SURFACE AREA ANALYZER



# micromeritics®

# **OPERATOR MANUAL**

239-42801-01 July 2021 (Rev E)



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# WARRANTY

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

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

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

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

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

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

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

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

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



# **CORPORATE PROFILE**

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



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#### **Micromeritics Learning Center**

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# ABOUT THIS MANUAL

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

- Calculations document (PDF)
- Error Messages document (PDF)
- Parts and Accessories



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

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.



<u>WARNING</u> — Warnings contain information to help prevent actions that may cause personal injury.



## GENERAL SAFETY



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

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

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



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



#### INTENDED USE

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

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

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

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



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



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

#### **TRAINING**

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



## **ENVIRONMENTALLY FRIENDLY USE PERIOD**

**Hazardous Substances Table** 

		Hazardous Substances				
Part Name	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Cover	x	О	О	О	О	o
Power Supplies	х	o	0	О	o	O
Printed Circuit Boards	x	o	0	O	o	0
Cables, Con- nectors & Transducers	x	o	O	O	o	0

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

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





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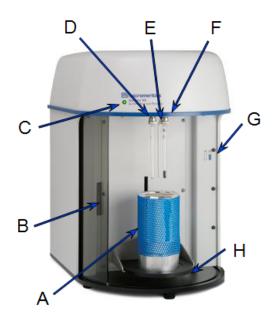
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# 1 ANALYZER COMPONENTS

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

## **FRONT COMPONENTS**



- A. Dewar
- B. Sample safety shield
- C. Power indicator light
- D. Balance port and balance tube
- E. P<sub>0</sub> port and tube
- F. Sample port and sample tube
- G. Tube position diagram
- H. Elevator

#### **Front Components**

Components	Description
Dewar	For housing cryogens used for analysis. The Dewar rests on an elevator.
Sample safety shield	Closes to cover the Dewar.
Power indicator light	Blinks when power is applied to the analyzer; illuminates when the analysis program is initiated and ready for operation.
Balance port and balance tube	The balance tube is identical to the sample tube. This design negates free-space errors introduced by thermal gradient variations or by initial mismeasurement of free space.
P <sub>0</sub> port and tube	Models 2390p and 2390t only. For measuring the saturation pressure.

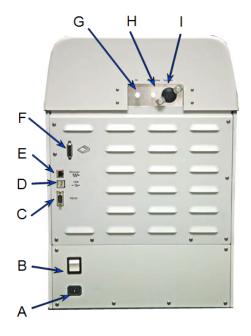


# **Front Components (continued)**

Components	Description
Sample port and sample tube	Holds the material to be analyzed.
Tube position diagram	Diagram of the positions of the balance and sample tubes. The empty tube represents the balance tube installed on the left port. The filled tube represents the sample tube installed on the right port.
Elevator	Allows placement of the Dewar around the sample and P <sub>0</sub> tubes. The elevator is raised automatically when the analysis is started and lowers automatically upon completion.



## REAR PANEL COMPONENTS



- A. Power connector
- B. Power switch
- C. RS-232 connector (for service personnel use)
- D. USB ports
- E. Ethernet port
- F. Keypad connection (used only with keypad configurations)
- G. Helium inlet port for helium gas supply
- H. Adsorptive gas inlet
- I. Vacuum pump connector

## **Rear Panel Components**

Components	Description
Adsorptive gas inlet	Attaches the analysis gas supply.
Ethernet port	Connects the analyzer to a network enabling notifications via e-mail of analysis completion, and to monitor analysis results using a web browser. A straight-through or shielded cross-over ethernet cable can be used since the analyzer will adapt to either type.
Helium inlet port	Attaches the helium gas supply.
Keypad connection	Connects the keypad to the analyzer.
Power connector	Connects the analyzer to the power supply.
Power switch	Powers the analyzer on and off.
RS-232 connector	Enables usage of a serial line for data transmission. This port can also be used to attach an analytical balance for automatic transmission of the sample mass. See <u>Analyze Functions on page 3-44</u> for details on transferring the mass value to the analyzer. See <u>RS-232 Operation on page C - 1</u> for the pin assignment for the RS-232 port.

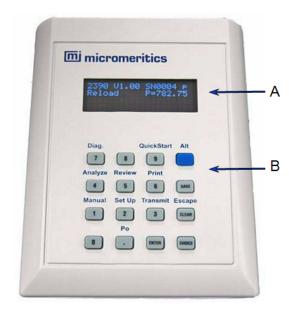


# **Rear Panel Components (continued)**

Components	Description
USB ports	Attaches external devices, such as printers, bar code readers, and keyboards. This connector is used to install software upgrades.
Vacuum pump connector	Attaches the vacuum pump hose.



## KEYPAD COMPONENTS



- A. Keypad window
- B. Keys



- A. Contrast adjustment
- B. Keypad cable port



# **EQUIPMENT OPTIONS AND UPGRADES**

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

Option	Description
Chiller Dewar	For Gemini model 2390t.
	A closed loop recirculating system that utilizes a high surface area copper coil to provide excellent heat transfer between the Dewar and the recirculating liquids. (missing or bad snippet) the Chiller Dewar Quick Start Guide [part number 025-42801-00].
FlowPrep	The FlowPrep applies both heat and a stream of inert gas to the sample to remove adsorbed contaminants from the surface and pores in preparation for analysis for up to six samples. Choose the temperature, gas, and flow rate best suited for the sample material. The FlowPrep is an independent unit and not controlled by the analyzer.
Smart VacPrep	The Smart VacPrep prepares samples by heating and evacuation. It contains six sample ports in which up to five temperatures, ramp rates, and soak times per sample are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. Samples can also be prepared, started, and completed independently. There is no need to wait for samples on other ports to finish. Front panel buttons allow a QuickStart operation with preprogrammed conditions.
	Up to three additional Smart VacPrep degassers can be connected to one computer permitting 24 preparation ports to be used. The Smart VacPrep is the recommended degassing unit.
VacPrep	The VacPrep offers two methods for removing contaminants. In addition to flowing gas, it provides vacuum to prepare samples by heating and evacuation of up to six samples. This combination provides preparation method options best suited to the material or application. Needle valves are also provided for introducing the vacuum slowly to prevent fluidization of samples. The VacPrep is an independent unit and not controlled by the analyzer.



Option	Description	
Vacuum Pump	The analyzer requires a vacuum pump for sample analysis. Vacuum pumps used must meet the following criteria:	
	<ul> <li>Achieve vacuum levels of 20 µmHg at the analyzer inlet</li> <li>Contain an anti-suckback valve to prevent vacuum pump oil from back streaming into the analyzer in the event of a vacuum pump failure</li> <li>Contain an NW16 inlet port for connection to the analyzer</li> </ul>	
	A device to reduce oil vapor backstreaming is recommended.	
	The vacuum pump must have an anti-suckback valve to prevent oil from being admitted into the analyzer should the power fail while the system is under vacuum. Pumps available from Micromeritics are equipped with an anti-suckback valve.	
	An oil-based or oil-free vacuum pump can be used with the analyzer. Appropriate vacuum pumps are available from Micromeritics.	



#### GAS REQUIREMENTS AND PURITY



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

Compressed gases are required for analyses. Gas cylinders or an outlet from a central source should be located near the analyzer.

Appropriate two-stage regulators which have been leak-checked and specially cleaned are required. Pressure relief valves should be set to no more than 30 psig (200 kPag). All gases should be of a purity listed below. Gas regulators can be ordered from Micromeritics. Parts and accessories can be found online at <a href="https://www.Micromeritics.com">www.Micromeritics.com</a>.

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

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

#### **CRYOGEN REQUIREMENTS**

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



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



# SPECIFICATIONS FOR THE GEMINI VII

## **Analysis**

Surface Area	From 0.1 m <sup>2</sup> , total From 0.01 m <sup>2</sup> /g, specific
Pore Volume	From $4 \times 10^{-6}$ cm <sup>3</sup> /g

#### **Electrical**

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

#### **Environment**

Humidity	20% to 80% relative, non-condensing		
Temperature	10 °C to 35 °C (50 °F to 95 °F), operating 0 °C to 50 °C (32 °F to 122 °F), non-operating		
Indoor/Outdoor Use	Indoor only (not suitable for wet locations) Altitude: 2000 m max Pollution degree of the intended environment: 2		
Degree of Ingress Protection	IPX0		

#### Gases

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



## **Physical**

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

#### **Pressure Measurement**

Range	0 to 950 mmHg		
P/P <sub>0</sub> Resolution	<10 <sup>-4</sup>		
Relative Pressure Range	0 to 1.0 P/P <sub>0</sub>		
Pressure Resolution	<0.1 mmHg		
Accuracy and Linearity (transducer manufacturer specification)	Better than ± 0.5% full scale		

#### Sample Tube and Dewar

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



#### Vacuum System

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

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

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

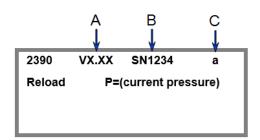


# KEYPAD WINDOW, KEYS AND FUNCTIONS

#### KEYPAD WINDOW

The keypad window provides information about the analyzer and the current operation. It consists of four lines.

- The first line of the display always contains the same information.
- The second, third, and fourth lines show different types of information, depending on the current operation.

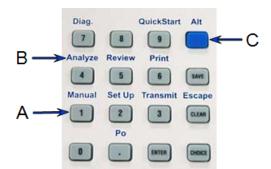


- A. Software version
- B. Analyzer serial number
- C. Gemini model: a, p, or t

#### **K**EYS

The keys on the keypad are used to execute commands and to enter information at prompts displayed. Some keys on the keypad perform a primary and an alternate function.

- The primary function is indicated by the number or command on the face of the key. Press only the key. For example, press 6 to enter the number 6 or **Save** to save the entry.
- The alternate function is indicated by the command above the key. Press the **Alt** key, then press the key associated with the function. For example, press **Alt+4** to start an analysis.



- A. Primary function
- B. Alternate function
- C. Alt key



# **Keypad Functions**

<b>Keypad Functions</b>	Description		
0-9	Enter the numbers 0-9.		
Character	Enter a decimal, a dash for the sample or instrument ID, a slash for the date, or a colon for time.		
Alt	Enter the alternate mode to perform commands printed above some keys. A plus sign (+) appears in the upper right corner of the display when alternate mode is active. Press <b>Alt</b> again to exit the alternate mode; the plus sign is removed from the display.		
CHOICE	Display the next message when at the <i>Reload</i> prompt. Display the next multiple choice item when in a command mode.		
CLEAR	Clear a message when at the <i>Reload</i> prompt. Clear an entry when in a command mode.		
ENTER	Completes an entry (when using the functions listed above) or begins an action (when using the functions listed below).		
SAVE	Saves the information entered and returns to the <i>Reload</i> prompt.		
Alt + 1 Manual	Enables manual mode in order to perform certain functions that may be requested by a service representative.		
Alt + 2 Set Up	Edits, copies, or transmits <i>Set Up</i> groups.		
Alt + 3 Transmit	Transmits analysis data over the serial line.		
Alt + 4 Analyze	Performs an analysis.		
Alt + 5 Review	Reviews or edits completed analysis data.		
Alt + 6 Print	Prints a report for the last analysis.		
Alt + 7 Diagnosis	Performs diagnostic tests and displays analyzer and calibration statistics.		
Alt + 9 QuickStart	Begins a <i>QuickStart</i> analysis.		
Alt + . (decimal) P_0	<ul> <li>Measures and records the saturation pressure when at the <i>Reload</i> prompt.</li> <li>Erases the previous keystroke when in a command mode.</li> </ul>		
Alt + CLEAR Escape	Performs any of the following functions:		
	<ul> <li>Discards all data entered in the current mode and return to the Reload prompt.</li> <li>Exits manual mode.</li> <li>Cancels an automatic operation in progress.</li> </ul>		
ALC - ENTER			
Alt + ENTER	Saves the current edit and return to the previous prompt.		



# KEYPAD AND KEYBOARD FUNCTIONS

#### **Standard Functions**

Keypad	Keyboard	Used To
CHOICE	Ctrl + N	<ul> <li>Displays the next message when in display mode.</li> <li>Displays the next multiple choice item when in a command mode.</li> </ul>
CLEAR	Ctrl + X	<ul><li>Clears a message when in display mode.</li><li>Clears an entry when in a command mode.</li></ul>
ENTER	Enter or Ctrl + M	Completes an entry or begin an action.
SAVE	Ctrl + W	Saves the entered information and return to display mode.

#### **Alternate Functions**

Function	Key Sequence		Used To
	Keypad	Keyboard	
Analyze	Alt + 4	Ctrl +A	Performs an analysis.
Diagnostics	Alt + 7	Ctrl + D	Performs certain diagnostics.
Escape	Alt + Clear	Esc	<ul> <li>Discards all data entered in the current mode and return to display mode</li> <li>Cancels an automatic operation in progress</li> <li>Exits manual mode.</li> </ul>
Manual	Alt + 1	Ctrl + Y	■ Enables manual mode. Press Alt + CLEAR to exit manual mode.
P <sub>0</sub>	Alt + . (decimal)	Ctrl + O	Measures the saturation pressure.
Print	Alt + 6	Ctrl + P	Prints an analysis or calibration report. If an automatic operation is in progress, prints a partial report.
QuickStart	Alt + 9	Ctrl + Q	Begins a series of analyses.
Review	Alt + 5	Ctrl + R	Reviews completed analysis data.



# **Alternate Functions (continued)**

Function	Key Sequence		Used To
	Keypad	Keyboard	
Set Up	Alt + 2	Ctrl + U	Displays or edit analysis parameters, report options, communication parameters, and system options.
Transmit	Alt + 3	Ctrl + T	Transmits analysis or calibration data over the serial line. Transmits a partial report if an automatic operation is in progress.



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# **2 OPERATIONAL PROCEDURES**

# **DEFINE SET UP GROUPS**

Analysis Conditions on page 3 - 14
Report Options on page 3 - 20
Communications on page 3 - 34
System Options on page 3 - 38
Set Up Commands on page 3 - 3

Alt + 2



Press **CHOICE** to display the options. Press **ENTER** to select.

