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Gemini VII 2390

Operator's Manual

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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.

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

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

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

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.

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One Micromeritics Drive • Norcross, GA 30093-1877 • Fax (770) 662-3696

Domestic Sales - (770) 662-3633
International Sales - (770) 662-3660
Domestic Repair Service - (770) 662-3666
Customer Service - (770) 662-3636

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1. GENERAL INFORMATION

This manual describes how to install, operate, and maintain the Gemini VII 2390 analyzer.

Organization of the Manual

The manual is organized as follows:

Chapter 1 **GENERAL INFORMATION**

Provides a general description and specifications of the analyzer.

Chapter 2 **INSTALLATION**

Describes how to install and verify operation of the Gemini analyzer.

Chapter 3 **USER INTERFACE**

Provides a description of the analyzer, its components, and software interface.

Chapter 4 **OPERATIONAL PROCEDURES**

Provides step-by-step operating procedures for the Gemini analyzer.

Chapter 5 **COMMANDS**

Provides a description of the commands available for the Gemini program.

Chapter 6 **TROUBLESHOOTING AND MAINTENANCE**

Provides troubleshooting and maintenance procedures.

Chapter 7 **ORDERING INFORMATION**

Provides information on ordering parts and accessories for the analyzer.

Appendix A **FORMS**

Contains a copy of the Sample Information Worksheet. This form is used to assist you in obtaining your sample mass.

Appendix B **ERROR MESSAGES**

Lists the error messages that may display in the keypad window and includes a cause and action for each.

Appendix C **CALCULATIONS**

Contains calculations used for producing report data.

Appendix D **DATA FORMAT**

Contains report formats for transmitted data.

Appendix E **SUPPORTED PRINTERS**

Provides information on the printers supported by the Gemini analyzer.

Appendix F **RS-232 OPERATION**

Provides information on RS-232 operation with the Gemini.

Appendix G **KEYBOARD INTERFACE**

Lists keyboard equivalents for the analyzer keypad.

Appendix H **ANALYZING SAMPLES WITH TOTAL SURFACE AREA OF 1.0 m2 OR LESS**

Provides information on analyzing samples with a total surface area of 1.0 m^2 or less.

Index **INDEX**

Provides quick access to a subject matter.

Conventions

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

Notes contain important information pertinent to the subject matter.

Warnings contain information that help you prevent actions that may cause personal injury.

Cautions contain information that help you prevent actions that may damage the analyzer.

Equipment Description

The Gemini VII Series analyzers are easy to use, fully automatic, and provide single- and multipoint surface area and pore size measurements. Three models are available; the Gemini 2390*a*, 2390*p*, and 2390*t*.

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

The Gemini 2390*t* 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 Po tube.

The Gemini analyzer contains a sample compartment and an attached keypad with a display window. The analyzer is controlled by commands entered through the keypad. The operational status of the analyzer can be continually monitored on the display window.

Most features are available on all three models, with the exception of a continuous Po measurement and some reports. Refer to Table 1-1 for a brief summary of model features; this table does not include all reports and features. Reports and features not specified in the table are available for all models.

The basic function of the Gemini analyzers is to determine the surface area of solid materials. Analyses are performed using a static volumetric technique in which the adsorptive flows into a tube containing the sample and into a balance tube, at the same time. The internal volume and the temperature surrounding both tubes are maintained at identical conditions. The only difference is the presence of the sample in the sample tube.

Table 1-1. Gemini VII Series Features

Feature	2390*a*	2390*p*	2390*t*
Single- and multipoint BET surface area	✓	✓	✓
Langmuir surface area	✓	✓	✓
Automatically generated pressure tables with user-selected points	✓	✓	✓
Total pore volume	✓	✓	✓
t-Plot method micropore volume and area	✓	✓	✓
Analysis log	✓	✓	✓
Horvath-Kawazoe data reduction	✓	✓	✓
BJH pore size distribution using adsorption isotherm		✓	✓
BJH pore size distribution using desorption isotherm			✓
Continuous measurement of saturation pressure (Po)		✓	✓

Analysis results can also be edited by specifying which points are to be included in surface area or t-Method calculations. The Gemini automatically recalculates the results and generates a new report. Reports remain available for viewing or printing until another analysis is started. Results can also be transmitted to a remote location or e-mailed automatically after analysis.

Operation

The sample and balance tubes are immersed in a single liquid nitrogen bath which maintains isothermal conditions for both tubes. The analysis gas is then delivered to the sample tube by a servo valve mechanism. The delivery rate of analysis gas flow into the balance tube is controlled by another servo valve connected to a differential pressure transducer. This differential pressure transducer measures the pressure imbalance between the sample and balance tubes, which is caused by the adsorption of the analysis gas onto the sample. As the sample adsorbs analysis gas, the pressure drops in the sample tube. The servo valve continuously restores the pressure balance between the two tubes by admitting more gas into the sample tube. The end result is that the Gemini maintains a constant pressure of analysis gas over the sample while varying the rate of analysis gas delivery to match exactly the rate at which the sample can adsorb the gas.

Gemini provides rapid and accurate sample analysis. As many as 50 data points can be collected and reported in the BET, Langmuir, or t-Method range. Additionally, all models of the Gemini VII Series can report discrete point adsorption isotherms consisting of up to 1,000 adsorption points, and up to 1,000 desorption isotherm points with the Gemini 2390*t*.

The Gemini requires only pure nitrogen as the analysis gas, even for most low surface area samples. The analyzer requires no flow regulator, gas mixer, or mixed gases to operate. Surface areas as low as 0.01 m^2/g are easily determined with excellent precision using nitrogen gas as the adsorbate. Optionally, helium may be used to determine any slight differences in volume between the balance tube and the sample tube (differential free space). The Gemini uses this information to automatically compensate even for these small differences in calculating analysis results.

The design of the Gemini avoids many errors which may occur with other surface area analysis equipment:

- Any free space associated with the sample tube is offset by an identical amount in the balance tube.
- Any thermal gradient in the coolant bath is the same for both the sample and balance tubes and produces a net effect of zero.
- Non-ideal gas behavior occurs in both the sample and balance tubes, cancelling negative effects on sample analysis.
- Gas-mixture separation, which may occur in flowing-gas systems due to thermal diffusion, cannot occur because mixed gases are not used.

Vacuum Pump

An external vacuum pump equipped with an anti-suckback valve is required for sample analysis with the Gemini analyzer. Any vacuum source achieving a vacuum better than 20 x 10^{-3} mmHg at the instrument inlet may be used.

An oil vapor trap to reduce oil vapor backstreaming is recommended.

The vacuum pump used with the Gemini must have an anti-suckback valve to prevent oil from being admitted to the instrument 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 Gemini analyzer. Appropriate vacuum pumps are available from Micromeritics. (Refer to **Ordering Information**, page **7-1** for ordering information.)

Cryogen

Liquid nitrogen is used as the cryogen to cool the sample during analysis. An easy-to-use liquid nitrogen transfer system, the Model 021, eliminates the need to use pressurized storage Dewars (refer to **Ordering Information**, page 7-1).

Gases

Compressed gases are required for sample analysis with the Gemini analyzer. Gas bottles and a rack for storage of these bottles should be located near the analyzer. As an alternative, outlets from a central source may be provided nearby. Gas sources must be able to supply flow rates of 4 liters per minute at 15 psig (103.5 kPa).

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. Gas regulators are available from Micromeritics (refer to **Ordering Information**, page 7-1 for ordering information).

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

Degasser

Micromeritics has available the following degassing units for preparing samples:

- **SmartPrep 065**
  The SmartPrep uses flowing gas to remove adsorbed contaminants from the surface and pores of your sample. It contains six sample ports, each one independently controlled for greater flexibility. It contains two serial ports, one for connecting to a computer and the other available for connecting an additional SmartPrep. You can connect up to four SmartPreps, one to the other, allowing the capability of up to 24 preparation ports at one time.

- **VacPrep 061**
  The VacPrep degasses up to six samples at up to 400 ºC with either flowing gas or evacuation.

- **FlowPrep 060**
  The FlowPrep degasses up to six samples at up to 400 ºC with flowing gas.

Refer to **Ordering Information**, page 7-1 for ordering information.

Internet Access

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

About Micromeritics	A brief history of Micromeritics, office locations, awards/certifications, career opportunities, and a virtual tour of its headquarters
Products	Product information and printable brochures
Applications	Application Notes, Product Bulletins, Tech Tips, Technical Articles/papers, and important application links
Online Catalog	Catalog of instruments and accessories, allowing you to place your order online
News and Press	Press releases, Events calendar, microReports, and latest Micromeritics news updates
Lab Service	Provides laboratory tips and access to the Micromeritics Analytical Services web site
Customer Support	Customer support contacts, product registration, instrument training information, Material Safety Data Sheets, and account registration
Grant Program	Details of the Grant Program established for non-profit organizations and universities
Contact Us	Contact information, office locations, maps and driving directions to the Micromeritics facility, and registration for the microReport newsletter

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

Specifications

The Gemini analyzer has been designed and tested to meet the specifications provided below.

Characteristic	Specification
Applicability	
Surface Area:	From 0.1 m^2, total; From 0.01 m^2/g, specific
Pore Volume:	From 4 x 10^{-6} cm^3/g
Pressure Measurement	
Pressure Measurement Range:	0 to 950 mmHg
P/Po Resolution:	< 10^{-4}
Relative Pressure Range:	0 to 1.0 P/Po (adsorption only)
Pressure Resolution:	< 0.1 mmHg
Accuracy and Linearity:	Better than ± 0.5% (full scale) (Transducer manufacturer's specification)
Vacuum System	
External:	Vacuum source achieving 20 x 10^{-3} mmHg (or better) at the instrument inlet for oil-based or oil-free pumps. For **oil-based pumps**: an anti-suckback valve is required to prevent oil from being admitted into the Gemini should there be a power failure. A device to reduce oil vapor backstreaming is also recommended.
Environment	
Temperature:	10 to 35 ºC (50 to 96 ºF) operating 0 to 50 ºC (32 to 122 ºF) non-operating
Humidity:	20% to 80% relative, non-condensing

Characteristic	Specification
Sample Tubes / Dewar	
Standard Tube:	Gemini VII 2390*a* and 2390*p*: 0.95-cm (3/8-in.) OD, 6.1 in.(15.5 cm) long with 6.5 cm^3 of volume. Sample capacity is approximately 2.0 cm^3 Gemini VII 2390*t:* 3/8-in. (0.95-cm) OD x 8.1-in. (20.5-cm) long with 8.9 cm^3 of volume. Sample capacity is approximately 2.0 cm^3
Dewar:	8 hours; Gemini VII 2390*a* and 2390*p* > 24 hours; Gemini VII 2390*t*
Electrical	
Voltage:	85 to 265 VAC
Frequency:	50/60 Hz
Power:	150 VA maximum
Gases	
Adsorbate:	Optimized for nitrogen in a liquid nitrogen bath. Gemini can be used with oxygen, argon, carbon dioxide, or other non-corrosive gases and butane, methane, or other light hydrocarbon vapors.
Physical	
Height:	23 in. (58 cm); Gemini VII 2390*a* and 2390*p* 29 in. (74 cm); Gemini VII 2390*t*
Width:	16 in. (40 cm)
Depth:	20 in. (51 cm)
Weight:	70 lbs (32 kg); Gemini VII 2390*a* and 2390*p* 74 lbs (34 kg); Gemini VII 2390*t*

2. INSTALLATION

This chapter describes how to install and verify operation of the Gemini analyzer. It also provides instructions for installing software upgrades.

Unpacking and Inspection

When you receive the shipping cartons, carefully compare the Packing List with the equipment actually received and check the equipment for any damage during shipment. Be sure to sift through all packing material before declaring equipment missing.

If you need to declare equipment as damaged or lost, save the shipping cartons. The claims investigator must examine the cartons in order to complete the inspection report.

Equipment Damage or Loss During Shipment

If equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The freight carrier, not Micromeritics, is responsible for all damage or loss occurring during shipment. If you discover damage or loss of equipment during shipment, report the condition to the carrier immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, a customer may receive equipment which is not in working order. When equipment has been damaged (either during shipment or in use) and you wish to return the equipment to Micromeritics for repair or replacement, please follow the steps below:

1. Pack the instrument in its original shipping carton if possible. If the original carton is unavailable, for a nominal fee, Micromeritics can provide another carton for your use.

Failure to package your instrument properly may result in shipping damage.

2. Tag or otherwise identify the defective equipment, noting the defect and, if possible, the circumstances under which the defect occurs.

3. Make reference to the sales order or purchase order for the equipment, and provide the date the equipment was received.

4. Notify a Micromeritics Service representative of the defect and request shipping instructions. The Service Department will assign a Return Material Authorization (RMA) number to your return and provide shipping information.

Installing the Analyzer

The analyzer should be checked to make sure it is operating properly before actual analyses are attempted. The remainder of this chapter describes how to install the analyzer, verify its operation, and install software upgrades.

Selecting a Location

When selecting the location of the analyzer, keep the following in mind:

- The analyzer performs best in a relatively constant temperature environment.
- It should be installed in a location free of drafts from sources such as a forced-air heating or cooling system.
- Do not place near an outside window; exposure to direct sunlight may cause the temperature to vary.

Connecting Gases

Use these guidelines when installing regulators and gas lines:

- Place gas bottles close to the analyzer. Using gas line extenders on gas bottles located in remote areas may degrade gas quality and reduce pressure.
- Use a retaining strap (or other appropriate tether) to secure the gas bottle.
- Carefully route the gas lines from the bottle to the analyzer, avoiding overlapping or entangling gas lines.
- Label the gas line at the instrument inlet for proper identification and maintenance.
- Ensure that the gas bottle is closed before connecting to the analyzer.

