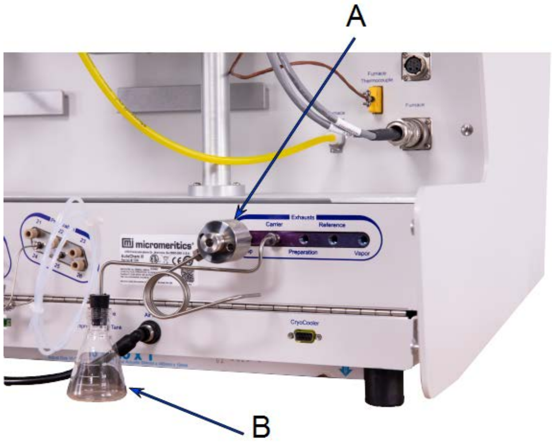
External Sampling Septum with Exhaust Manifold Assembly



A. External sampling septum
B. Exhaust manifold assembly

1. Remove both ends of the tubing from the Loop port of the exhaust manifold assembly.
2. Insert the external sampling septum into the Loop port of the exhaust manifold assembly.

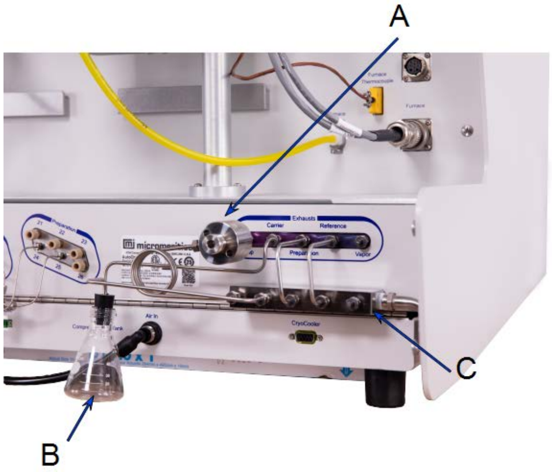
Condensate Trap without Exhaust Manifold Assembly



A. Stainless steel tubing
B. Condensate trap

1. Insert the condensate trap into the Carrier port of the exhaust manifold.

Condensate Trap with Exhaust Manifold Assembly



A. Stainless steel tubing
B. Condensate trap
C. Exhaust manifold assembly

1. Remove both ends of the tubing from the Carrier port of the exhaust manifold assembly.
2. Insert the condensate trap into the Carrier port of the exhaust manifold assembly.

SEPTUM REPLACEMENT

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

1. Turn the knurled nut counter-clockwise 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.

CAUTION 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.

SORPTION TRAP

Heating Mantle on page 8-14

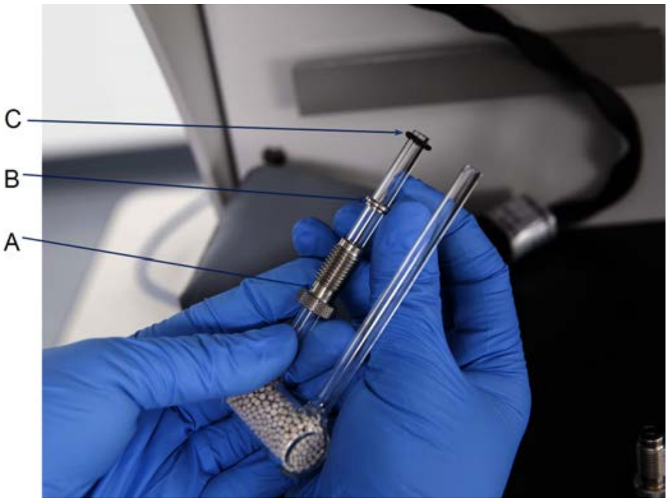
Parts and accessories are located on the Micromeritics web page.

The Sorption Trap is used to trap water produced from Temperature Programmed Reduction (TPR) analyses or other temperature programmed experiments in which moisture needs to be removed from the flow path before reaching the detector.

Sorption trap inside heating mantle

Sorption Trap

1. On both ends of the tube, insert the retaining nut (knurled end first), the ferrule (small end first), and O-ring. Leave the O-rings close to the top of the tube.

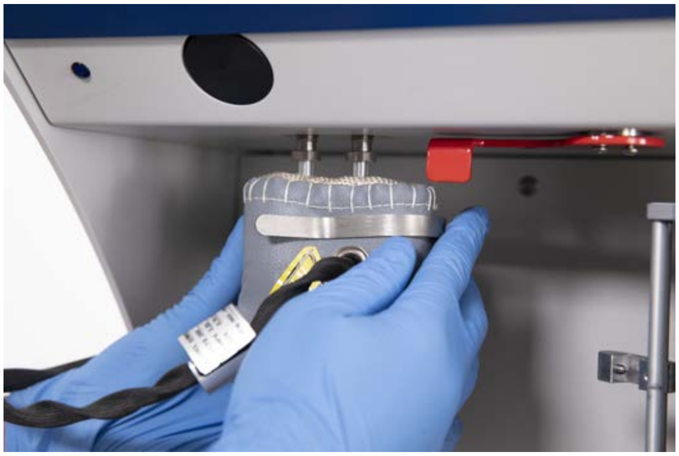


A. Retaining nut (insert knurled end first)
B. O-ring
C. Ferrule (insert small end first)

2. Insert the open ends of the tube into the sorption trap ports under the upper panel of the analyzer.
3. Press the tube up into the analyzer until it comes to a stop. Hold the tube in place and tighten the retaining nuts by hand. Tighten the nuts only enough to hold the tube securely in place and prevent leaks.

CAUTION Do not overtighten the retaining nuts. A sufficient seal is achieved when the fittings are finger tight.

4. Insert the heating mantle plug into the sorption trap port on the front of the analyzer.
5. Slide the heating mantle over the sorption trap tube and push the heating mantle up until the sorption trap touches the bottom of the mantle. Secure the mantle with the heating mantle clip.



CHANGE OR CLEAN THE SORPTION TRAP

Insert Experiment Steps on page 4 - 8

When using a sorption trap, insert a *Trap Regeneration* step into the experiment.

VAPOR GENERATOR INSTALLATION

Use caution in the areas where this symbol is displayed on the instrument.Use the cotton gloves provided in the accessory kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

Topics in this section are applicable only if the vapor generator option is installed on the analyzer

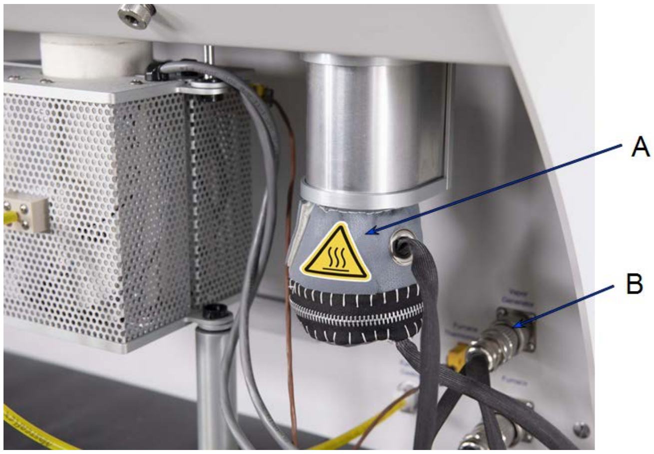
Perform a Leak Test on page 9-14

Clean the vapor generator before changing to a different liquid.

The majority of the vapor generator's components are internal to the analyzer and are installed and calibrated by a Micromeritics Service Representative.

CAUTION

Use appropriate safety measures to prevent injury from contact with hazardous liquids used in the vapor generator.



A. Heating mantle
B. Heating mantle connector

1. Fill the Erlenmeyer flask to a depth of at least 50 mm (2 in.).

2. Unzip the heating mantle, place it around the flask; do not zip it closed. Screw the flask into the port on the underside of the front panel. The metal cylinder that extends down from the vapor generator port is the aerator. When the flask is properly installed, the aerator should extend down into the flask and its contents. Zip the mantle closed.
3. Plug the heating mantle connector into the appropriate connector on the recessed front panel.
4. Twist the ring to secure the connector.
5. The valve that controls the flow of gas from the vapor generator has two states: *Bypass* and *Vapor*. In the sample file, select *Vapor* for the state of the vapor generator valve during the portion(s) of analyses to flow vapor through the system. The loop gas is the gas that flows through the vapor generator; ensure the gas used for vapor generation is connected to one of the loop gas inlet valves.

CLEAN THE VAPOR GENERATOR

Enable Manual Control on page 10-4

It is necessary to clean the vapor generator before changing the liquid.

1. Fill the flask with Isopropyl Alcohol (IPA).
2. Install the flask on the analyzer.
3. Flow an inert gas through the vapor generator and analyzer for approximately 20 minutes. Such cleaning can be performed using Manual Control.

9 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).

- CryoCooler II and CryoCooler III Operator Manual (PDF)
- Error Messages document (PDF)
- Parts and Accessories

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.

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are 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 AutoChem III weighs approximately 87 kg (130 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.

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 service or 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 5 - 3***.

Protective measures for these risks:

- **Electrical**. The electrical components operate at low voltage (24V or less) and pose low risk when energized. However, maintenance, troubleshooting, and repairs should be performed with the instrument de-energized whenever possible, in accordance with standard electrical safety guidelines.

Verification of the safe state of the instrument after repair:

- Disable the TCD and insert a blank tube into the port.
- Set all heat zones back to the default temperature.
- Set the vapor generator temperature to ambient temperature.

GUIDELINES FOR CONNECTING GASES

Regulator Pressure Settings

Analyzer	Gauge should indicate
AutoChem	14 - 16 psig (95 - 110 kPag)
AutoCool	100 - 120 psig (690 - 830 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. If expansion kits or other accessories are used in the lab, 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 SDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories are 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.

  Long gas lines, such as those used with gas cylinders placed in remote areas, must be purged 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 600 psi or 4100 kPa on the high-pressure gauge. Contaminants adsorbed to the walls of the cylinder will desorb as the pressure decreases.

CONNECT GAS TO ANALYZER

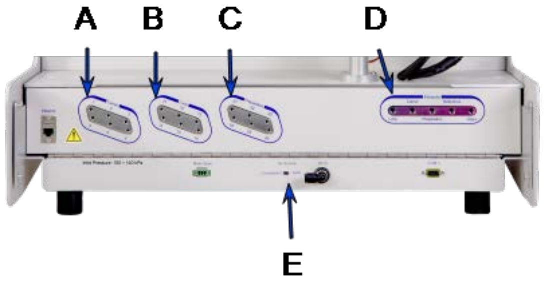
Regulator Pressure Settings

Analyzer	Gauge should indicate
AutoChem	14 - 16 psig (95 - 110 kPag)
AutoCool	100 - 120 psig (690 - 830 kPag)

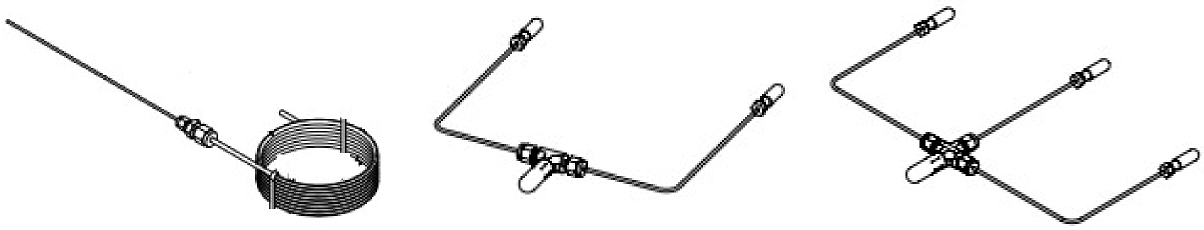
Exceeding the maximum recommended pressure could cause personal injury or damage the instrument.

Ensure the gas cylinder is closed before connecting the gas line to the analyzer.

The 18 gas inlet ports are located underneath the front cover.



A. Carrier gas inlets (6)
B. Loop gas inlets (6)
C. Preparation gas inlets (6)
D. Exhaust ports
E. *Air Source* selector and *Air In* port



Port

Gas Type	Port Label
Carrier	1 through 6
Loop	11 through 16
Preparation	21 through 26
Exhaust	One port each for Loop, Carrier, Preparation, Reference, Vapor

1. Attach the gas line to the appropriate port.
2. Set the regulator pressure as described in the Regulator Pressure Settings table.
3. Repeat steps 1 and 2 to connect all required gases.
4. Ensure any unused gas inlet ports are plugged.
5. Record the gas installed in the port in the analysis program (***Unit [n] > Gas Connections***) after the software is installed. See ***Gas Connections on page 2 - 14***.

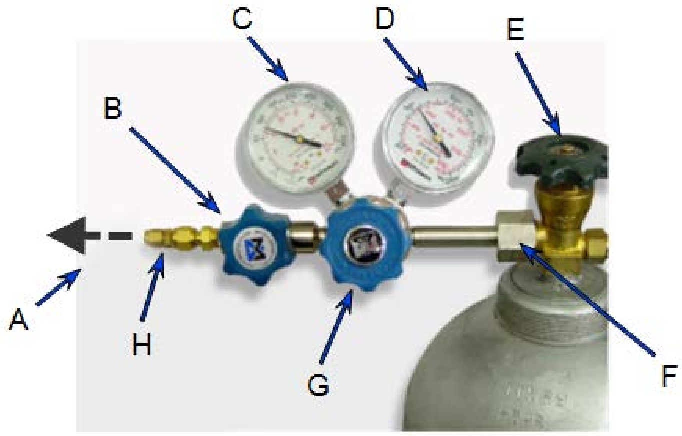
REPLACE A GAS CYLINDER

Regulator Pressure Settings

Analyzer	Gauge should indicate
AutoChem	14 - 16 psig (95 - 110 kPag)
AutoCool	100 - 120 psig (690 - 830 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. Gas tubing to instrument
B. Gas regulator shut-off valve
C. Low pressure gauge
D. High pressure gauge
E. Gas cylinder shut-off valve
F. Regulator connecter nut
G. Regulator control knob
H. Brass reducer fitting

Disconnect a Depleted Gas Cylinder

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

Connect a Gas Cylinder

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.

CAUTION

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.

AIR FILTER REPLACEMENT

The air filter located below the analyzer should be replaced, as needed. A standard 16" x 20" 1" filter is used.

There are two methods for replacing the air filter:

- Sliding the filter under the analyzer
- Removing the front cover of the analyzer

REPLACE THE FILTER BY SLIDING IT UNDER THE ANALYZER

1. Remove the existing filter.
2. Slide the new filter into the tray under the analyzer.
3. Push in the filter until it stops.

REPLACE THE FILTER BY REMOVING THE COVER

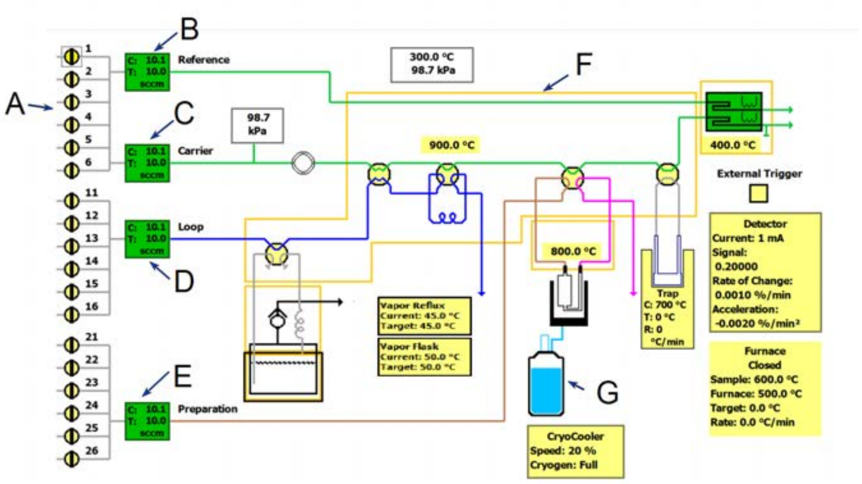
1. Grasp the top and bottom of the front cover.

2. Pull the cover forward and remove it.

3. Remove the existing filter by sliding it out of the tray.

4. Slide the new filter into the tray until it stops.
5. Replace the cover by setting it back on the latches and pushing forward until it locks into place.

PERFORM A LEAK TEST



A. Inlet valves
B. Reference gas MFC
C. Carrier gas MFC
D. Loop gas MFC
E. Preparation gas MFC
F. Valve heat zone
G. CryoCooler

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. Prepare for the leak test:
    a. Set all heat zones to ambient temperature.
    b. Set all four MFCs to 50 sccm.
    c. Allow the analyzer to cool to room temperature.
    d. Connect helium to an inlet of each gas path to be tested.

This test must be performed in the order provided to ensure the analyzer is fully tested.

PREPARATION GAS PATH LEAK TEST

This tests all fitting connections in the gas flow path and part of the analysis valve.

1. Right-click the analysis valve and select *Analysis*.
2. Right-click the helium preparation valve and select *Open*.
3. Plug the preparation gas exhaust outlet.
4. Observe the preparation gas MFC reading as it slowly drops to zero. A flow reading of zero indicates the flow path between the preparation gas MFC and the preparation exhaust does not leak.
5. To fix a leak that may be indicated on the preparation gas MFC, tighten the fittings in the gas flow path and observe the reading for any decrease that would indicate that the leak has stopped.

> Any leak detected in the remaining steps of this procedure may be fixed by using the same process mentioned in Step 5.

6. Right-click the preparation gas MFC and select *Close*.
7. Close the helium preparation valve.
8. Remove the plug from the preparation gas exhaust outlet.

REFERENCE GAS PATH LEAK TEST

This tests all fitting connections in the gas flow path and the reference side of the thermal conductivity detector.

Valve positions need not be preset for this test.

1. Right-click the helium reference valve and select *Open*.
2. Plug the reference gas exhaust outlet.
3. Observe the reference gas MFC reading as it slowly drops to zero. A flow reading of zero indicates the flow path between the reference gas MFC and the reference gas exhaust does not leak.
4. Right-click the reference gas MFC and select *Close*.
5. Close the helium reference valve.
6. Remove the plug from the reference gas exhaust outlet plug.