A Set Up group can be defined by:

- Editing an unused Set Up group
- Copying the values of a *Set Up* group and editing them
- Resetting a Set Up group to factory-defined Set Up group

In addition, either of the following can be performed:

- Viewing the settings for a Set Up group
- Printing or transmitting a Set Up group



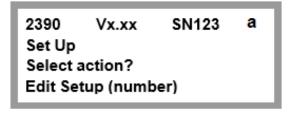
#### **EDIT**

2390 Vx.xx SN123 a Set Up Select Setup: (number) ? (set up description)

The number of the *Set Up* group last selected is displayed on the third line.

The fourth line contains one of the following:

- If an ID was entered, the ID of the Set Up group is displayed.
- If the Set Up group has been defined, but no ID was entered, used is displayed.
- If the Set Up group has not been defined, unused is displayed.
- If the Set Up group is being used and has been reset to a default configuration, factory defaults is displayed.
  - 1. Enter the Setup number or select an existing number.
  - 2. Select Edit Setup.

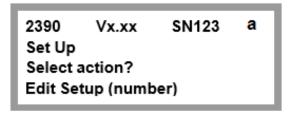


- 3. Select Analysis Conditions, Report Options, Communications, or System Options.
- 4. Enter the information, as prompted.

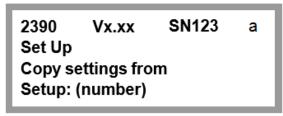


## COPY

- 1. Enter the Setup number or select an existing number.
- 2. Select Edit Setup (number).



3. At Copy settings from, enter the Setup (number) to copy from.

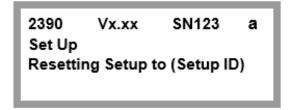


4. At Copying settings, enter the Setup (no) to copy to.

```
2390 Vx.xx SN123 a
Set Up
Copying settings
Setup (no.) to Setup (no.)
```

## RESET

- 1. Enter the Setup number or select an existing number.
- 2. Select Resetting Setup to (Setup ID).

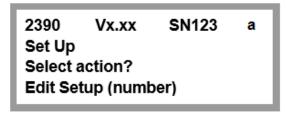


3. When the reset is complete, press **SAVE**.



#### **VIEW SETTINGS**

- 1. Enter the *Setup number* to view.
- 2. Select Edit Setup.



3. Make the applicable selections as prompted.

# PRINT OR TRANSMIT



A Set Up group cannot be printed or transmitted while it is being edited.

Press Alt + 6 to print the Set Up group or Alt + 3 to transmit the Set Up group.



## PREPARE FOR ANALYSIS

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

#### VERIFY REGULATOR PRESSURE

Verify that the regulator pressure for the nitrogen and helium cylinders are set to a level between 15-18 psig (103.4 - 124.1 kPa).



Check the gas cylinder pressure to ensure that it is greater than 200 psig. Pressures less than 200 psig may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.

#### **VERIFY VACUUM**

The vacuum level must be better than  $20 \times 10^{-3}$  mmHg at the instrument inlet. Most two-stage vacuum pumps, such as the one available from Micromeritics, will provide a vacuum level of about  $5 \times 10^{-3}$  mmHg.



#### 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.

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



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







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



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

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



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

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



### **DETERMINE THE SAMPLE MASS**

<u>Sample Data Worksheet for Gas Adsorption on page E - 1</u>

Analyze Samples with a Total Surface Area of 1.0 m2 or Less on page D - 1



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

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

- If there is between 0.1 m² and 1 m² in the tube, follow the recommendations for balancing the free space using glass beads, along with filler rods, and measure the resulting free space difference.
- If there is between 1 m² and 5 m² of total surface under test, measure the free space difference between the two tubes as a part of each analysis.
- If there is between 5 m² and 30 m², use the option to calculate the free space difference between the two tubes using the density of the sample in the tube, along with the mass entered. There are some special considerations when using the tubes with the cylindrical bulb on the end as opposed to the straight-walled tubes.
- If there is more than 30 m² in the tube total, it may not be necessary to determine the free space difference between the two tubes as a part of the analysis.

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

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

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

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

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





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

- 5. Place a sample container on the balance. Tare the balance and allow it to stabilize to zero.
- 6. Slowly pour the specified amount of sample into the sample container.
- 7. Remove the rubber stopper from the sample tube.
- 8. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.
- 9. On the Sample Data Worksheet, record the following:
  - Mass of the sample tube set with the sample.
  - Subtract the Mass of empty sample tube set from the Mass of sample tube set plus sample.

### **DEGAS THE SAMPLE**

### Sample Data Worksheet for Gas Adsorption on page E - 1

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

After degassing is complete, perform the following steps:

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



### SAMPLE TUBE INSTALLATION



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

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



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



If analyzing samples with a surface area of 1.0 m<sup>2</sup> or less, use filler rods in the sample and balance tubes. See <u>Analyze Samples with a Total Surface Area of 1.0 m<sup>2</sup> or Less on page D - 1</u>.

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



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



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





### FILL AND INSTALL THE DEWAR

### Dewar Precautions on the facing page

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



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

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



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



## **Dewar Precautions**



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



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



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

When handling Dewars containing liquefied gases or cryogenic liquids:

- Wear protective equipment:
  - o goggles or face shield
  - o 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.



### START ANALYSIS

System Options on page 3 - 38
Analyze Functions on page 3 - 44

Alt + 4

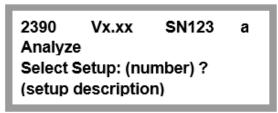


Press CHOICE to display the options. Press ENTER to select.

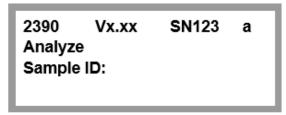
The following steps describe how to start an analysis responding to the prompts that appear if all the prompts have been selected to display. To disable any of the prompts before every analysis, make the change in *System Options*.

To start an analysis using the default analysis parameters:

1. At *Select Setup*, enter the *Set Up* group to use for the current analysis (by default, the number for the previous analysis is displayed).



2. Enter a *Sample ID* (up to 20 numbers and dashes). Using sample IDs helps to keep track of data from various analyses.



3. Enter the sample mass from 0.0001 to 999.9999 g.

2390	Vx.xx	SN123	а
Analyz	е		
Sample mass:			
1.0000 g			





If the sample mass is to be calculated using the empty tube mass and the tube plus sample, two prompts display, allowing for entry of these two values.

4. If *Entered* for *Saturation Pressure* was selected in *System Options*, enter a saturation pressure from *500.00* to *900.00* mmHg.

2390 Vx.xx SN123 a Analyze Saturation pressure: 760 mmHg



If *During analysis* or *Previously measured for Saturation Pressure* was selected in *System Options*, the previous prompt is not displayed.

5. Enter the temperature of the bath liquid from 0 to 999.999 K.

2390 Vx.xx SN123 a Analyze Bath temperature:

6. Enter the evacuation rate from 1.0 to 1000.0 mmHg/min.

2390 Vx.xx SN123 a Analyze Evacuation rate: 500.0 mmHg/min



Use a lower evacuation rate when analyzing powder samples to avoid possibly contaminating the sample port filter and manifold with sample particles. Higher evacuation rates can safely be used with samples consisting of large, solid pieces.

7. Press **ENTER** twice to start the analysis.



### ANALYSIS RESULTS

Report Options on page 3 - 20



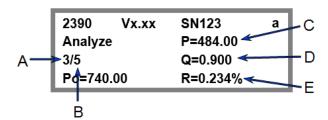
Press CHOICE to display the options. Press ENTER to select.

There are several methods for working with analysis results:

- View
- Print
- Transmit
- E-mail

### **VIEW ANALYSIS RESULTS**

As the analysis is performed, operational status messages are displayed. An example message is shown below:



- A. Number of points collected
- B. Number of points requested
- C. Current pressure (mmHg)
- D. Quantity adsorbed (cm<sup>3</sup> STP)
- E. Rate of change for quantity adsorbed

During analysis, the quantity (Q) value equals the quantity adsorbed by the sample in cm<sup>3</sup> STP. At all other times, the Q value equals the reading of the differential transducer in cm<sup>3</sup> STP. This reading indicates the amount of gas in the balance side of the system above the amount in the sample side of the system. This value is usually near zero (0) when an analysis is not in progress.

When the analysis is complete, the analyzer beeps three times. If data have been collected and surface area or pore volume calculations have been selected, press **CHOICE** to view the results.



A few examples of the display report formats:

2390 Vx.xx SN123 a Analyze P=484.0 BET Multi-pt Area: nnnnn.nnnn m2/g

2390 Vx.xx SN123 a Analyze P=484.0 t-Method Volume: nnnn.nnnn cm3

2390 Vx.xx SN123 a Analyze P=484.0 Total Pore Volume: nnnnn.nnnn m2/g

Total pore volume is calculated and reported if the last data point collected, other than saturation, was at a relative pressure  $(P/P_0)$  greater than or equal to 0.5000. The Calculations document can be found on the Micromeritics web page (<a href="www.Micromeritics.com">www.Micromeritics.com</a>). Press **CHOICE** to cycle through any additional report calculations and error messages.

#### PRINT ANALYSIS RESULTS

A report is automatically printed if *Printer* is selected as the report destination in *Report Options*. If not, print a report by pressing **Alt + 6** during or after an analysis.

### TRANSMIT ANALYSIS RESULTS

A report can be transmitted through the RS-232 serial port by pressing **Alt + 3** during or after an analysis. Or *Transmission line* as a report destination can be selected in *Report Options* to have results transmitted automatically after the analysis.

#### E-MAIL ANALYSIS RESULTS

Analysis results can be e-mailed automatically at the completion of an analysis when the Gemini is connected to a network.



### Measure Saturation Pressure

### Alt + . (decimal)



Press **CHOICE** to display the options. Press **ENTER** to select.

The saturation pressure can be measured in the:

- Sample tube (all Gemini VII 2390 models)
- Po tube (Gemini VII 2390*p* and 2390*t* only)

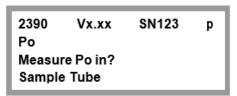
To measure the saturation pressure (P\_0):

1. Attach an empty sample tube to the analysis port. This sample tube must be the same size as the tube attached to the balance port. Do not place sample in the tube; an erroneous Po reading occurs when sample is in the tube.



Sample tubes on the ports do not have to be installed if measuring the saturation pressure in the Po tube but should be plugged if tubes are not installed.

- 2. Add liquid nitrogen (LN<sub>2</sub>) to the Dewar.
- 3. Place the Dewar support on the elevator, then place the Dewar on the support. A Dewar support is not necessary for the larger Dewar used with the Gemini VII 2390*t*. Place the Dewar directly on the elevator.
- 4. If adding LN<sub>2</sub> to an empty Dewar, allow the temperature of the LN<sub>2</sub> to stabilize (approximately 30 minutes).
- 5. Press Alt + . (decimal).



- 6. Select Sample Tube or Po Tube.
- 7. Press **ENTER** twice to begin the measurement or press **Alt + CLEAR** at any time to cancel the measurement.

When the measurement is complete, the saturation pressure is stored in memory and used in report calculations.



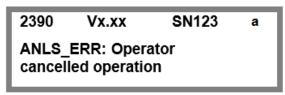
## CANCEL AN AUTOMATIC OPERATION

#### Alt + Clear



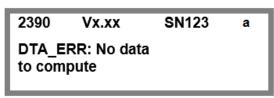
Press CHOICE to display the options. Press ENTER to select.

1. To cancel the operation, press **ENTER**. Messages are displayed indicating the termination is in progress. One of the following prompts is displayed:



2390 Vx.xx SN123 a
P0\_ERR: Operator
cancelled operation

2. Press **CLEAR** to remove the message from the keypad window. If an analysis is being canceled and enough data have been collected for calculation, results are displayed. If there were not enough data collected, the following prompt is displayed:



3. Press **CLEAR** to remove the message from the keypad window. To ignore the request for cancellation and resume the operation, press **Alt + CLEAR**.



### DATA RESULTS

Communications on page 3 - 34

Alt + 2



Press CHOICE to display the options. Press ENTER to select.

Data results for the last five analyses are saved in the analyzer's memory. These results can be viewed using a web browser. To use this feature, connect a computer to the instrument's Ethernet port.

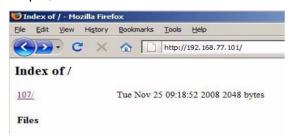


These steps assume an IP address has already been assigned. If an address has not been assigned, follow the steps in *Communications* to assign one.

- 1. Open a web browser.
- 2. Enter the *IP address* in the *Address* field of the web browser. This must consist of numbers only, separated by a decimal.

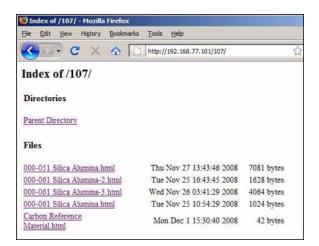


3. Press **Enter** on the keypad. The analyzer serial number displays in the window—for example, 107.



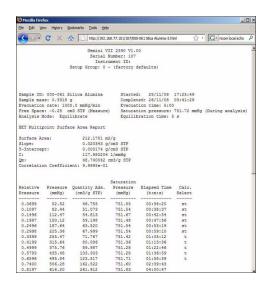


4. Click the serial number hyperlink. The last five analyses display in the window.



The .html format are assigned the name used in the Sample ID. If sample identifications are not used, the name *Untitled* is assigned and is appended numerically for each file.

5. Click the applicable file to view the data results. Or click the file for a current operation to monitor its progress.