1. Remove the protective cap from the gas bottle.

Place the cap in a secure location, you will need to recap the gas bottle when it is depleted and replaced.

2. Attach an appropriate two-stage regulator to the gas supply bottle. Hand-tighten the nut, then use an appropriate wrench to tighten an additional 3/4 turn.

Leave the gas bottle shut-off valve closed until instructed otherwise.

3. Attach the gas inlet line to the regulator, reducer fitting, or regulator expansion:

It is very important to use the inlet tubing supplied with the analyzer. Gas lines made of materials other than copper or stainless steel can cause operational problems as well as inaccurate data.

If regulator has	Then .....
1/4-in outlet	Attach the reducer fitting to the outlet of the regulator shut-off/isolation valve.
	Tighten the nut finger-tight, then 1-1/4 turns with a wrench.
	Continue with the steps given for the 1/8-in outlet.
1/8-in outlet	Insert the gas tubing into the fitting.
	Make sure the tubing is seated fully inside the fitting.
	Tighten the nut finger-tight.
	While holding the fitting body steady, tighten the nut with a wrench 3/4 turn.

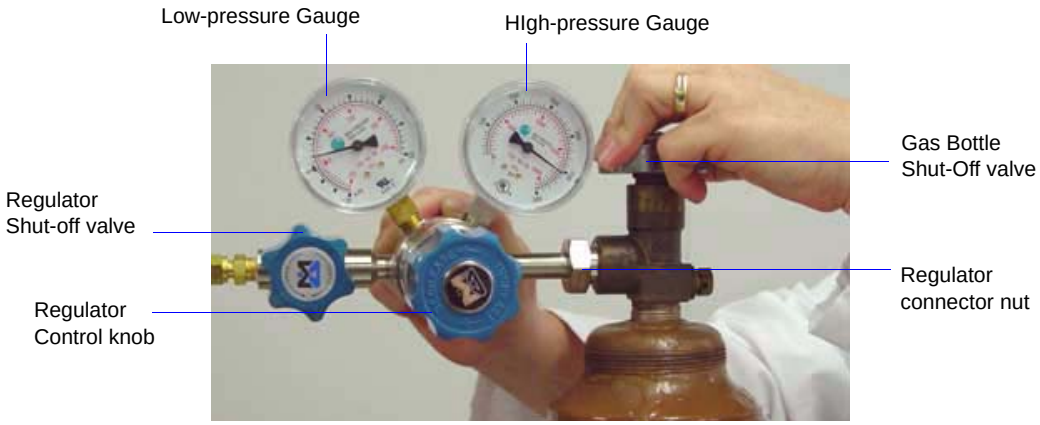
Do not overtighten the fittings. Doing so can collapse the brass ferrule and cause a leak.

4. Remove the plug from the **Adsorptive** gas port on the rear panel of the analyzer.

5. Insert the other end of the copper tubing into the port. Hand-tighten the connector nut, then use an appropriate wrench to tighten an additional 1/4 turn.

6. If you are using helium for free-space measurement, repeat steps 1 through 5 for the helium gas bottle.

7. Purge the regulator and inlet tubing; this is important to prevent contamination of the analysis gas.



   a. Close the regulator shut-off valve; turn it fully clockwise.

   b. Turn the regulator control knob fully counterclockwise.

   c. Slowly open the gas bottle shut-off valve; turn it counterclockwise, then quickly close it.

   d. Observe the high-pressure gauge.

      • If the pressure is stable, proceed with the next step.
      • If the pressure decreases, tighten the regulator connector nut until it becomes stable.

8. Set the instrument pressure.

   a. Turn the regulator control knob clockwise until the low-pressure gauge reads 15- 18 psig (103 - 124 kPag).

   b. Open the regulator shut-off valve.

   c. Open the gas bottle shut-off valve and flow gas for 10 to 30 seconds.

   d. Close the gas bottle shut-off valve.

Installing the Vacuum Pump

You can use an oil-based or oil-free vacuum pump with the Gemini analyzer. Both types are available from Micromeritics. The instructions provided below are for pumps ordered from Micromeritics.

You can also provide your own vacuum pump as long it meets required specifications (refer to **Vacuum System**, page **1-9**). Refer to the manufacturer's manual for installation instructions.

Oil-Based Pump

The oil-based pump is the standard pump shipped with the analyzer. It is strongly recommended that an oil vapor trap to reduce oil vapor backstreaming be installed on the oil-based vacuum pump.

Preparing the Oil Vapor Trap

1. Turn on a drying oven; preheat to 300 °C.
2. Pour approximately 180 grams of fresh alumina into a glass or metal container (approximately 250 mL if a graduated beaker is used).
3. Place the container in the oven; bake for two hours.

4. Remove the baked alumina from the oven and allow it to cool.
5. Insert an o-ring into each of the end fittings.

6. Screw one of the end fittings onto the trap body.

7. Be sure the trap body is dry and the alumina is lukewarm; pour the alumina pellets into the trap until they are just below the threads of the trap body.

8. Screw the other end fitting back onto the trap and tighten securely by hand.

9. Lightly tap both ends of the trap body on the work surface. This will remove any remaining dust from the pellets.

10. Place the trap on the work table and proceed with connecting the vacuum pump; you will install the trap onto the pump during that procedure.

Connecting the Vacuum Pump

If your pump was not purchased from Micromeritics, these instructions may vary. Refer to the documentation provided with your vacuum pump if necessary.

1. Place the vacuum pump on a work table.

2. Turn the ballast valve completely clockwise to close it. The ballast valve should remain closed during operation to obtain the best vacuum.

3. Install the oil vapor trap onto the intake port of the vacuum pump.

   a. Lift up and remove the cap on the intake port (see location above).

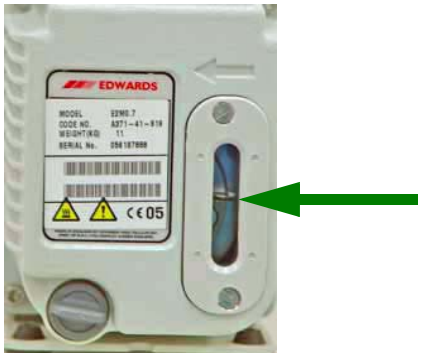
   b. Be sure there is a centering ring on the intake port; if not, place a centering ring with the small opening on the port. Place the oil vapor trap (containing the prepared alumina) onto the port.

Use this type of centering ring at the intake port.

   c. Place a clamp around the flange of the intake port and the flange of the trap.

   d. Swing the clamp fastening screw towards the intake port until it fits into the slot on the other half of the clamp. Tighten the wing nut securely by hand.

4. Observe the oil level window on the vacuum pump; ensure that the oil level is midway between the indicator lines.



Proceed with Step 5 if the oil-level is appropriate; if not, add oil before proceeding:

a. Remove the oil-fill plug.

b. Using the funnel provided in the accessories kit, slowly add oil until it is at the appropriate level, then replace the oil-fill plug.

5. Install the exhaust port filter.

   a. Loosen the wing nut of the clamp on the exhaust port. Swing the clamp fastening screw outward and remove the clamp; do not remove the centering ring.

   b. Place the filter on the centering ring.

   c. Place the clamp around the flange of the exhaust port and the flange of the exhaust filter.

   d. Swing the clamp fastening screw towards the exhaust port until it fits into the slot on the other half of the clamp. Tighten the wing nut securely by hand.

6. Connect the vacuum pump hose to the vacuum pump:

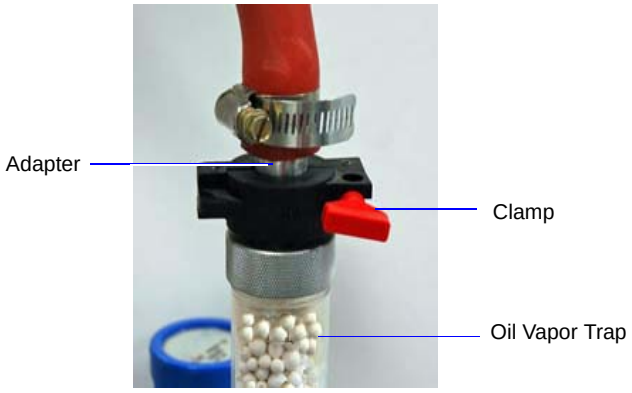
   a. Place a centering ring on the top of the oil vapor trap, then place the vacuum pump hose adapter on the centering ring.

   Use this type of centering ring for the trap body.

   b. Place the clamp around the flange of the oil vapor trap and the flange of the adapter.

   c. Close the clamp and tighten the wing nut securely by hand.

d. Attach one end of the vacuum pump hose to the adapter. Attach a hose clamp and tighten with a flat-head screwdriver.



7. Connect the vacuum pump hose to the analyzer:

a. Loosen the screws on the **Vacuum** port on the rear panel of the analyzer; remove the port cap and the centering ring.

b. Place the centering ring on the vacuum adapter, then insert the adapter into the vacuum port. Tighten the port screws with an appropriate wrench

c. Place the other end of the vacuum hose onto the adapter. Attach a hose clamp and tighten with a flat-head screwdriver.

8. Plug the vacuum pump power cord into an appropriate power source.

Oil-free Vacuum Pump

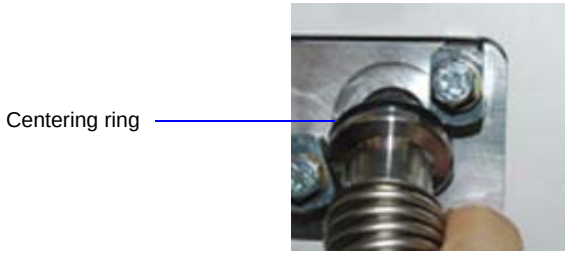
The oil-free/high-vacuum pump is available as an option.

1. Select the line voltage and prepare the pump using the instructions in the manufacturer's operator's manual.

2. Place an unopened clamp around the hose connection. Swing the clamp fastening screw around until it fits into the slot on the other half of the clamp, then tighten the wing nut.

3. Loosen the screws on the **Vacuum** port on the rear panel of the analyzer; remove the port cap and the centering ring.

4. Place the centering ring removed in step 3 on the hose flange; insert the hose flange into the vacuum port.

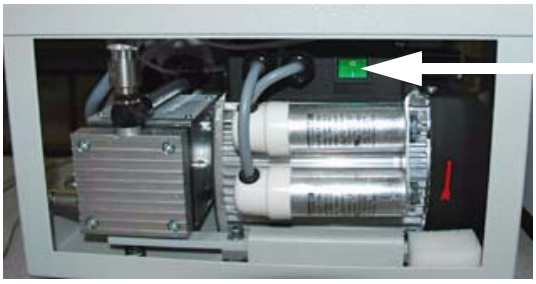


5. Tighten the two retaining screws with an adjustable wrench.

6. Plug the vacuum pump power cord into an appropriate power source.

7. Place the high-vacuum pump power switch on the back of the pump enclosure in the ON position.

8. Place the vacuum pump power switch on the front of the pump enclosure in the ON position.

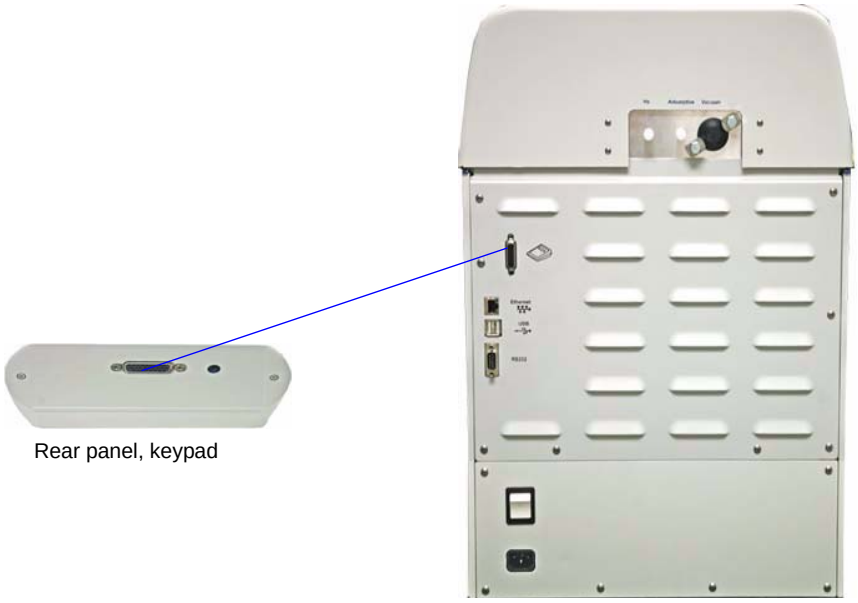


Connecting the Keypad

For the keypad to work properly, it must be connected before the analyzer is turned on.

1. Insert one end of the keypad cable into the connector on the rear panel of the keypad; tighten the retaining screws finger-tight.

2. Insert the other end of the keypad cable into the connector labeled with a keypad icon on the rear panel of the analyzer; tighten the retaining screws finger-tight.



Rear panel, keypad

Connecting the Power Cord

Attach the analyzer power cord to the power connector on the rear panel of the analyzer and into an appropriate power source, then turn the analyzer on

Rear panel, lower left

Connecting Optional Devices

This section provides instructions for connecting optional devices to the analyzer:

- Printer, current page
- Computer keyboard, current page
- Analytical balance, page **2-17**
- Serial device for data transmission, page **2-17**
- Bar code reader, page **2-17**
- Network cable, page **2-17**

If you are not connecting any of these devices, proceed to **Verifying Operation**, page **2-18**.