CARRIER GAS PATH LEAK TEST

1. Right-click the trap valve and select *Bypass*.
2. Right-click the analysis valve and select *Preparation*.
3. Right-click the blend valve and select *Bypass*.
4. Right-click the loop valve and select *Fill*.
5. Set the helium regulator to less than 5 psi (34.5 kPag).
6. Right-click the helium carrier valve and select *Open*.
7. Plug the analysis gas exhaust outlet.
8. Observe the carrier gas MFC reading as it slowly drops to zero. A flow reading of zero indicates the flow path between the carrier gas flow MFC and carrier gas exhaust does not leak.
9. Right-click the loop valve and select *Inject*.
10. Observe the carrier gas MFC reading as it slowly drops to zero or remains at zero from the previous test. A flow reading of zero indicates the flow path between the carrier gas MFC and the carrier gas exhaust outlet does not leak. This tests all fitting connections in the gas flow path, the remaining parts of the loop valve, and the loop.
11. Install an empty sample tube on the sample port.
12. Right-click the analysis valve and select *Analysis*
13. Observe the carrier gas MFC reading as it slowly drops to zero or remains at zero from the previous test. A flow reading of zero indicates the flow path between the carrier gas MFC and the carrier gas exhaust outlet does not leak. This tests all fitting connections in the gas flow path, the sample tube, and the remaining parts of the analysis valve.
14. Install a sorption trap, cold trap, or delay line on the trap port.
15. Right-click the trap valve and select *Trap*.
16. Observe the carrier gas MFC reading as it slowly drops to zero or remains at zero from the previous test. A flow reading of zero indicates the flow path between the carrier gas MFC and the carrier gas exhaust outlet does not leak. This tests all fitting connections in the gas flow path, the remaining parts of the trap valve, and the trap.
17. Right-click the helium carrier gas MFC and select *Close*.
18. Close the helium carrier valve.
19. Remove the plug from the carrier exhaust outlet.
20. Reset the helium regulator to its original pressure.

VAPOR GENERATOR GAS PATH LEAK TEST

> This test is applicable only if the vapor generator option is installed on the analyzer.

1. Right-click the loop valve and select *Inject*.
2. Right-click the vapor valve and select *Bypass*.
3. Right-click blend valve and select *Bypass*.
4. Plug the loop gas exhaust outlet.
5. Plug the vapor generator gas exhaust outlet.
6. Right-click the helium loop valve and select *Open*.
7. Observe the loop gas MFC reading as it slowly drops to zero. A flow reading of zero indicates the flow path between the loop gas MFC and loop gas exhaust outlet does not leak.
8. Install an empty flask onto the vapor generator port.
9. Right-click the vapor valve and select *Vapor*.
10. Observe the loop gas MFC reading as it slowly drops to zero or remains at zero from the previous test. A flow reading of zero indicates the flow path between the loop gas MFC and the loop gas exhaust does not leak. This tests all fitting connections in the gas flow path, the remaining portions of the vapor generator valve, and the relief valve in the vapor generator.

LOOP GAS PATH LEAK TEST

1. Right-click the loop valve and select *Inject*.
2. Right-click the blend valve and select *Bypass*.
3. Plug the loop gas exhaust outlet.
4. Right-click the helium loop valve and select *Open*.
5. Observe the loop gas MFC reading as it slowly drops to zero or remains at zero from the previous test. A flow reading of zero indicates the flow path between the loop gas MFC and the loop gas exhaust outlet does not leak. 1This tests all fitting connections in the loop gas flow path and the tubing to the loop valve.
6. Right-click the loop gas MFC and select *Close*.
7. Close the helium loop valve.
8. Remove the loop and vapor exhaust plugs.

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.

Procedure	3 months or 75 runs	6 months or 150 runs	9 months or 225 runs	12 months or 300 runs
Change septum		U-R		F
Change Injection loop		U-I-A		F
Foam air filter				F
Sample tube filter		U-R		F-R
Clean analyzer exterior		U-I-A		F
Adjust sample thermocouple		U-I-A		F
Clean the sorption trap or cold trap	U-I-A	U-I-A	U-I-A	F
Replace TCD		U-I-A		F
Clean Dewar	U-I-A	U-I-A	U-I-A	F
Change sample O-rings				F-R
Perform leak check		U-I-A		F
Check temperature stability				F
Check sample thermocouple calibration				F-A
Perform calibration run				F

Legend:

U	User performed
F	Factory / Micromeritics Service procedure
I	Inspect
R	Replace
A	As necessary

CHECK AND CLEAN THE DEWAR

When handling Dewars, follow the precautions outlined in ***Dewar Precautions on page 5 - 3***.

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.

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

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

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

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

CAUTION

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

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

CLEAN THE INSTRUMENT

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

CAUTION

Do not allow liquid to penetrate the casing of the instrument. Doing so could result in damage to the unit.

POWER INSTRUMENT ON AND OFF

CAUTION

Do not connect or disconnect cables when the instrument is powered ON.

The AutoChem III does not have a power switch. To power the instrument on or off, the instrument must be connected to or disconnected from the power source. The blue LED light on the front panel indicates power has been applied to the instrument.

When the system is powered on for the first time or after a period of non-use, allow it to warm up for at least three hours before analyses. During this time, a purge carrier gas (such as helium or nitrogen) should be flowed through the system at about 50 cm^3/min. The heat zones should be set at 110 °C.

PURGE THE SYSTEM

When changing the gas that is flowing through the analyzer, or when the type of gas connected to a port is changed, purge the analyzer of the previous gas by flowing the new gas. The system should also be purged if the loop is changed.

If changing gases 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). In some cases, purge one gas by flowing an inert gas for a period of time before starting to flow another gas. This may be useful for avoiding undesirable combinations of gases within the analyzer.

To flow an inert gas between incompatible gases, insert a *Change Gas Flows* 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.

Purge one line at a time. It is the operator’s responsibility to ensure that dangerous combinations of gases are not created while using Manual Control. It may be necessary to flow an inert gas through the system between flows of incompatible gases.

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. Set the valve and Detector heat zone temperatures to 110 °C.
    a. Move the cursor over temperature reading for each zone until the cursor changes to a thermometer.

    When the cursor is on a heat zone, it appears as a thermometer. If it appears as a handing turning a valve, move the cursor slightly.

    b. Right-click and select Set temperature.
    c. Enter *110*.
    d. Click **OK**.
3. Wait until the temperature reaches the 110 °C target.

4. Purge all air from the gas lines. These steps should be performed for each inlet valve for the preparation, carrier, and loop gas lines where a gas cylinder is connected.
    a. Right-click a Mass Flow Controller (MFC)
    b. Select *Set Flow Rate* and enter *50*.
    c. Click **OK**.
    d. Open an inlet valve that supplies the MFC.
    e. Allow the gas to flow for approximately 20 minutes or until the lines have been purged of air.
    f. Repeat Step 4 for each valve that has a gas cylinder connected.

Change the Gas Flow During an Analysis

When changing gases 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). Purge one gas (by flowing an inert gas for a period of time) before starting to flow another gas. This may be useful for avoiding undesirable combinations of gases within the analyzer.

To flow an inert gas between incompatible gases, insert a *Change Gas Flows* 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.

CAUTION

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.

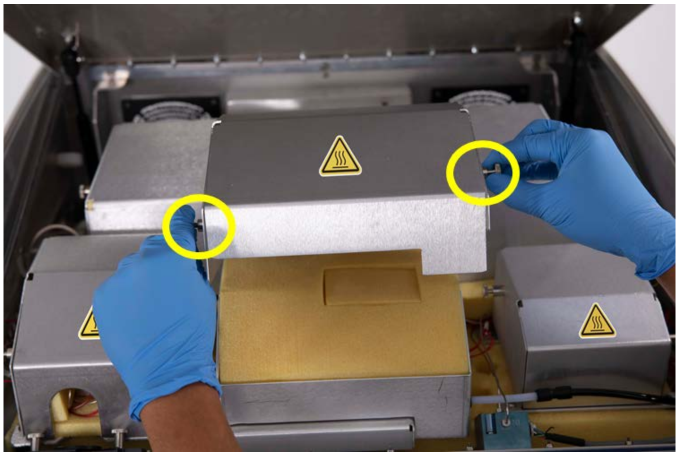
CHANGE THE INJECTION LOOP

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

1. Ensure that no heat is being applied to the analyzer. If necessary, enable manual control and set the target temperature for the heat zones to ambient. Wait for the analyzer to cool.
2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle. Use the handle to open the hinged top panel. It is not necessary to remove the top panel. Pull down the upper front panel.

Depending upon the state of the analyzer, the internal components may be hot. Use caution.

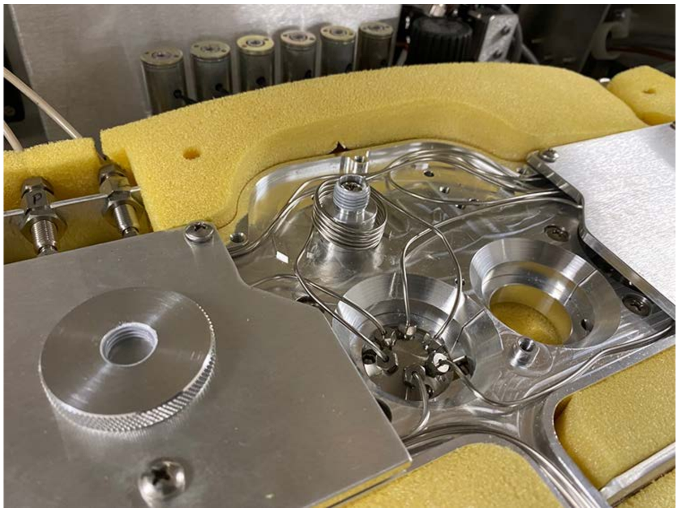
3. The *Fill/Inject* (Loop) valve cover is held in place by two retaining plungers. Remove the valve cover by pulling the plungers outward until the *Fill/Inject* valve cover is released. Pull the cover up and out.



4. Remove the insulation from the *Fill/Inject* valve.

5. Unscrew the loop nut and lay it aside. It may be necessary to loosen the screw in the middle of the assembly in order to free the loop nut.

6. Remove the enclosure ring.
7. Use a 1/4 in. open-end wrench to loosen the nuts on the injection loop.



8. Pull up and remove the injection loop and enclosure ring from the mounting plate.

Ensure the body of the loop is positioned in the temperature-controlled enclosure of the loop valve. This orientation keeps the temperature (and volume) of the loop constant. Failure to keep the full loop at a constant temperature may cause variations in the quantity of gas contained in the loop.

9. Install the replacement injection loop by placing the ends of the loop into the sides of the valve and turning the nuts finger tight.
10. Push the replacement loop down to contact the mounting plate and place the loop enclosure ring firmly into place.
11. Screw the loop nut down finger tight. Tighten the screw in middle of the assembly if it was loosened earlier.
12. Tighten the loop nuts securely, using a 1/4 in. open-end wrench.
13. Check the injection loop for leaks. If a leak is detected, the injection loop is probably not installed properly. Repeat the installation and check for leaks again. See ***Perform a Leak Test on page 9-14***.
14. After checking for leaks, move the insulation back into position and reinstall the *Fill/Inject* valve cover.
15. Close the top panel and the lower front panel.
16. After installing a new injection loop, the loop must be calibrated. Go to ***Unit [n] > Unit Configuration*** to update the loop volume.

The smaller loops connect to the same openings in the valve, but rest in a different position than the larger loop.

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10 TROUBLESHOOTING

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

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

- CryoCooler II and CryoCooler III Operator Manual (PDF)
- Error Messages document (PDF)
- Parts and Accessories

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:

Does not work when powered ON

Cause A: Power cord is not fully inserted at one of the ends

Action A: Insert power plug firmly into outlet socket. Insert unit connector into power connector opening.

Cause B: No power at outlet.

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

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

Action C: Gently move power plug at the outlet while watching the status LEDs on the analyzer front panel. If at least one LED becomes illuminated, have an electrician replace the outlet or the plug.

Cause D: Power cord is damaged.

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

Cause E: Loose internal connection or broken wire.

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

Liquid nitrogen boils away too quickly to complete an analysis.

Cause A: Dewar damaged.

Action A: Replace Dewar.

Cause B: Lengthy analysis.

Action B: Refill Dewar during analysis.

Specified temperature not reached or not maintained.

Cause: Thermocouple or another internal component is damaged or disconnected.

Action: Contact the appropriate service personnel.

Gas drained from gas cylinder.

Cause: Leaks in the gas line connection.

Action: Replace the gas cylinder; then pressurize the system. Close, then open the cylinder valve. If the needle on the pressure gauge on the gas cylinder jumps abruptly, a leak in the gas line connection may be indicated. Check all gas line connections.

Too much heat supplied to analyzer.

Cause: Thermocouple or another internal component is damaged or disconnected.

Action: Contact the appropriate service personnel.

A stable TCD baseline cannot be maintained.

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

Action A: Contact your Micromeritics service technician about replacing or cleaning the TCD.

Cause B: Possible leak in carrier path.

Action B: Perform leak test in the carrier path. See ***Perform a Leak Test on page 9-14*** .

Cause C: Faulty RTD in one of the temperature zones.

Action C: Access manual mode and check for temperature stability in the temperature zones.

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: Contact your Micromeritics service technician about replacing or cleaning the TCD.

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

Action B: Repeat the analysis using a higher 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: Contact your Micromeritics service technician about replacing or cleaning the TCD.

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

Action B: Ensure the TCD is re-enabled after all run suspensions.

Furnace will not heat.

Cause A: Furnace not closed and latched.

Action A: Close and latch the furnace.

Cause B: The furnace is not plugged in.

Action B: Plug in the furnace connector.

Cause C: Thermocouple damaged or not plugged in.

Action C: If unplugged, plug in the thermocouple. Replace the thermocouple if necessary.

Pressure will not rise or readings very unstable.

Cause A: Furnace not closed and latched.

Action A: Close and latch the furnace.

Cause B: The furnace is not plugged in.

Action B: Plug in the furnace connector.

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

Show Instrument Schematic on page 2 - 26

Use *Enable Manual Control* 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***.

POWER

The AutoChem is designed to operate with a universal input power supply (100/120/230 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 120 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 the computer, monitor, printer, and any other 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.

CAUTION

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.

This instrument does not have a power switch and must be disconnected from the wall outlet when powering off; therefore it is important to position the instrument where the wall outlet is easily accessible.

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.

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.

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11 ANALYSIS TUTORIALS

BET SURFACE AREA ANALYSIS TUTORIAL

The BET Surface Area analysis evaluates total surface area of the catalyst before and after chemisorption. Loss of activity, which may occur due to the blocking of pores during the chemisorption reaction, as well as the occurrence of sintering of the support, can be studied.

After outgassing the sample, a mixture of 30% nitrogen and 70% helium is applied to the sample which is immersed in a liquid nitrogen (LN_2) bath. The amount of nitrogen adsorbed (usually measured on desorption) at LN_2 temperatures is used to calculate total surface area.

Because nitrogen uptake is a function of pore size, sample sintering can cause results to show reduced surface area. Therefore, it may be helpful to perform BET as the first and last experiments in the analysis in order to check for sintering of the support.

PREPARATION

Pretreatment	Flow helium over the sample to remove impurities, usually at the maximum temperature tolerated by the sample (for example, the temperature at which the sample was calcined).
Analysis	Flow 30% N_2/He, using a Dewar of liquid nitrogen, to measure the uptake of nitrogen. Then remove the LN_2 Dewar and replace it immediately with a Dewar of water at ambient temperature. The amount of N_2 desorbed is measured and the BET equation is used to calculate the active surface area.
Trap	Install the external long delay path. Do not use a cold trap Dewar.
Pressure regulator	For BET analysis, gas cylinders should be set to a level between 14 and 16 psig (95 and 110 kPag).
Furnace temperature	During pretreatment, select a temperature high enough to remove contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Hold the temperature long enough to remove contaminants or moisture. Maintain the target temperature for at least 30 minutes to ensure adequate degassing.

CALIBRATION

BET experiments require a separate calibration. If using a pre-blended nitrogen/helium cylinder, the calibration consists of three manual injections of nitrogen through the septum. The optional external septum should be connected to the loop vent port to collect the required gas. If using the internal blend valve with pure nitrogen and helium, a standard gas calibration must be performed. Calibrations can be performed either before or after the BET analysis. As with all TCD calibrations, use the Peak Editor to associate the calibration file with the analysis file. For this reason, it is necessary that the data on the manual injections are collected in a separate experiment from the BET experiment. The injections may be included in the same analysis as the BET experiment, or they may be performed as a completely separate analysis. Use the same recording options (zero, invert) for the Calibration experiment and the BET experiment.

The following method includes the calibration injections as a separate experiment which follows the BET experiment in the analysis.

INJECTION SIZE

Inject three volumes of gas with the goal of approximating the volume of gas taken up by the sample. Use one injection slightly larger than the expected volume, one injection slightly smaller than the expected volume, and one injection between these two volumes. The largest injection should be two to three times greater than the smallest injection.

Because the same syringe must be used for all three injections, select a syringe that can accommodate all three injection volumes. The volume of gas uptake can be estimated if the approximate surface area (SA) of the sample is known. Use the following formula to determine the approximate volume (V_m) to inject.

$$V_m(cm^3 STP) = \frac{SA(m^2)}{(6.023\times 10^{23} molecule/mole)(16.21\times 10^{-20} m^2/molecule)} \times 22414 cm^3/mole$$

So:

$$V_m(cm^3 STP) = SA(m^2) \times 0.229$$

For example, a sample of 0.1 g of 50 m^2/g material has a surface area of 5 m^2. Therefore, the volume of the gas which will be adsorbed is approximately 1.145 cm^3 STP.

PROCEDURE

> Before performing an analysis, ensure the sample and analyzer are adequately prepared.

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer.
2. Ensure the *Delay Path* is installed in the trap port.
3. Create a sample file containing the appropriate analysis conditions and report options.
    a. Go to ***File > New Sample***.
    b. Complete the *Sample Description* window using appropriate values.
    c. Select the *Analysis Conditions* tab.
    d. Select *AutoChem III* in the *View conditions for* drop-down box.
    e. Insert the following experiment steps. After each step, click **OK** to close each window to move to the next step.

Experiment Steps:

- **Insert > *Experiment***

Description	Enter a description of the experiment
Type of Analysis	Select *Physisorption Surface Area*

- **Insert > *Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Helium
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	● Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	°C	110 °C	110 °C	☐ °C	°C/min
Flask:	°C			● Return to ambient temperature ☐ Disable temperature control	

● Disable vapor heating

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- *Insert* > ***Temperature Ramp***

  Sample ramp
  - End temperature: *350 °C*
  - Ramp rate: *50.0 °C/min*
  - Hold time: *0 min*

- *Insert* > ***Temperature Ramp***

  Return to ambient.

- ***Insert* > Instrument Settings**

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Nitrogen-Helium	None	None
50 cm^3 STP/min	cm^3 STP/min	cm^3 STP/min

☐ Different reference rate

cm^3 STP/min

FlowPath

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
☑ Bypass	☑ Bypass	☑ Fill	☐ Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	☑ Analyze	☑ Trap

Temperature

	Vapor	Valves	Sample port	Sample	Rate
Reflux:	°C	110 °C	60 °C	☐ °C	°C/min
Flask:	°C			☑ Return to ambient temperature	
				☐ Disable temperature control	

☑ Disable vapor heating

Detector

	Block temperature	100	°C
☑	Enable detector		
	Filament temperature	245	°C

- ***Insert* > *Wait***

  Wait until baseline and temperatures are stable.