6. Use the *File > Print* option to print a copy of the results.



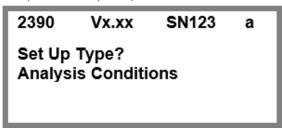
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# 3 COMMAND PROMPTS

## **MULTIPLE CHOICE**

A fixed set of responses and is followed by a question mark (?). This *Set Up Type* example is a multiple-choice prompt:

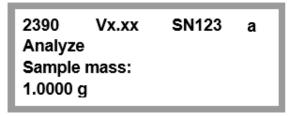


There are four *Set Up* types: *Analysis Conditions*, *Report Options*, *Communications*, and *System Options*.

## DATA ENTRY

A data entry prompt is followed by a colon (:), prompting to make an entry. The type of entry requested is displayed on the third line. The fourth line is used for the entry.

The following prompt displays when performing an analysis and requests a value for the sample mass. Use the keypad to enter the mass. Press **ENTER** to save the entry and advance to the next prompt.





If applicable, attach a keyboard to a USB port on the analyzer and use the keyboard to make entries.



## **ACTION**

An action prompt indicates how to perform an action. The following prompt displays when pressing **Alt +**. (decimal) to measure the saturation pressure (Po). Press **ENTER** to start the measurement or **Escape** (**Alt + CLEAR**) to cancel and return to the *Reload* prompt.

2390 VX.XX SN123 a
Po
[ENTER] to Start
[ESCAPE] to Cancel



### SET UP COMMANDS

Analysis Conditions on page 3 - 14
Report Options on page 3 - 20
Communications on page 3 - 34
System Options on page 3 - 38

Use the Set Up function to:

- Edit a Set Up group
- Copy the values of one Set Up group to another one, then modify the values
- Reset the values of a Set Up group to a default set

A *Set Up* group is a user-defined set of conditions to perform analyses, report data, and transmit data. It also contains system options such as instrument ID. *Set Up* groups contain the following condition sets.

### **Set Up Function**

Function	Description
Analysis Conditions	Specify the evacuation rate, evacuation time, free-space measurement, saturation pressure, target relative pressures, and analysis mode.
Report Options	Specify the report destination, the starting and ending points for collecting data to calculate surface area, the surface area calculations to report BET multipoint, Langmuir, BET single-point, t-method report options, the area of the adsorbate gas molecule, and the density conversion factor to use for calculating total pore volume.
Communications	Specify transmission parameters and internet criteria.
System Options	Specify criteria for the system (date, time, etc.) and for <i>Set Up</i> groups (sample ID, sample mass, etc.).

Up to 10 *Set Up* groups may be defined. Each group has a number, 0 to 9, associated with it. When an analysis is performed, the keypad window displays a prompt to enter the number of the *Set Up* group to be used for the analysis. The default *Set Up* groups 0 and 4 through 9 set to factory default values.

- Set Up group 0 has been predefined with a sample identification (predefined with factory defaults).
- Set Up groups 1 through 3 also have been predefined.
- Set Up groups 4 through 9 are unused.

All values in the Set Up groups can be edited or reset back to factory defaults.



# SET UP GROUP 0 DEFAULT VALUES, FACTORY DEFAULTS

## **Analysis Conditions Parameters**

Field	Default
Evacuation time	1:00
Free space	None
Sample density	1.00 g/cm <sup>3</sup>
Pressure table	Replace
First rel. pressure	0.1000 P/Po
Last rel. pressure	0.3000 P/Po
Number of points	5
Adsorb pressure 1	0.1000 P/Po
Adsorb pressure 2	0.1500 P/Po
Adsorb pressure 3	0.2000 P/Po
Adsorb pressure 4	0.2500 P/Po
Adsorb pressure 5	0.3000 P/Po
Analysis mode	Equilibrate
Equilibration time	5 s
Scan rate	10 min/analysis

## **Report Option Parameters**

Field	Default
Print Report	No
Transmit Report	No
E-mail Report	No
Report BET multi-pt	Yes
Minimum area	0.0000 m <sup>2</sup> /g
Maximum area	1.0000 m <sup>2</sup> /g
Report Langmuir	No
Report BET 1-point	No
Report t-method	No



## **Report Option Parameters (continued)**

Field	Default
Report BJH	No
Report H-K	No
Molecular area	0.162 nm <sup>2</sup>
Density conversion	0.0015468
Nonideality	5.0 %/atm

# **System Option Parameters**

Field	Default
ID for Setup 1	(factory defaults)
Request sample ID	Yes
Request sample mass	Yes
Request Sat. Press.	During analysis
Request Evac. Rate	Yes



# SET UP GROUP 1 DEFAULT VALUES, N300-700

## **Analysis Conditions Parameters**

Field	Default
Evacuation time	1:00
Free space	Calculate
Sample density	1.90 g/cm <sup>3</sup>
Pressure table	Replace
First rel. pressure	0.1000 P/Po
Last rel. pressure	0.5000 P/Po
Number of points	5
Adsorb pressure 1	0.1000 P/Po
Adsorb pressure 2	0.2000 P/Po
Adsorb pressure 3	0.3000 P/Po
Adsorb pressure 4	0.4000 P/Po
Adsorb pressure 5	0.5000 P/Po
Analysis mode	Equilibrate
Equilibration time	2 s
Scan rate	10 min/analysis

## **Report Option Parameters**

Field	Default
Print Report	No
Transmit Report	No
E-mail Report	No
Report BET multi-pt	Yes
Minimum area	0.0000 m <sup>2</sup> /g
Maximum area	1.0000 m <sup>2</sup> /g
Report Langmuir	No
Report BET 1-point	No
Report t-method	Yes



## **Report Option Parameters (continued)**

Field	Default
t-method range from	0.1900 P/Po
t method range to	0.5100 P/Po
Thickness curve	Magee-STSA
M-STSA parameter 1	13.9900
M-STSA parameter 2	0.0340
M-STSA parameter 3	0.5000
Minimum thickness	4.200 A
Maximum thickness	6.500 A
Area correction	1.000
Ads. pore volume	No
Report BJH	No
Report H-K	No
Molecular area	0.162 nm <sup>2</sup>
Density conversion	0.0015468
Nonideality	5.0 %/atm

# **System Options Parameters**

Field	Default
ID for Setup 1	N300-700
Request sample ID	Yes
Request sample mass	Yes
Request Sat. Press.	During analysis
Request Evac. Rate	Yes



# SET UP GROUP 2 DEFAULT VALUES, N100-200

## **Analysis Conditions Parameters**

Field	Default
Evacuation time	1:00
Free space	Calculate
Sample density	1.900 g/cm <sup>3</sup>
Pressure table	Replace
First rel. pressure	0.0500 P/Po
Last rel. pressure	0.5000 P/Po
Number of points	7
Adsorb pressure 1	0.0500 P/Po
Adsorb pressure 2	0.0750 P/Po
Adsorb pressure 3	0.1000 P/Po
Adsorb pressure 4	0.2000 P/Po
Adsorb pressure 5	0.3000 P/Po
Adsorb pressure 6	0.4000 P/Po
Adsorb pressure 7	0.5000 P/Po
Analysis mode	Equilibrate
Equilibration time	2 s
Scan rate	10 min/analysis

## **Report Options Parameters**

Field	Default
Print Report	No
Transmit Report	No
E-mail Report	No
Report BET multi-pt	Yes
Minimum area	0.0000 m <sup>2</sup> /g
Maximum area	1.0000 m <sup>2</sup> /g
Report Langmuir	No



# **Report Options Parameters (continued)**

Field	Default
Report BET 1-point	No
Report t-method	Yes
t-method range from	0.1900 P/Po
t method range to	0.5100 P/Po
Thickness curve	Magee-STSA
M-STSA parameter 1	13.9900
M-STSA parameter 2	0.0340
M-STSA parameter 3	0.5000
Minimum thickness	4.200 A
Maximum thickness	6.500 A
Area correction	1.000
Ads. pore volume	No
Report BJH	No
Report H-K	No
Molecular area	0.162 nm <sup>2</sup>
Density conversion	0.0015468
Nonideality	5.0 %/atm

# **System Options Parameters**

Field	Default
ID for Setup 0	N100-200
Request sample ID	Yes
Request sample Mass	Yes
Request Sat. Press.	During analysis
Request Evac. Rate	Yes



# SET UP GROUP 3 DEFAULT VALUES, OVER 130

## **Analysis Conditions Parameters**

Field	Default
Evacuation time	1:00
Free space	Calculate
Sample density	1.90 g/cm <sup>3</sup>
Pressure table	Replace
First rel. pressure	0.1000 P/Po
Last rel. pressure	0.5000 P/Po
Number of points	7
Adsorb pressure 1	0.0500 P/Po
Adsorb pressure 2	0.0750 P/Po
Adsorb pressure 3	0.1000 P/Po
Adsorb pressure 4	0.2000 P/Po
Adsorb pressure 5	0.3000 P/Po
Adsorb pressure 6	0.4000 P/Po
Adsorb pressure 7	0.5000 P/Po
Analysis mode	Equilibrate
Equilibration time	2 s
Scan time	10 min/analysis

## **Report Options Parameters**

Field	Default
Print Report	No
Transmit Report	No
E-mail Report	No
Area points from	0.0400 P/Po
Area points to	0.1100 P/Po
Report BET multi-pt	Yes
Minimum area	0.0000 m <sup>2</sup> /g
Maximum area	1.0000 m <sup>2</sup> /g



## **Report Options Parameters (continued)**

Field	Default
Report Langmuir	No
Report BET 1-point	No
Report t-method	Yes
t-method range from	0.1900 P/Po
t method range to	0.5100 P/Po
Thickness curse	Magee-STSA
M-STSA parameter 1	13.990
M-STSA parameter 2	0.0340
M-STSA parameter 3	0.500
Minimum thickness	4.200 A
Maximum thickness	6.500 A
Area correction	1.000
Ads. pore volume	No
Report BJH	No
Report H-K	No
Molecular area	0.162 nm2
Density conversation	0.0015468
Nonideality	5.0%/atm

## **System Options Parameters**

Field	Default
ID for Setup 0	Over 130
Request sample ID	Yes
Request sample mass	Yes
Saturation Pressure	During analysis



Press **Alt + 2** on the keypad to access the *Set Up* function.



Set Up groups may be selected by pressing **CHOICE** until the applicable number is displayed or by entering the group number. A colon and a question mark prompt for entry of the Set Up group number at the Select Setup and Use Setup prompts.

## **Set Up Functions**

Function	Description
Edit Setup	Edits the selected Set Up group.
	2390 Vx.xx SN123 a Setup Group (number) Select Action? Edit Setup
	Choices:
	<ul> <li>Edit Setup</li> <li>Copy another setup</li> <li>Reset to default</li> <li>Reset to N300-700</li> <li>Reset to N100-200</li> <li>Reset to Over 130</li> </ul>
Copy settings from	Copies the settings from another <i>Set Up</i> group to the current one. Enter the number of the <i>Set Up</i> group from which to copy the values.
	2390 Vx.xx SN123 a Setup Group (number) Copy settings from Setup: (Setup ID)



# **Set Up Functions (continued)**

Function	Description	
Resetting Setup	Resets the current Set Up group to the selected default number (see the table defaults detailed in the previous sections). When resetting a Set Up group, only group-specific parameters are reset.	
	2390 Vx.xx SN123 a Set Up Resetting Setup to (Setup ID)	
Setup Type	Selects the setup type.	
	Choices: Analysis Conditions, Report Options, Communications, System Options	



### **ANALYSIS CONDITIONS**

Report Options on page 3 - 20 System Options on page 3 - 38



Press **CHOICE** to display the options. Press **ENTER** to select.

## **Analysis Condition Prompts**

Field or Button	Description
Empty tube mass	Displays if <i>Tube and tube+sample</i> for the sample mass were selected when specifying <i>System Options</i> .  Enter the mass of the empty sample tube. Range: 0.0000 to 999.9999 g
	2390 Vx.xx SN123 a Analysis Conditions Empty tube mass: 10.0000 g
Evacuation Time	Length of time that the sample will be evacuated after reaching an evacuation rate of less than 0.1 mmHg per 30 second interval.  Enter the evacuation time. Range: 0.1 to 999.9 min
	2390 Vx.xx SN123 a Analysis Conditions Evacuation time: 1.0 min



Field or Button	Description
Free space	Typically, materials having surface areas greater than 25-50 m <sup>2</sup> /g can be analyzed accurately without free-space measurement.  Select the method for handling free-space measurement.  Choices: <i>None, Previous, Measure, Calculate</i> 2390 Vx.xx SN123 a
	Analysis Conditions Free space? None
Previous	Uses the free spaced applied to the previous sample.
Measure	Provides a more accurate, but slower, analysis. If analyzing a series of samples with similar volumes, measure the first sample, then select <b>Previous</b> for the remaining samples.
Calculate	Provides high accuracy without the additional time required for free-space measurement. This method also includes a balance volume correction and a nonideality correction.  The free-space correction is calculated by dividing the sample mass by the sample density to get the sample volume. When Calculate is selected, the following prompt is displayed for entry of the sample density. Enter the density of the sample to be analyzed. (Pycnometers are available from Micromeritics for automatic density measurement.)  Range: 0.000 to 99.999 g/cm³  2390 Vx.xx SN123 a Analysis Conditions Sample density: 1.000 g/cm3  An approximate value is adequate to provide an accurate free-space correction.



Field or Button	Description
Pressure table	Selects the means for changing or preserving the currently stored target relative pressure table. Choices are: Replace, Preserve, Edit
	2390 Vx.xx SN123 a Analysis Conditions Pressure table? Replace
	The highest adsorption point prior to saturation should be 0.9990 P/Po. If saturation pressure has decreased significantly since the previous measurement, target pressures close to 1.0 P/Po can result in premature saturation.
Replace	Replaces the currently stored pressure table with a new table of evenly spaced points.  Enter the First rel. pressure for data collection.  Range: 0.00001 to 1.0000 P/Po  2390 Vx.xx SN123 a  Analysis Conditions  First rel. pressure: 0.1000 P/Po  Enter the Last rel. pressure for data collections.  Range: First rel. pressure to 1.0000 P/Po
	2390 Vx.xx SN123 a Analysis Conditions Last rel. pressure: 0.3000 P/Po
	Enter the same value entered for <i>First rel. pressure</i> if performing a single-point analysis. The analyzer collects the specified number of data points at evenly spaced pressure intervals during the period of analysis. Enter 1 if performing a single-point analysis.