Printer

You can connect a printer to one of the USB ports for printing reports.

Refer to Supported Printers on page E-1 to verify that your printer driver is supported by the Gemini analyzer.

Connect one end of the printer cable to one of the USB 2.00 connectors on the rear panel of the analyzer. Connect the other end to the input connector on the printer.

Keyboard

A computer keyboard can be connected to one of the USB ports, enabling input from a standard keyboard. Having a computer keyboard connected does not affect operation of the keypad; you can use them interchangeably. For example, you may wish to use the operational commands using the keypad functions and a keyboard for entering a sample identification or description.

Operational commands can also be executed using the computer keyboard if you prefer to use a keyboard for all functions. Refer to **Keyboard Interface**, page **G-1** for the keyboard equivalents to keypad functions.

Insert the keyboard cable into one of the USB 2.00 connectors on the rear panel of the analyzer.

Analytical Balance or Serial Line Communication

The RS-232 port can be used to:

- connect a serial line for data transmission
- connect an analytical balance, allowing direct transfer of the sample mass to the analyzer software

1. Be sure the pin assignment of the RS-232 port matches the pin assignment of the output device for serial line transmission, or for the analytical balance. Refer to **RS-232 Operation**, page **F-1** for the pin assignment of the RS-232 port.
2. Check that the serial port settings (baud rate, stop bits, etc.) of the analyzer coincide with those of the attaching device. Refer to **Communications**, page **5-29** for information on viewing and editing current settings.
3. Connect one end of the RS-232 cable to the connector labeled **RS232** on the back of the analyzer and the other end to the attaching device.

Bar Code Reader

A bar code reader can be connected to a USB port and used to scan bar code numbers directly to the analyzer software, avoiding entry errors. The number is scanned into the field displayed at the time of the scan, typically, the **Sample ID** field is used for this information.

Network Cable

You can use the ethernet port on the rear panel of the analyzer to connect a network cable, enabling you to:

- e-mail results automatically after an analysis
- use a web browser to view analysis results; refer to **Using a Web Browser**, page **4-23**

1. Connect your network cable to the ethernet connector on the rear panel of the analyzer.
2. Press **Alt + 2**; the **Select Setup** prompt is displayed.
3. Press **ENTER** to accept the current Setup number.
4. Press **CHOICE** until **Edit Setup** is displayed, then press **ENTER**.
5. Press **CHOICE** until **Communications** is displayed, then press **ENTER**.
6. Obtain an IP address (refer to **Communications**, page **5-29**).

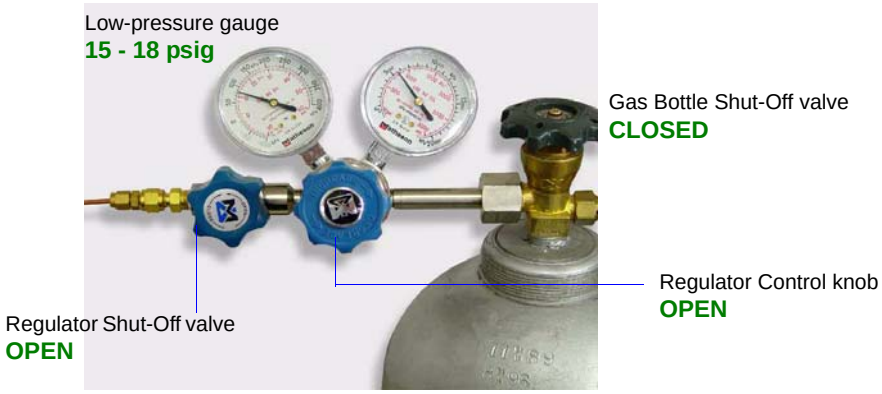
Verifying Operation

It is recommended that you read Chapter 3, User Interface to familiarize yourself with the keypad and prompts before verifying operation.

Cleaning and Verifying the Gas Lines

A feature has been added to the software that enables you to clean gas lines and verify there are no leaks at any connection before beginning operation. This test examines the gas line from the instrument to the gas bottle, then from the instrument to the regulator shut-off valve. A report is generated at the completion of the test verifying that it has passed or failed.

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



1. Check the adsorptive gas line for leaks:

   a. Press **Alt + 7** on the keypad; the Diagnostic prompt is displayed. Press **CHOICE** until **Adsorp. Line Test** is displayed, then press **ENTER**.

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

   b. A series of prompts displays each time you press **ENTER**. Some prompts may request that you perform a task. Always perform the task before you press **ENTER**.

   Refer to **Adsorptive Line Test**, page **5-46** for a description of the types of prompts that display in the keypad window.

c. 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.

2. Check the helium gas line for leaks:

If you are not attaching a Helium gas supply, you are ready to perform a reference analysis (next page).

a. Press **Alt + 7** on the keypad; the Diagnostic prompt is displayed.

b. Press **CHOICE** until **Helium Line Test** displays, then press **ENTER**.

```
2390    Vx.xx    SN123    a
Diagnostic
Diagnostic Type?
Helium Line Test
```

c. A series of prompts displays each time you press **ENTER**. Some prompts may request that you perform a task. Always perform the task before you press **ENTER**.

These are the same type of prompts that displayed for the **Adsorptive Line Test**.

d. At the end of the test, a prompt indicating **Pass** or **Fail** 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 Helium gas and restart the test.
- If **Passed** displays in the window, you are ready to perform a reference analysis (next page).

Performing a Reference Analysis

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

1. Clean and label your balance tube and sample tube (refer to **Cleaning and Labeling Sample and Balance Tubes**, page **4-8**).

2. Determine your sample mass before degassing (refer to **Determining the Sample Mass**, page **4-11**).

3. After the sample has been prepared, install the sample tube (containing sample) on the sample port (refer to **Installing the Sample Tube**, page **4-14**).

4. Install a clean, empty sample tube (of the same size) on the balance port.

5. 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. Refer to **Preparing the Analysis Dewar**, page **4-15**.

Always handle Dewars with care. Observe the precautions given on page 4-15 when preparing the Dewar.

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

Be sure 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 cause personal injury or damage to the equipment.

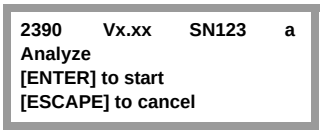
7. Press **Alt** + **4** to; the following prompt is displayed

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

8. Press **CHOICE** until Setup Group 1 (**N300-700** is Sample ID) is displayed. The parameters specified in this Setup Group are appropriate for the reference material you are analyzing.

9. Press **ENTER;** a series of prompts is displayed. Verify or enter information as requested at each prompt, pressing **ENTER** after each one to access the next one.

10. Press **ENTER** to start the analysis when the following prompt is displayed:



11. After the analysis is complete, the **Reload** prompt is displayed. Shown in the display window are 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 weight is correct. If the analysis results still fail to match, contact a Micromeritics Service representative.

Installing Software Updates

1. Place the On/Off switch on the rear panel in the Off (O) position.

2. Insert the USB media containing the software update into one of the USB connectors on the rear panel.

3. Place the On/Off switch in the On (|) position.

4. The software starts to load after initialization (approximately 1-2 minutes); the following prompt is displayed:

Updating Application
from USB Media

5. The following prompt displays after the software update is loaded:

Update Successful
Remove USB Media

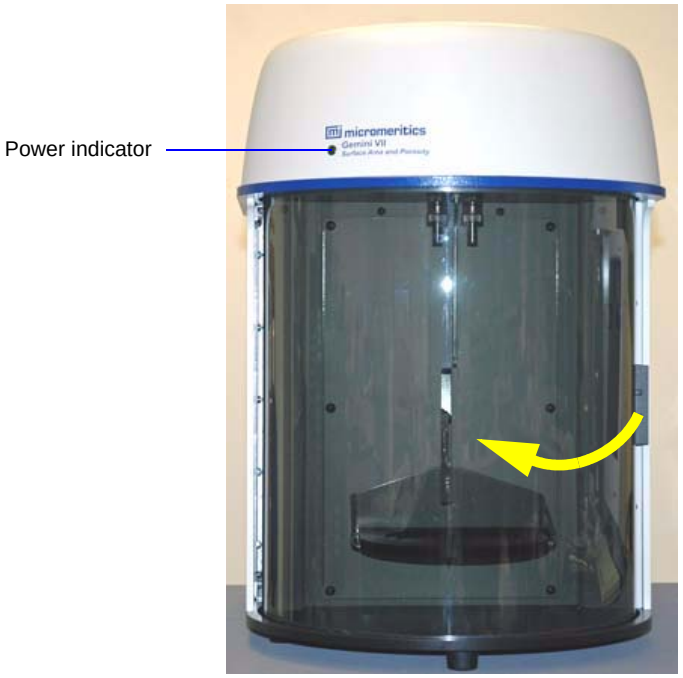
6. Remove the USB media and store in a secure location.

3. USER INTERFACE

This chapter contains information to familiarize you with the hardware and software components of the Gemini VII 2390. It is recommended that you read this chapter before attempting to operate the Gemini system.

Instrument Components and Connectors

Front Panel

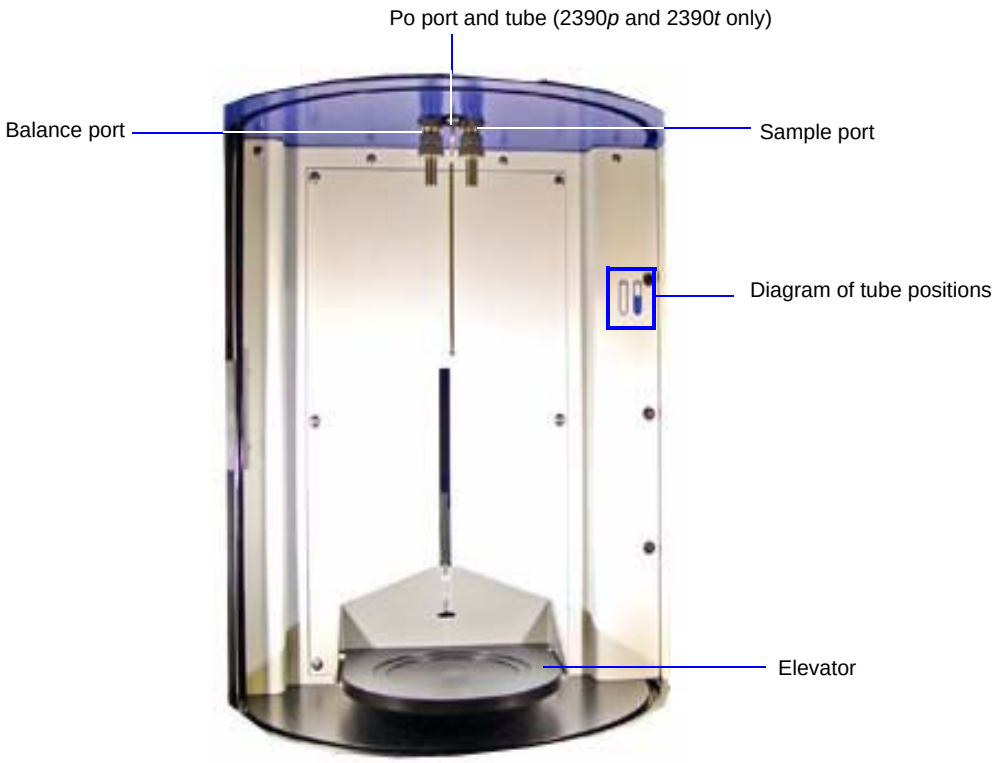


Power indicator — Blinks when power is applied to the analyzer; illuminates when analysis program is initiated and ready for operation.

Sample Compartment door — Sliding door which encloses the sample compartment.

This door should remain closed during analysis.

Sample Compartment



Sample port — For installing the sample tube.

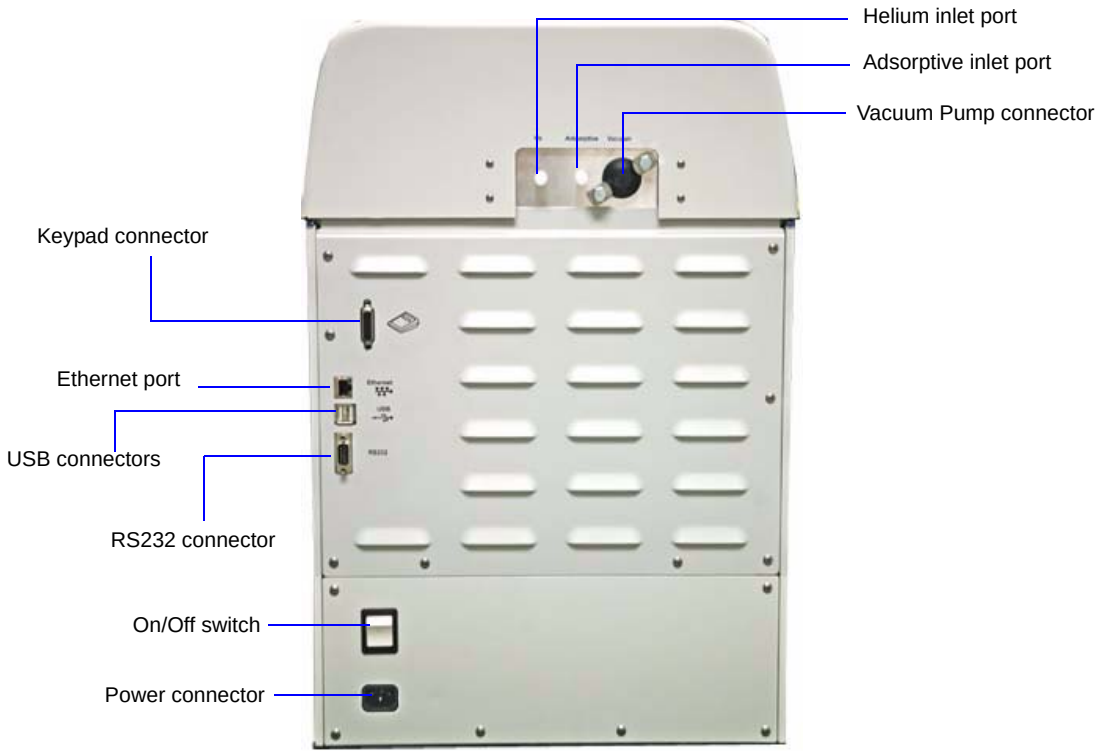
Balance port — For installing the balance tube.