- ***Insert* > *Wait***

  Wait for operator and add note *Click OK to proceed with physisorption measurement.*

- ***Insert* > *Start Recording***

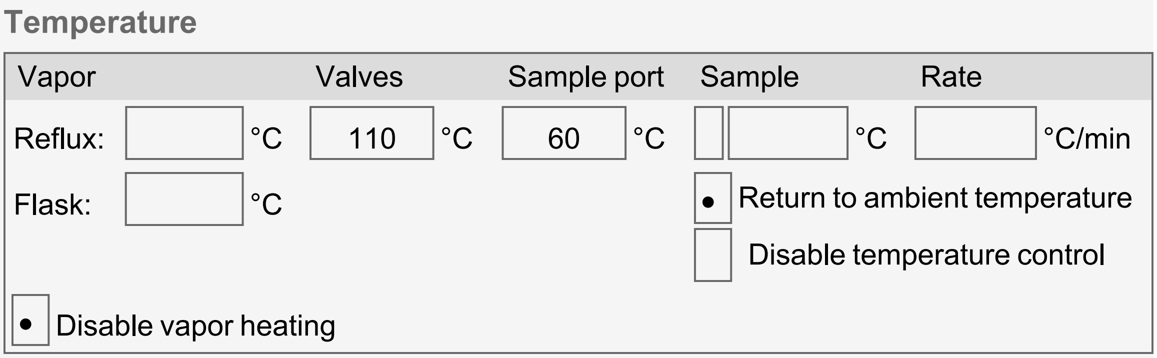
  One measurement every *0.1* s.

- ***Insert* > *Dose***

  After injection: Wait for 2 minutes, then wait for a return to baseline.

The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that a *Dose* step and a *Temperature Ramp* step is inserted within the *Start/Stop Recording* loop.

- **Termination**: Double-click *Termination* step in the experiment list box (or click *Termination*, then click **Edit**).



Temperature

	Vapor	Valves	Sample port	Sample	Rate
Reflux:	°C	110 °C	60 °C	☐ °C	°C/min
Flask:	°C			● Return to ambient temperature	
				☐ Disable temperature control	

● Disable vapor heating

To include the calibration data collection (three manual injections through the septum) within this analysis but as a separate experiment, also insert the following steps (before the *Dose* step):

- *Insert* > ***Experiment***

Description	Enter a description of the experiment
Type of analysis	*Other*

- *Insert* > ***Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Nitrogen-Helium	None	None
50 cm^3 STP/min	cm^3 STP/min	cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	☐ Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	● Analyze	● Trap

Temperature

	Vapor	Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature	
				☐ Disable temperature control	
● Disable vapor heating					

Detector

Block temperature	100	°C
● Enable detector		
Filament temperature	245	°C

- ***Insert*** **>** ***Wait***

  Wait until baseline and temperatures are stable.

- ***Insert*** **>** ***Start Recording***

  One measurement every *0.1* s.

- ***Insert*** **>** ***Start Repeat***

  Repeat *3* times

- ***Insert*** **>** ***Wait***

  Wait for operator. In text box enter: *Prepare manual* N_2 *injection. Click OK.*

- ***Insert*** **>** ***Dose***

  Injection type Manual injection

- The application automatically inserts a *Stop Repeat* step when a *Start Repeat* step is inserted. Ensure that a *Wait* step is inserted within the *Start/Stop Repeat* loop.
- The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Dose* step and the *Start Repeat/Wait/Dose/Stop Repeat* step is inserted within the *Start/Stop Record* loop.

   f. Select the *Report Options* tab and modify the values as needed.
   g. Click **Save**, then click **Close**.

4. Start the analysis.
   a. Go to ***Unit [n] > Sample Analysis*** and select the sample file that was just created.
   b. Edit the sample file as needed, but for this example, editing is not required. Click **Next**.
   c. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select *None*.
   d. Click **Next**.
   e. Read the cautionary window and make any necessary changes. Click **Start** to start the analysis. During the analysis, change the Dewar when prompted.

Change the Dewar rapidly. It is recommended to hold the plastic beaker in one hand and remove the LN_2 Dewar with the other to minimize the time required for the change.

5. When the analysis ends, remove the sample tube from the analyzer, place the caps on the tube, and dry off the outside of the tube with a paper towel.
6. Weigh the sample tube, sample, and caps. Subtract this weight from the weight obtained for the sample tube and caps. The resulting weight is the dry sample weight (the *after analysis weight*). This weight will be used when calculating the BET surface area in the following sections.

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

GENERATE THE BET REPORT

The following tasks must be performed before generating the BET report:

- Edit the sample file and the calibration file to ensure that peaks are properly marked
- Create a TCD calibration file
- Associate the TCD calibration file with the sample file

After completion of these tasks, go to ***Reports > Start Report***. Verify that *BET Surface Area* is selected.

EDIT THE PEAKS IN THE SAMPLE FILE

1. Open the Peak Editor.
2. Select the *Peak Editor* experiment from the view selector drop-down list at the bottom of the window.
3. Select *Edit Peaks* in the *View Type* group box.
4. Click **Find All Peaks**.
5. If needed, edit the peak markers of the desorption peak:

> The desorption peak is most often used to determine the surface area because the desorption process starts with the adsorbate equilibrated on the surface.

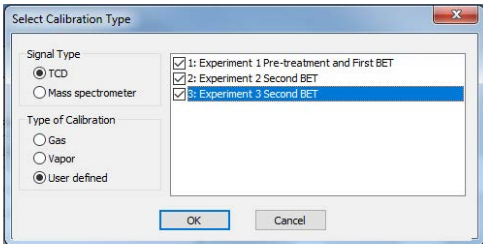
   a. Drag the cursor over the entire baseline of the peak to enlarge the editing area.
   b. Click on the baseline of the left side of the peak, right click and select *Mark left edge*.
   c. Repeat step b for the right side of the peak.
6. As the peak edges are adjusted, the values associated with the peak (listed in the peak table) are also adjusted. When satisfied with the appearance of the peak, right click and select **Save**.

EDIT THE PEAKS IN THE CALIBRATION FILE

1. Click **Calibration**.
2. Select the calibration file used with the analysis. Click **OK**.
3. Edit the peaks in the same manner as described for the sample file.

Create the TCD calibration file

1. Go to ***Options > Signal Calibration > New***.
2. Select *User defined* and enable an Experiment. Click **OK**.

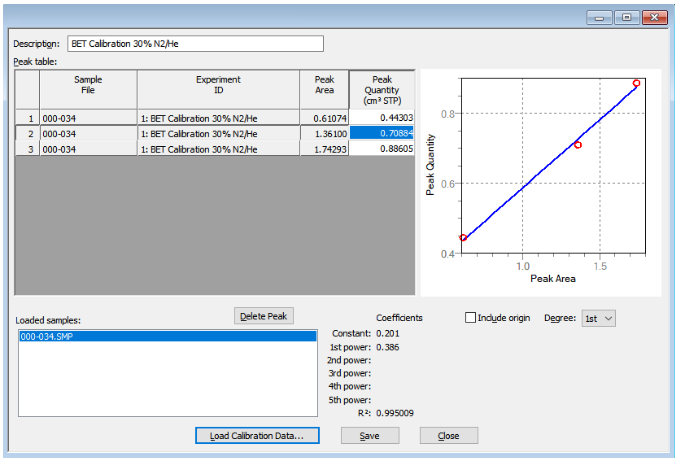


Select Calibration Type

Signal Type
● TCD
○ Mass spectrometer

Type of Calibration
○ Gas
○ Vapor
● User defined

☑ 1: Experiment 1 Pre-treatment and First BET
☑ 2: Experiment 2 Second BET
☑ 3: Experiment 3 Second BET



Description: BET Calibration 30% N2/He

Peak table:

	Sample File	Experiment ID	Peak Area	Peak Quantity (cm³ STP)
1	000-034	1: BET Calibration 30% N2/He	0.61074	0.44303
2	000-034	1: BET Calibration 30% N2/He	1.36100	0.70884
3	000-034	1: BET Calibration 30% N2/He	1.74293	0.88605

Loaded samples:
000-034.SMP

Coefficients
Constant: 0.201
1st power: 0.386
2nd power:
3rd power:
4th power:
5th power:
R²: 0.995009

☐ Include origin
Degree: 1st

3. Click **Load Calibration Data**.
4. Select the sample file containing the calibration data. Click **OK**. The data in the file are inserted into the appropriate fields of the Signal Calibration window.
5. The values in the *Peak Quantity* column are calculated based on temperatures and pressures measured by the instrument. Values for the volume of each injection at STP can be verified using the formula:

$$V_{\mathrm{STP}} = V(injected)\left(\frac{273.15}{273.15+ambient\ \ temp}\right) \times \left(\frac{ambient\ \ pressure}{760mmHg}\right)$$

6. Assess the Peak Area plot, Goodness of Fit, and Coefficients to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, use a calibration file with a very low value for Goodness of Fit (less than 0.1), when 1st or 2nd Degree is specified.

Assess the Peak Area plot, Goodness of Fit, and Coefficients to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, use a calibration file with a very low value for Goodness of Fit (less than 0.1), when 1st or 2nd Degree is specified.

To delete peaks that are outliers, highlight the peak data in the table and click **Delete Peak**. Evaluate the results when changing the degree (select a different degree from the drop-down list).

7. Enter a new description into the *Description* field.
8. When satisfied with all values in the file, click **Save**, then click **Close**

Associate the Calibration file with the Sample file

1. Open the Peak Editor.
2. Select the *Peak Editor* experiment from the view selector drop-down list at the bottom of the window.
3. Select the experiment and click the **Calibration** button.
4. Select the down-arrow to the right of the *Calibration* field. Select the calibration file just created. Click **OK**.
5. Repeat steps 3 and 4 for each experiment.
6. Click **Save**, then click **Close**.

LOOP CALIBRATION FOR TCD ANALYZERS TUTORIAL

Septum on page 8-27

Each 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***.

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**.
4. Inject 80% of the loop volume. For example, if calibrating the 1 cm^3 loop, inject as close to 0.8 cm^3 of gas as possible.

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 on page 6 - 2

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. Go to ***File > Open > [.SMP]***. Open the .SMP file containing the calibration experiment. Verify the defined peaks are properly marked (or correct them) using the *Peak Editor*.
2. On the *Reports* tab, select only the *Loop Calibration* report option. Click *Preview* to view the results, including the *Loop Volume*.
3. Go to ***Unit [n] > Unit Configuration***. In the *Loop Volume* field, enter the *Loop Volume*.

GENERATE THE PULSE CHEMISORPTION REPORT

Peak Editor on page 6 - 2

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

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

- 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.

1. Go to ***File > Open > .SMP file***.
2. Select the sample file used with this analysis. Click **OK**.
3. On the Sample Description tab, click **Active Metals**.
4. Verify that the *Stoichiometry Factor* and the *% of Sample Mass* values are correct.
5. Click **OK**.
6. Open the *Peak Editor* and click **Calibration**.
7. Verify that the values for the loop (or manual injection) volume, ambient temperature, atmospheric pressure, and active concentration are accurate.
8. Go to ***Report Options > Pulse Chemisorption*** to set the report options. Click **OK**, then click *Preview* to preview the report, or go to ***Reports > Start Report > SMP file*** to generate the report.

LOOP VOLUME

It is preferred for the sample to require at least two doses of gas, but no more than ten doses, before the reaction ends — depending on the environment. Some factors that influence the number of doses required are sample size, the active surface area per unit of sample, and the size of the loop. If the pulse chemisorption analysis requires more doses or fewer doses, change either the sample size or the loop size.

Three loops of different sizes are provided with the analyzer — each loop must be calibrated prior to use. As an alternative to replacing the loop, the loop volume may be changed by adjusting its temperature. If the loop volume is changed by changing its temperature, results will be more accurate if the loop volume is recalibrated at the new temperature.

LOOP CALIBRATION

Before data from an analysis that uses an injection loop can be reduced, a loop calibration experiment must be performed. An independent loop calibration can be performed either 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.
Analysis	Pulse the loop gas over the sample until the peak area remains constant.
Trap	No trap is needed during pretreatment. During the analysis, use the trap fixture with a Dewar of water in place as a short delay path. This delay compensates for the flow disturbance caused by each loop injection.
Pressure regulator	Gas cylinders should be set to a level between 14 and 16 psig (95 and 110 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 *Done* step is set to return the sample temperature to ambient.

PULSE CHEMISORPTION ANALYSIS TUTORIAL

A Pulse chemisorption analysis determines active surface area, percent metal dispersion, and active particle size by applying measured pulses of reactant gas to the sample, depending upon what technique is used. Pulse chemisorption is performed in the same manner as TPD, except that the sample is dosed with the analysis gas using the injection loop until each active site has reacted. After the active sites have completely reacted, the discretely injected gas volumes emerge unchanged. If TPD is performed after Pulse chemisorption, additional information about the distribution of active sites and the strength of active sites is collected.

The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that did not react with the active sites of the sample. The size of each pulse is determined by the loop, which is located on an electrically operated valve.

PROCEDURE

In the following example, Pulse chemisorption is performed on a sample of Pt/Al_2O_3 with CO. Alter the analysis conditions to accommodate a different analysis.

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See *Prepare for Analysis on page 5 - 5*.

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file containing the appropriate analysis conditions and report options.
   a. Go to ***File > New Sample***.
   b. Complete the *Sample Description* window using appropriate values.
   c. Select the *Analysis Conditions* tab.
   d. Select *AutoChem III* in the *View conditions for* drop-down box.
   e. Insert the following experiment steps. After each step, click **OK** to close each window to move to the next step.

Experiment Steps:

- ***Insert > Experiment***

Description	Enter a description of the experiment
Type of Analysis	Select *Pulse*

- ***Insert > Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
None	None	Helium
cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
☑ Bypass	☑ Bypass	☑ Fill	☑ Prepare	☑ Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

	Vapor	Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	110 °C	☐ °C	°C/min
Flask:	20 °C			☐ Disable temperature control	
☑ Disable vapor heating					

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- ***Insert* > *Wait***

  Wait 5 *minutes*.

- ***Insert* > *Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
None	None	Hydrogen
cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

Vapor		Valves		Sample port		Sample		Rate	
Reflux:	20 °C	110	°C	110	°C	☐	°C		°C/min
Flask:	20 °C					☐ Disable temperature control			
● Disable vapor heating									

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- ***Insert* > *Temperature Ramp***

Sample ramp	○ End temperature: *400 °C* ○ Ramp rate: *10.0 °C/min* ○ Hold time: *30 min*

- ***Insert* > Instrument Settings**

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Helium
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
☑ Bypass	☑ Bypass	☑ Fill	☑ Prepare	☑ Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			☐ Return to ambient temperature	
☑ Disable vapor heating				☐ Disable temperature control	

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- ***Insert* > Wait**

Wait 30 minutes.

- *Insert* > ***Temperature Ramp***

Sample ramp
- End temperature: 35 °*C*
- Ramp rate: *10 °C/min*
- Hold time: *10 min*

- *Insert* > ***Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	CO/He	None
50 cm^3 STP/min	25 cm^3 STP/min	cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
☑ Bypass	☑ Bypass	☑ Fill	☐ Prepare	☑ Bypass
☐ Vapor	☐ Blend	☐ Inject	☑ Analyze	☐ Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	60 °C	☐ 35 °C	5 °C/min
Flask: 20 °C			☐ Return to ambient temperature	
☑ Disable vapor heating			☐ Disable temperature control	

Detector

Block temperature	100	°C
☑ Enable detector		
Filament temperature	245	°C

- ***Insert* > *Wait***

  Wait 5 minutes.

- ***Insert* > *Wait***

  Wait until baseline and temperature are stable.

- ***Insert* > *Wait***

  Wait 5 minutes.

- ***Insert* > *Start Recording***

  One measurement every 0.1 *s*

- **Insert > *Start Repeat***

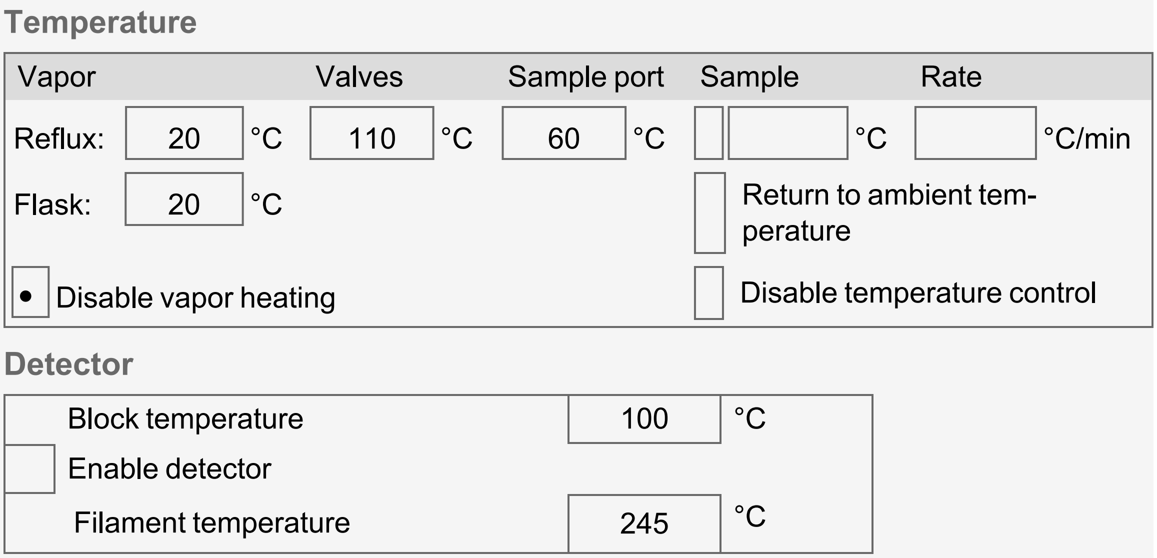
  Repeat until peaks are equal or 20 *times.*

- ***Insert* > *Dose***

  Wait for 2 *minutes.*

- The application automatically inserts a *Stop Repeat* step when a *Start Repeat* step is inserted. Ensure that a *Dose* step is inserted within the *Start/Stop Repeat* loop.
- The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Dose* step is inserted within the *Start/Stop Record* loop.

- **Termination**: Double click *Termination* step in the experiment list box (or click *Termination*, then click **Edit**).



Temperature

	Vapor		Valves		Sample port		Sample		Rate	
Reflux:	20	°C	110	°C	60	°C	☐	°C		°C/min
Flask:	20	°C								

☐ Return to ambient temperature

● Disable vapor heating

☐ Disable temperature control

Detector

Block temperature 100 °C

☐ Enable detector

Filament temperature 245 °C

f. Select the *Report Options* tab and modify values as needed.
g. Click **Save**, then click **Close**.

3. Start the analysis.
   a. Go to ***Unit > Sample Analysis***. From the *Files* list box, select the sample file created in the previous step. Edit the file as needed. Click **Next**.
   b. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select *None*. Click **Next**.