Description
Enter the number of data points to be collected. Range: 1 to 1000
2390 Vx.xx SN123 a Analysis Conditions Number of points: 5
Preserves the currently stored pressure table. No further pressure table question prompts are displayed.
Allows for viewing and editing of the current pressure table. This is useful if an unevenly spaced sequence of data points is applicable; for example, when a five-point surface area is to be combined with a single-point total pore volume.  Enter the next applicable target relative pressure or accept the default and press <b>ENTER</b> . Entries must be in ascending order. This request is made for each pressure in the table or until 1000 points have been entered. An entry of 0.0000 terminates the table. An entry of 1.0000 specifies a saturation pressure measurement and terminates the table. If a saturation pressure measurement is specified, the stored Po value is updated automatically when the measurement is made.
Range: 0.0001 plus the previous target pressure to 1.0000  2390 Vx.xx SN123 a Analysis Conditions Adsorb pressure (number): 0.1000 P/Po  This following prompt displays only for the Gemini VII 2390t. Follow the instructions given for the Adsorb pressure prompt, except the entries must be in descending order for desorption.  Range: 0.9999 plus the previous target pressure to 0.0



Field or Button	Description
	2390 Vx.xx SN123 t Analysis conditions Desorb pressure: (number) 0.1000 P/Po
Equilibrate	Selects the analysis mode. Choices: Equilibrate, Scan
	2390 Vx.xx SN123 a Analysis Conditions Analysis mode? Equilibrate
Equilibration time	Increases the pressure in steps to the next specified level for data collection. It maintains this pressure and monitors the volume adsorbed. When the rate of adsorption (volume adsorbed during the equilibration time divided by total volume adsorbed) falls below 0.01% and the variance from 11 consecutive readings falls below 0.1%, equilibration is assumed to have occurred. The analyzer then introduces the next adsorbate gas dose.  Enter the equilibration time. Range: 1 to 1000 sec
	2390 Vx.xx SN123 a Analysis Conditions Equilibration time: 5 s



Field or Button	Description
Scan time	Raises the adsorptive gas pressure at a constant rate based on the pressures to be achieved and the length of the analysis.
	Enter the scan time (length of time for the analysis). Range: 1 to 10000 min/analysis
	2390 Vx.xx SN123 a Analysis Conditions Scan time: 10 min/analysis



### **REPORT OPTIONS**

Data Format on page A - 1
Supported Printers on page B - 1



Press **CHOICE** to display the options. Press **ENTER** to select.

## **Report Options**

Option	Description
Print Report	Generates the report automatically after analysis. Choices: Yes, No If Yes is selected, select the type of printer.
	2390 Vx.xx SN123 a Report Options Print Report? No
Transmit Report	Transmits the analysis data automatically after the operation via serial line communication. Choices: Yes, No
	2390 Vx.xx SN123 a Report Options Transmit Report? No
E-mail Report	E-mails the analysis results automatically after the analysis concludes. Choices: Yes, No If Yes is selected, specify an e-mail address and server.
	2390 Vx.xx SN123 a Report Options E-mail Report? No



Option	Description
Printer	Selects the printer to use for generating reports. This is a system option and does not have to be specified for each analysis. Select the applicable printer.
	2390 Vx.xx SN123 a Report Options Printer? Epson ESCP Raster
	Choices:  Epson ESCP Raster  Epson ESCP  HP PCL 6XL  Epson ESCP2  Postscript  HP PCL 3  Canon Bubble Jet
Transmission Format	Selects the format to be used when transmitting reports. Choices: Single Column, Spreadsheet.  2390 Vx.xx SN123 a Report Options Transmission Format?
Area points from	Single Column  Enter the relative pressure at which to start selecting collected data
Tiea hours nou	points for calculating surface area. Range: 0.0 to 1.0000 P/Po
	2390 Vx.xx SN123 a Report Options Area points from: 0.0400 P/Po



Option	Description
Area points to	Enter the relative pressure at which to stop selecting collected data points for calculating surface area. If the range specified includes more than 50 data points, only the first 50 points are used to calculate surface area. Individual points after analysis may be selected or deselected.
	Range: Area points from value to 1.0000 P/Po
	2390 Vx.xx SN123 a Report Options Area points to: 0.3100 P/Po
Report BET multi-pt	Choices: Yes, No, Pass/Fail
	2390 Vx.xx SN123 a Report Options Report BET multi-pt? Yes
	If Pass/Fail is selected, specify a range.
	Range: 0.0000 to 9999.9999 m2/g (for both prompts)
	2390 Vx.xx SN123 a BET multi-pt Mlnimum area: 0.0000 m2/g
	2390 Vx.xx SN123 a BET multi-pt Maximum area: 1.0000 m2/g



Option	Description	
Report Langmuir	Choices: Yes, No, Pass/Fail If Pass/Fail is selected, enter the appropriate range.	
	2390 Vx.xx SN123 a Report Options	
	Report Langmuir?	
Report BET 1-pt	Choices: Yes, No, Pass/Fail  If Pass/Fail is selected, enter the appropriate range.	
	2390 Vx.xx SN123 a Report Options Report BET 1-pt? No	
Report t-method	Choices: Yes, No, Pass/Fail If Pass/Fail is selected, specify ranges for external area, micropore area, and volume.	
	2390 Vx.xx SN123 a t-method Ext. area pass/fail? No	
	2390 Vx.xx SN123 a t-method Mic. area pass/fail? No	



# Option 2390 Vx.xx SN123 a t-method Volume pass/fail? No

Each prompt has a Yes/No response. If Yes is selected at any of these prompts, enter the appropriate ranges.

After specifying Pass/Fail criteria, the prompts that display for the Yes choice is shown. If Yes is selected for the t-method report:

Enter the relative pressure at which to *start* selecting collected data points for calculating t-method micropore volume.

Range: 0.0 to 1.0 P/Po Default: 0.0 P/Po

> 2390 Vx.xx SN123 a t-method t-method range from: 0.0000 P/Po

Enter the relative pressure at which to *stop* selecting collected data points for calculating t-method micropore volume.

Range: *t-method range from value to 1.0 P/Po* Default: 0.7 or *t-method range from P/Po* 

2390 Vx.xx SN123 a t-method t-method range to: 0.7000 P/Po

If the range specified includes more than 50 data points, only the first 50 points are used in t-method calculations. Individual points after analysis may be selected or deselected.



Option	Description		
	2390 Vx.xx t-method Thickness cur Harkins and Jura		
	Choices: Harkins and J	ura, Halsey, Magee-ST	SA
Harkins and Jura	$t = [rac{13.990}{0.0340 - log(1)}]$	$[P/Po]^{0.500}$	
	The Harkins and Jura <sup>1</sup>	equation has three ed	
	Parameter	Range	Default
	1	0.0001 to 9999.00	13.9900
	2	0.0001 to 9999.00	0.0340
	3	0.010 to 1.000	0.500
Halsey	Halsey $t=3.540\left[rac{-5.000}{1n\left(rac{P}{Po} ight)} ight]^{0.333}$ The Halsey $^2$ ) equation has three editable parameters:		
			ameters:
	Parameter	Range	Default
		0.0001 to 9999.00	3.540
	1	0.0001 to 9999.00	3.540
	2	0.0001 to 9999.00 0.0001 to 9999.00	5.000

<sup>1)</sup> Harkins, W.D. and Jura, G., "An Adsorption Method for the Determination of the Area of a Solid without the Assumption of a Molecular Area and the Area Occupied by N2 Molecules on the Surface of Solids," J. Chem. Phys. 11, 431-432 (1943). <sup>2</sup>) Halsey, G., J. Chem. Phys 16, 931-932 (1948)



Option	Description			
Magee-STSA	$t=0.88\left(rac{P}{Po} ight)^2+$	$6.45\left(rac{P}{Po} ight) + 2.98$		
	The Magee-STSA <sup>1</sup> )	The Magee-STSA <sup>1</sup> ) equation has three editable parameters:		
	Parameter	Range	Default	
	1	0.0001 to 9999.00	0.88	
	2	0.0001 to 9999.00	6.45	
	3	0.0001 to 9999.00	2.98	
	Enter the <i>minimum thi</i> calculations. Range: 0.0 to 99.99 at Default: 3.5 A	ckness for points to be us	ed in t-method	
	2390 Vx.xx t-method Minimum thic 3.5 A			
	calculations.	ickness for points to be us		
	2390 Vx.xx t-method Maximum thic 5.000 A			
	l l	nds a surface area corrects to get the BET area and of zeolite.		
	Enter the surface area	correction factor to be us	sed in t-method	

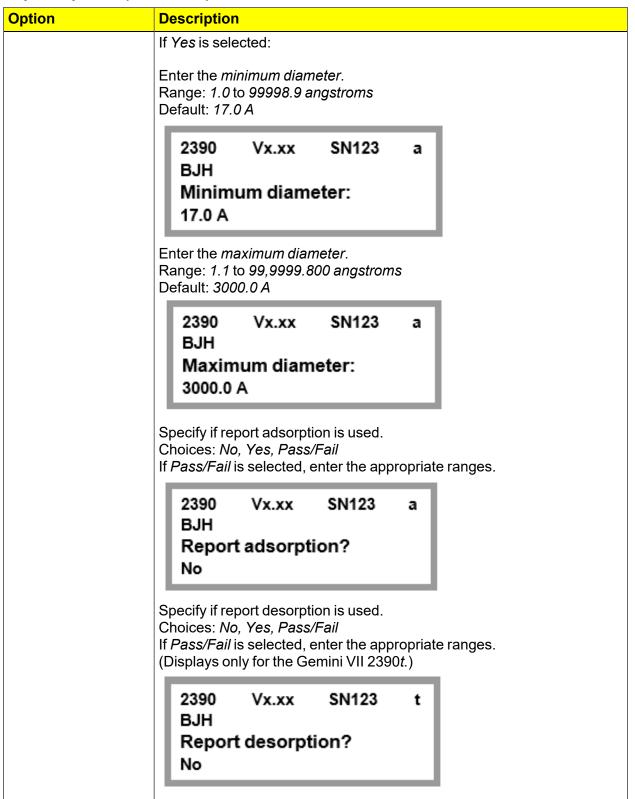
<sup>1)</sup> Magee, Ricky, Columbian Chemicals Company (personal communications).

<sup>&</sup>lt;sup>2)</sup> Marvin F.L. Johnson, "Estimation of the Zeolite Content of a Catalyst from Nitrogen Adsorption Isotherms," Journal of Catalysis 52, 425-431 (1978).



Option	Description	
	calculations. Range: 0.100 to 1.000 Default: 1.000	
	2390 Vx.xx SN123 a t-method Area correction: 1.000	
Ads. pore volume	Calculates the pore volume from the highest point of the adsorption isotherm. Choices: No, Yes, Pass/Fail If Pass/Fail is selected, enter the appropriate ranges.	
	2390 Vx.xx SN123 a Report Options Ads. pore volume? No	
Des.pore volume	Calculates the pore volume from the highest point of the desorption isotherm. (Displays only for the Gemini VII 2390t.) Choices: No, Yes, Pass/Fail If Pass/Fail is selected, enter the appropriate ranges.	
	2390 Vx.xx SN123 t Report Options Des. pore volume? No	
Report BJH	BJH reporting assumes nitrogen is the adsorptive. Choices: Yes, No	
	2390 Vx.xx SN123 a Report Options Report BJH? No	







Option	Description	
	Select the type of thickness curve. Choices: Halsey, Harkins and Jura, Magee-STSA Default: Halsey	
	2390 Vx.xx SN123 a BJH Thickness curve? Halsey	
	See the t-method report for an explanation of the thickness curves.	
Report H-K	Choices: No, Yes, Pass/Fail	
	2390 Vx.xx SN123 a Report Options Report H-K? No	
	If Pass/Fail is selected, specify ranges for volume and width. Choices: Yes, No	
	2390 Vx.xx SN123 a Horvath-Kawazoe Volume pass/fail? No	
	2390 Vx.xx SN123 a Horvath-Kawazoe Width pass/fail? No	
	If Yes is selected at any of the Pass/Fail prompts, enter the appropriate ranges.	
	If Yes is selected, enter the H-K range from and H-K range to pressure ranges.	
	Range: 0.0001 to 1.0 P/Po (both prompts)	



Option	Description
	2390 Vx.xx SN123 a Horvath-Kawazoe H-K range from: 0.0000 P/Po
	2390 Vx.xx SN123 a Horvath-Kawazoe H-K range to: 1.0800 P/Po
Pore geometry	Select the shape which best represents the physical geometry of the micropores in the sample material. Choices: Slit, Cylinder, Sphere
	2390 Vx.xx SN123 a Horvath-Kawazoe Pore geometry: Slit
	If Slit or Cylinder is selected, enter the Interaction param. to be used for report calculations.
	Input into this field is slightly different from that of other fields. The significand and the exponent can be edited. For example, the prompt shown above displays 3.49 as the significand. Enter 2.34 to replace 3.49; the next keystroke automatically replaces the exponent.
	Range: 1.00e-46 to 9.99e-40 erg cm <sup>4</sup> Default: 3.49e-43 erg cm <sup>4</sup>
	2390 Vx.xx SN123 a Horvath-Kawazoe Interaction param.: 3.49e-43 erg cm4



Report Options (cor			
Option	Description		
Adsorptive diam.	Diameter of the adsorptive molecule. Edit or accept the default. Range: 0.0 to 99.999 angstroms Default: 3.0 A (N2)		
	2390 Vx.xx SN123 a Horvath-Kawazoe Adsorptive diam.: 3.0 A		
Adsorbent diam.	Diameter of the sample adsorbate atom. Edit or accept the default. Range: 0.0 to 99.999 angstroms Default: 3.04 (Zeolite)		
	2390 Vx.xx SN123 a Horvath-Kawazoe Adsorbent diam.: 3.040 A		
Mean zero diam.	If <i>Slit</i> is selected for Pore geometry, enter the mean zero diameter. Range: 0.0 to 99.9999 angstroms Default: 2.5915 A		
	2390 Vx.xx SN123 a Horvath-Kawazoe Mean zero diam.: 2.5915 A		
	The mean zero diameter is the gas-solid nuclear separation at zero interaction energy,		
	$\sigma = rac{Z_S + Z_A}{2}$		
	where $Z_S$ is the sample equilibrium diameter at zero interaction energy and $Z_A$ the adsorptive zero interaction energy diameter.		
	If Sphere is selected for Pore geometry:		
	Enter the <i>adsorptive</i> spherical parameters.		