Po port and tube — The Po (saturation pressure) tube comes installed on the Gemini II 2390*p* and 2390*t*.

Diagram — Provides a pictorial representation of the positions of the balance and sample tubes. The empty tube represents the balance tube and is installed on the left port; the filled tube represents the sample tube and is installed on the right port.

Elevator — Allows placement of the Dewar around the sample and Po tubes. The elevator raises automatically when the analysis is started and lowers automatically when the analysis is finished. During analysis, the elevator also lowers after the free-space measurement to allow evacuation, then raises and continues the analysis.

Rear Panel

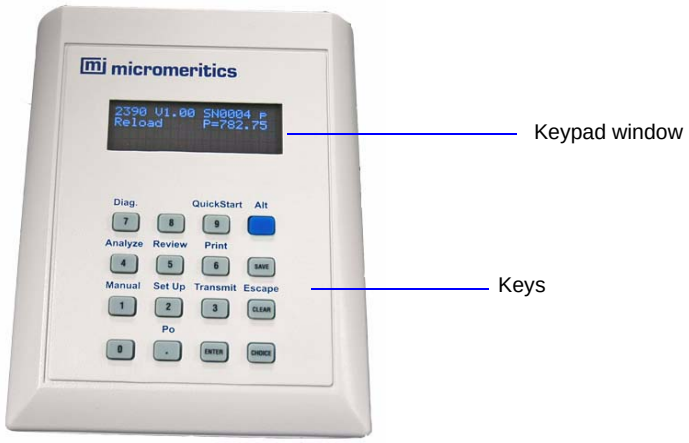


Adsorptive	For attaching the analysis gas supply.
Helium	For attaching the helium gas supply.
Vacuum Pump connector	For attaching the vacuum pump hose.
USB connectors	For attaching external devices, such as a printer, bar code reader, or keyboard. This connector is also used to install software upgrades.
Ethernet port	This port can be used to connect to a network enabling you to be notified via e-mail of analysis completion, and to monitor analysis results using a web browser. A straight-through or cross-over ethernet cable can be used since the analyzer will adapt to either type.

RS232 connector	Enables you to use a serial line for data transmission. This port can also be used to attach an analytical balance for automatic transmission of the sample mass. Refer to page **5-37** for details on transferring the mass value to the analyzer. See **RS-232 Operation**, page **F-1** for the pin assignment for the RS-232 port.
Keypad connector	For connecting the keypad.
On/Off switch	For turning the analyzer on and off.
Power connector	For connecting the analyzer to the power supply.

Keypad

Front Panel



Keypad window For viewing system operations, as well as data input.

Keys For entering and executing commands.

Rear Panel

26-pin connector For connecting the communications cable from the analyzer.

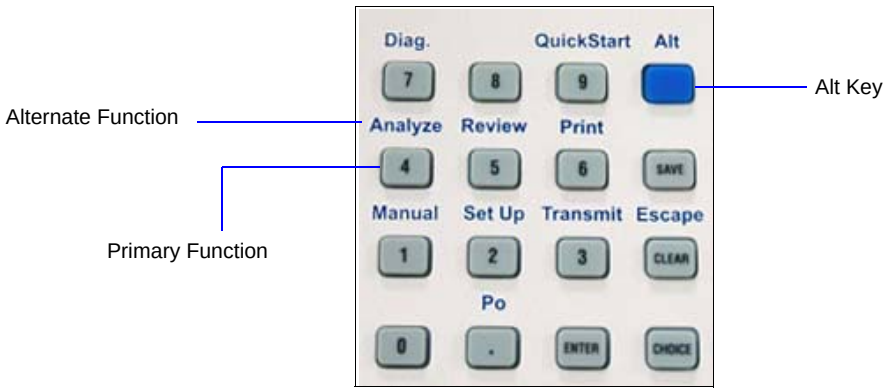
Contrast adjustment Enables you to adjust the contrast of the display window.

Insert a small-blade screwdriver into the opening. Rotate the screwdriver until you feel it engage in the slot; turn clockwise to darken and counterclockwise to lighten.

Using the Software

The keys and the window on the keypad are used in conjunction to enter and execute commands.

Keys



The keys on the keypad are used to execute commands and to enter information at prompts displayed in the software. 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. The alternate function is indicated by the command above the key.

For the:

- **primary** function, press only the key. For example, press **6** to enter the number **6** or **Save** to save an entry.
- **alternate** function, press the **Alt** key, then the key associated with the function. For example, press **Alt** + **4** to start an analysis..

Table 3-1 provides the functions for the keys on the keypad, as well as key combinations for entering such characters as a dash or slash. You may wish to print this page and keep it close to your Gemini for quick reference.

Refer to **COMMANDS**, page **5-1** for a description of each command on the keypad.

Table 3-1. Key Functions

Key(s)	Used To
0 through **9**	Enter the numbers 0 through 9.
. (decimal)	Enter a decimal point, a dash for sample or instrument ID, a slash for 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 the **Alt** key again to exit the alternate mode; the plus sign is removed from the display.
CHOICE	Display the next message when at the **Reload** prompt.
	Display the next multiple choice item when in a command mode.
CLEAR	Clear a message when at the **Reload** prompt.
	Clear an entry when in a command mode.
ENTER	Complete an entry or begin an action.
SAVE	Save the information you entered and return to the **Reload** prompt.
Alt + **1** **Manual**	Enables manual mode, allowing you to perform certain functions that may be requested by your service representative.
Alt + **2** **Set Up**	Edit, copy, or transmit Setup Groups.
Alt + **3** **Transmit**	Transmit analysis data over the serial line.
Alt + **4** **Analyze**	Perform an analysis.
Alt + 5 **Review**	Review or edit completed analysis data.
Alt + 6 **Print**	Print a report for the last analysis.
Alt + 7 **Diag.**	Perform diagnostic tests and view analyzer and calibration statistics.
Alt + 9 **QuickStart**	Begin a QuickStart analysis.
Alt + **.** (decimal) **Po**	Measure and record the saturation pressure when at the **Reload** prompt.
	Erase the previous keystroke when in a command mode.

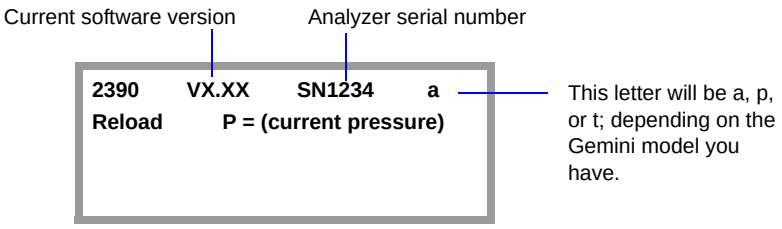
***Table 3-1. Key Functions** (continued)*

Key(s)	Used To
Alt + **CLEAR Escape**	Discard all data entered in the current mode and return to the **Reload** prompt.
	Cancel an automatic operation in progress.
	Exit manual mode.
Alt + **ENTER**	Save the current edit and return to the previous prompt.

Keypad Window

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

This is an example of the display window when the analyzer is in an idle state, referred to in this documentation as the **Reload** prompt.



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

Command Prompts

When you initiate a command, a prompt is displayed. There are three types of prompts used in the software:

- Multiple choice
- Data entry
- Action

Multiple Choice

A multiple choice prompt contains a fixed set of responses and is followed by a question mark (?).

This is an example of a multiple choice prompt:

```
2390        SN1234    10 cm3

Set Up type?
Analysis Conditions
```

The prompt shown here is displayed when you access Set Up. There are four Set Up parameters: **Analysis Conditions**, **Report Options**, **Communications**, and **System Options**. Press **CHOICE** until the desired parameter is displayed, then press **ENTER** to select it.

Data Entry

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

This is an example of a data entry prompt.

```
2390      Vx.xx      SN123      a
Analyze
Sample mass:
1.000 g
```

The prompt shown here displays when performing an analysis and requests a value for the sample mass. Use the keys on the keypad to enter the mass, then press **ENTER** to save the entry and advance to the next prompt.

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

Action

An action prompt simply instructs you to perform an action. This is an example of an action prompt.

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

The prompt shown here displays when you press **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.

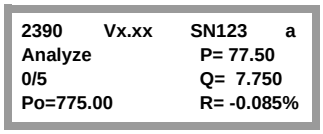
Messages

There are two types of messages that display in the software:

- Status
- Error

Status Messages

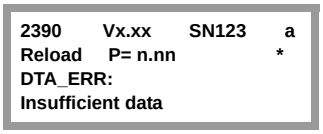
Status messages display when an operation is in progress. The following is an example of a status message.



The message shown here displays during an analysis. No action is required for a status message.

Error Messages

After an automatic operation is complete, the **Reload** prompt is displayed. If error messages occurred during the operation, they are placed in a queue along with report data and are indicated with an asterisk (*) on the second line. The first error message is shown in the third and fourth lines. The following is an example of an error message.



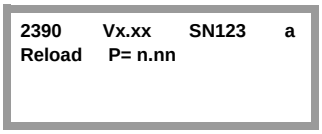
Press **CHOICE** to navigate through the messages. To delete a message, press **CLEAR** while the message is displayed. If you do not understand the message, refer to **Error Messages**, beginning on page **B-1** for the cause/action before deleting. Any messages that remain in the queue are deleted when the next operation is started.

Turning On and Off the Analyzer

To turn on the analyzer, place the power ON/OFF switch on the rear panel in the ON (|) position. The green indicator light on the front panel illuminates when the analyzer is on; the pump will turn on automatically if it is plugged into the electrical outlet.

Do not turn off the analyzer while initialization is in progress. Doing so may damage the instrument.

The **Reload** prompt is displayed in the keypad window when the system is fully initialized.



Allow approximately two hours for the pump to warm before performing analyses. For analyses that require very precise results, allow the analyzer to warm up while the sample is being prepared.

It is recommended that you leave the analyzer on at all times. If it does become necessary to turn it off, perform the following steps.

1. Allow any analyses in progress to complete.
2. Place the printer ON/OFF switch in the OFF position.
3. Place the analyzer ON/OFF switch in the OFF position

4. OPERATIONAL PROCEDURES

This chapter contains brief step-by-step instructions for operations with the Gemini analyzer. Chapter 5 contains details on the prompts used in these procedures. Use the index to assist you in locating the appropriate prompt.

Defining a Setup Group

A Setup Group can be defined by:

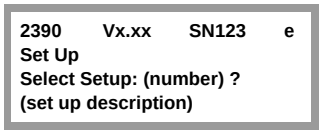
- editing an unused Setup group
- copying the values of a Setup group and editing them
- resetting a Setup group to factory-defined Setup group

Refer to **Set Up**, page **5-3** for details on the prompts for this procedure.

Editing a Setup Group

To edit a Setup Group:

1. Press **Alt** + **2** at the **Reload** prompt; the following prompt is displayed:



The number of the Setup Group last selected is displayed on the third line. The fourth line contains one of the following:

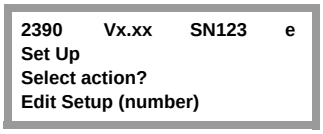
- The **ID** of the Setup Group if an ID was entered.
- (**used**) if the Setup Group has been defined, but no ID was entered.
- (**unused**) if the Setup Group has not been defined.
- (**factory defaults**) if the Setup Group is being used and has been reset to a default configuration.

2. Choose one of the following:

   - Enter the number of the desired Setup Group and press **ENTER**.
   - Press **CHOICE** until the number of the desired Setup Group is shown, then press **ENTER**.

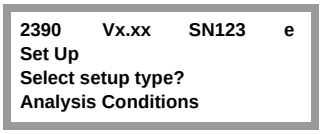
   The ID of the Setup Group is displayed if one has been entered. If not, either **Used** or **Unused** is displayed.

3. Press **ENTER**; the following prompt is displayed:



   This prompt enables you to choose the manner in which you wish to define your Setup Group. The choices are **Edit Setup** (n), **Copy another Setup**, **Reset** (n) **to** (n) defaults.

4. Press **CHOICE** until the **Edit** function displays, then press **ENTER**; the following prompt is displayed:



   This prompt enables you to select the type of Setup Group to edit. The choices are **Analysis Conditions**, **Report Options**, **Communications**, and **System Options**.

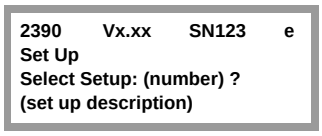
5. Press **CHOICE** until the desired parameter is displayed, then press **ENTER**.