> Calibration files can also be associated with a sample file after analysis using **Calibration** button in the Peak Editor.

   c. Read the cautionary window and make any necessary changes.
   d. Click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the optional CryoCooler or AutoCool.

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

When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

TEMPERATURE PROGRAMMED DESORPTION ANALYSIS TUTORIAL

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 5 - 5*.

Pretreatment	Degas by flowing inert gas — such as helium, argon, or nitrogen — over the sample while ramping the temperature.
Analysis	Choose a gas mixture from the following table based on the needed results. Flow the gas over the sample, then ramp the temperature beginning at ambient.
Trap	Not required

Needed Results	**Carrier Gas**	**Analysis Gas**
Active Metal Surface Area H_2 Chemisorption	argon	hydrogen
Active Metal Surface Area CO Chemisorption	helium (or nitrogen)	carbon monoxide
Acidity	helium	ammonia (15% maximum NH_3, blended in helium)

Before performing an analysis, ensure the sample and analyzer have been adequately prepared.

PROCEDURE

These instructions analyze zeolite with ammonia (use 15% maximum NH_3 blended in helium) at 3 to 20 ºC/min. Adjust the values in the example to accommodate the analysis you are performing.

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file containing the appropriate analysis conditions and report options.
    a. Go to ***File > New Sample***.
    b. Complete the *Sample Description* window using appropriate values.
    c. Select the *Analysis Conditions* tab.
    d. Select *AutoChem III* in the *View conditions for* drop-down box.
    e. Insert the following experiment steps. After each step, click **OK** to close each window to move to the next step.

Experiment Steps:

- ***Insert > Experiment***

Description	Enter a description of the experiment
Type of Analysis	Select *Other*

- ***Insert > Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Helium
50 cm^3 STP/min	☐ cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
☐ cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

Vapor		Valves		Sample port		Sample		Rate	
Reflux:	20 °C	110	°C	110	°C	☐	°C		°C/min
Flask:	20 °C					●	Return to ambient temperature		
						☐	Disable temperature control		
● Disable vapor heating									

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- *Insert* > ***Temperature Ramp***

Sample ramp

  - End temperature: 350 °C
  - Ramp rate: *20 °C/min*
  - Hold time: *10 min*

- *Insert* > ***Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Helium
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

	Vapor	Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature	
				☐ Disable temperature control	
● Disable vapor heating					

Detector

	Block temperature	100 °C
☐	Enable detector	
	Filament temperature	245 °C

- ***Insert* > *Temperature Ramp***

  Return to ambient

- ***Insert* > *Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Ammonia
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			☑ Return to ambient temperature	
				☐ Disable temperature control	

☑ Disable vapor heating

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- *Insert* > ***Wait***

  Wait **60** *minutes*.

- ***Insert* > Instrument Settings**

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	None
50 cm^3 STP/min	cm^3 STP/min	cm^3 STP/min
☐ Different reference rate cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	☐ Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	● Analyze	☐ Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature	
				☐ Disable temperature control	
● Disable vapor heating					

Detector

	Block temperature	100	°C
●	Enable detector		
	Filament temperature	245	°C

- ***Insert* > *Wait***

  Wait **30** *minutes*.

- ***Insert* > *Wait***

  Wait until baseline and temperatures are stable.

- ***Insert* > *Start Recording***

  One measurement every **1.0** *s*

- ***Insert* > *Temperature Ramp***

  Sample ramp

  - End temperature: 500 *°C*
  - Ramp rate: *10 °C/min*
  - Hold time: *30 min*

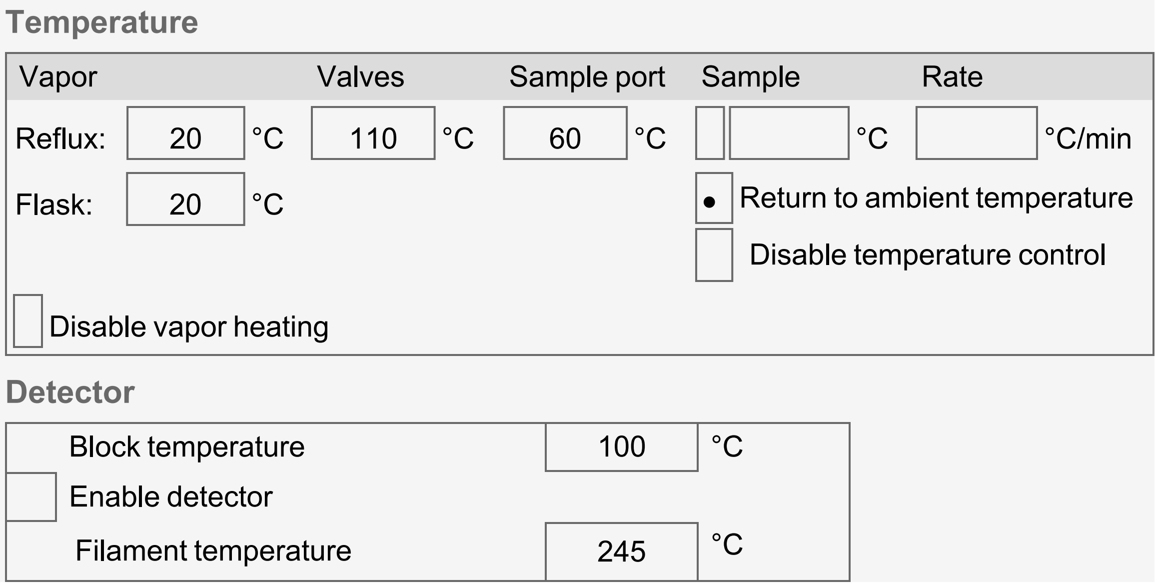
The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Temperature Ramp* step is inserted within the *Start/Stop Record* loop.

- ***Insert* > *Temperature Ramp***

  Return to ambient.

Create a new experiment and repeat the steps from the yellow highlighted step (shown previously) to here for each repetition..

- **Termination**: Double click *Termination* step in the experiment list box (or click *Termination*, then click **Edit**).



Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature	
				☐ Disable temperature control	

☐ Disable vapor heating

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

   f. Select the *Report Options* tab and set the values. Click **Save** then click **Close**.

3. Start the analysis.
   a. Go to ***Unit > Sample Analysis***. From the *Files* list box, select the sample file created in the previous step. Edit the file as needed. Click **Next**.
   b. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select *None*. Click **Next**.
   c. Read the cautionary window and make any necessary changes.
   d. Click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the optional CryoCooler or AutoCool. Remove the sample tube when the analysis is complete.

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

TEMPERATURE PROGRAMMED OXIDATION ANALYSIS TUTORIAL

> 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 C - 1***.

PREPARATION

Pretreatment	TPR
Analysis	2 to 5% oxygen/helium is flowed through the sample while temperature is ramped, beginning at ambient temperature.
Trap	Not required.

> Before performing an analysis, ensure the sample and analyzer are adequately prepared. See ***Prepare for Analysis on page 5 - 5***.

PROCEDURE

1. Obtain the sample weight, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file containing the appropriate analysis conditions and report options.
    a. Go to ***File > New Sample***.
    b. Complete the *Sample Description* window using appropriate values.
    c. Select the *Analysis Conditions* tab.
    d. Select *AutoChem III* in the *View conditions for* drop-down box.
    e. Insert the following experiment steps. After each step, click **OK** to close each window to move to the next step.

Experiment Steps:

- ***Insert > Experiment***

Description	Enter a description of the experiment
Type of Analysis	Select *Temperature Programmed*

- ***Insert > Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Hydrogen-Argon	None	Helium
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	● Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	60 °C	☐ °C	°C/min
Flask: 20 °C			☐ Return to ambient temperature ☑ Disable temperature control	
☑ Disable vapor heating				

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- ***Insert* > *Temperature Ramp***

  Return to ambient.

- ***Insert* > *Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Hydrogen-Argon	None	None
50 cm^3 STP/min	cm^3 STP/min	10 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
☑ Bypass	☑ Bypass	☑ Fill	☐ Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	☑ Analyze	☑ Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	110 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature	
				☐ Disable temperature control	
● Disable vapor heating					

Detector

	Block temperature	100	°C
●	Enable detector		
	Filament temperature	245	°C

- *Insert* > ***Wait***

  Wait for operator. Add text: *Setup trap and close furnace*.

- *Insert* > ***Wait***

  Wait until baseline and temperatures are stable.

- *Insert* > ***Start Recording***

  One measurement every 1.0 s.

- *Insert* > ***Temperature Ramp***

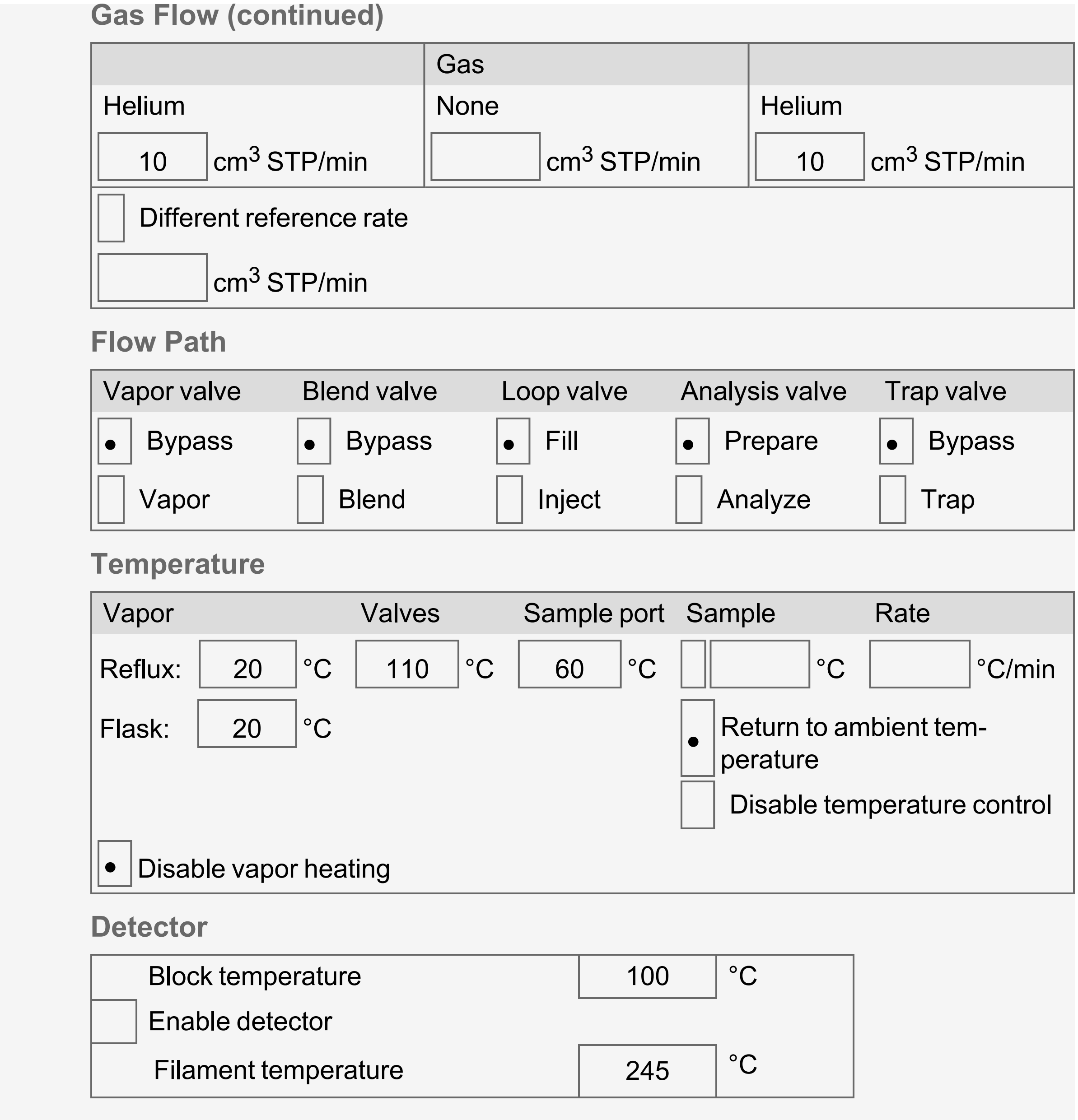
Sample ramp	◦ End temperature: *980 0 °C* ◦ Ramp rate: *10.0 °C/min* ◦ Hold time: *0.0 min*

The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Temperature Ramp* step is inserted within the *Start/Stop Record* loop.

- *Insert* > ***Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend	Preparation Gas



Gas Flow (continued)

	Gas	
Helium	None	Helium
10 cm^3 STP/min	cm^3 STP/min	10 cm^3 STP/min

☐ Different reference rate

cm^3 STP/min

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	☐ Trap

Temperature

Vapor		Valves	Sample port	Sample	Rate
Reflux:	20 °C	110 °C	60 °C	☐ °C	°C/min
Flask:	20 °C			● Return to ambient temperature ☐ Disable temperature control	

● Disable vapor heating

Detector

Block temperature	100 °C
☐ Enable detector	
Filament temperature	245 °C

- ***Insert* > Experiment**

Description	Enter a description of the experiment
Type of Analysis	Select *Temperature Programmed Oxidation*

- ***Insert* > Instrument Settings**

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Oxygen-Helium	None	None
50 cm^3 STP/min	cm^3 STP/min	cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	☐ Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	● Analyze	☐ Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	60 °C	☐ °C	°C/min
Flask: 20 °C			● Return to ambient temperature	
			☐ Disable temperature control	
● Disable vapor heating				

Detector

Block temperature	100	°C
● Enable detector		
Filament temperature	245	°C

- *Insert* > ***Wait***

  Wait until baseline and temperatures are stable.

- *Insert* > ***Sttart Recording***

  One measurement every **1.0** *s*

- *Insert* > ***Temperature Ramp***

  Sample ramp
  - End temperature: *980 °C*
  - Ramp rate: *20.0 °C/min*
  - Hold time: *0.0 min*

The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Temperature Ramp* step is inserted within the *Start/Stop Record* loop.

- *Insert* > ***Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Helium	None	Helium
10 cm^3 STP/min	cm^3 STP/min	10 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	☐ Prepare	● Bypass
☐ Vapor	☐ Blend	☐ Inject	● Analyze	☐ Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	60 °C	☐ °C	°C/min
Flask: 20 °C			● Return to ambient tem-	

Temperature (continued)

☐ perature

☐ Disable temperature control

☑ Disable vapor heating

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

- **Termination**: Double click *Termination* step in the experiment list box (or click *Termination*, then click **Edit**).

Temperature

Vapor			Valves		Sample port		Sample		Rate	
Reflux:	20	°C	110	°C	60	°C	☐	°C		°C/min
Flask:	20	°C					☑ Return to ambient temperature			
							☐ Disable temperature control			
☑ Disable vapor heating										

Detector

Block temperature	100	°C
☐ Enable detector		
Filament temperature	245	°C

f. Select the *Report Options* tab and set the values.
g. Click **Save**, then click **Close**.

3. Start the analysis.
    a. Go to ***Unit > Sample Analysis***. From the *Files* list box, select the sample file created in the previous step. Edit the file as needed. Click **Next**.
    b. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select *None*. Click **Next**.
    c. Read the cautionary window and make any necessary changes.
    d. Click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the optional CryoCooler or AutoCool. Remove the sample tube when the analysis is complete.

4. As the temperature increases, the sample is oxidized, and the application calculates the volume of oxygen taken up.
5. Allow the TCD signal to return to the initial baseline after the peak has been displayed
6. 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 kit when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

TEMPERATURE PROGRAMMED REDUCTION ANALYSIS TUTORIAL

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

Some reactions begin at temperatures below ambient. In such cases, a Dewar containing an appropriate coolant should be used instead of the furnace at the beginning of the experiment. For example, reduction of PtO should begin at approximately -50 °C, because the reaction begins at about -30 °C. Alternatively, the optional CryoCooler can be used.

Temperature Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which reduction occurs. An important aspect of TPR analyses is that the sample need not have special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) over the sample, usually starting 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.

PREPARATION

Pretreatment	Oxidize by flowing O_2 over the sample.
Analysis	Flow 5-10% hydrogen/argon 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. Argon is recommended over nitrogen because the resultant peak(s) show no reaction between sample and gas.
Trap	A sorption or cold trap is required to remove traces of water formed as a product of the reduction.

Before performing an analysis, ensure the sample and analyzer are adequately prepared. See ***Prepare for Analysis on page 5 - 5***.

PROCEDURE

1. Obtain the sample mass then install the loaded sample tube on the analyzer. If the analysis begins below ambient, either place a Dewar of coolant around the sample tube or close the furnace around the sample tube and install the CryoCooler. If the analysis begins at ambient, close the furnace around the sample tube.
2. Ensure that the zeolite adsorbent has been regularly activated and that it is at room temperature.
3. If using the optional cold trap instead of the soprtion trap, install it then place a Dewar filled with coolant around the cold trap. Ensure that the Dewar contains sufficient coolant to cover the cold trap loops.

   A mixture of isopropyl alcohol (IPA) and liquid nitrogen (LN_2) is the recommended coolant for this experiment. Place the isopropyl alcohol in a Dewar and slowly pour LN_2 into the Dewar while stirring the mixture. Continue to add and stir the mixture until it becomes a slush. The mixture must be capable of achieving a temperature of about -90 ºC.

Extreme caution should be used when mixing the IPA/LN_2. See ***Dewar Precautions on page 5 - 3***.

4. Create a sample file containing the appropriate analysis conditions and report options.
   a. Go to ***File > New Sample***.
   b. Complete the *Sample Description* window using appropriate values.
   c. Select the *Analysis Conditions* tab.
   d. Select *AutoChem III* in the *View conditions for* drop-down box.
   e. Insert the following experiment steps. After each step, click **OK** to close each window to move to the next step.

Experiment Steps:

- ***Insert > Experiment***

Description	Enter a description of the experiment
Type of Analysis	Select *Temperature Programmed*

- ***Insert* > Instrument Settings**

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Hydrogen-Argon	None	Helium
50 cm^3 STP/min	cm^3 STP/min	50 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	● Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	☐ Analyze	● Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	110 °C	☐ °C	°C/min
Flask: 20 °C			● Return to ambient temperature	
			☐ Disable temperature control	
● Disable vapor heating				

Detector

Block temperature	100 °C
☐ Enable detector	
Filament temperature	245 °C

- ***Insert* > Wait**

Wait for operator. Insert text: *Add sample and setup trap*.

- ***Insert* > *Instrument Settings***

Gas Flow

Carrier/Reference Gas	Loop, Injection, or Blend Gas	Preparation Gas
Hydrogen-Argon	None	None
50 cm^3 STP/min	cm^3 STP/min	10 cm^3 STP/min
☐ Different reference rate		
cm^3 STP/min		

Flow Path

Vapor valve	Blend valve	Loop valve	Analysis valve	Trap valve
● Bypass	● Bypass	● Fill	☐ Prepare	☐ Bypass
☐ Vapor	☐ Blend	☐ Inject	● Analyze	● Trap

Temperature

Vapor	Valves	Sample port	Sample	Rate
Reflux: 20 °C	110 °C	60 °C	☐ °C	°C/min
Flask: 20 °C			● Return to ambient temperature	
			☐ Disable temperature control	
● Disable vapor heating				

Detector

	Block temperature	100	°C
●	Enable detector		
	Filament temperature	245	°C

- ***Insert* > *Wait***

  Wait until baseline and temperatures are stable.