Option	Description
	Range: 1.0e-40 to 9.99999e-37 cm <sup>3</sup> Default: 4.25146e-38 cm <sup>4</sup>
	2390 Vx.xx SN123 a Horvath-Kawazoe Adsorp. sp. param.: 4.25146e-38 cm3
	Enter the <i>adsorbent</i> spherical parameters. Range: 1.0e-40 to 9.99999e-37 cm <sup>3</sup> Default: 6.05156e-38 cm <sup>3</sup>
	2390 Vx.xx SN123 a Horvath-Kawazoe Adsorb. sp. param.: 6.05156e-38 cm3
	For more details on the spherical parameters, refer to the Calculations document found on the Micromeritics web page (www.Micromeritics.com).
Cheng-Yang corr.	Choices: Yes, No
	2390 Vx.xx SN123 a Horvath-Kawazoe Cheng-Yang corr.? No
	If Yes is selected, the Cheng/Yang correction is applied to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath- Kawazoe derivation.



Option	Description	
Smooth diff?	Smooths the differential calculation, eliminating variations in the differential computation caused by noise in the input data. Choices: Yes, No	
	2390 Vx.xx SN123 a Horvath-Kawazoe Smooth diff.? No	
Molecular area	Enter the molecular cross-sectional area of the adsorptive gas.	
	2390 Vx.xx SN123 a Report Options Molecular area: 0.162 nm2	
	Range: 0.001 to 1.000 nm2	
Density conversion	Enter the adsorptive gas-to-liquid density conversion factor.	
	2390 Vx.xx SN123 a Report Options Density conversion: 0.0015468	
	Range: 0.0000001 to 1.0000000	
Nonideality	Nonideality is calculated when a free-space value (other than none) is selected. Enter the nonideality correction factor for the adsorptive gas.	
	2390 Vx.xx SN123 a Report Options Nonideality: 5.0 %/atm	
	Range: 0.0 to 99.9 %/atm	



#### **COMMUNICATIONS**



Press **CHOICE** to display the options. Press **ENTER** to select.

These settings apply to all Set Up groups.

## **Communications Functions**

Function	Description
Set-up Type	Choices: Yes, No
	2390 Vx.xx SN123 a Set-up Type? Communications
IP Address	Select how the IP address is assigned. An IP address provides access to a web browser to view analysis results. Results from 5 analyses are stored on the controller and can be viewed in a web browser.
	2390 Vx.xx SN123 a Communications IP Address? DHCP
	The IP address is assigned automatically. When requesting the DHCP address for the first time, this prompt displays:
	2390 Vx.xx SN123 a Communications Requesting Address [ENTER] to continue
	The system automatically tries to assign an IP address. If the system can determine an address, the address is displayed.



**Communications Functions (continued)** 

Function	Description
	2390 Vx.xx SN123 a Communications Assigned Address: (assigned address)
	If an address cannot be determined, the system requests an address.
	2390 Vx.xx SN123 a Communications Requesting Address [ENTER] to continue
	Press <b>Alt + CLEAR</b> to cancel the operation and return to the <i>Reload</i> prompt. Either contact your IT department for assistance or use the <i>Specify</i> option.
IP Address, Subnet Mask, and Gateway	Use to specify a user supplied IP address. Enter the numerical IP address (include periods) [nnn.nnn.nnn].
,	2390 Vx.xx SN123 a Communications IP Address: (user-entered)
	Enter the subnet Mask number using the same format as the IP address.
	2390 Vx.xx SN123 a Communications Subnet Mask: (user-entered)
	The Gateway address is used for communicating outside of a local network. This address is often the same as the instrument's IP address with a 1 after the last dot instead of the last three characters [nnn.nnn.nnn.1]. Enter the Gateway address.



# **Communications Functions (continued)**

Function	Description
	2390 Vx.xx SN123 a Communications Gateway: (user-entered)
E-mail Address	When connected to a network via an Ethernet connection, analysis results can be e-mailed automatically upon completion. Enter the email address to receive these reports. A keyboard must be used to complete this function. If not using DHCP and the e-mail address is outside the local network, a gateway address must be specified.  Enter the e-mail address.
	2390 Vx.xx SN123 a Communications E-mail Address: (user-entered)
E-mail Server	Enter the numerical address of the SMTP server (the computer that is going to deliver the results).
	2390 Vx.xx SN123 a Communications E-mail Server: (user-entered)
	Your IT Department may need to configure the server to accept e-mail from the analyzer. For proper configuration, the following information may be required:
	<ul> <li>IP address for the Gemini</li> <li>Sender line: Micromeritics_2390</li> </ul>



**Communications Functions (continued)** 

Function	Description
Baud Rate	Baud rate specifies the rate at which data are transmitted via the RS-232 port.  2390 Vx.xx SN123 a Communications Baud Rate? 9600
Data Bits	Choices: 110, 150, 300, 600, 1200, 4800, 9600, 19200  At the Data Bits prompt, select either 7 or 8.
	2390 Vx.xx SN123 a Communications Data Bits?
Stop Bits	At the Stop Bits prompt, select either 1 or 2.
	2390 Vx.xx SN123 a Communications Stop Bits?
Parity	At the Parity prompt, select either None, Even, or Odd.
	2390 Vx.xx SN123 a Communications Parity? None
Xon/Xoff Protocol	At the Xon/Xoff Protocol prompt, select either Disabled or Enabled.
	2390 Vx.xx SN123 a Communications Xon / Xoff Protocol? Disabled



#### SYSTEM OPTIONS

Analysis Conditions on page 3 - 14



Press **CHOICE** to display the options. Press **ENTER** to select.

The parameters specified for most of these prompts apply to all *Set Up* groups. The parameters specific to a *Set Up* group are marked accordingly.

## **System Options Functions**

Function	Description
Language	Choices: English, Deutsch, Francais, Espanol, Italiano
	2390 Vx.xx SN123 a System Options Language? English
	After selecting a language, the change is effective immediately: there is no need to press <b>SAVE</b> . For example, the next prompt displays in Italian if <i>Italiano</i> was selected. However, any error messages that may be in the message queue remain in English (or the current selected language) are not translated. Error messages that occur after selecting the language are shown in that language.
	Keypad overlays containing translations for the alternate functions printed above the keys for each language are available on the Micromeritics web page ( <a href="https://www.Micromeritics.com">www.Micromeritics.com</a> ).
ID for Setup*	Enter the ID for the Set Up group. Press . (decimal) on the keypad to enter a dash (-).
	2390 Vx.xx SN123 a System Options ID for Setup (number): (description)



Function	Description
Instrument ID	The instrument ID is a unique identifier of the analyzer. It can contain 1 to 20 numbers or dashes. Press . (decimal) on the keypad to enter a dash (-). If there are several analyzers, use this field to identify each analyzer on printed or transmitted reports.
	2390 Vx.xx SN123 a System Options Instrument ID: (user-entered)
Date (DD/MM/YY)	Range: Day: 1 to 31 Month: 1 to 12 Year: 00 to 99  Press . (decimal) on the keypad to enter a slash (/).  2390 Vx.xx SN123 a System Options Date (DD/MM/YY):
Time (MM:HH:SS)	Range: Hours: 0 to 23 Minutes: 0 to 59 Seconds: 0 to 59  Press . (decimal) on the keypad to enter a colon (:).  2390 Vx.xx SN123 a System Options Time (HH:MM:SS):



Function	Description
Request Sample ID*	The sample ID is a unique identifier of the sample. It can contain 1 to 20 numbers or dashes. Using sample IDs helps to keep track of data from various analyses. Choices: Yes, No  If No is selected, this prompt does not appear when using the Analyze and Review functions.
	2390 Vx.xx SN123 a System Options Request Sample ID? Yes
Request Sample Mass*	Prompts can be shown in the <i>Analyze</i> function for entering the sample mass, or for entering criteria to have it calculated automatically.  Choices: Yes, Tube and tube+sample, No  2390 Vx.xx SN123 a System Options Request Sample Mass? Yes
	Yes displays a prompt for entering the sample mass.  Tube and tube+sample displays two prompts for entering the mass of the empty tube and the mass of the tube with sample. The sample mass will be calculated automatically using these two entries and used in report calculations.
	No does not display a prompt. The last sample mass entered will be used in report calculations.



Function	Description
Request Saturation Pressure*	<ul> <li>During analysis. The saturation pressure is measured for each data point taken; applicable only for the 2390p and 2390t.</li> <li>Previously measured. The saturation pressure from the last data point of the previous analysis is used.</li> <li>Entered. A prompt is displayed when the analysis is started allowing for entry of a saturation pressure.</li> <li>2390 Vx.xx SN123 a System Options Request Sat. Press.?</li> </ul>
Request Bath Temperature*	Choices: Yes, No  If No is selected, this prompt does not appear when using the Analyze and Review functions.  2390 Vx.xx SN123 a System Options Request Bath Temperature?
Request Evacuation Rate*	Choices: Yes, No  If No is selected, this prompt does not appear when using the Analyze and Review functions.  2390 Vx.xx SN123 a System Options Request Evac. Rate?



Function	Description
Volume Correction	The volume correction is only used when calculated free space is selected. It accounts for any difference in volume between the balance tube and the empty sample tube. Perform a liquid nitrogen analysis using two empty sample tubes of the same size. (In <i>Analysis Conditions</i> , select <i>Measure</i> as the free-space option.)
	After the analysis, record the reported free-space difference and use it for these two tubes only for future calculated runs.
	Enter the difference in gas capacity between the balance volume and the sample tube. Press <b>CHOICE</b> to change the plus or minus sign.
	2390 Vx.xx SN123 a System Options Volume Correction: +0.000 cm3 STP
	Range: -99.990 to +99.990
Amount Ads. Unit	Choices: cm <sup>3</sup> STP, mmol
	2390 Vx.xx SN123 a System Options Amount Ads. Unit? cm3 STP
Length Unit	Choices: A (Angstrom), nm (nanometer)  2390 Vx.xx SN123 a System Options Length unit?
	A



Function	Description
Pore dimension	Choices: Diameter, Width, Radius
	2390 Vx.xx SN123 a System Options Pore dimension? Diameter
Pressure unit	Choices: mmHg, kPa, mbar
	2390 Vx.xx SN123 a System Options Pressure unit? mmHg
Pressure symbol	Choices: P, Po, p, po
	2390 Vx.xx SN123 a System Options Pressure symbol? P, Po

<sup>\*</sup> This prompt is specific to a Set Up group.



## **ANALYZE FUNCTIONS**

System Options on page 3 - 38

#### At + 4



Press **CHOICE** to display the options. Press **ENTER** to select.

## **Analyze Functions**

Function	Description
Select Setup (factory defaults)	Enter the number of the <i>Set Up</i> group to use for the current analysis. Choices: <i>0</i> through 9
	2390 Vx.xx SN123 a Analyze Select Setup: (number) ? 0 (factory defaults)
Sample ID (user entered)	Displays if Request Sample ID is set to Yes. Enter the sample ID.  Press . (decimal) to insert a dash, if required.  Range: 1 to 20 numbers and dashes
	2390 Vx.xx SN123 a Analyze Sample ID: (user-entered)



## **Analyze Functions (continued)**

Function	Description
Sample mass	Displays if Request Sample Mass is set to Yes.
	This field will also accept input from an analytical balance. The analytical balance must be connected to the RS-232 port. While this prompt is displayed, press the appropriate button on the analytical balance to transfer the mass. Most balances use the <b>ENTER</b> button (refer to the manufacturer's manual for information on transferring the value).  Enter the sample mass. Range: 0.0001 to 999.9999 g Default: 1.0000 g
	2390 Vx.xx SN123 a Analyze
	Sample mass: 1.0000 g
Empty tube mass Tube+sample mass	These two prompts display in sequence when Request Sample Mass is set to Tube and tube+sample.
	Enter the mass of the empty tube.
	2390 Vx.xx SN123 a Analyze Empty tube mass: 10.0000 g
	Enter the mass of the Tube plus sample.
	2390 Vx.xx SN123 a Analyze Tube+sample mass: 11.0000 g
	Range: 0.0001 to 999.9999 g Default: 1.0000 g



# **Analyze Functions (continued)**

Function	Description
Saturation pressure	Displays if Saturation Pressure is set to Entered.  If the saturation pressure was measured using Gemini's Po function, the measured value is displayed here. If not, enter the saturation pressure.  Range: 500.00 to 900.00 mmHg Default: Measured value, 760.00 mmHg or previous entry
	2390 Vx.xx SN123 a Analyze Saturation pressure: 760 mmHg
Bath temperature	Enter the bath temperature. Range: 0 to 999.999 kelvins  2390 Vx.xx SN123 a Analyze Bath temperature:
Evacuation rate	Enter the evacuation rate. Range: 1.0 to 1000.0 mmHg/min Default: 500.0 mmHg/min or previous entry  2390 Vx.xx SN123 a Analyze Evacuation rate: 500.0 mmHg/min



## **QUICKSTART**

#### Analyze Functions on page 3 - 44

#### Alt + 9

QuickStart performs a series of analyses using the same Setup number. Like the Analyze function, the prompts shown with QuickStart are based on the Setup parameters specified for the Setup number used.