6. Enter the information as prompted.

Copying a Setup Group

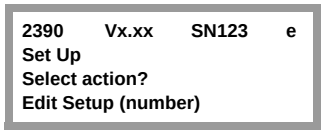
To copy the contents of a Setup Group:

1. Press **Alt** + **2** at the **Reload** prompt; the following prompt is displayed:

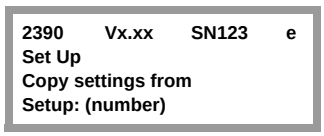


2. Enter the Setup number or press **CHOICE** until the desired Setup number is displayed (this is the Setup group you will be copying to).

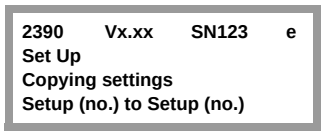
3. Press **ENTER**; the following prompt is displayed:



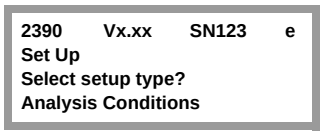
4. Press **CHOICE** until **Copy another setup** is displayed, then press **ENTER**; the following prompt is displayed:



5. Enter the number of the **copy from** Setup Group, then press **ENTER**; the following prompt is displayed:



6. When the copy is complete, the following prompt is displayed:



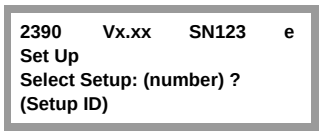
7. Choose one of the following:

   - Press **SAVE** to store the Setup Group and return to the **Reload** prompt.
   - Select a setup type to edit or view the settings.

Resetting a Setup Group to a Defined Setup Group

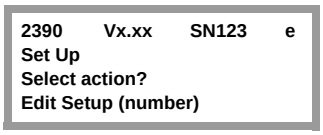
This procedure provides instructions for resetting a Setup group to a defined Setup group. This defined group may be one that you have defined or one that was included with the software (refer to Tables 5-1 through 5-4 beginning on page 5-4 for factory-defined groups).

1. Press **Alt** + **2** at the **Reload** prompt; the following prompt is displayed:



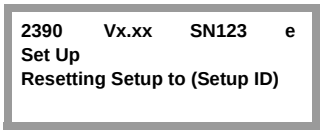
2. Enter the Setup number you wish to reset to a defined set, or press **CHOICE** until the desired Setup number is displayed.

3. Press **ENTER**; the following prompt is displayed.

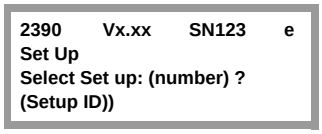


4. Press **CHOICE** until **Reset to (Setup ID)** is displayed.

5. Press **ENTER** to accept this defined set or press **CHOICE** until the desired set is displayed, then press **ENTER**; the following prompt is displayed:



6. When the reset is complete, the following prompt is displayed:

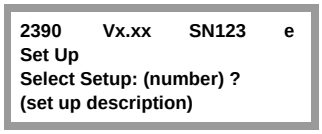


7. Choose one of the following:

   - Press **SAVE** to store the Setup Group and return to the **Reload** prompt.
   - Select a setup type to edit or view the settings.

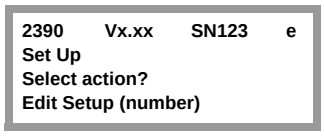
Viewing the Settings in a Setup Group

1. Press **Alt** + **2** at the **Reload** prompt; the following prompt is displayed:

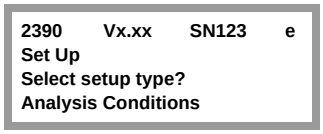


2. Enter or select the Setup Group you wish to view.

3. Press **ENTER**; the following prompt is displayed:



4. Press **ENTER**; the following prompt is displayed:

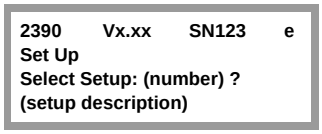


5. Press **CHOICE** to select the setup type you wish to view, then press **ENTER**.

6. Press **ENTER** at each prompt to browse through the settings. When you return to the **Select setup type?** prompt, you may select another setup type or press **Alt + CLEAR** to return to the **Reload** prompt.

Printing or Transmitting a Setup Group

1. Press **Alt** + **2** at the **Reload** prompt; the following prompt is displayed:



2. Enter or select the Setup Group you wish to print (or transmit).

3. Press:

   - **Alt** + **6** to print the Setup Group
   - **Alt** + **3** to transmit the Setup Group

A Setup Group cannot be printed or transmitted while it is being edited.

Preparing for Analysis

The table shown here outlines the tasks necessary to properly prepare for an analysis, as well as the location of the procedure for performing the task. It is best to perform the tasks in the order given in the table.

Task	Name and Location
Verify regulator pressure	**Verifying Regulator Pressure**, this page
Verify vacuum level	**Verifying Vacuum Level**, this page
Clean the sample and balance tubes	**Cleaning and Labeling Sample and Balance Tubes**, page **4-8**
Specify setup parameters	**Editing a Setup Group**, page **4-1**
Weigh your sample	**Determining the Sample Mass**, page **4-11**
Degas your sample	**Degassing the Sample**, page **4-13**
Load sample on sample port	**Installing the Sample Tube**, page **4-14**
Fill Dewar	**Preparing the Analysis Dewar**, page **4-15**

Verifying Regulator Pressure

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

Check the gas tank 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.

Verifying Vacuum

The vacuum level must be better than 20 x 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 x 10^{-3} mmHg.

Cleaning and Labeling Sample and Balance Tubes

After you have cleaned a balance tube and attached it to the analyzer, you may leave it in place for repeated analyses. You need to change the balance tube only if it becomes contaminated or if you change the size of the sample tube being used. The balance tube size must always match the sample tube size.

Table 4-1. Materials Required to Clean and Weigh Sample and Balance Tubes

Supplied by Micromeritics	Supplied by User
Sample tube	Drying oven
Filler rod (if used)	Ultrasonic cleaning unit
Sample tube brush	Detergent
Stopper for sample tube	Rubber gloves or lint-free cloth
Sample tube rack	Acetone or isopropyl alcohol
Sample weighing support	Safety glasses
	Waste container
	Analytical balance

Clean the tubes and filler rods as follows.

1. Turn on the drying oven used for heating the sample tubes and filler rods and set the temperature to 110 ºC.

2. Check the bowl of the ultrasonic cleaning unit to make sure it is clean.

3. Using 5 grams of Alconox® (or other suitable detergent) per 500 mL of warm water, fill the reservoir of the ultrasonic unit with enough water to cover the sample tubes and filler rods. Make sure the detergent is dissolved before placing the sample tubes and filler rods into the water. If too much detergent is used, it may be difficult to rinse from the sample tubes.

4. Fill the tubes with warm water and place them in the reservoir of the ultrasonic cleaning unit. Place the filler rods in the bowl also. Turn on the ultrasonic cleaning unit for approximately 15 minutes.

5. Using rubber gloves, remove the sample tubes and filler rods from the reservoir.

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

7. Rinse the sample tubes and filler rods thoroughly with hot water, then with isopropyl alcohol or acetone.

If isopropyl alcohol or acetone is not available, deionized water may be used to rinse the sample tubes.

8. Stand the sample tubes on the sample tube rack and place the filler rods in a basket or in the rack. Place the tubes and filler rods in the preheated drying oven (set in Step 1) for two hours.

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

10. Wipe a rubber stopper with a lint-free cloth. Label the tube and stopper for identification.

11. Fill the tube with helium or nitrogen, then install the stopper. This must be done quickly to prevent air, dust, or moisture from entering the tube.

To obtain an accurate weight of a degassed sample, the same gas type must be present in the sample tube during both weighings; that is, when weighed without and with sample present. Buoyancy differences can cause significant errors when helium is used inconsistently.

Determining the Sample Mass

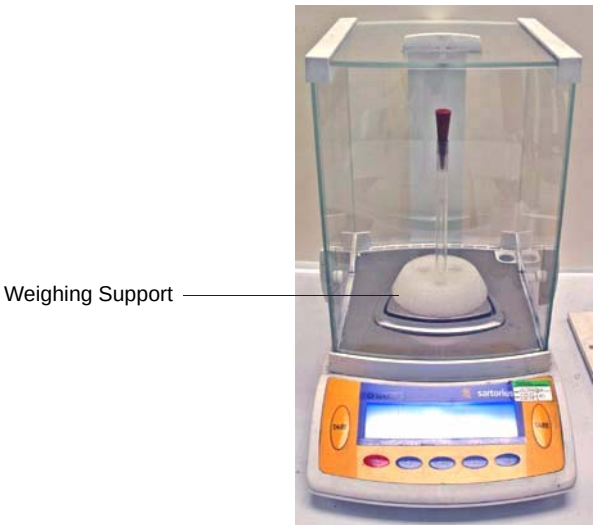
Analysis results are expressed in units of surface area per gram of sample; therefore, it is important the true sample mass be known. The true mass is best calculated as follows:

- Weigh the Sample Tube Set (sample tube and stopper) *before degas*
- Weigh the Sample Tube Set with sample *before degas*; subtract the weight of the Sample Tube Set
- Weigh the Sample Tube Set with sample *after degas*; subtract the weight of the Sample Tube Set (this is the value you should enter at the **Sample mass** prompt)
- Weigh the Sample Tube Set with sample *after analysis*; subtract the weight of the Sample Tube Set

For your convenience, a **Sample Data Worksheet** for recording the weights and calculating the mass is included in Appendix A. You may make copies as needed.

Make a copy of the Sample Data Worksheet, then proceed with the instructions provided below.

1. Write 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 sample tube with stopper onto the sample weighing support.



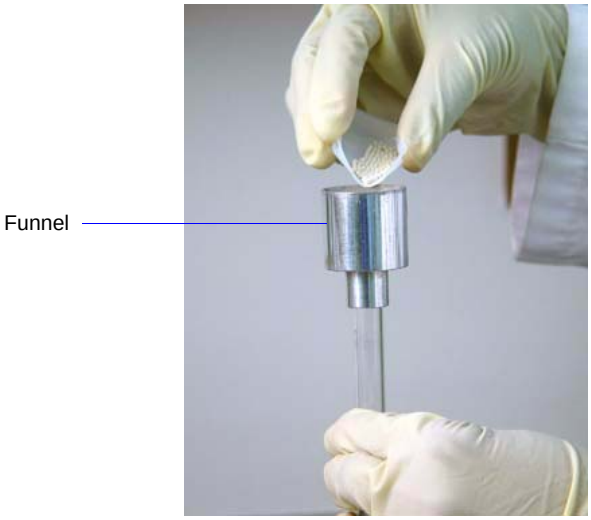
4. Record the stabilized weight on the Sample Data Worksheet as *Mass for empty sample tube set*. Remove the sample weighing support and sample tube set from the balance.
5. Place a sample container on the balance; allow the balance to stabilize.

6. Slowly add the sample to the weighing container.

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

7. Remove the rubber stopper from the sample tube.

8. Using the sample tube funnel (provided in the accessories kit), pour the sample from the weighing container into the sample tube.



If your sample has a total surface area of 1.0 m^2 or less, filler rods should be used in the balance and sample tubes. Refer to Appendix H, page H-1 for instructions.

9. Replace the rubber stopper.

10. Weigh the sample tube set containing the sample; record the value on the Sample Data Worksheet as *Sample tube set plus sample mass (Before Degas)*.

11. Subtract the *Mass for empty sample tube set* from the *Mass of sample tube set plus sample*; record this value as the *Sample mass (Before Degas)*.

Degassing the Sample

After the sample has been weighed, use a degassing unit to remove any contaminants which may have adsorbed to the surface or pores of your sample. Appropriate degassing units are available from Micromeritics. Refer to **Ordering Information**, beginning on page **7-1** for ordering information.

After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample; record the weight on the Sample Data Worksheet as *Sample tube set plus sample mass (After Degas)*.

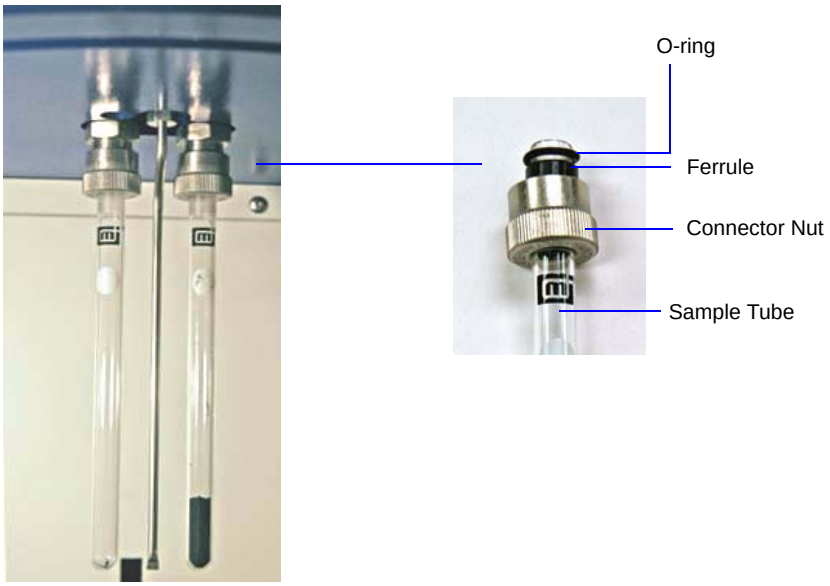
2. Subtract the *Mass for empty sample tube set (Before Degas)* from the *Sample tube set plus sample mass (After Degas)* to obtain the sample's mass; record this value as *Sample mass (After Degas)*.