- ***Insert* > *Start Recording***

  One measurement every **1.0** *s*

- *Insert* > ***Temperature Ramp***

Sample ramp	◦ End temperature: 400 *°C* ◦ Ramp rate: *10 °C/min* ◦ Hold time: *30 min*

The application automatically inserts a *Stop Recording* step when a *Start Recording* step is inserted. Ensure that the *Temperature Ramp* step is inserted within the *Start/Stop Record* loop.

- **Termination**: Double click *Termination* step in the experiment list box (or click *Termination*, then click **Edit**).

Temperature

Vapor			Valves		Sample port		Sample			Rate	
Reflux:	20	°C	110	°C	60	°C	☐		°C		°C/min
Flask:	20	°C					●	Return to ambient temperature			
							☐	Disable temperature control			
●	Disable vapor heating										

Detector

	Block temperature	100	°C
☐	Enable detector		
	Filament temperature	245	°C

f. Select the *Report Options* tab and modify the values as needed.
g. Click **Save**, then click **Close**.

5. Start the analysis.
   a. Go to ***Unit > Sample Analysis***. From the *Files* list box, select the sample file created in the previous step. Edit the file as needed. Click **Next**.
   b. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select *None*. Click **Next**.

Calibration files can also be associated with a sample file after analysis using *Set Calibration in the Peak Editor.*

c. Read the cautionary window and make any necessary changes.
d. Click **Start** to start the analysis.

As the temperature increases, the copper oxide is reduced, the water produced by the reaction is collected in the trap (if used), and the amount of hydrogen consumed is detected and transmitted to the application. Use the *Results* view to display a chromatogram of the hydrogen consumed from the detector signal as a function of the ramping temperature.

A hydrogen consumption peak, which corresponds to the reduction capacity of copper oxide, is displayed. The maximum peak should occur at approximately 280 ºC. This temperature varies highly, depending on the CuO particle size. Larger particle size shifts the temperature upward to 330 °C or more. Under certain combinations of sample, hydrogen concentration, and flow rate, two peaks may appear due to the transition state of Cu^{2+} to Cu^{+} to Cu.

GAS CONCENTRATION CALIBRATION

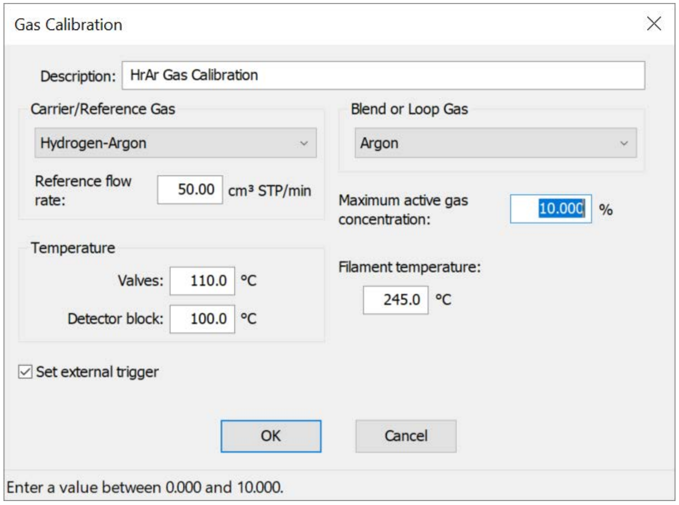
After performing an analysis, it may be necessary to convert the peak area data it yielded to volume data. The analyzer can do this automatically, but it must first have some additional information.

In some cases (such as in TPR analyses), the analyzer needs to know how to correlate the signal readings collected in the analysis with the volume of gas uptake at any given point in the analysis. If the analyzer is provided with a series of known gas concentrations, and it records the signal associated with each known concentration, then it can use this information to calculate the concentrations associated with the signals it recorded during the analysis. From that point, it can also calculate the volume of gas associated with each peak in the data.

- Prepare a clean, empty sample tube and install it on the analyzer.
- Make sure the correct gases are connected, and verify that the Gas Selections table (Unit menu) accurately reflects the current gas connections.

CREATE THE GAS CONCENTRATION CALIBRATION SAMPLE FILE

1. Open the AutoChem III application, and select ***File > New Sample***.
2. Enter a *Sample Name* and any other relevant information.
3. Click the **Analysis Conditions** tab.
4. Click **Insert** and select **Gas Calibration**. The Gas Calibration dialog box is displayed.
5. Enter a *Description*.
6. Choose *Hydrogen-Argon* for the *Carrier/Reference Gas* and *Argon* for *Blend or Loop Gas*.
7. Enter *50.00* for the *Reference Flow Rate*.
8. Enter the *Maximum active gas concentration* (found on the gas cylinder).
9. Enter the *Temperature (Valves, Detector block and Filament temperature)* as shown below.
10. Select *Set external trigger*.



Description: HrAr Gas Calibration

Carrier/Reference Gas: Hydrogen-Argon

Reference flow rate: 50.00 cm^3 STP/min

Blend or Loop Gas: Argon

Maximum active gas concentration: 10.000 %

Temperature

Valves: 110.0 °C

Detector block: 100.0 °C

Filament temperature: 245.0 °C

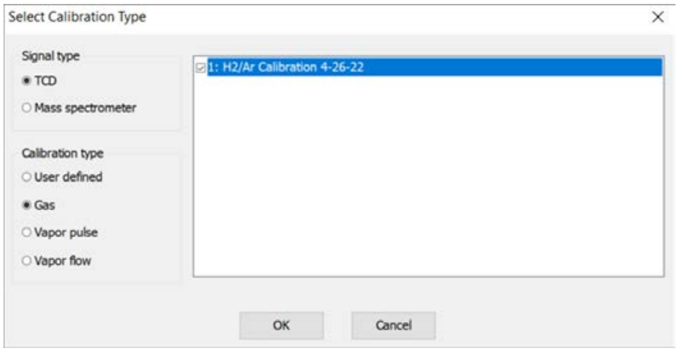
☑ Set external trigger

Enter a value between 0.000 and 10.000.

11. Click **OK**.
12. Click **Save As**, name the file, and click **Close**.
13. Select ***Unit > Sample Analysis***, then click **Browse** to select the sample file created.
14. Click **Start** then **Next**.

EDIT THE GAS CONCENTRATION CALIBRATION FILE

1. When the analysis is complete, select ***Options > Signal Calibration > New***.
2. Select the analyzed file. The Select Calibration Type dialog box is displayed.
3. For *Signal Type*, select **TCD** and for *Calibration Type*, select **Gas**.
4. Select the calibration in the list, and click **OK**.



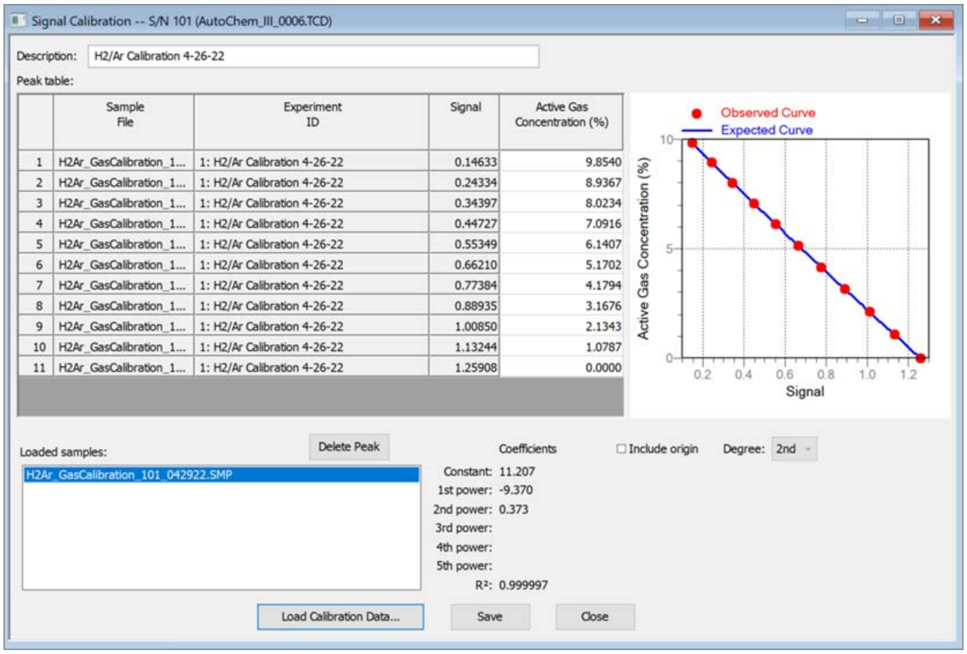
Select Calibration Type

Signal type
● TCD
○ Mass spectrometer

Calibration type
○ User defined
● Gas
○ Vapor pulse
○ Vapor flow

☑ 1: H2/Ar Calibration 4-26-22

5. In the Signal Calibration window, enter a *Description*, select **1st** or **2nd** for the *Degree*, and click **Save**.



Signal Calibration -- S/N 101 (AutoChem_III_0006.TCD)

Description: H2/Ar Calibration 4-26-22

Peak table:

	Sample File	Experiment ID	Signal	Active Gas Concentration (%)
1	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.14633	9.8540
2	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.24334	8.9367
3	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.34397	8.0234
4	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.44727	7.0916
5	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.55349	6.1407
6	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.66210	5.1702
7	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.77384	4.1794
8	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	0.88935	3.1676
9	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	1.00850	2.1343
10	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	1.13244	1.0787
11	H2Ar_GasCalibration_1...	1: H2/Ar Calibration 4-26-22	1.25908	0.0000

Loaded samples:
H2Ar_GasCalibration_101_042922.SMP

Coefficients
Constant: 11.207
1st power: -9.370
2nd power: 0.373
3rd power:
4th power:
5th power:
R²: 0.999997

☐ Include origin
Degree: 2nd

6. Assess the gas concentration plot, Goodness of Fit, and Coefficients to determine if the calibration file is acceptable. based upon the laboratory's standards for determining the level of linearity. As a general guideline, however, use a calibration file with a very low value for Goodness of Fit (less than 1/2% of the maximum concentration), when 1st or 2nd Degree is selected. Also, delete any data point that deviates significantly from linear, and evaluate the results when changing the degree (making a selection from the drop-down list).
7. To generate a report, select ***Options > Signal Calibration > Report*** and select the applicable file.

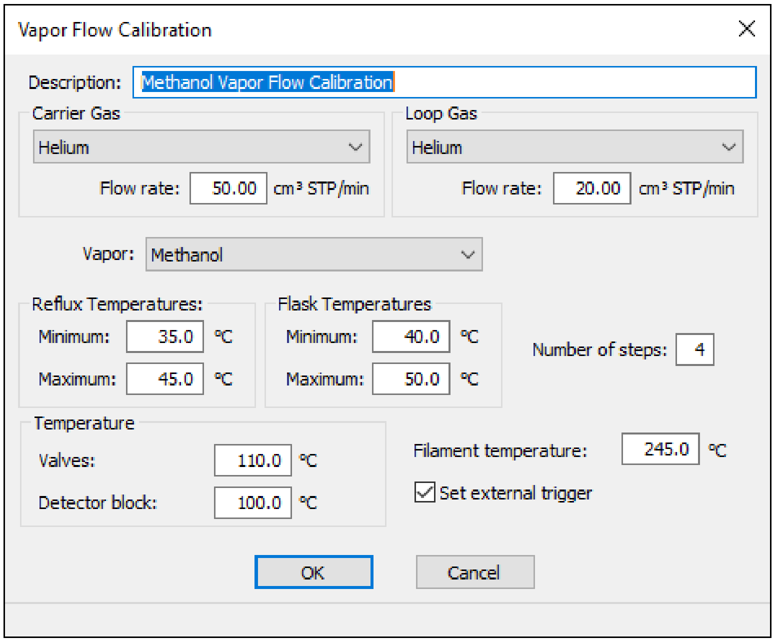
VAPOR FLOW CALIBRATION

When you use the optional Vapor Generator, you must calibrate it for the type of vapor you are using. Associating your analysis data with a Vapor Generator calibration file enables you to convert your peak area data to volume data.

You can perform the Vapor Generator calibration either before or after performing your analysis. A single calibration file can be used with multiple analysis files.

CREATE THE VAPOR FLOW CALIBRATION SAMPLE FILE

1. Open the AutoChem III application, and select ***File > New Sample***.
2. Enter a *Sample Name* and any other relevant information.
3. Click the **Analysis Conditions** tab.
4. Click **Insert** and select **Vapor Flow Calibration**. The Vapor Flow Calibration dialog box is displayed.
5. Enter a *Description*.
6. Choose *Helium* for both the *Carrier Gas* and *Loop Gas*.
7. Enter *50.00* for the carrier *Flow Rate* and *20.00* for the loop *Flow rate*.
8. Select *Methanol* for the *Vapor*.
9. Enter the *Temperatures* as shown below.



Description: Methanol Vapor Flow Calibration

Carrier Gas
Helium
Flow rate: 50.00 cm³ STP/min

Loop Gas
Helium
Flow rate: 20.00 cm³ STP/min

Vapor: Methanol

Reflux Temperatures:
Minimum: 35.0 °C
Maximum: 45.0 °C

Flask Temperatures
Minimum: 40.0 °C
Maximum: 50.0 °C

Number of steps: 4

Temperature
Valves: 110.0 °C
Detector block: 100.0 °C

Filament temperature: 245.0 °C

☑ Set external trigger

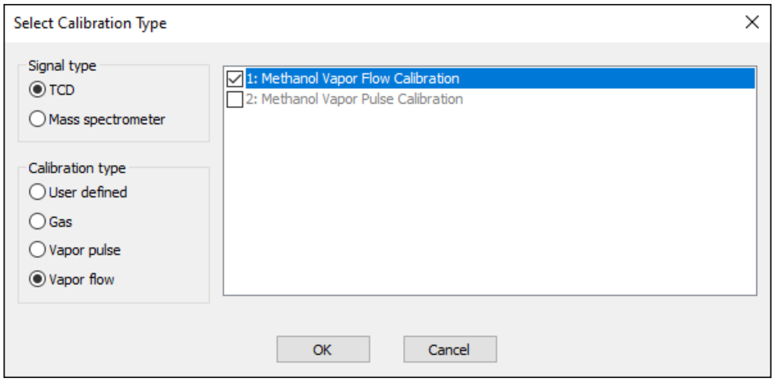
10. Click **OK**.
11. Click **Save As**, name the file, and click **Close**.

12. Select ***Unit > Sample Analysis***, then click **Browse** to select the sample information file created.
13. Click **Start** then **Next**.
14. When the displayed sample temperature reaches the ambient temperature, open the furnace and, using gloves, remove the sample tube.

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

EDIT THE VAPOR FLOW CALIBRATION FILE

1. When the analysis is complete, select ***Options > Signal Calibration > New***.
2. Select the analyzed file. The Select Calibration Type dialog box is displayed.
3. For *Signal Type*, select *TCD* and for *Calibration Type*, select *Vapor flow*.
4. Select a calibration in the list, and click **OK**.



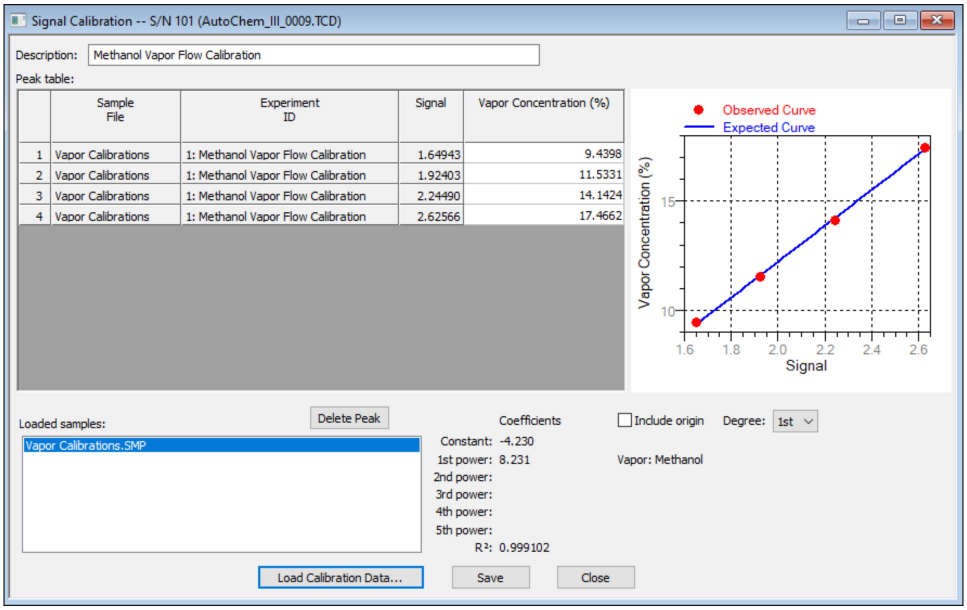
Select Calibration Type

Signal type
● TCD
○ Mass spectrometer

Calibration type
○ User defined
○ Gas
○ Vapor pulse
● Vapor flow

☑ 1: Methanol Vapor Flow Calibration
☐ 2: Methanol Vapor Pulse Calibration

5. In the Signal Calibration window, enter a *Description*, select *1st* or *2nd* for the *Degree*, and click **Save**.
6. Assess the plot, Goodness of Fit, and Coefficients to determine if the calibration file is acceptable, based upon the laboratory's standards for determining the level of linearity. As a general guideline, however, use a calibration file with a very low value for Goodness of Fit (less than 1/2% of the maximum concentration), when 1st or 2nd Degree is selected. Also, delete any data point that deviates significantly from linear, and evaluate the results when changing the degree (making a selection from the drop-down list).



Description: Methanol Vapor Flow Calibration

Peak table:

	Sample File	Experiment ID	Signal	Vapor Concentration (%)
1	Vapor Calibrations	1: Methanol Vapor Flow Calibration	1.64943	9.4398
2	Vapor Calibrations	1: Methanol Vapor Flow Calibration	1.92403	11.5331
3	Vapor Calibrations	1: Methanol Vapor Flow Calibration	2.24490	14.1424
4	Vapor Calibrations	1: Methanol Vapor Flow Calibration	2.62566	17.4662

Loaded samples: Vapor Calibrations.SMP

Coefficients
Constant: -4.230
1st power: 8.231
2nd power:
3rd power:
4th power:
5th power:
R²: 0.999102

☐ Include origin

Degree: 1st

Vapor: Methanol

7. Click **Load Calibration Data** and select the sample file.
8. To generate a report, select ***Options > Signal Calibration > Report*** and select the applicable file.