The parameters for the last *Set Up* group used will be applied to the *QuickStart* analyses.

The information displayed on the third and fourth lines of this prompt and subsequent prompts depends on the parameters specified for the *Set Up* group.

2390 Vx.xx SN123 a QuickStart (user-requested) (user-entered)

Press **ENTER** to advance through the prompts, entering information as requested until this prompt is displayed:

2390 Vx.xx SN123 a QuickStart [ENTER] to Start [ESCAPE] to Cancel

Press **ENTER** to start the analysis.



## **REVIEW FUNCTIONS**

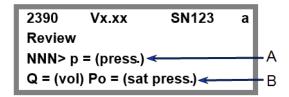
Use to review and edit the results of the last analysis.

#### Alt + 5



Press CHOICE to display the options. Press ENTER to select.

Displays for all collected data points.



- A. Current data point
- B. For each point if measured during analysis

#### **Review Functions**

Function	Description
Sample ID	Displays when <i>Request Sample ID</i> is set to Yes. The sample ID entered in the <i>Analyze</i> function is displayed. Enter a new sample ID, if applicable.
	2390 Vx.xx SN123 a Review Sample ID: (user-entered)
Sample Mass	Displays when Request Sample Mass is set to Yes. The value entered for the sample mass in the Analyze function is displayed. Enter a new sample mass, if applicable.
	2390 Vx.xx SN123 a Review Sample mass: 1.0000 g



## **Review Functions (continued)**

Function	Description
Empty tube mass Tube+sample mass	These two prompts display in sequence when Request Sample Mass is set to Tube and tube+sample. Displays the values entered for these prompts in the Analyze function.
	2390 Vx.xx SN123 a Review Empty tube mass: 10.0000 g
	2390 Vx.xx SN123 a Review Tube+sample mass: 11.0000 g
Saturation pressure	Displays if Saturation Pressure is set to Previously measured or Entered. The last measured saturation pressure entered in the Analyze function is displayed. Enter a new saturation pressure, if applicable.
	2390 Vx.xx SN123 a Review Saturation pressure: 760.00 mmHg
Analyze Bath temperature	Displays the temperature of the cryogen bath.  2390 Vx.xx SN123 a Analyze Bath temperature:



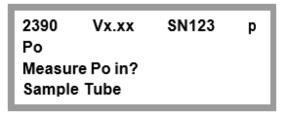
#### Po Command

#### Alt + . (decimal)



Press CHOICE to display the options. Press ENTER to select.

Use to measure the saturation pressure (Po). Applicable for the 2390*p* or 2390*t* models only. Choices: *Sample Tube, Po Tube* 



If Sample Tube is selected, ensure that empty sample tubes of the same size are installed on the analysis port and balance port. It is not necessary to install tubes on both ports if Po tube is selected.

Select the preferred method for measuring the saturation pressure.

This prompt displays when using the 2390a model:



Before beginning, ensure that empty sample tubes of the same size are installed on the analysis port and balance port. To begin measuring the saturation pressure, press **ENTER**.

Status messages display while the saturation pressure is being measured, and the saturation pressure is stored in memory and used in report calculations.



#### PRINT

Report Options on page 3 - 20 Communications on page 3 - 34 Data Results on page 2 - 20

#### Alt + 6

Use to print a report from the last analysis. A printed report contains more information than the report generated to the keypad display. A report can be printed either to a printer or through a serial transmission line. Reports are also sent to the window, regardless of the specified destination.

Reports are generated after analysis and remain available for viewing or printing until another analysis is performed. When performing an analysis, reports from the previous analysis are deleted. Data for up to five analyses, however, are stored in the instrument. These data can be viewed using a web browser when connected to a network via an Ethernet connection.



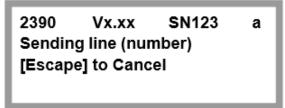
#### **TRANSMIT**

<u>Data Format on page A - 1</u> RS-232 Operation on page C - 1

#### AIt + 3

The RS-232 interface transmits report data to a computer using the standard ASCII data format. When captured with an asynchronous serial communications program, the report data can be used in spreadsheet and data manipulation programs.

This window indicates that data is being transmitted.



To cancel the transmission, press Alt + Clear (Escape).



#### MANUAL MODE OPERATIONS

#### Alt + 1

Some manual operations have been included in the software. These operations are used for system testing and diagnostic purposes and should only be performed at the request of a service representative.

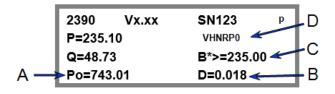


Do not use this mode of operation without direction from a service representative. Some operations when used incorrectly may cause damage to the instrument or one of its components.

This template shows the keys used for manual mode functions.



When performing the manual control functions, pertinent information is displayed in the keypad window. The following example shows the type of information that displays. A service representative may request this information.



- A. Current Po reading (not applicable for the 2390a)
- B. Differential pressure
- C. Servo valves
- D. System valves



# **Manual Mode Keypad Functions**

Functions	Description
Raw/True	Displays the raw A/D readings for the sample, balance, Po transducers or quantity adsorbed transducer, or the true pressure and volume readings.
B/S	Disables/enables the balance servo.
	A <i>B</i> in the display window indicates that the balance servo and sample servo are enabled. In the example on the previous page:
	* indicates the valves are on
	<ul><li>&gt; is the servo direction</li></ul>
	■ 234.56 is the target pressure
	An S indicates the sample servo is enabled, and the balance servo is disabled.
	For this command, press <b>Alt + 8</b> .
Servo valves	The 4, 5, and 6 keys operate servo valves.
	<b>ON/OFF</b> . Turns the servo valves on and off. An asterisk (*) appears next to the servo valve letter (B or S) when the servo valves are on.
	<b>UP/DOWN ARROWS</b> . Press ↑ ↓ to change the direction of the servo. > displays when in dose mode. < displays when in evacuate mode.
	TARGET. Use to edit the sample servo target pressure.
	2390 Vx.xx SN123 a P=760.000 VR Target Pressure: (current target pressure)
	The fourth line defaults to the current target pressure. Press <b>CLEAR</b> to clear the field of its entry or begin to enter the new value. Use <b>Alt +</b> . (decimal) to erase the previous keystroke.



## **Manual Mode Keypad Functions (continued)**

Functions	Description
System valves	The 0, dot, 1, 2, and 3 keys operate system valves.
	Choices are:
	■ V = Vacuum
	■ H = Helium
	<ul><li>N = Nitrogen</li><li>R = Reservoir</li></ul>
	■ P = Port
	■ 0 = Po valve (not applicable for Gemini 2390 <i>a</i> )
	<b>Po / VACUUM</b> . Opens and closes the Po or vacuum valve. For the Po value (not applicable for the Gemini 2390a), press <b>Alt + 1</b> . For the vacuum valve, press <b>1</b> .
	<b>HELIUM</b> . Opens and closes the helium valve.
	NITROGEN. Opens and closes the nitrogen valve.
	<b>RESERVOIRS</b> . Opens and closes the balance and sample reservoir valve in unison.
	<b>PORTS</b> . Opens and closes the balance and sample port valves in unison.
System values	When the character displays, the valve is open. When a dash (–) displays, the valve is closed.



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## **4 DIAGNOSTICS**

Data Results on page 2 - 20
Unit Configuration on the next page
Adsorptive Line Test on page 4 - 3
Helium Line Test on page 4 - 5
System Leak Test on page 4 - 7
System Check on page 4 - 9
Zero Test on page 4 - 10

#### AIt + 7



Press **CHOICE** to display the options. Press **ENTER** to select.

#### Use to:

- View analyzer and calibration statistics
- Clean and verify the gas line for the adsorptive gas
- Clean and verify the gas line for helium
- Check the system for leaks (service request only)
- Verify that the analyzer is operating properly (service request only)
- Zero transducers to stored offsets (service request only)

Some of the diagnostics require operator attention and a beep sounds at that time. The beeping continues at intervals until the operator performs the task shown in the display window. Do not press **ENTER** until the task has been performed. After a test has completed, a report is generated automatically to the destination(s) specified in *Report Options*.



It is not recommended that diagnostic reports be transmitted via serial line communication. Diagnostic results in this form produce values only and will require interpretation by a Service representative.

2390 Vx.xx SN123
Diagnostic
Diagnostic Type?
Unit Configuration

Choices: Unit Configuration, Adsorptive Line Test, Helium Line Test, System Leak Test, System Check, Zero Test



## **UNIT CONFIGURATION**

#### AIt + 7

Use to view instrument and calibration statistics. The information contained in the prompts cannot be edited.

2390 Vx.xx SN123 a Diagnostic Diagnostic Type? Unit Configuration

- IP Address
- Controller Boot
- Last Measured Po
- Sample Offset
- Sample Slope
- Adsorbed Offset
- Adsorbed Slope
- Balance Offset
- Balance Servo
- Sample Servo Low
- Sample Servo High
- Po Offset
- Po Slope

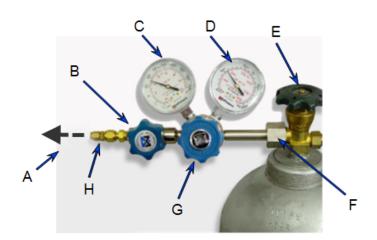


## ADSORPTIVE LINE TEST

Guidelines for Connecting Gases on page 5 - 6
Clean and Verify the Gas Line on page 5 - 7

This test helps to clean and verify the gas line for the adsorptive; first from the instrument to the regulator shut-off valve, then from the instrument to the gas cylinder shut-off valve. This test should be performed every time the adsorptive gas cylinder is replaced.

Always confirm the state for valves and the low-pressure gauge reading.



- A. Gas tubing to instrument
- B. Gas regulator shut-off valve OPEN
- C. Low pressure gauge (15-18 psig)
- D. High pressure gauge
- E. Gas cylinder shut-off valve CLOSED
- F. Regulator connecter nut
- G. Regulator control knob OPEN
- H. Brass reducer fitting

2390 Vx.xx SN123 a Diagnostic Diagnostic Type? Adsorp. Line Test

A series of prompts displays each time **ENTER** is pressed. Some prompts may request a task to be performed. Always perform the task before pressing **ENTER**.

**Informative prompt**. Indicates how long a test will take or when an operator should open or close a valve. Read the message, then press **ENTER**.



2390 Vx.xx SN123 a Operator action in 10 min. [ENTER] to continue.

**Operator action prompt**. Instructs the operator to perform an action. Perform the action, then press **ENTER**.

2390 Vx.xx SN123 a Open gas bottle shut-off valve. [ENTER] to continue.

At the end of the test, a Passed or Failed prompt is displayed.

2390 Vx.xx SN123 a Diagnostic Passed [ENTER] to continue.

- If *Failed* displays in the window, tighten all gas line connections for the adsorptive gas and restart the test.
- If *Passed* displays in the window, continue with the next step.

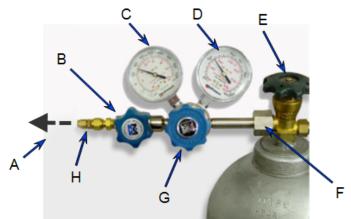


### HELIUM LINE TEST

Guidelines for Connecting Gases on page 5 - 6
Clean and Verify the Gas Line on page 5 - 7

This test helps to clean and verify the Helium gas line; first from the instrument to the regulator shut-off valve, then from the instrument to the gas cylinder shut-off valve.

Always confirm the state for valves and the low-pressure gauge reading.



2390 Vx.xx SN123 a Diagnostic Diagnostic Type?

**Helium Line Test** 

- A. Gas tubing to instrument
- B. Gas regulator shut-off valve OPEN
- C. Low pressure gauge (15-18 psig)
- D. High pressure gauge
- E. Gas cylinder shut-off valve CLOSED
- F. Regulator connecter nut
- G. Regulator control knob OPEN
- H. Brass reducer fitting

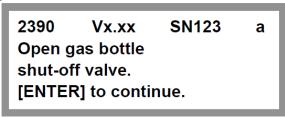
A series of prompts displays each time **ENTER** is pressed. Some prompts may request a task to be performed. Always perform the task before pressing **ENTER**.

**Informative prompt**. Indicates how long a test will take or when an operator should open or close a valve. Read the message, then press **ENTER**.

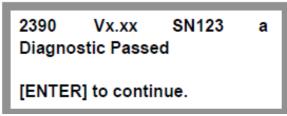
2390 Vx.xx SN123 a Operator action in 10 min. [ENTER] to continue.



**Operator action prompt**. Instructs the operator to perform an action. Perform the action, then press **ENTER**.



At the end of the test, a *Passed* or *Failed* prompt is displayed.



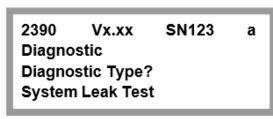
- If *Failed* displays in the window, tighten all gas line connections for the adsorptive gas and restart the test.
- If *Passed* displays in the window, continue with the next step.



## SYSTEM LEAK TEST

#### Guidelines for Connecting Gases on page 5 - 6

This test checks for atmospheric leaks among instrument components and typically is performed only when requested by a service representative. Sample tubes or plugs must be installed on the sample and balance ports prior to performing this test.



A series of prompts displays each time **ENTER** is pressed. Some prompts may request a task to be performed. Always perform the task before pressing **ENTER**.

■ Informative prompt. Indicates how long a test will take or how long before a valve should be opened or closed. Read the message, then press ENTER.

2390 Vx.xx SN123 a Operator action in 10 min. [ENTER] to continue.

Operator Action prompt. Instructs the operator to perform an action. Perform the action, then press ENTER.

2390 Vx.xx SN123 a Open gas bottle shut-off valve. [ENTER] to continue.



At the end of the test, a prompt indicating *Passed* or *Failed* is displayed.

2390 Vx.xx SN123 a Diagnostic Passed [ENTER] to continue.

- If *Failed* displays in the window, tighten all gas line connections for the adsorptive gas and restart the test.
- If *Passed* displays in the window, continue with the next step.



## SYSTEM CHECK

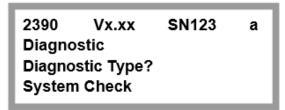
#### Adsorptive Line Test on page 4 - 3

This test verifies that all instrument components are operating properly. This test requires operator attention. The instrument will beep when attention is required.

Before beginning this test, ensure that the:

- sample tubes or plugs are installed on the sample and balance ports.
- helium gas inlet is plugged or if helium is attached, the regulator shut-off valve is closed.
- adsorptive gas in attached to its inlet port.
- vacuum pump is installed.

Press **ENTER** to start the test.



When the test is finished, press **ENTER**.

2390 Vx.xx SN123 a
Diagnostic Complete

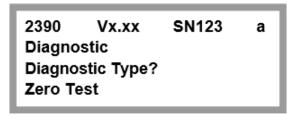
[ENTER] to continue.



## ZERO TEST

Use to compare and zero transducers to stored offsets. This test should be performed only when requested by a service representative. This test does not require operator attention.

Before beginning this test, ensure that clean, empty sample tubes are installed on the sample and balance ports



Verify that clean, empty sample tubes of the same size are installed on the sample and balance ports.

2390 Vx.xx SN123 a Verify port tubes are installed. [ENTER] to continue.

This prompt indicates how long the test will take to complete. This test takes 60 minutes, and no operator attention is required.

2390 Vx.xx SN123 a Verify port tubes are installed. [ENTER] to continue.

When the test is complete, press **ENTER**.

2390 Vx.xx SN123 a
Diagnostic Complete

[ENTER] to continue.



# **5 TROUBLESHOOTING**

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

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

- Error Messages document (PDF)
- Parts and Accessories
- Vacuum Pump Guide (PDF)

Most operational problems are caused by:

- Leaks (commonly found at the sample tube O-ring at the analysis port)
- Sample weighing errors
- Use of too much analysis bath fluid in the Dewar at the start of an analysis
- Entry of incorrect system volume for analysis
- Impure gas supply

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

#### Elevator cannot be raised or lowered.

Cause: Dewar elevator stuck.

Action: Check for possible obstruction to elevator movement.

#### Elevator is noisy.

Cause: The elevator screw may need greasing.

Action: Contact your Micromeritics Service Representative.

#### Expected results are not within range.

Cause A: Sample improperly degassed.

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

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



Action B: Replace gas cylinder. See Replace a Gas Cylinder on page 5 - 8.

Cause C: Undetermined.

Action C: Do the following:

- Perform an empty tube analysis. See <u>System Leak Test on page 4 -</u>
   Print the results.
- Perform a reference material analysis. See <u>Reference Analysis on</u> page 5 - 9. Print the results.
- 3. Contact your Micromeritics Service Representative.

#### Specified pressure not reached.

Cause A: Sample or balance tube not properly attached.

Action A: Reattach sample and balance tubes. Ensure both are securely attached to ports. Replace O-rings if defective. See <u>Sample Tube O-ring Replacement</u> on page 5 - 14.

Cause B: Leak in gas line.

Action B: Perform the Adsorptive and Helium Line tests to determine if a leak exists. See *Diagnostics on page 4 - 1*.

### Unable to reach a satisfactory vacuum.

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

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

Cause B: Vacuum pump is leaking.

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

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

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

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

Cause: Leak in the gas line connection.

Action: Perform the Adsorptive (or Helium) Line test to determine the location of the

leak. See Diagnostics on page 4 - 1.

#### Unit does not work when powered ON.



- Cause A: Power cord not fully inserted at one end.
- Action A: Insert power plug firmly into power source and analyzer power connecter.
- Cause B: No power at outlet.
- Action B: Plug in another electrical device to test the outlet. If there is no power, contact an electrician.
- Cause C: Plug prongs bent so that contact not made at outlet.
- Action C: Gently move power plug at outlet while watching power indicator light. If the indicator light comes on, have electrician replace outlet or plug.
- Cause D: Power cord damaged.
- Action D: Have electrician check cord using a test meter. Replace the cord if defective.
- Cause E: Loose internal connection or broken wire.
- Action E: Call a Micromeritics Service Representative for repair or replacement information.

#### Valves cannot be operated.

Cause: Cable from computer to the instrument is loose.

Action: Reconnect the cable.

#### Vacuum pump is noisy.

- Cause A: Sample tube connector is loose.
- Action A: Tighten fitting. Replace O-ring.
- Cause B: Sample tube O-ring is worn or cracked.
- Action B: Replace O-ring. See Sample Tube O-ring Replacement on page 5 14.
- Cause C: Sample tube is cracked.
- Action C: Replace with new sample tube.
- Cause D: No sample tube loaded on a selected port.
- Action D: Install plug or empty sample tube.
- Cause E: Gas inlet valve open while vacuum valve open.
- Action E: Close gas inlet valve.



## **POWER**

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



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



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

## PARTS AND ACCESSORIES

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



### SAFE SERVICING



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

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

Product specific risks that may affect service personnel:

- **Electrical**. Servicing or repair could require opening the outer panels and exposing energized electrical components.
- Liquid nitrogen. See <u>Dewar Precautions on page 2 13</u>.
- **Elevator.** Could pose a pinching hazard when lowering. Maintenance may require the elevator screw to be greased. The service engineer can use a manual switch on the elevator assembly to cycle the elevator to distribute the grease to permit safe servicing and continued safety of the equipment after servicing.

#### Protective measures for these risks:

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

Verification of the safe state of the instrument after repair:

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



## GUIDELINES FOR CONNECTING GASES



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



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

- Place gas cylinders within 6 feet (2 m) of the gas inlets of the analyzer. Place the cylinders close enough to allow for proper connection at the analyzer inlet.
  - Using gas line extenders on gas cylinders located in remote areas may degrade gas quality and reduce pressure. Gas lines are typically five to six feet long.
  - Long gas lines, such as those used with gas cylinders placed in remote areas, must be evacuated for an extended period of time to remove ambient gases. When possible, avoid placing gas cylinders in remote locations. It is always best to have gas cylinders located near the analyzer.
- Use a retaining strap (or other appropriate tether) to secure the gas cylinder.
- Always use the gas lines provided with the analyzer. It is very important that proper gas lines are used with the analyzer.
  - Do not use polymer tubing for the gas line.
  - <u>Do not use</u> flexible gas lines. Some flexible lines may appear to be appropriate, such as those with a herringbone covering, but the line may be coated internally with a polymer.
- Carefully route the gas lines from the cylinder to the analyzer avoiding overlapping or entangling gas lines. This will help avoid confusion when maintenance is required.
- Label the gas line at the analyzer inlet for proper identification and maintenance.
- Replace gas cylinders before gas is depleted. It is best to replace a gas cylinder when the pressure reads approximately 500 psi (3500 kPa) on the high-pressure gauge. Contaminants absorbed to the walls of the cylinder will desorb as the pressure decreases.
- Ensure the gas cylinder is closed before connecting to the analyzer.



#### CLEAN AND VERIFY THE GAS LINE

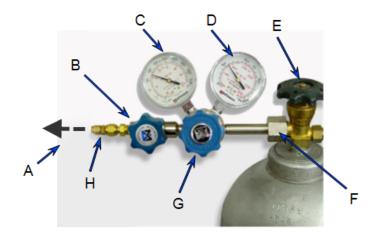
Adsorptive Line Test on page 4 - 3
Helium Line Test on page 4 - 5



Press CHOICE to display the options. Press ENTER to select.

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

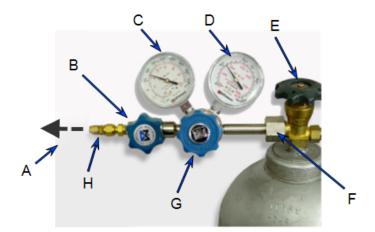
Always confirm the state for valves and the low-pressure gauge reading.



- A. Gas tubing to instrument
- B. Gas regulator shut-off valve OPEN
- C. Low pressure gauge (15-18 psig)
- D. High pressure gauge
- E. Gas cylinder shut-off valve CLOSED
- F. Regulator connecter nut
- G. Regulator control knob OPEN
- H. Brass reducer fitting



#### REPLACE A GAS CYLINDER



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

## Disconnect a Depleted Gas Cylinder

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

## Connect a Gas Cylinder

#### **Regulator Pressure Settings**

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



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



### REFERENCE ANALYSIS

Clean and Label Sample Tubes on page 2 - 6

Determine the Sample Mass on page 2 - 8

Sample Tube Installation on page 2 - 10

Fill and Install the Dewar on page 2 - 12

Perform an analysis using the carbon black reference material included in the accessories kit to verify that the instrument is operating properly. See the booklet included with the reference material when preparing for the reference analysis.

- 1. Clean and label the balance tube and sample tube.
- 2. Determine the sample mass before degassing.
- 3. Degas the sample.
- 4. Install the sample tube (containing sample) on the sample port.
- 5. Install a clean, empty sample tube (of the same size) on the balance port.
- 6. Fill the Dewar with liquid nitrogen to about 2 cm (3/4 in.) from the top for the small Dewar and 5 cm (2 in.) for the large Dewar.



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.

7. Close the sample compartment door and allow the Dewar to equilibrate to ambient conditions (approximately 30 minutes).



Ensure the sample compartment door is closed before beginning an analysis. If an abnormal condition causes the analyzer to operate at an excessive pressure, the sample or balance tube could dislodge from its port, possibly breaking the Dewar and causing personal injury or damage to the equipment.

8. Press **Alt + 4**. The following prompt is displayed:

2390 Vx.xx SN123 a Analyze Select Setup: (number) ? 0 (factory defaults)

- 9. Press **CHOICE** until Setup *Group 1* (*N300-700* is Sample ID) is displayed. The parameters specified in this *Set Up* group are appropriate for the reference material being analyzed.
- 10. At the prompts, verify or enter information at each prompt.



11. Press **ENTER** to start the analysis.

2390 Vx.xx SN123 a Analyze [ENTER] to Start [ESCAPE] to Cancel

- 12. After the analysis is complete, the window shows the results. Compare the BET results in the window with those shown on the label of the reference material bottle.
  - If the analysis results match those shown within the tolerance level on the bottle, the analyzer is ready to use.
  - If the analysis results are not within the tolerance level, repeat steps 2 through 10 to analyze the sample again. Verify that the value entered for the mass is correct. If the analysis results still fail to match, contact a Micromeritics Service representative.



## PREVENTIVE MAINTENANCE

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

Maintenance Required	Frequency
Analyzer	Clean outside as required or every 6 months
Analysis Dewar	Clean weekly
Port Frit and O-ring	Replace every 30 days
Vacuum pump oil	Inspect every 3 months. Change as required.
Alumina in oil vapor trap	Replace as required
Vacuum pump exhaust filter	Replace as required or every 12 months
Tube analysis	Blank as required

#### **CLEAN THE INSTRUMENT**

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



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



#### CHECK AND CLEAN THE DEWAR



When handling Dewars, follow the precautions outlined in <u>Dewar Precautions on</u> page 2 - 13.



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.
- Pour out liquid nitrogen into an appropriate cryogenic container. Do not re-use liquid nitrogen.



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

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



### 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.

## Sample Port Frit Replacement



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

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



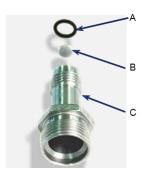
Use the proper size frit for the sample port. The analyzer will not operate properly if an incorrect size is used.

1. Use a wrench to remove the connecting nut from the sample port. Pry out the filter (frit) and O-ring.



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

2. Place a new frit into the connecting nut.



- A. O-ring
- B. Filter (frit)
- C. Sample tube fitting
- 3. Replace the filter and O-ring. Carefully reassemble the sample tube fitting and reinstall on the sample port. Tighten by hand, then with a wrench to prevent leaks.



## Sample Tube O-ring Replacement



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

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced.



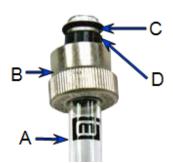
Before removing (or installing) a sample tube, ensure that the *Reload* prompt is displayed. Observe the analyzer schematic to verify valve status.



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

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

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



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



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



If the O-ring remains inside the sample port, use the O-ring removal tool from the accessory kit *[tool part number 004-54618-00]*.

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



## Perform a Blank Analysis for Diagnostic Purposes

Analysis Conditions on page 3 - 14
System Options on page 3 - 38

AIt + 2



Press CHOICE to display the options. Press ENTER to select.

Perform a blank analysis to help pinpoint operational problems. Leaks, as well as electronic problems, will result in an unsatisfactory blank analysis. Perform this test before beginning detailed troubleshooting. The data produced during the blank analysis can be helpful in identifying the cause of a problem. For this reason, perform a blank analysis before contacting a Service Representative.

Helium is required for performing a blank analysis since the free space needs to be measured. If helium is unavailable, contact a Micromeritics service representative for an alternative method of performing this procedure.



The following procedure is performed using water at ambient temperature in the Dewar.

- 1. Install empty, straight-wall sample tubes (free of cracks or chips) of the same size in both the sample and balance ports. Ensure the O-rings are present and are in good condition.
- 2. Place the Dewar support on the elevator, then place a Dewar of water at ambient temperature on the Dewar support.



A Dewar support is not necessary for the larger Dewar used with the Gemini VII 2390t. Place the Dewar directly on the elevator.

- 3. Press Alt + 2.
- 4. At the Setup prompt, select with an unused number or existing Setup.
- 5. Continue through the Select Action and Edit Setup prompts.



6. Access Analysis Conditions and enter the following parameters:

Parameter	Entry
Evacuation time	3 minutes
Free space	Measured
First rel pressure	0.100 P/Po
Last rel pressure	0.90 P/Po
Number of points	9
Analysis mode	Equilibrate
Equilibration time	3 seconds

- 7. Access System Options and select Request Sat. Press?.
- 8. Press **Alt + 4** and enter the following parameters.