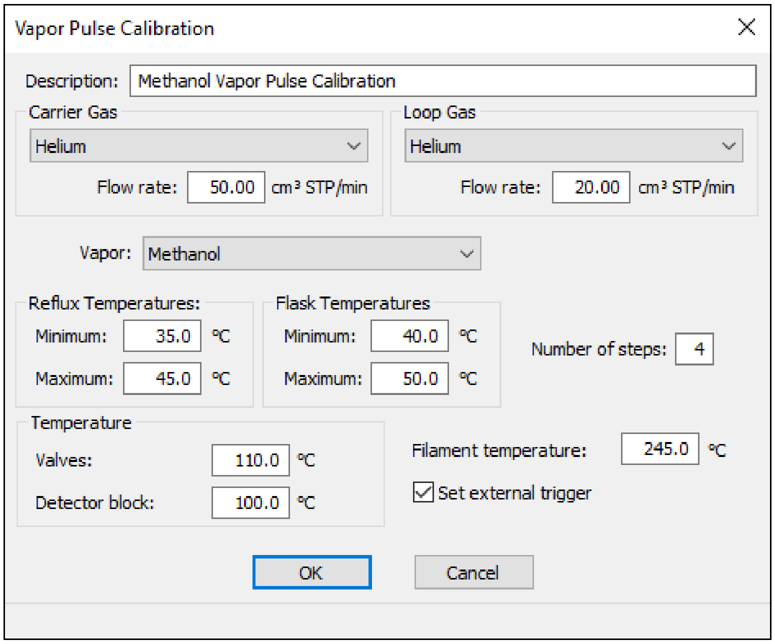
VAPOR PULSE CALIBRATION

When you use the optional Vapor Generator, you must calibrate it for the type of vapor you are using. Associating your analysis data with a Vapor Generator calibration file enables you to convert your peak area data to volume data.

You can perform the Vapor Generator calibration either before or after performing your analysis. A single calibration file can be used with multiple analysis files.

CREATE THE VAPOR PULSE CALIBRATION SAMPLE FILE

1. Open the AutoChem III application, and select ***File > New Sample***.
2. Enter a *Sample Name* and any other relevant information.
3. Click the **Analysis Conditions** tab.
4. Click **Insert** and select **Vapor Pulse Calibration**. The Vapor Pulse Calibration dialog box is displayed.
5. Enter a *Description*.
6. Choose *Helium* for the *Carrier Gas* and *Loop Gas*.
7. Enter *50.00* for the carrier *Flow Rate* and *20.00* for the loop *Flow rate*.
8. Select *Methanol* for the *Vapor*.
9. Enter the *Temperatures* as shown below.



Description: Methanol Vapor Pulse Calibration

Carrier Gas: Helium

Flow rate: 50.00 cm³ STP/min

Loop Gas: Helium

Flow rate: 20.00 cm³ STP/min

Vapor: Methanol

Reflux Temperatures:
Minimum: 35.0 °C
Maximum: 45.0 °C

Flask Temperatures
Minimum: 40.0 °C
Maximum: 50.0 °C

Number of steps: 4

Temperature
Valves: 110.0 °C
Detector block: 100.0 °C

Filament temperature: 245.0 °C

☑ Set external trigger

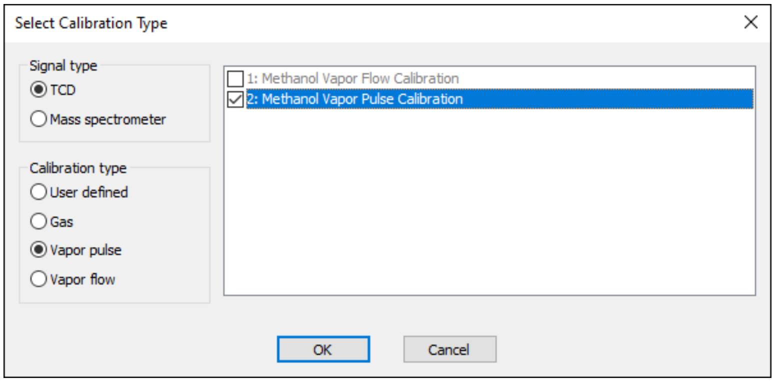
10. Click **OK**.
11. Click **Save As**, name the file, and click **Close**.

12. Select ***Unit > Sample Analysis***, then click **Browse** to select the sample information file created.
13. Click **Start** then **Next**.
14. When the displayed sample temperature reaches the ambient temperature, open the furnace and, using gloves, remove the sample tube.

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

EDIT THE VAPOR PULSE CALIBRATION FILE

1. When the analysis is complete, select ***Options > Signal Calibration > New***.
2. Select the analyzed file. The Select Calibration Type dialog box is displayed.
3. For *Signal Type*, select *TCD* and for *Calibration Type*, select *Vapor pulse*.
4. Select a calibration in the list, and click **OK**.



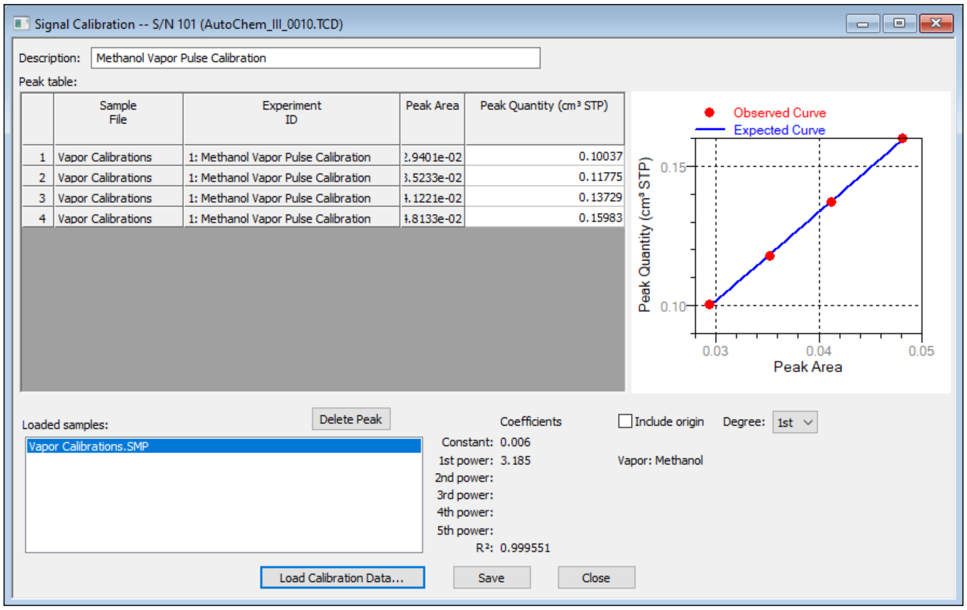
Select Calibration Type

Signal type
● TCD
○ Mass spectrometer

Calibration type
○ User defined
○ Gas
● Vapor pulse
○ Vapor flow

☐ 1: Methanol Vapor Flow Calibration
☑ 2: Methanol Vapor Pulse Calibration

5. In the Signal Calibration window, enter a *Description*, select *1st* or *2nd* for the *Degree*, and click **Save**.
6. Assess the plot, Goodness of Fit, and Coefficients to determine if the calibration file is acceptable, based upon the laboratory's standards for determining the level of linearity. As a general guideline, however, use a calibration file with a very low value for Goodness of Fit (less than 1/2% of the maximum concentration), when 1st or 2nd Degree is selected. Also, delete any data point that deviates significantly from linear, and evaluate the results when changing the degree (making a selection from the drop-down list).



Signal Calibration -- S/N 101 (AutoChem_III_0010.TCD)

Description: Methanol Vapor Pulse Calibration

Peak table:

	Sample File	Experiment ID	Peak Area	Peak Quantity (cm³ STP)
1	Vapor Calibrations	1: Methanol Vapor Pulse Calibration	2.9401e-02	0.10037
2	Vapor Calibrations	1: Methanol Vapor Pulse Calibration	3.5233e-02	0.11775
3	Vapor Calibrations	1: Methanol Vapor Pulse Calibration	4.1221e-02	0.13729
4	Vapor Calibrations	1: Methanol Vapor Pulse Calibration	4.8133e-02	0.15983

Loaded samples: Vapor Calibrations.SMP

Coefficients
Constant: 0.006
1st power: 3.185
2nd power:
3rd power:
4th power:
5th power:
R²: 0.999551

☐ Include origin
Degree: 1st
Vapor: Methanol

6. Click **Load Calibration Data** and select the sample file.
7. To generate a report, select ***Options > Signal Calibration > Report*** and select the applicable file.

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12 DIAGNOSTICS

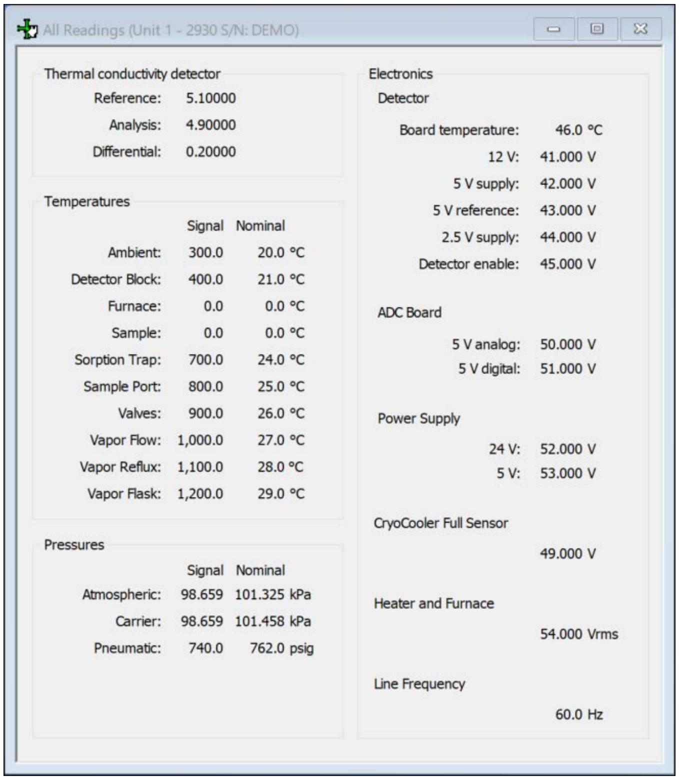
Unit [n] > Diagnostics

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

SHOW ALL READINGS

Unit [n] > Diagnostics > Show All Readings

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



Thermal conductivity detector
Reference: 5.10000
Analysis: 4.90000
Differential: 0.20000

Temperatures

	Signal	Nominal
Ambient:	300.0	20.0 °C
Detector Block:	400.0	21.0 °C
Furnace:	0.0	0.0 °C
Sample:	0.0	0.0 °C
Sorption Trap:	700.0	24.0 °C
Sample Port:	800.0	25.0 °C
Valves:	900.0	26.0 °C
Vapor Flow:	1,000.0	27.0 °C
Vapor Reflux:	1,100.0	28.0 °C
Vapor Flask:	1,200.0	29.0 °C

Pressures

	Signal	Nominal
Atmospheric:	98.659	101.325 kPa
Carrier:	98.659	101.458 kPa
Pneumatic:	740.0	762.0 psig

Electronics

Detector
Board temperature: 46.0 °C
12 V: 41.000 V
5 V supply: 42.000 V
5 V reference: 43.000 V
2.5 V supply: 44.000 V
Detector enable: 45.000 V

ADC Board
5 V analog: 50.000 V
5 V digital: 51.000 V

Power Supply
24 V: 52.000 V
5 V: 53.000 V

CryoCooler Full Sensor
49.000 V

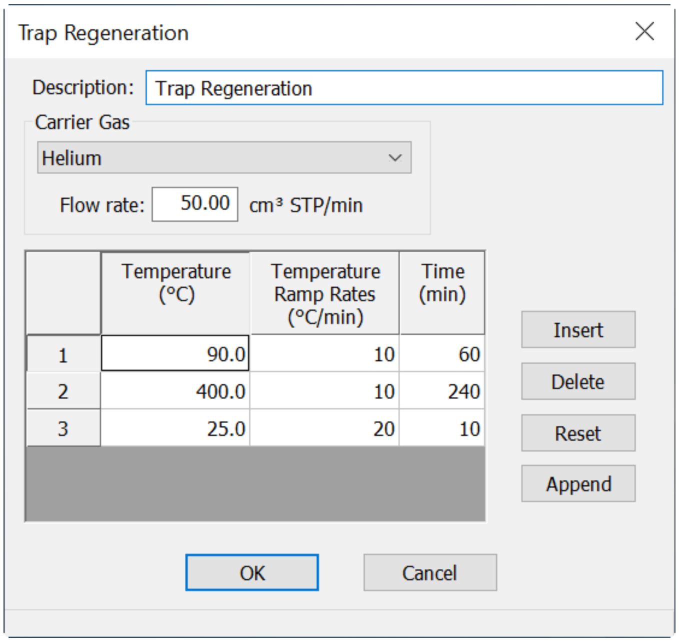
Heater and Furnace
54.000 Vrms

Line Frequency
60.0 Hz

TRAP REGENERATION CONDITIONS

Unit [n] > Diagnostics > Trap Regeneration Conditions

Diagnostics > Trap Regeneration details should only effect the available Trap Regeneration diagnostic test. Default analysis conditions tasks will not be affected.



Trap Regeneration

Description: Trap Regeneration

Carrier Gas

Helium

Flow rate: 50.00 cm^3 STP/min

	Temperature (°C)	Temperature Ramp Rates (°C/min)	Time (min)
1	90.0	10	60
2	400.0	10	240
3	25.0	20	10

Use to regenerate (degas) the optional sorption trap. The specified gas flows over the trap while the trap heater is subjected to the specified temperature profile.

Trap Regeneration Conditions

Selections	Description
Description [*text box*]	A description for this step.
Carrier Gas [*drop-down box*]	Select the carrier gas and enter the flow rate.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

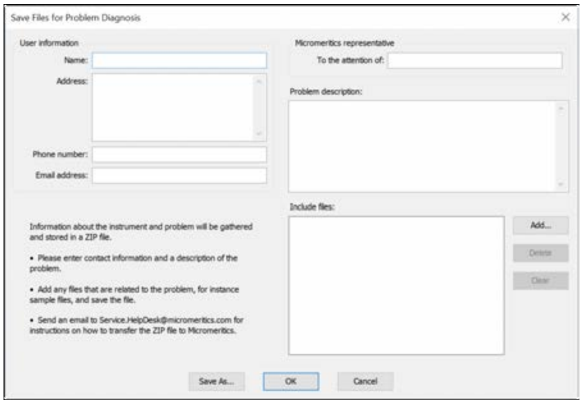
DIAGNOSTIC TEST REPORT

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.



Information about the instrument and problem will be gathered and stored in a ZIP file.

- Please enter contact information and a description of the problem.
- Add any files that are related to the problem, for instance sample files, and save the file.
- Send an email to Service.HelpDesk@micromeritics.com for instructions on how to transfer the ZIP file to Micromeritics.

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 [*button***]**	■ **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).

Save Files for Problem Diagnostics (continued)

Selections	Description
User Information **[***text box***]**	Enter information for the person to be contacted by a Micromeritics 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 - 6*.

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13 CALIBRATION

Unit [n] > Calibration > Calibrate

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.

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 CALIBRATION

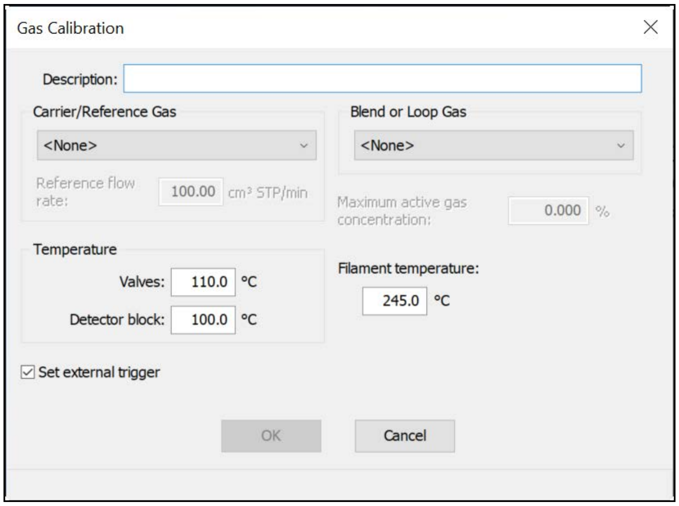
Sample Files on page 3 - 2

Use to calibrate the TCD so that peak area data can be converted to volume data. During a gas calibration, a series of known gas mixtures flows through the analyzer and the resultant signal readings are recorded. The analyzer can then use these data to calculate the concentrations of unknown mixtures flowing past the detector during subsequent analyses.

In some cases (such as in TPR analyses), the analyzer needs to know how to correlate the signal readings collected in the analysis with the volume of gas uptake at any given point in the analysis. If the analyzer is provided with a series of known gas concentrations, and it records the signal associated with each known concentration, then it can use this information to calculate the concentrations associated with the signals it recorded during the analysis. From that point, it can also calculate the volume of gas associated with each peak in the data.

Zeroing all Mass Flow Controllers is recommended prior to gas calibration to ensure its accuracy. Consider inserting a Zero MFCs task to the analysis conditions list. See Experiment Steps, Zero Mass Flow Controllers.

1. Prepare and install a clean, empty sample tube.
2. Ensure the correct gases are connected and the *Adsorptive Properties* file has been updated.
3. Create the sample file:
   a. Go to ***File > New Sample***.
   b. Complete the fields on the *Sample Description* tab, as needed.
4. Select the *Analysis Conditions* tab.
5. Click **Insert** and select *Gas Calibration*.
6. Click **OK**.



Gas Calibration

Description:

Carrier/Reference Gas: <None>

Reference flow rate: 100.00 cm³ STP/min

Blend or Loop Gas: <None>

Maximum active gas concentration: 0.000 %

Temperature

Valves: 110.0 °C

Detector block: 100.0 °C

Filament temperature: 245.0 °C

☑ Set external trigger

7. Complete the window entering the appropriate information for the sample being used.

> Specific selections and entries shown in this table are only applicable when using Micromeritics samples. Make the necessary adjustments when using non-Micromeritics samples.

Selections	Description
Blend or Loop Gas [*drop-down box***]**	Select *Hydrogen-Argon* and enter *10* for the *Maximum active gas concentration* percentage. The concentration is listed on the Certificate of Analysis on the gas cylinder.
Carrier/Reference Gas [*drop-down box***]**	Select *H_2/Ar* and enter *50* as the *Reference flow rate*. Select Ar as the Blend.
Description [*text box***]**	Enter a description for this calibration.
Filament temperature [*selection***]**	Enter the temperature between 20 to 250 °C for the filament. A typical setting is 245 °C.
Set external trigger [*check box***]**	If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected.
Temperature [*group box***]**	Enter a value between 20.0 and 150.0 °C for the *Valves* and *Detector block*. A typical setting is 110 °C for the valves and 100 °C for the block.

8. Click **OK** again to close the Calibration window.
9. Click **Save As**, provide a new file name, then click **Close**.
10. Go to ***Unit [n] > Sample Analysis***.
11. Click **Browse** to locate sample file previously created.
12. Click **Start** to start the analysis. Use the other views of the analysis window to observe the progress of the analysis. During the automatic analysis, the analyzer decreases the proportion of the analysis gas in 10% increments, beginning with 100% and ending with 0%. The resultant data should appear as a series of ten stepwise changes in the TCD signal.
13. When the analysis ends, close the *Analysis* window.
14. When the displayed sample temperature reaches the ambient temperature, open the furnace and remove the sample tube.
15. Go to ***Options > Signal Calibration > New***. Select the calibration file, then click **Open**.
16. Click **Load Calibration Data** then select the sample file for this calibration, then click **OK**.
17. Click **OK**. The data in the sample file are automatically inserted into the appropriate fields of the *Signal Calibration* window.

18. Assess the gas concentration plot, Goodness of Fit, and Coefficients to determine if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, use a calibration file with a very low value for Goodness of Fit (less than 1/2% of the maximum concentration) when 1st or 2nd Degree is specified.

    Data points that deviate significantly from linear can be deleted. Evaluate the results when the degree is changed (select a different degree from the drop-down list).

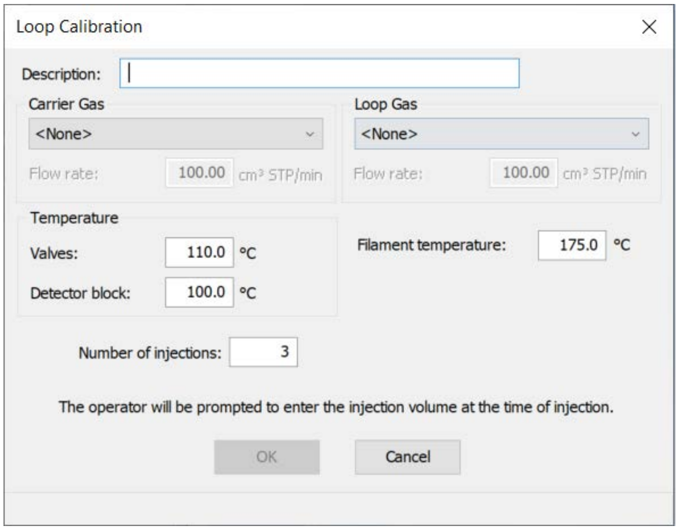
19. When satisfied with the calibration file, enter a descriptive name in the *Description* field.
20. Click **Save**, then click **Close**.
21. Go to ***Options > Signal Calibration > Report*** to select a report to generate.

LOOP CALIBRATION

Sample Files on page 3 - 2

Use to calibrate the gas injection loop's volume under current conditions so that precise volume injections can be made in analyses that perform loop injections, such as Pulse Chemisorption experiments. During a loop calibration, the operator repeatedly injects a known volume of gas through the analyzer septum using a syringe, then the analyzer repeatedly injects gas from the loop. This results in a series of recorded peaks in the TCD signal. After the operation completes, the operator can adjust the peaks in the peak editor and make a loop calibration report. This calculates the loop volume by comparing the average area of the manual injection peaks to the average area of the automatic injection peaks. The operator then enters the calculated loop volume under ***Unit [n] > Unit Configuration*** for use in subsequent analyses.

1. Install the correct injection loop.
2. Prepare and install a clean, empty sample tube.
3. Ensure the correct gases are connected.
4. Create the sample file:
   a. Go to ***File > New Sample***.
   b. Complete the fields on the *Sample Description* tab, as needed.
5. Select the *Analysis Conditions* tab.
6. Click **Insert** and select *Loop Calibration*.
7. Click **OK**.



Carrier Gas: <None>
Flow rate: 100.00 cm³ STP/min
Loop Gas: <None>
Flow rate: 100.00 cm³ STP/min
Valves: 110.0 °C
Detector block: 100.0 °C
Filament temperature: 175.0 °C
Number of injections: 3

The operator will be prompted to enter the injection volume at the time of injection.

8. Complete the dialog box entering the appropriate information for the sample being used.

Specific selections and entries shown in this table are only applicable when using Micromeritics samples. Make the necessary adjustments when using non-Micromeritics samples.

Selections	Description
Carrier Gas **[***drop-down box***]**	Select *Helium* and enter *50* for the **flow rate**.
Description [*text box***]**	Enter a description for this calibration.
Filament temperature **[***selection***]**	Enter the temperature between 20 to 250 degrees for the filament.
Loop Gas **[***drop-down box***]**	Select *Argon* and enter *50* for the **flow rate**.
Number of injections **[***text box***]**	Enter the number of automatic injections (3-10) to be done with the loop during calibration.
Temperature **[***group box***]**	Enter a value between 20.0 and 150.0 degrees for the **Valves** and **Detector block**.

9. Click **OK** again to close the Calibration window.
10. Click **Save As**, provide a new file name, then click **Close**.
11. Go to ***Unit [n] > Sample Analysis***.
12. Click **Browse** to locate sample file previously created.
13. Click **Start** to start the analysis. Use the other views of the analysis window to observe the progress of the analysis. During the first half of the analysis, the analyzer displays a prompt before each manual injection. Enter the syringe's physical injection volume (which should be approximately 80% of the nominal loop volume), click **Continue**, then inject gas through the analyzer septum from the syringe. During the second half of the analysis, the analyzer automatically injects gas from the loop. The resultant data should appear as a series of peaks in the TCD signal.
14. When the analysis ends, close the *Analysis* window.
15. When the displayed sample temperature reaches the ambient temperature, open the furnace and remove the sample tube.

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

16. Go to ***File > Open > [.SMP]***. Open the .SMP file containing the calibration experiment. Verify the defined peaks are properly marked (or correct them) using the *Peak Editor*.

17. On the *Reports* tab, select only the *Loop Calibration* report option. Click **Preview** to view the results, including the Loop Volume.
18. Go to ***Unit [n] > Unit Configuration***. In the *Loop Volume* field, enter the *Loop Volume*.

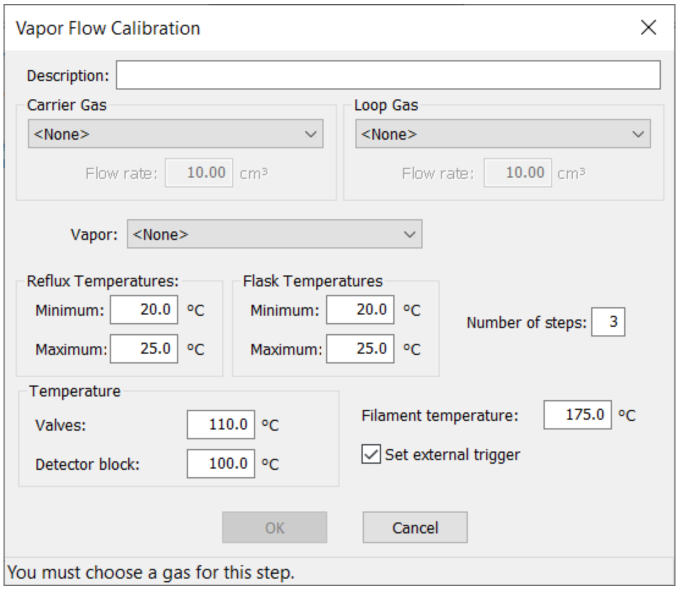
VAPOR FLOW CALIBRATION

Use to calibrate the type of vapor to be used when using the optional vapor generator. Associating the analysis data with a vapor generator calibration file allows the conversion of peak area data to volume data. The calibration can be performed either before or after performing the analysis. A single calibration file can be used with multiple analysis files.

1. Go to ***File > Open > Sample information***.
2. Enter a calibration file name (.CAL).
3. Click **OK**, then click **Yes** to confirm.
4. Complete the remaining fields on the *Sample Description* window, as needed.

If the window displays in *Basic* presentation, click *Advanced* in the view selector drop-down at the bottom of the window.

5. Select the *Analysis Conditions* tab.
6. Select *AutoChem III* in the *View conditions for* drop-down list.
7. Click **Insert**, then select *Vapor Flow Calibration*.



Description:

Carrier Gas
<None>
Flow rate: 10.00 cm³

Loop Gas
<None>
Flow rate: 10.00 cm³

Vapor: <None>

Reflux Temperatures:
Minimum: 20.0 °C
Maximum: 25.0 °C

Flask Temperatures
Minimum: 20.0 °C
Maximum: 25.0 °C

Number of steps: 3

Temperature
Valves: 110.0 °C
Detector block: 100.0 °C

Filament temperature: 175.0 °C

☑ Set external trigger

You must choose a gas for this step.

8. Complete the dialog box according to this table:

Selections	Description
Carrier Gas/Loop Gas [*drop-down box*]	Select the gases to be used for this step and enter the **Flow rate**.
Description [*text box*]	Description for this calibration.
Flask Temperatures [*group box*]	Enter a value between 20.0 and 120.0 degrees for the **Minimum** and **Maximum**.
Filament temperature [*selection*]	Enter the temperature for the filament.
Number of steps [*text box*]	Enter the number of steps (between 3 and 7) for this calibration.
Reflux Temperatures [*group box*]	Enter a value between 20.0 and 100.0 degrees for the **Minimum** and **Maximum**.
Set external trigger [*check box*]	If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected.
Temperature [*group box*]	Enter a value between 20.0 and 150.0 degrees for the **Valves** and **Detector block**.
Vapor [*drop-down box*]	Select the vapor to be used.

9. Click **OK**.
10. Go to ***Unit [n] > Sample Analysis***.
11. Click **Browse** and select the sample file just created. Make edits as required.
12. Click **Start**.
13. Select an option from the *View* drop-down list to observe the progress of the analysis.
14. When the analysis completes, close the *Analysis* window.
15. When the displayed sample temperature reaches the ambient temperature, open the furnace. Use gloves and remove the sample tube. These fields do not require editing; the *Reflux* and *Flask* temperatures entered on the *Vapor Calibration Experiment* window are used.

16. To edit the calibration file, go to ***Options > Signal Calibration > New***.

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

   a. Select *Load Calibration Data* and select the sample file for this calibration. The data contained in the file are automatically loaded.
   b. Access the *Calculations* document on the Micromeritics web page (www.micromeritics.com) for information on calculating the volume of each vapor injection, then enter the values for each peak.
   c. Assess the plot, *Goodness of Fit*, and *Coefficients* to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, use a calibration file with a very low value for *Goodness of Fit* (less than 1 to 2% of the signal), when 1st or 2nd Degree is specified.
   d. To delete any peaks that are outliers, highlight the peak data in the table and click **Delete Peak**.
   e. Evaluate the results when the degree is changed (select a different degree from the drop-down list).
   f. When satisfied with the calibration file, click **Save** then click **Close**.

VAPOR PULSE CALIBRATION

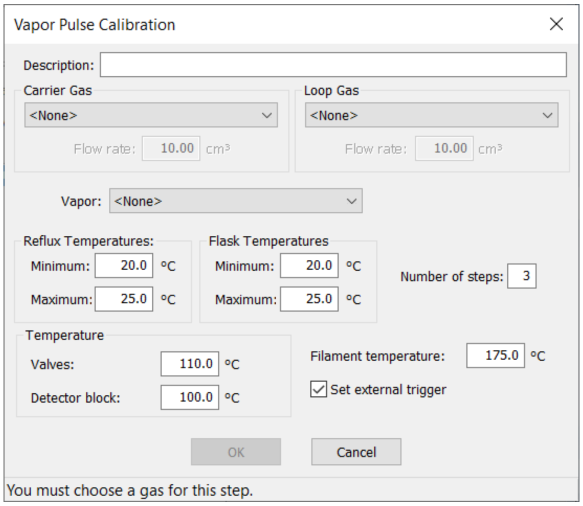
Use to associate analysis data with a vapor pulse calibration file. This allows the conversion of pulse peak areas to the quantity of vapor adsorbed for each peak. The calibration can be performed either before or after performing the analysis. A single calibration file can be used with multiple analysis files.

Specific selections and entries shown in this table are only applicable when using Micromeritics samples. Make the necessary adjustments when using non-Micromeritics samples.

1. Go to ***File > Open > Sample information***.
2. Enter a calibration file name (.CAL).
3. Click **OK**, then click **Yes** to confirm.
4. Complete the remaining fields on the *Sample Description* window, as needed.

If the window displays in *Basic* presentation, click *Advanced* in the view selector drop-down at the bottom of the window.

5. Select the *Analysis Conditions* tab.
6. Select *AutoChem III* in the *View conditions for* drop-down list.
7. Click **Insert**, then select *Vapor Pulse Calibration*.



Vapor Pulse Calibration

Description:

Carrier Gas: <None>; Flow rate: 10.00 cm³

Loop Gas: <None>; Flow rate: 10.00 cm³

Vapor: <None>

Reflux Temperatures: Minimum: 20.0 °C; Maximum: 25.0 °C

Flask Temperatures: Minimum: 20.0 °C; Maximum: 25.0 °C

Number of steps: 3

Temperature: Valves: 110.0 °C; Detector block: 100.0 °C

Filament temperature: 175.0 °C

☑ Set external trigger

You must choose a gas for this step.

8. Complete the dialog box according to this table:

Selections	Description
Carrier Gas/Loop Gas [*drop-down box***]**	Select the gases to be used for this step and enter the **Flow rate**.
Description [*text box***]**	Description for this calibration.
Flask Temperatures [*group box***]**	Enter a value between 20.0 and 120.0 degrees for the **Minimum** and **Maximum**.
Filament temperature [*selection***]**	Enter the temperature between 20 to 250 degrees for the filament.
Number of steps [*text box***]**	Enter the number of steps (between 3 and 7) for this calibration.
Reflux Temperatures [*group box***]**	Enter a value between 20.0 and 100.0 degrees for the **Minimum** and **Maximum**.
Set external trigger [*check box***]**	If selected, the contact closure used to trigger an external mass spectrometer will be activated. If deselected, the contact closure will be deselected.
Temperature [*group box***]**	Enter a value between 20.0 and 150.0 degrees for the **Valves** and **Detector block**.
Vapor [*drop-down box***]**	Select the vapor to be used.

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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A ADVANCED REPORTS - PYTHON MODULE

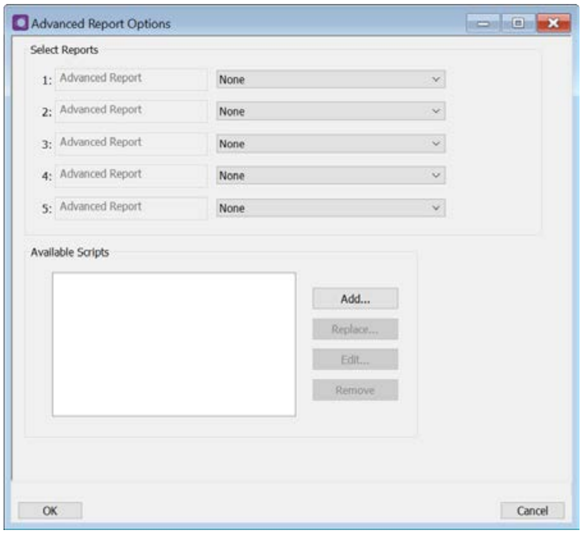
- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data. Tabular reports are created 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 REPORTS

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 - 11*** 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.



1: Advanced Report None
2: Advanced Report None
3: Advanced Report None
4: Advanced Report None
5: Advanced Report None

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.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 6*.

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. 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.
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.

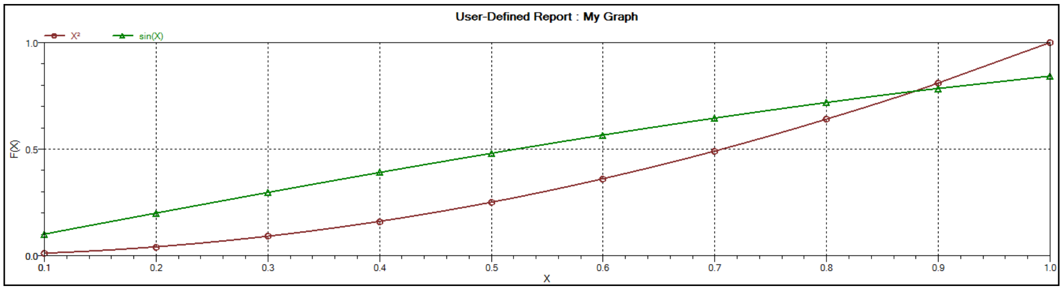
PYTHON REPORTS

Graphic Report

This script is an example of the mic module producing a graph with two curves:

```python
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:

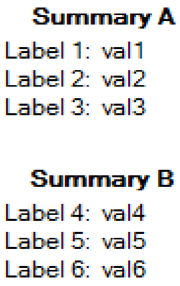


Summary Report

This script produces a summary report with two summaries:

```python
import mic
import numpy as np

mic.summary( "My Summaries" )
mic.summary.add( "Summary A",
                 ["Label 1:", "Label 2:", "Label 3:"],
                 ["val1", "val2", "val3"] )
mic.summary.add( "Summary B",
                 ["Label 4:", "Label 5:", "Label 6:"],
                 ["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

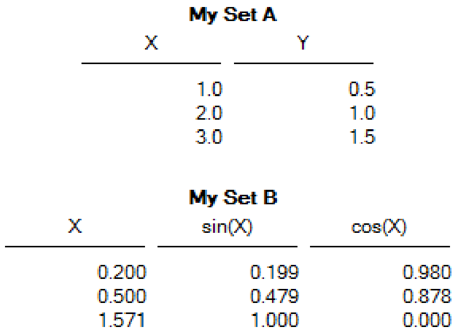
Tabular Report

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.

```python
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

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.

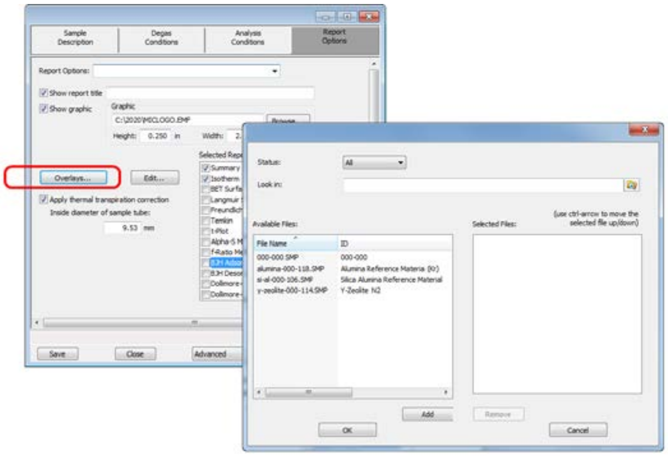
Note the calls to *mic_chem.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```python
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',
```

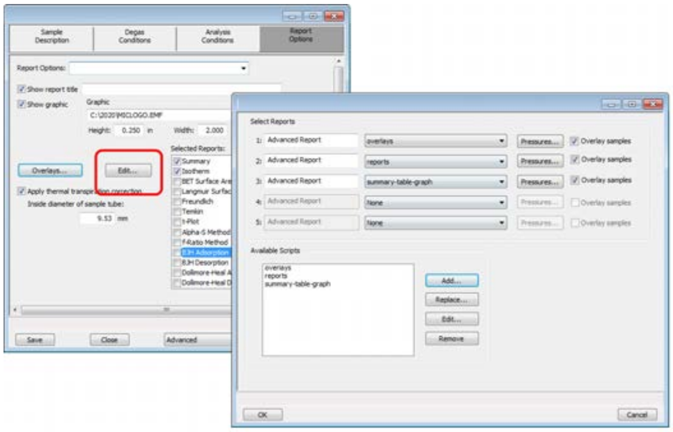
```
                   mic.sample_information('sample description'),
                   '{:8.3f}'.format(mic.sample_information('analysis temperature')) + ' K',
                   '{:8.3f}'.format(mic.sample_information('sample density')) + ' g/cm³' ] )

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 ] )
```

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 - 3***.