Parameter	Entry	
Sample ID	(user-entered)	
Sample mass	1.00 g	
Saturation pressure	760 mmHg	
Evacuation rate	1000 mmHg/min	

- 9. Press **ENTER** to begin the analysis.
- 10. Review the analysis results for acceptability. They should be within the tolerances shown below. If these results are not achieved, contact a Service Representative.

P/Po	Tolerance in cm3
0.1	±0.008
0.2	±0.010
0.3	±0.012
0.4	±0.014
0.5	±0.016
0.6	±0.018
0.7	±0.020
0.8	±0.022
0.9	±0.024



#### CLEAN THE GAS DELIVERY TUBES

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

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

## LUBRICATE THE ELEVATOR DRIVE ASSEMBLY

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

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

## POWER INSTRUMENT ON AND OFF



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

#### Power ON the equipment in the following order:

- 1. Analyzer.
- External vacuum pump (the pump must warm approximately two hours before performing analyses).

## Power OFF the equipment in the following order

- 1. Analyzer.
- 2. External pump.



# A DATA FORMAT

Data can be transmitted in one of two user-selectable formats: single column or spreadsheet. The data are in ASCII-delimited format. Spreadsheet format is suitable for direct import into many popular spreadsheets.

In all tables, units are:

Date: DD/MM/YY Time: HH:MM:SS

Pressure: mmHg Elapsed

Time: seconds

For all Pass/Fail reports:

0 = Fail

1 = Pass or Reported-only

## REPORT FORMAT - SINGLE COLUMN

Record Number	Information Conveyed	Form
1	Software version number	21 characters
2	Instrument ID	20 characters
3	Set Up group number	1 integer
4	Setup ID	20 characters
5	Sample ID	20 characters
6	Sample mass	1 floating point
7a	Start date	8 characters
7b	Start time	8 characters
8a	End date	8 characters
8b	End time	8 characters
9	Evacuation rate	1 floating point
10	Evacuation time	1 integer
11	Free space method: 0 = none 1 = previous 2 = measured 3 = calculated	1 integer



12	Free space (cm <sup>3</sup> STP/760 mmHg)	1 floating point
13	Nonideality	1 floating point
14	Sample density	1 floating point
15	Saturation pressure (initial point if <b>During analysis</b> selected)	1 floating point
16	Analysis mode 0 = equilibrate 1 = scan	1 integer
17	Equilibration time	1 floating point
18	Scan time	1 integer
t-Method F	Parameters	
19	Thickness curve 0 = Harkins and Jura 1 = Halsey 2 = Magee-STSA	1 integer
20	Thickness parameter 1	1 floating point
21	Thickness parameter 2	1 floating point
22	Thickness parameter 3	1 floating point
23	Minimum thickness	1 floating point
24	Maximum thickness	1 floating point
25	Area correction	1 floating point
BJH Paran	neters	'
26	Thickness curve 0 = Harkins and Jura 1 = Halsey 2 = Magee-STSA	1 integer
27	Thickness parameter 1	1 floating point
28	Thickness parameter 2	1 floating point
29	Thickness parameter 3	1 floating point
30	Minimum diameter	1 floating point
31	Maximum diameter	1 floating point



22	Pore geometry	1 integer
32	0 = Slit	i iiilegei
	1 = Cylinder	
	2 = Sphere	
33	Cheng-Yang correction	1 integer
	0 = Not requested 1 = Requested	
34	Smooth differentials	1 integer
	0 = Yes	
	1 = No	1 floating point
35	Molecular cross sec area	1 floating point
36	Density conversion factor	1 floating point
37	BET multipoint surface area	1 floating point
38	BET multipoint surface area Pass/Fail result	1 integer
39	Langmuir surface area	1 floating point
40	Langmuir surface area Pass/Fail result	1 integer
41	BET single point surface area	1 floating point
42	BET single point surface area Pass/Fail results	1 integer
43	t-method micropore volume	1 floating point
44	t-method micropore volume Pass/Fail result	1 integer
45	t-method micropore area	1 floating point
46	t-method micropore area Pass/Fail result	1 integer
47	t-method external surface area	1 floating point
48	t-method external surface area Pass/Fail result	1 integer
49	Adsorption total pore volume	1 floating point
50	Adsorption total pore volume Pass/Fail result	1 integer
51	Desorption total pore volume	1 floating point
52	Desorption total pore volume Pass/Fail result	1 integer
53	BJH adsorption total pore volume	1 floating point
54	BJH adsorption total pore volume Pass/Fail result	1 integer
55	BJH desorption total pore volume	1 floating point
56	BJH desorption total pore volume Pass/Fail result	1 integer



57	Horvath-Kawazoe maximum pore volume	1 floating point
58	Horvath-Kawazoe maximum pore volume Pass/Fail result	1 integer
59	Horvath-Kawazoe relative pressure at maximum pore volume	1 floating point
60	Horvath-Kawazoe median pore width	1 floating point
61	Horvath-Kawazoe median pore width Pass/Fail result	1 integer
62	Number of points collected	1 integer
63	Absolute pressure (number of points)*	1 floating point
64	Quantity adsorbed (number of points)*	1 floating point
65	Saturation pressure* (if <b>During analysis</b> selected)	1 floating point
66	Elapsed time in minutes*	1 integer
67	Report selections (number of points)*  0 = Not selected  1 = Selected from surface area  2 = Selected for t-method  3 = Selected for both  End of transmission	1 integer

<sup>\*</sup> Equals the number of points collected, for example, if five points were collected, there are five records for each field.



# REPORT FORMAT - SPREADSHEET

Record Number	Information Conveyed	Form
1	Software version number	
2	Instrument ID	
3	Set Up group number	1 integer
4	Setup ID	20 characters
5	Sample ID	20 characters
6	Sample mass	1 floating point
7a	Start date	8 characters
7b	Start time	8 characters
8a	End date	8 characters
8b	End time	8 characters
9	Evacuation rate	1 floating point
10	Evacuation time	1 integer
11	Free space method: 0 = none 1 = previous 2 = measured 3 = calculated	1 integer
12	Free space (cm <sup>3</sup> STP/760 mmHg)	1 floating point
13	Nonideality	1 floating point
14	Sample density	1 floating point
15	Saturation pressure (initial point if <b>During analysis</b> selected)	1 floating point
16	Analysis mode 0 = equilibrate 1 = scan	1 integer
17	Equilibration time	1 floating point
18	Scan time	1 integer



t-Method P	arameters	
19	Thickness curve 0 = Harkins and Jura 1 = Halsey 2 = Magee-STSA	1 integer
20	Thickness parameter 1	1 floating point
21	Thickness parameter 2	1 floating point
22	Thickness parameter 3	1 floating point
23	Minimum thickness	1 floating point
24	Maximum thickness	1 floating point
25	Area correction	1 floating point
BJH Paran	neters	<u>'</u>
26	Thickness curve 0 = Harkins and Jura 1 = Halsey 2 = Magee-STSA	1 integer
27	Thickness parameter 1	1 floating point
28	Thickness parameter 2	1 floating point
29	Thickness parameter 3	1 floating point
30	Minimum diameter	1 floating point
31	Maximum diameter	1 floating point
Horvath-Ka	awazoe Parameters	'
32	Pore geometry 0 = Slit 1 = Cylinder 2 = Sphere	1 integer
33	Cheng-Yang correction 0 = Not requested 1 = Requested	1 integer
34	Smooth differentials 0 = Yes 1 = No	1 integer
35	Molecular cross sec area	1 floating point
36	Density conversion factor	1 floating point
37	BET multipoint surface area	1 floating point
38	BET multipoint surface area Pass/Fail result	1 integer



39	Langmuir surface area	1 floating point
40	Langmuir surface area Pass/Fail result	1 integer
41	BET single point surface area	1 floating point
42	BET single point surface area Pass/Fail results	1 integer
43	t-method micropore volume	1 floating point
44	t-method micropore volume Pass/Fail result	1 integer
45	t-method micropore area	1 floating point
46	t-method micropore area Pass/Fail result	1 integer
47	t-method external surface area	1 floating point
48	t-method external surface area Pass/Fail result	1 integer
49	Adsorption total pore volume	1 floating point
50	Adsorption total pore volume Pass/Fail result	1 integer
51	Desorption total pore volume	1 floating point
52	Desorption total pore volume Pass/Fail result	1 integer
53	BJH adsorption total pore volume	1 floating point
54	BJH adsorption total pore volume Pass/Fail result	1 integer
55	BJH desorption total pore volume	1 floating point
56	BJH desorption total pore volume Pass/Fail result	1 integer
57	Horvath-Kawazoe maximum pore volume	1 floating point
58	Horvath-Kawazoe maximum pore volume Pass/Fail result	1 integer
59	Horvath-Kawazoe relative pressure at maximum pore volume	1 floating point
60	Horvath-Kawazoe median pore width	1 floating point
61	Horvath-Kawazoe median pore width Pass/Fail result	1 integer
62	Number of points collected	1 integer
63	Space separator	
63a	Carriage return/line feed	
63b	Carriage return/line feed	
63c	Carriage return/line feed	
64	Collected data, one record for each point collected (separated with commas) for the following:	



- Elapsed time
- Report selection
  - 0 = Not selected
  - 1 = Selected for surface area
  - 2 = Selected for t-method
  - 3 = Selected for both
- Absolute pressure
- Quantity adsorbed
- Saturation pressure (if *During Analysis* is selected)



# **B SUPPORTED PRINTERS**

The Gemini analyzer provides support for a USB printer and includes the following printer drivers:

- Canon Bubble Jet
- Epson ESCP
- Epson ESCP Raster
- Epson ESCP2
- HP PCL 3
- HP PCL 6XL
- Postscript

Printers capable of interfacing with the Gemini analyzer:

- must be USB 2.00 (or newer)
- must have a printer language supported by one of the printer drivers listed above
- cannot be host-based

Printers that do not meet these criteria will not interface properly with the analyzer. The printer driver is selected using **Set Up > Report Options**. Press **ENTER** until the **Printer?** prompt is displayed, then **CHOICE** until the appropriate printer driver is displayed.





### C RS-232 OPERATION

The Gemini analyzer is a standard DTE device. The RS-232 port on the back panel of the analyzer can be used to attach an analytical balance for transfer of sample mass, or for transmitting data through a serial line to a computer.

Ensure the receiving device is configured to interface with the assignments in the *Pin Assignment* table. Any signals not listed will be ignored by the analyzer.

### **Pin Assignment**

Pin	Signal	Description	Data Direction	
2	RXD	Receive data	To analyzer	
3	TXD	Transmit data	From analyzer	
4	DTR	Data terminal ready	From analyzer	
5	GND	Ground	N/A	
6	DSR	Data set ready	To analyzer	

The Gemini uses the DTR and DSR signals for hardware flow control. Ensure the serial device being used provides these signals. For example; if attaching to a computer (also a DTE device), use a null modem cable which includes the signals designated in the above table. If there is a problem with transmission, ensure that the signals are set up properly. If the signals are correctly configured, contact the receiving device manufacturer for assistance.





# D Analyze Samples with a Total Surface Area of 1.0 $M^2$ or Less

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

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

This technique can be performed by the following methods:

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

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

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

### USING GLASS BEADS

This method typically produces the best results.

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

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

where

m = mass of sample (g)

ρ = density of sample (g/cm<sup>3</sup>); if density is unknown, refer to your laboratory handbook.



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

$$n=rac{v}{0.014cm^3}$$

where

 $0.014 \text{ cm}^3$  = approximate volume of one bead

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

$$rac{ ext{free space } cm^3 imes 2.515 \ g/cm^3}{3.53} = ext{mass of glass beads(g)}$$

where

 $2.515 \,\mathrm{g/cm^3}$  = density of glass beads

3.53 = thermal correction (no units)



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

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



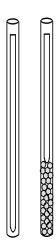
For subsequent samples of the same material, use the same mass of sample that was used for the initial sample so that the original bead quantity may be left undisturbed on the balance port.



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

### **USING FILLER RODS**

- 1. Clean the sample tube, balance tube, and filler rods.
- 2. Label the sample and balance tubes.
- 3. Prepare the sample and place it into the sample tube.
- 4. Insert the shorter filler rod into the sample tube. If using bulbous sample tubes, use the same size filler rod as used in the balance tube.
- 5. Ensure that the filler rods are equidistant from the top of each tube. If they are not, add or remove sample until they are the same distance from the top of the tube.





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

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



### USING GLASS BEADS AND FILLER RODS

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

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



# E SAMPLE DATA WORKSHEET FOR GAS ADSORPTION

Use this worksheet to record the values necessary to calculate the sample mass.							
Sam	ole tube identification	:					
Sam	ple Mass (g)						
			Before Degas	After Degas	After Analysis		
1.	. Mass of empty sample tube set		g				
2.	Mass of sample tube set plus sample		g	g	9		
3.	Mass of sample (step 2 minus step 1)		g	g	g		
Dega	s Information						
Dega	s apparatus						
Tem	perature (°C)						
Time	(hours)						
Actu	al time started						
Actu	al time finished						
Dega	s Notes:						





### **EU DECLARATION OF CONFORMITY**

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

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

Hereby declares that the product:

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

is in conformity with the following EU harmonization legislation:

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

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

### 2014/35/EU (LVD)

**EN 61010-1:2010/A1:2019 -** Safety requirements for electrical equipment for measurement, control, and laboratory use — Part 1: General requirements.

**EN 61010-2-081:2020** - Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes

#### 2014/30/EU (EMC)

**EN 61326-1:2013** - Electrical equipment for measurement, control and laboratory use — EMC requirements — Part 1: General requirements

**EN 61000-3-2:2014** - Part 3-2: Limits — Limits for harmonic current emissions (equipment input current  $\leq$  16 A per phase)

**EN 61000-3-3:2014** - Part 3-3: Limits — Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current <= 16 A per phase and not subject to conditional connection

#### 2011/65/EU (RoHS)

EN 63000:2018 - Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances

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Title: Vice President, R & D

Signature:

Date of issue: <u>04/30/2021</u>

Location: Norcross, GA USA





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