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 con-
vertible)
  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 exper-
```

```
iment
  data) through n where n is the number of secondary experiments (e.g.,
  mass spec data).
```

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 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.
```

B ATOMIC WEIGHTS AND CROSS-SECTIONAL AREAS

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 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

Gas Conversion Constants for the MFC (continued)

Gas	**Symbol**	**MFC Conversion Constant (H_2 = 1.000)**
Cyanogen	C_2N_2	0.4950
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	$CCl_2F\text{-}CClF_2$	0.2277
Freon 114	$CCl_2F_4\text{-}CClF_2$	0.2554
Freon 115	$CClF_2\text{-}CF_3$	0.2713

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Freon 116	CF_3-CF_3	0.2277
Germane	GeH_4	0.6436
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

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Nitrogen Trioxide	N_2O3	0.4356
Nitrogen Trifluoride	NF_3	0.5446
Nitrous Oxide	N_2O	0.7426
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

Gas Conversion Constants for the MFC (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Xenon	Xe	1.3762

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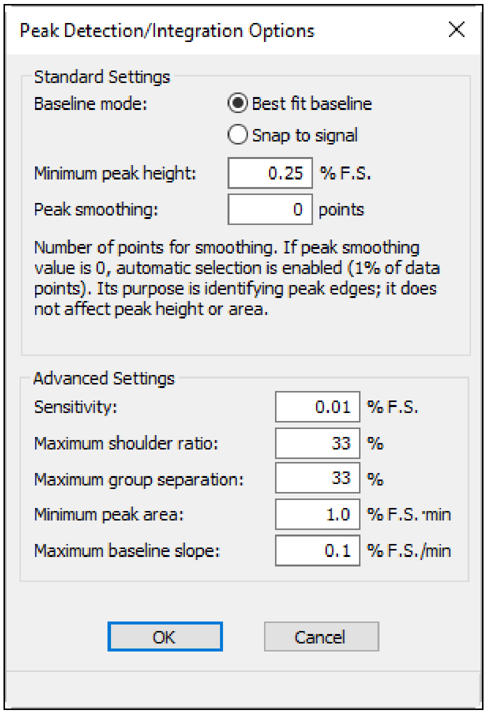
D PEAK DETECTION / INTEGRATION OPTIONS

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. peaks are found using events that occurred during the analysis — such as the time an injection started — instead of peak detection options.



Peak Detection/Integration Options

Standard Settings

Baseline mode: ● Best fit baseline ○ Snap to signal

Minimum peak height: 0.25 % F.S.

Peak smoothing: 0 points

Number of points for smoothing. If peak smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges; it does not affect peak height or area.

Advanced Settings

Sensitivity: 0.01 % F.S.

Maximum shoulder ratio: 33 %

Maximum group separation: 33 %

Minimum peak area: 1.0 % F.S.·min

Maximum baseline slope: 0.1 % F.S./min

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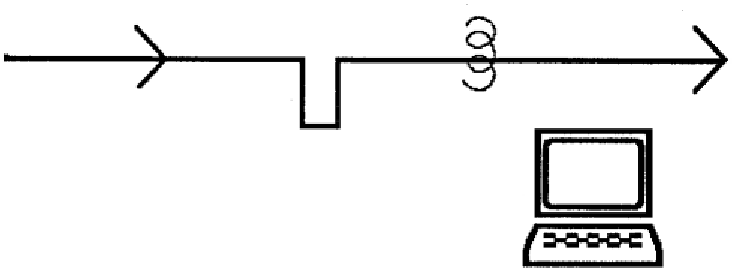
E TEMPERATURE PROGRAMMED ANALYSES

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.

Data that are Collected

The detector reports the amount of electricity (in volts) required to keep its temperature constant during the analysis.

---

[1] The thermal conductivity of a gas is its ability to conduct heat. Each gas has a distinct thermal conductivity.

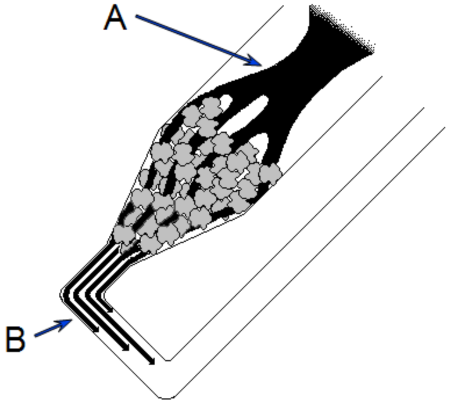
The Role of the Sample Temperature

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.

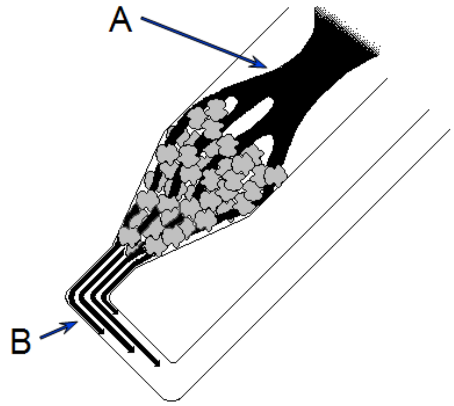
For example, consider the example of a Temperature-Programmed Reduction (TPR). During the TPR, a metal oxide is reacted with hydrogen to form a pure metal. This reaction is referred to as "reducing" the metal; for example, TPR of a catalyst containing Platinum. Argon, which has a very low thermal conductivity, 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.



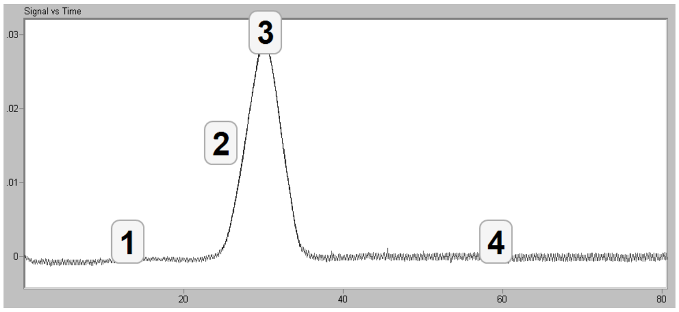
A. Gas flows into the sample tube and through the sample.
B. Before the reaction begins, the concentration of gases exiting the sample tube is unchanged after flowing through the sample.

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 sorption or 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.

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F WORKSHEETS

Worksheets in this section may be copied as needed.

micromeritics®

AUTOCHEM III GAS CONNECTIONS WORKSHEET

Use this form to record the connected gases. Use this form as a checklist against the gases identified in the analyzer application.

Unit number (S/N): ______________________

Date: ______________________

Operator: ______________________

Preparation Gas	Carrier / Reference Gas	Loop Gas

Copy and use this worksheet as needed

MANUAL INJECTIONS WORKSHEET FOR AUTOCHEM

Use this worksheet to list the planned injection volumes, then check indicate if completed.

Injection	Comments/Volume	Completed
1		☐
2		☐
3		☐
4		☐
5		☐
6		☐
7		☐
8		☐
9		☐
10		☐
11		☐
12		☐
13		☐
14		☐
15		☐
16		☐
17		☐
18		☐
19		☐
20		☐

Copy and use this worksheet as needed

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:

Copy and use this worksheet as needed

G EXPORTED DATA EXAMPLE

This exported data has been truncated for this manual.

```
Sample Information

                    Method:    $ CuO H2Ar TPR
                    Sample:    CuO H2Ar TPR
                  Operator:    PK
                 Submitter:    Performance Test
                 Mass type:    Calculated
                Empty tube:    26.5221 g
             Sample + tube:    26.5843 g
               Sample mass:    0.0622 g
                   Density:    1.000 g/cm³
              Type of data:    Automatically collected
           Instrument type:    2930
  Original instrument type:    2930
                  Comments:    Performance Test Run1
CuO Lot SZBG3070H
Sample+Tube mass after analysis: 26.5749g

Analysis Conditions
```

```
  Analysis conditions:    Analysis Conditions
  View conditions for:    AutoChem III 2930

             Baseline Options

          Stable Baseline
         Slope threshold:    0.010 %/min
                Duration:    5.00 min
     Change from Baseline
  Acceleration threshold:    0.200 %/min²
                Duration:    0.10 min
       Return to Baseline
  Acceleration threshold:    0.050 %/min²
                Duration:    1.00 min
                                                                                                                        -
        Tasks
              Description                                                                                               -
                          Details

  1.01    Experiment            --- CuO TPR 50 sccm H2Ar ---
Temperature Programmed

  1.02    Instrument Settings   --- Instrument Settings ---
Carrier: H2Ar @ 50.00 (None @ 100.00)
```

```
Loop: None @ 10.00 (None @ 100.00)
Prep.: None @ 10.00 (None @ 100.00)

(Bypass)  (Bypass)  (Fill)  (Analyze)  Trap

Filaments:245.0 °C (Disabled)

  1.03    Wait                   --- Wait ---
  Wait 10.00 min.
  1.04    Wait                   --- Wait ---
  Wait until baseline and temperatures are stable.
  1.05    Start Recording        --- Start Recording ---
  One measurement every 0.20 s
  Set external trigger: No
  1.06    Temperature Ramp       --- Temperature Ramp ---
  Type: Sample
  Temperature: 400.0 °C
  Ramp rate: 10.0 °C/min
  Hold time: 5.00 min
  1.07    Stop Recording           --- Stop Recording ---

          Termination            --- Termination ---
Carrier:   Ar @ 10.00 (H2Ar @ 50.00)
```

```
(Bypass)  (Bypass)  (Fill)  Prepare  (Trap)

Port:      60.0 °C (110.0 °C)
Sample:      Ambient (400.0 °C)
Rate:      Ambient (10.0 °C/min)

Filaments:Disabled (245.0 °C)

Adsorptive Properties

                      Adsorptive:    Hydrogen-Argon (H2Ar)
       Maximum manifold pressure:    123.323 kPa
  Molecular cross-sectional area:    0.123 nm²
      Adsorbate molecular weight:    2.02
   Relative thermal conductivity:    1.40
                       Gas blend:    Yes
                       Inert gas:    Argon
                      Active gas:    Hydrogen
           % Active Concentration    9.998 %
                Fluid properties:    H:\FPI\fpiv7\hydrogen.fpi

  Adsorptive:    Argon (Ar)
```

```
Report Options

  Report options:    Report Options

        Summary: Yes
        BET Surface Area: No
        Pulse Chemisorption: No
        First Order Kinetics: No
        Loop Calibration: No
        Active Surface Area: No
        Signal and Temperature vs. Time: Yes

   Signal and Temperature vs. Time

         Show peak marks:    Yes
                  X-axis:    Time
               Autoscale:    Yes
                  Y-axis:    Signal
               Autoscale:    Yes
             Experiments:
  1: CuO TPR 50 sccm H2Ar    Yes
```

```

      Active Gas Flow Rate vs. Temperature 1: Yes

    Active Gas Flow Rate vs. Temperature 1

       Show peak marks:    Yes
                X-axis:    Temperature
             Autoscale:    Yes
                Y-axis:    Active Gas Flow Rate
             Autoscale:    Yes
           Experiments:
1: CuO TPR 50 sccm H2Ar    Yes

      Signal vs. Temperature 1: No
      Active Gas Flow Rate vs. Time 1: No
      Active Gas Flow Rate vs. Temperature 2: No
      Signal vs. Time: No
      Signal vs. Temperature 2: No
      Active Gas Flow Rate vs. Time 2: No
      Active Gas Flow Rate vs. Temperature 3: No
      Signal and Active Gas Flow Rate vs. Time: No
      Advanced Reports: No
      Options Report: No
      Sample Log: Yes
```

```
        Peak Detection/Integration Options

             Baseline mode:    Best fit baseline
       Minimum peak height:    0.25 % F.S.
            Peak smoothing:    0 points
               Sensitivity:    1.0e-02 % F.S.
    Maximum shoulder ratio:    33%
  Maximum group separation:    33%
         Minimum peak area:    1.0 % F.S.·min
    Maximum baseline slope:    0.1 % F.S./sec


Collected Data

                  Collected Data

         Serial number:    002
            Start time:    12/10/2020 4:30:04 PM
             Stop time:    12/10/2020 5:29:57 PM
   Ambient temperature:    25.0 °C
  Atmospheric pressure:    101.3 kPa
              p° of N2:    103.3249 kPa
```

```
 Tables have been truncated to 1000 entries for preview.
Consider restricting the time range, using a larger time interval, or exporting directly to a file.

                               CuO TPR 50 sccm H2Ar

                     Experiment ID:    1: CuO TPR 50 sccm H2Ar
                       Calibration:    (AutoChem_III_0002) 10% H2/Ar 50cc 121020
                     Analysis type:    Temperature Programmed Desorption
                Measured flow rate:    50.00000 cm³ STP/min
  Active concentration % at time 0:    9.998 %
         Physical injection volume:     1.0000 mL
                     Use fit peaks:    No
                        Start time:    0.00 min
                         Stop time:    43.15 min

                  CuO TPR 50 sccm H2Ar

                              Temperature
            Start    Stop         at
   Peak     Time     Time       Maximum      Quantity
  Number    (min)    (min)       (°C)        (cm³ STP)
-----
```

```
          1     3.55    33.93         207.42    15.383484

                                       CuO TPR 50 sccm H2Ar

                                                          Carrier       Loop
                                                            Flow        Flow
                                                            Rate        Rate       Reflux
      Time     Temperature     Signal (a     Injection      (cm³        (cm³      Temperature
     (min)        (°C)            u )          Event      STP/min)    STP/min)       (°C)
   -------
     0.0000           27.3    0.020317575    0               50.00        0.03           24.3
     0.0033           27.3    0.020331932    0               50.00        0.03           24.3
     0.0067           27.3    0.020329252    0               50.00        0.03           24.3
     0.0100           27.3    0.020342696    0               50.00        0.03           24.3
     0.0133           27.3    0.020332888    0               50.00        0.03           24.3
     0.0167           27.3    0.020323444    0               50.00        0.03           24.3
     0.0200           27.3    0.020320816    0               50.00        0.03           24.3
     0.0233           27.3    0.020332487    0               50.00        0.03           24.3
     0.0267           27.3    0.020331409    0               50.00        0.03           24.3
     0.0300           27.3    0.020303261    0               50.00        0.03           24.3
     0.0333           27.3    0.020322692    0               50.00        0.03           24.3
     0.0367           27.3    0.020317623    0               50.00        0.03           24.3
     0.0400           27.3    0.020327993    0               50.00        0.03           24.3
```

```
0.0433           27.3    0.020315453    0               50.00        0.03           24.3
0.0467           27.3    0.020320794    0               50.00        0.03           24.3
0.0500           27.3    0.020323554    0               50.00        0.03           24.3
0.0533           27.3    0.020323807    0               50.00        0.03           24.3
0.0567           27.3    0.020352364    0               50.00        0.03           24.3
0.0600           27.3    0.020323513    0               50.00        0.03           24.3
0.0633           27.3    0.020334244    0               50.00        0.03           24.3
0.0667           27.3    0.020322597    0               50.00        0.03           24.3
0.0700           27.3    0.020311031    0               50.00        0.03           24.3
0.0733           27.3    0.020342128    0               50.00        0.03           24.3
0.0767           27.3    0.020313352    0               50.00        0.03           24.3
0.0800           27.3    0.020326018    0               50.00        0.03           24.3
0.0833           27.3    0.02031542     0               50.00        0.03           24.3
0.0867           27.3    0.020323347    0               50.00        0.03           24.3
0.0900           27.3    0.020325938    0               50.00        0.03           24.3
0.0933           27.3    0.020327948    0               50.00        0.03           24.3
0.0967           27.3    0.020308368    0               50.00        0.03           24.3
0.1000           27.3    0.020344859    0               50.00        0.03           24.3
```

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:

AutoChem III Automated Catalyst Characterization System Model 2930
Includes variant models with Cold trap option, Vapor Generator Accessories and ECR

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 61010-2-010:2019 - *Particular requirements for laboratory equipment for the heating of materials.*
EN 61010-2-081:2020 - *Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes*

2014/30/EU (EMC)
EN 61326-1:2020 - *Electrical equipment for measurement, control and laboratory use — EMC requirements — Part 1: General requirements*
EN 61000-3-2:2018 - *Part 3-2: Limits — Limits for harmonic current emissions (equipment input current ≤ 16 A per phase)*
EN 61000-3-3:2013;+A1:2017 - *Part 3-3: Limits — Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current <= 16 A per phase and not subject to conditional connection*

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

Name: John McCaffrey, Ph.D. **Title:** Vice President, R & D

Signature: **Date of issue:** 08/12/2022

Location: Norcross, GA USA

micromeritics®

UK 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:

AutoChem III Automated Catalyst Characterization System 2930
Includes variant models with Cold trap option, Vapor Generator Accessories and ECR

is in conformity with the following UK legislation:

Electrical Equipment (Safety) Regulations 2016
Electromagnetic Compatibility Regulations 2016
Restriction of the Use of Certain Hazardous Substances in E&E Equipment Regulations 2012

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

Electrical Equipment (Safety) Regulations 2016

EN 61010-1:2012/A1:2019 - *Safety requirements for electrical equipment for measurement, control, and laboratory use — Part 1: General requirements.*
EN 61010-2-010:2019 - *Particular requirements for laboratory equipment for the heating of materials.*
EN 61010-2-081:2020 - *Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes*

Electromagnetic Compatibility Regulations 2016

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

Restriction of the Use of Certain Hazardous Substances in E&E Equipment Regulations 2012

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.

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