Installing the Sample Tube

Follow the steps below to attach the sample tube to the analysis port and the balance tube to the balance port. Minimize sample exposure to air by completing the steps rapidly.

1. Remove the sample tube stopper.

2. Place the connector nut, ferrule, and O-ring onto the sample tube stem.



The sample tube ferrule is tapered slightly on one end. The ferrule may be installed onto the sample tube with the tapered end in the up or down position.

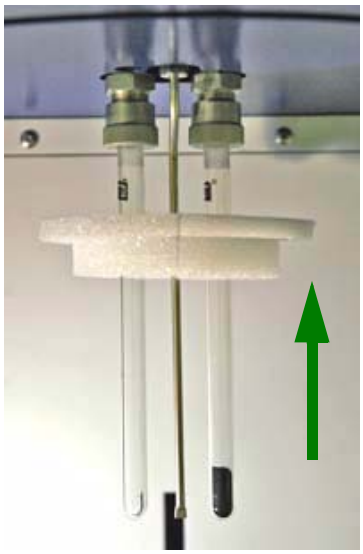
If you are analyzing samples with a surface area of 1.0 m^2 or less, you should use filler rods in the sample and balance tubes. Refer to Appendix H, page H-1 for additional information and instructions.

3. Attach the sample tube to the analysis port. Make sure it is fully in the port. Secure it in place by screwing the connector nut onto the analysis port. Hand-tighten the connector nut.

4. If the balance tube is not installed, attach it to the balance port following procedures similar to those described above. If you used a filler rod in the sample tube, you must use one in the balance tube.

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

5. Place the Dewar cover under the sample and balance tubes and slide it toward the top of the tube.



Preparing the Analysis Dewar

Always handle Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. Always observe the precautions listed below.

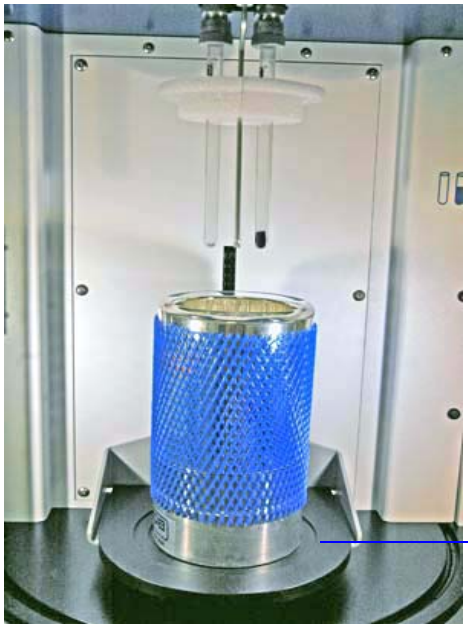
Observe the following precautions when handling Dewars containing liquefied gases:

- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.
- When pouring liquefied gases from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the liquefied gas slowly to prevent splashing, and 3) vent the receiving container to the atmosphere.
- Use a plastic stirring rod when stirring substances in a glass Dewar containing liquefied gases (or other materials suitable for extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some type of soft protective coating.
- Do not remove the mesh covering from a glass Dewar flask. This covering enables you to maintain a more secure grip on the Dewar.
- Do not handle heavy objects above a glass Dewar. If unavoidable, place a protective cover over the Dewar's opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

Prepare the Dewar as follows:

1. Fill the Dewar with the analysis bath liquid to approximately 2 cm (3/4 in.) from the top for the small Dewar and 5 cm (2 in.) for the large Dewar.

2. 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.

3. Close the sample compartment door.

4. Allow approximately 30 minutes (for best results) for the temperature of the Dewar to stabilize with that of the bath liquid.

Be sure to close the door to the sample station before beginning an analysis. If the analyzer is operated at an excessive pressure, the sample or balance tube could become dislodged from its port, possibly causing personal injury or damage to the equipment.

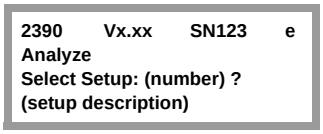
Starting the Analysis

The following steps describe how to start an analysis responding to the prompts that appear if all of the System Options prompts have been selected to display. If you want to disable any of the prompts so they do not appear before every analysis, refer to **System Options**, page **5-33**.

Refer to **Analyze**, page **5-37** for details on the prompts used for this procedure.

To start an analysis using the default analysis parameters:

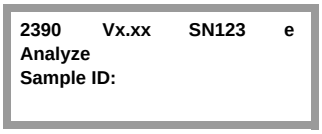
1. Press **Alt** + **4**; the following prompt is displayed:



   The number of the Setup Group used for the previous analysis is displayed.

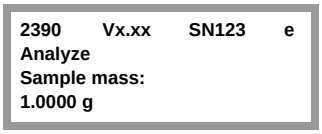
2. Enter the number of the Setup Group you wish to use for the current analysis, or press **CHOICE** until it is displayed.

3. Press **ENTER**; the following prompt is displayed:



4. Enter an ID (you can use up to 20 numbers and dashes). Using sample IDs can help you keep track of data from various analyses.

5. Press **ENTER**; the following prompt is displayed.



   Enter the sample mass; the range of valid entries is 0.0010 to 999.9990 g.

If you requested the sample mass to be calculated using the empty tube mass and the tube plus sample, two prompts allowing you to enter these two values are displayed.

6. Press **ENTER**; the following prompt is displayed if you selected **Entered** for **Saturation Pressure** in System Options.

```
2390    Vx.xx    SN123    e
Analyze
Saturation pressure:
760 mmHg
```

Enter the saturation pressure; the range is 500.00 to 900.00 mmHg.

You do not see the prompt in this step if you selected *During analysis* or *Previously measured* for Saturation Pressure; the prompt in the next step is displayed.

7. Press **ENTER**; the following prompt is displayed.

```
2390    Vx.xx    SN123    e
Analyze
Bath temperature:
```

Enter the temperature of the bath liquid; the range is 0 to 999.999 K.

8. Press **ENTER**; the following prompt is displayed.

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

Enter the evacuation rate; the range is 1.0 to 1000.0 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.

9. Press **ENTER**; the following prompt is displayed.

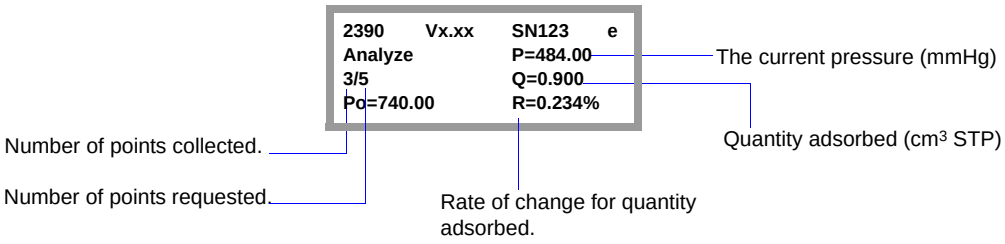
```
2390    Vx.xx    SN123    e
Analyze
[ENTER] to Start
[ESCAPE] to Cancel
```

- Press **ENTER** to start the analysis
- Press **Alt + CLEAR** to cancel and return to the **Reload** prompt

Viewing, Printing, or Transmitting Analysis Results

Viewing Analysis Results

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



During analysis, the quantity (Q) value equals the quantity adsorbed by the sample in cm^3 STP. At all other times, the Q value equals the reading of the differential transducer in cm^3 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, you can press **CHOICE** to view the results. A few examples of the display report formats are shown below:

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

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

```
2390    Vx.xx    SN123    e
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/Po) greater than or equal to 0.5000. Refer to Appendix C, page **C-18** for the formula used to calculate total pore volume.

Press **CHOICE** to cycle through any additional report calculations and error messages. When the **Reload** prompt is displayed, you may begin another operation.

Printing Analysis Results

A report is automatically printed if you selected **Printer** as a Report destination in Report Options (refer to **Report Options**, page **5-16**). If not, you may print a report by pressing **Alt** + **6** during or after an analysis.

Transmitting Analysis Results

You can transmit a report through the RS-232 serial port by pressing **Alt** + **3** during or after an analysis. You can also select **Transmission line** as a Report destination in Report Options (refer to **Report Options**, page **5-16**) if you wish to have results transmitted automatically after the analysis.

E-mailing Analysis Results

Analysis results can be e-mailed automatically at the completion of an analysis when the Gemini is connected to a Network. Refer to **Report Options**, page **5-16** for information on e-mailing results.

Measuring the Saturation Pressure

The saturation pressure can be measured in the:

- sample tube (all Gemini II 2390 models)
- Po tube (Gemini II 2390*p* and 2390*t* only)

Perform the following steps to measure the saturation pressure (Po):

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

You do not have to install sample tubes on the ports if you are measuring the saturation pressure in the Po tube, but they should be plugged if tubes are not installed.

2. Add liquid nitrogen (LN_2) 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 you are adding LN_2 to an empty Dewar, allow the temperature of the LN_2 to stabilize (approximately 30 minutes).

5. Press **Alt** + **.** (decimal); the following prompt is displayed:

```
2390    Vx.xx    SN123    p
Po
Measure Po in?
Sample Tube
```

6. Press **CHOICE** to choose **Sample Tube** or **Po Tube**, then press **ENTER**; the following prompt is displayed:

```
2390    Vx.xx    SN123    e
Po
[ENTER] to Start
[ESCAPE] to Cancel
```

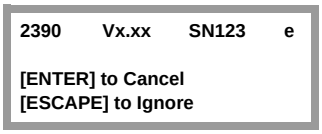
7. Press **ENTER** to begin the measurement. You may press **Alt** + **CLEAR** at any time to cancel the measurement.

   When the measurement is complete, the **Reload** prompt is displayed. The saturation pressure is stored in memory and used in report calculations.

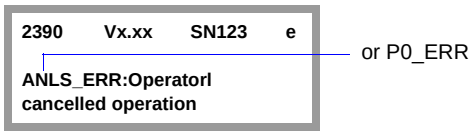
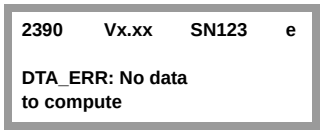
Canceling an Automatic Operation

Perform the following steps to cancel an analysis or a Po measurement while in progress.

1. Press **Alt** + **CLEAR**; the following prompt is displayed:



2. Press:

ENTER	To cancel the operation and return to the **Reload** prompt. Messages are displayed indicating that termination is in progress, then the following prompt is displayed:  Press **CLEAR** to remove the message from the keypad window.
	If an analysis is being cancelled and enough data have been collected for calculation, results are displayed. Press **CHOICE** to navigate through the results. If there were not enough data collected, the following prompt is displayed:  Press **CLEAR** to remove the message from the keypad window.
Alt + **CLEAR**	To ignore your request for cancellation and resume the operation.

Using a Web Browser

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, you must be connected to a network via an ethernet cable and have a keyboard attached to a USB port.

These steps assume an IP address has already been assigned. If an address has not been assigned, refer to Communications, page 5-29 for information on obtaining one.

1. Make a note of the IP address:

   a. Press **Alt + 2** to access **Set Up**.

   b. Press **CHOICE** until **Communications** is displayed, then press **ENTER**. The following prompt is displayed:

   ```
   2390    Vx.xx    SN123    e
   Communications
   IP Address?
   DHCP
   ```

   The fourth line may show **Specify**, depending on the manner in which the IP address was assigned (refer to **Communications**, page **5-29** for additional information on this prompt).

   c. Press **ENTER** to view the assigned address:

   ```
   2390    Vx.xx    SN123    e
   Communications
   Assigned Address:
   (assigned address)
   ```

   If the address was specified, the third line of the display shows **IP Address?** and the fourth line shows the entered address. This line is editable since it is specified and not assigned automatically. An assigned address cannot be edited.

   d. Press **Alt + CLEAR** to return to the **Reload** prompt.

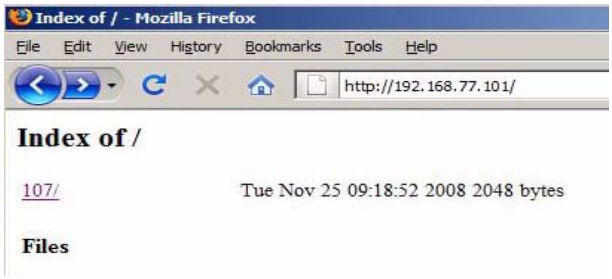
2. Access your web browser.

3. Enter your IP address in the **Address** field of the web browser.

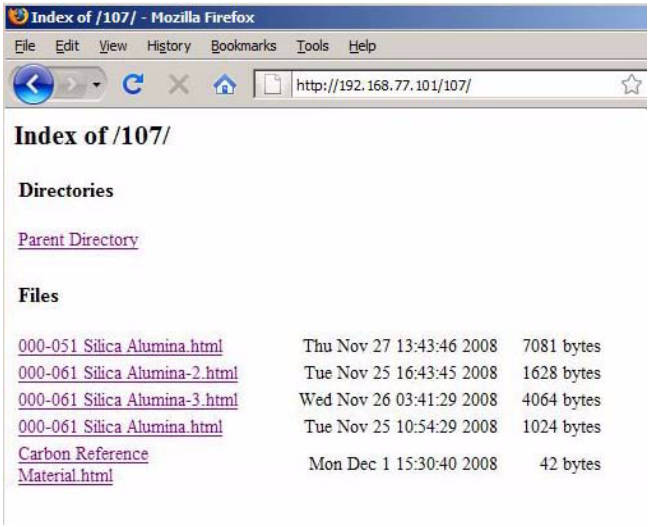
   

   This will consist of 12 numbers in the following format: **nnn.nnn.nnn.nnn**

4. Press **Enter** on your computer keyboard; a window containing the serial number of the attached unit is displayed.

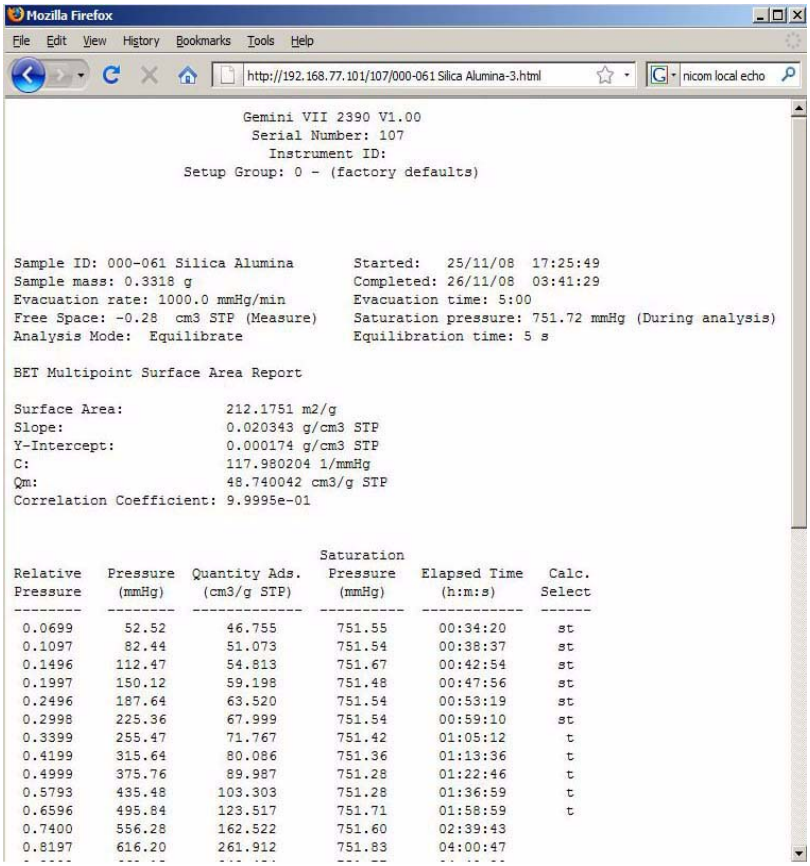


5. Click on the serial number; a window containing the data files for the last five analyses is displayed.



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

6. Click on the desired file to view the data results.



Gemini VII 2390 V1.00
Serial Number: 107
Instrument ID:
Setup Group: 0 - (factory defaults)

Sample ID: 000-061 Silica Alumina
Sample mass: 0.3318 g
Evacuation rate: 1000.0 mmHg/min
Free Space: -0.28 cm3 STP (Measure)
Analysis Mode: Equilibrate

Started: 25/11/08 17:25:49
Completed: 26/11/08 03:41:29
Evacuation time: 5:00
Saturation pressure: 751.72 mmHg (During analysis)
Equilibration time: 5 s

BET Multipoint Surface Area Report

Surface Area:	212.1751 m2/g
Slope:	0.020343 g/cm3 STP
Y-Intercept:	0.000174 g/cm3 STP
C:	117.980204 1/mmHg
Qm:	48.740042 cm3/g STP
Correlation Coefficient:	9.9995e-01

Relative Pressure	Pressure (mmHg)	Quantity Ads. (cm3/g STP)	Saturation Pressure (mmHg)	Elapsed Time (h:m:s)	Calc. Select
0.0699	52.52	46.755	751.55	00:34:20	st
0.1097	82.44	51.073	751.54	00:38:37	st
0.1496	112.47	54.813	751.67	00:42:54	st
0.1997	150.12	59.198	751.48	00:47:56	st
0.2496	187.64	63.520	751.54	00:53:19	st
0.2998	225.36	67.999	751.54	00:59:10	st
0.3399	255.47	71.767	751.42	01:05:12	t
0.4199	315.64	80.086	751.36	01:13:36	t
0.4999	375.76	89.987	751.28	01:22:46	t
0.5793	435.48	103.303	751.28	01:36:59	t
0.6596	495.84	123.517	751.71	01:58:59	t
0.7400	556.28	162.522	751.60	02:39:43	
0.8197	616.20	261.912	751.83	04:00:47	

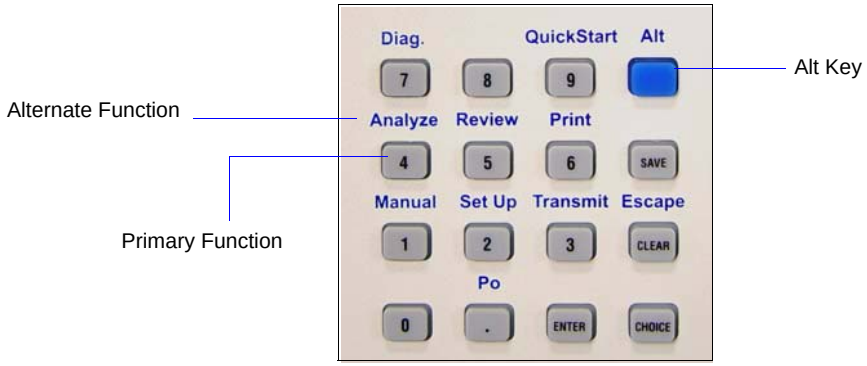
You can also click on the file for a current operation to monitor its progress.

7. Use the **File > Print** option if you wish to print a copy of the results.

5. COMMANDS

Analyzer operations are performed using the alternate commands on the keypad. This chapter contains a description of the prompts associated with each command.

Description



Diag.	**Alt + 7**	Enables you to perform user diagnostic tests. Page **5-44**.
QuickStart	**Alt + 9**	Enables you to begin a series of analyses using the same Setup conditions. Page **5-39**.
Analyze	**Alt + 4**	Enables you to perform an analysis. Page **5-37**.
Review	**Alt + 5**	Enables you to review and/or edit analysis data. Page **5-40**.
Print	**Alt + 6**	Enables you to print reports. Page **5-43**.
Manual	**Alt + 1**	Enables you to manually control valves. Page **5-51**.
Set Up	**Alt + 2**	Enables you to specify analysis conditions, report options, and system defaults. Page **5-3**.

Transmit	**Alt + 3**	Enables you to transmit data. Page **5-43**.
Po	**Alt + .**	Enables you to measure the saturation pressure. Page **5-42**.

Set Up

The Set Up function enables you to:

- edit a Setup group
- copy the values of one Setup group to another one, then modify the values
- reset the values of a Setup group to a default set

A Setup group is a user-defined set of conditions used by the Gemini to perform analyses, report data, and transmit data. It also contains system options such as instrument ID. Setup groups contain the condition sets shown below.

- **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 Setup groups (sample ID, sample mass, etc.)

You may define up to 10 Setup groups. Each group has a number, 0 to 9, associated with it. When you perform an analysis, you are prompted to enter the number of the Setup group to be used for the analysis.

The Gemini is shipped with Setup groups 0 and 4 thru 9 set to factory default values (See Table 5-1). Setup group 0 has been predefined with a sample identification of (factory defaults); 4 thru 9 are unused. For your convenience, Setup groups 1 through 3 also have been predefined (Tables 5-2, 5-3, and 5-4). The values in all Setup groups can be edited, even those that have been predefined. An option is provided in the software allowing you to reset any Setup group to factory defaults.

Table 5-1. Setup Group 0 Default Values, Factory Defaults

Parameter	Field	Default
Analysis Conditions	Evacuation time Free space Sample density Pressure table First rel. pressure Last rel. pressure Number of points Adsorb pressure 1 Adsorb pressure 2 Adsorb pressure 3 Adsorb pressure 4 Adsorb pressure 5 Analysis mode Equilibration time Scan rate	1:00 None 1.00 g/cm3 Replace 0.1000 P/Po 0.3000 P/Po 5 0.1000 P/Po 0.1500 P/Po 0.2000 P/Po 0.2500 P/Po 0.3000 P/Po Equilibrate 5 s 10 min/analysis
Report Options	Print Report Transmit Report E-mail Report Report BET multi-pt Minimum area Maximum area Report Langmuir Report BET 1-point Report t-method Report BJH Report H-K Molecular area Density conversion Nonideality	No No No Yes 0.0000 m2/g 1.0000 m2/g No No No No No 0.162 nm2 0.0015468 5.0 %/atm
System Options	ID for Setup 1 Request sample ID Request sample mass Request Sat. Press. Request Evac. Rate	(factory defaults) Yes Yes During analysis Yes

Table 5-2. Setup Group 1 Default Values, N300-700

Parameter	Field	Default
Analysis Conditions	Evacuation time Free space Sample density Pressure table First rel. pressure Last rel. pressure Number of points Adsorb pressure 1 Adsorb pressure 2 Adsorb pressure 3 Adsorb pressure 4 Adsorb pressure 5 Analysis mode Equilibration time Scan rate	1:00 Calculate 1.90 g/cm3 Replace 0.1000 P/Po 0.5000 P/Po 5 0.1000 P/Po 0.2000 P/Po 0.3000 P/Po 0.4000 P/Po 0.5000 P/Po Equilibrate 2 s 10 min/analysis
Report Options	Print Report Transmit Report E-mail Report Report BET multi-pt Minimum area Maximum area Report Langmuir Report BET 1-point Report t-method t-method range from t method range to Thickness curve M-STSA parameter 1 M-STSA parameter 2 M-STSA parameter 3 Minimum thickness Maximum thickness Area correction Ads. pore volume Report BJH Report H-K Molecular area Density conversion Nonideality	No No No Yes 0.0000 m2/g 1.0000 m2/g No No Yes 0.1900 P/Po 0.5100 P/Po Magee-STSA 13.9900 0.0340 0.5000 4.200 A 6.500 A 1.000 No No No 0.162 nm2 0.0015468 5.0 %/atm
System Options	ID for Setup 1 Request sample ID Request sample mass Request Sat. Press. Request Evac. Rate	N300-700 Yes Yes During analysis Yes

Table 5-3. Setup Group 2 Default Values, N100-200

Parameter	Field	Default
Analysis Conditions	Evacuation time	1:00
	Free space	Calculate
	Sample density	1.900 g/cm3
	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	Print Report	No
	Transmit Report	No
	E-mail Report	No
	Report BET multi-pt	Yes
	Minimum area	0.0000 m2/g
	Maximum area	1.0000 m2/g
	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 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 nm2
	Density conversion	0.0015468
	Nonideality	5.0 %/atm

Table 5-3. Setup Group 2 Default Values, N100-200 (continued)

Parameter	Field	Default
System Options	ID for Setup 0 Request sample ID Request sample mass Request Sat. Press. Request Evac. Rate	N100-200 Yes Yes During analysis Yes

Table 5-4. Setup Group 3 Default Values, Over 130

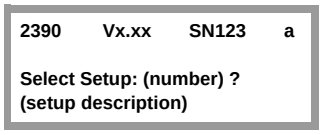
Parameter	Field	Default
Analysis Conditions	Evacuation time Free space Sample density Pressure table First rel. pressure Last rel. pressure Number of points Adsorb pressure 1 Adsorb pressure 2 Adsorb pressure 3 Adsorb pressure 4 Adsorb pressure 5 Adsorb pressure 6 Adsorb pressure 7 Analysis mode Equilibration time Scan time	1:00 Calculate 1.90 g/cm3 Replace 0.1000 P/Po 0.5000 P/Po 7 0.0500 P/Po 0.0750 P/Po 0.1000 P/Po 0.2000 P/Po 0.3000 P/Po 0.4000 P/Po 0.5000 P/Po Equilibrate 2 s 10 min/analysis
Report Options	Print Report Transmit Report E-mail Report Area points from Area points to Report BET multi-pt Minimum area Maximum area Report Langmuir Report BET 1-point Report t-method t-method range from t method range to	No No No 0.0400 P/Po 0.1100 P/Po Yes 0.0000 m2/g 1.0000 m2/g No No Yes 0.1900 P/Po 0.5100 P/Po

Table 5-4. Setup Group 3 Default Values, Over 130 *(continued)*

Parameter	Field	Default
Report Options *(continued)*	Thickness curve M-STSA parameter 1: M-STSA parameter 2: M-STSA parameter 3: Minimum thickness Maximum thickness Area correction Ads. pore volume Report BJH Report H-K Molecular area Density conversion Nonideality	Magee-STSA 13.9900 0.0340 0.5000 4.200 A 6.500 A 1.000 No No No 0.162 nm2 0.0015468 5.0 %/atm
System Options	ID for Setup 0 Request sample ID Request sample mass Saturation Pressure	Over 130 Yes Yes During analysis

Press **Alt** + **2** on the keypad to access the **Set Up** function; the following prompt is displayed.

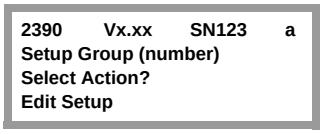
Setup Groups **may be selected by pressing CHOICE until the desired number is displayed or by entering the group number. Therefore, you will see a colon and a question mark when you are prompted to enter the Set Up Group number at the** ***Select Setup*** **and** ***Use Setup*** **prompts.**



The third line displays the number of the Setup group last selected. The fourth line displays one of the following:

- identification of the Setup group (if one was entered)
- **used** if the Setup group has been defined, but an identification was not entered
- **unused** if the Setup group has not been defined
- **factory default** if the Setup group is being used and has been reset to the default

Enter the number of the desired Setup group you wish to edit (alternatively, press **CHOICE** until the desired Setup group is displayed), then press **ENTER**.



This prompt enables you to choose the desired function.

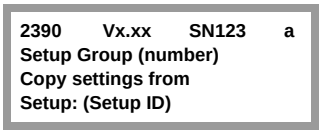
Choices: Edit Setup, Copy another setup, Reset to default, Reset to N300-700 Reset to N100-200, Reset to Over 130

Edit Setup

This option enables you to edit the selected Setup Group; press **ENTER** to display the **Setup type?** prompt.

Copy another setup

Allows you to copy the settings from another Setup group to the current one. Press **ENTER**; the following prompt is displayed:



Enter the number of the Setup group from which to copy the values, then press **ENTER** to display the **Setup type?** prompt.

Reset to default
Reset to (Setup ID)

Allows you to reset the current Setup group to the chosen default number (see Tables 5-1 through 5-4 for details); the following prompt is displayed:

```
2390    Vx.xx    SN123    a
Setup Group (number)
Resetting Setup (number)
to (Setup ID)
```

When resetting a Setup group, only group-specific parameters are reset. (Refer to **Communications**, page **5-29** and **System Options**, page **5-33** for parameters not specific to groups.)

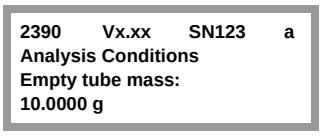
```
2390    Vx.xx    SN123    a
Setup Group (number)
Setup Type?
Analysis Conditions
```

Press **CHOICE** to display the parameter of the Setup group you wish to edit.

Choices: *Analysis Conditions, Report Options, Communications, System Options*

Analysis Conditions

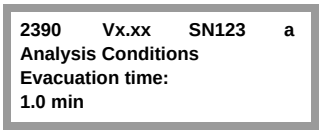
The following prompts are displayed for **Analysis Conditions**:



Displays if you selected **Tube and tube+sample** for the sample mass when specifying System Options (see page **5-34**).

Range: 0.0000 to 999.9999 g

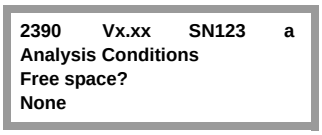
Enter the mass of the empty sample tube, then press **ENTER**.



Enter the evacuation time and press **ENTER**.

Range: 0.1 to 999.9 min

This is the length of time that the sample will be evacuated after reaching an evacuation rate of less than 0.1 mmHg per 30-second interval.



Select the method for handling free-space measurement and press **ENTER**.

Choices: None, Previous, Measure, Calculate

Typically, materials having surface areas greater than 25 to 50 m^2/g can be analyzed accurately without free-space measurement.

Previous

Uses the free space applied to the previous sample.

Measure

This option provides a more accurate, but slower, analysis. If you are analyzing a series of samples with similar volumes, you may want to measure the first sample, then select **Previous** for the remaining samples.

Calculate

This method of free space provides high accuracy without the additional time required for free-space measurement. This method also includes a balance volume correction (entered in **System Options**) and a nonideality correction (entered in **Report Options**).

The free-space correction is calculated by dividing the sample weight by the sample density to get the sample volume. When you select **Calculate**, the following prompt allowing you to enter the sample density is displayed:

Calculate

(continued)

```
2390    Vx.xx    SN123    a
Analysis Conditions
Sample density:
1.000 g/cm3
```

Enter the density of the sample to be analyzed and press **ENTER**. (Pycnometers are available from Micromeritics for automatic density measurement. Refer to Ordering Information, page 7-1 for ordering information.)

Range: *0.010 to 99.990 g/cm^3*

An approximate value is adequate to provide an accurate free-space correction.

```
2390    Vx.xx    SN123    a
Analysis Conditions
Pressure table?
Replace
```

Select the means for changing or preserving the currently stored target relative pressure table and press **ENTER**.

Choices: *Replace, Preserve, Edit*

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; the following prompts display:

```
2390    Vx.xx    SN123    a
Analysis Conditions
First rel. pressure:
0.1000 P/Po
```

Enter the first relative pressure for data collection and press **ENTER**.

Range: *0.0001 to 1.0000 P/Po*

```
2390    Vx.xx    SN123    a
Analysis Conditions
Last rel. pressure:
0.3000 P/Po
```

Enter the last relative pressure for data collection and press **ENTER**.

Range: *First relative pressure to 1.0000 P/Po*

Enter the same value you entered for **First rel. pressure** if you are performing a single-point analysis.

Replace

(continued)

```
2390      Vx.xx      SN123      a
Analysis Conditions
Number of points:
5
```

Enter the number of data points to be collected and press **ENTER**.

Range: 1 to 1000

The analyzer collects the specified number of data points at evenly spaced pressure intervals during the period of analysis.

Enter **1** if you are performing a single-point analysis.

Preserve

Preserves the currently stored pressure table. No further pressure table questions prompts are displayed.

Edit

Allows you to view and edit the current pressure table. This is useful if an unevenly spaced sequence of data points is desired; for example when a five-point surface area is to be combined with a single-point total pore volume. The following prompt is displayed:

```
2390      Vx.xx      SN123      a
Analysis Conditions
Adsorb pressure (number):
0.1000 P/Po
```

Enter the next desired target relative pressure or accept the default and press **ENTER**. 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

Edit

(continued)

```
2390 Vx.xx SN123 t
Analysis conditions
Desorb pressure: (number)
0.1000 P/Po
```

Displays only for the Gemini II 2390*t*.

This prompt displays only for the Gemini II 2390*t*. 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

```
2390 Vx.xx SN123 a
Analysis Conditions
Analysis mode?
Equilibrate
```

Select the analysis mode and press **ENTER**.

Choices: Equilibrate, Scan

Equilibrate

The analyzer 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.

The following prompt displays:

```
2390 Vx.xx SN123 a
Analysis Conditions
Eqilibration time:
5 s
```

Enter the equilibration time and press **ENTER**.

Range: 1 to 1000 sec

Scan

The analyzer continuously raises the adsorptive gas pressure at a constant rate based on the pressures to be achieved and the length of the analysis.

The following prompt displays:

```
2390    Vx.xx    SN123    a
Analysis Conditions
Scan time:
10 min/analysis
```

Enter the scan time (length of time for the analysis) and press **ENTER**.

Range: 1 to 10000 min/analysis

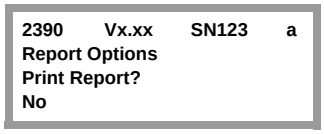
```
2390    Vx.xx    SN123    a

Setup type?
Analysis Conditions
```

Displays again; you may:

- Press **SAVE** to save the information you entered and return to the **Reload** prompt.
- Press **ENTER** to review or edit analysis conditions.
- Select a different Setup type.
- Press **Alt + CLEAR** to cancel any changes, delete all input, and return to the **Reload** prompt.

Report Options

The following prompts are displayed for **Report Options**:

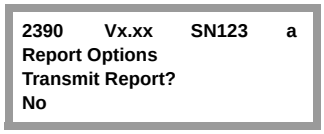


This prompt enables you to have a report generated automatically after an analysis.

Choices: *Yes, No*

The type of printer is selected at the **Printer?** prompt shown below.

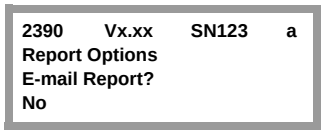
Press **CHOICE** to select **Yes** or **No**, then press **ENTER**.



This prompt enables you to transmit analysis data automatically after the operation via serial line communication. Refer to **Data Format**, page **D-1** for the format in which data are transmitted.

Choices: *Yes, No*

Press **CHOICE** to select **Yes** or **No**, then press **ENTER**.

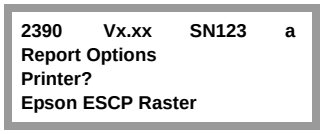


This prompt enables you to e-mail analysis results automatically after the analysis concludes.

Choices: *Yes, No*

If you choose **Yes**, an e-mail address and server must be specified (refer to **Communications**, page **5-29**).

Press **CHOICE** to select **Yes** or **No**, then press **ENTER**.

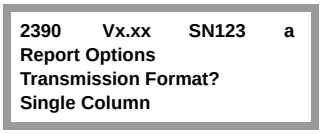


This prompt enables you to select the printer you wish to use for generating reports. This is a system option and does not have to be specified for each analysis.

Choices: *Epson ESCP Raster, Epson ESCP, HP PCL 6XL, Epson ESCP2, Postscript, HP PCL 3, Canon Bubble Jet*

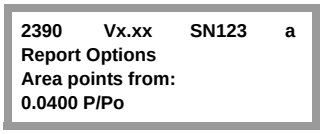
Refer to **Supported Printers**, page **E-1** for additional information on printers.

Press **CHOICE** to select the appropriate printer, then press **ENTER**.



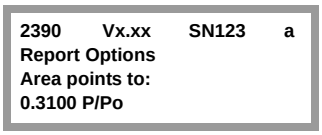
Select the format to be used when transmitting reports. Refer to Appendix D, page **D-1** for a description of the formats.

Choices: Single Column, Spreadsheet



Enter the relative pressure at which to start selecting collected data points for calculating surface area and press **ENTER**.

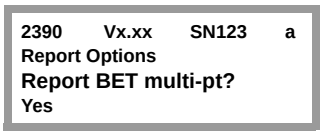
Range: 0.0 to 1.0000 P/Po



Enter the relative pressure at which to stop selecting collected data points for calculating surface area and press **ENTER**.

Range: ***Area points from value*** *to 1.0000 P/Po*

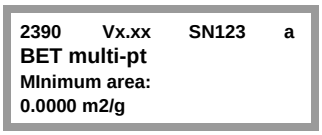
If the range you specify includes more than 50 data points, only the first 50 points are used to calculate surface area. You may select (or deselect) individual points after analysis.

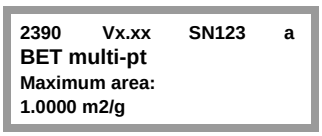


Press **CHOICE** to select one of the three options and press **ENTER.**

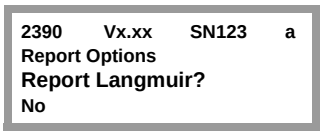
Choices: No, Yes, Pass/Fail

If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed.





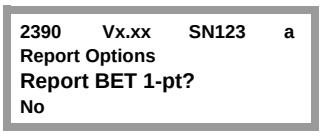
Range: 0.0000 to 9999.9999 m^2/g (for both prompts)



Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: No, Yes, Pass/Fail

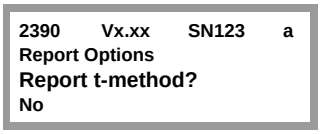
If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.



Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: No, Yes, Pass/Fail

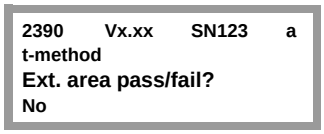
If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.

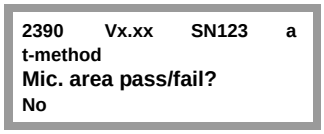


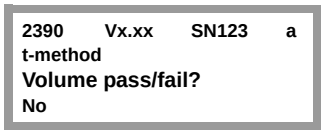
Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: No, Yes, Pass/Fail

If you select **Pass/Fail**, you can choose to specify ranges for external area, micropore area, and volume; the following prompts display:

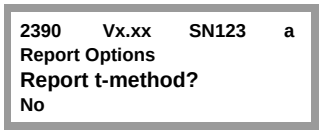






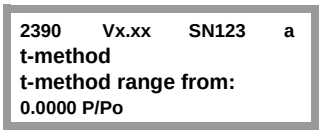
Each prompt has a **Yes**/**No** response. If you choose **Yes** at any of these prompts, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.

After specifying **Pass**/**Fail** criteria, the prompts that display for the **Yes** option are shown (see **Yes** response) next.



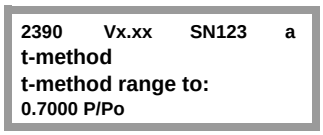
(continued)

If you choose **Yes** for the t-method report, the following prompts are displayed.



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

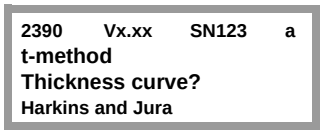
Range: 0.0 to 1.0 P/Po
Default: 0.0 P/Po



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

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

If the range you specify includes more than 50 data points, only the first 50 points are used in t-method calculations. You may select (or deselect) individual points after analysis.



Press **CHOICE** to select the desired thickness curve and press **ENTER.**

Choices: Harkins and Jura, Halsey, Magee-STSA

```
2390    Vx.xx    SN123    a
Report Options
Report t-method?
No
```

(continued)

Harkins and Jura

$$t = \left[\frac{①\ 13.9900}{0.0340 - \log\left(\frac{P}{Po}\right)\ ②} \right]^{③\ 0.500}$$

The Harkins and Jura* equation has three editable parameters; a prompt is displayed for each. The range and default for each parameter is as follows:

Parameter	Range	Default
1	0.0001 to 9999.00	13.990
2	0.0001 to 9999.00	0.0340
3	0.010 to 1.000	0.500

*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).

Halsey

$$t = {①}\ 3.540 \left[\frac{②\ -5.000}{\ln\left(\frac{P}{Po}\right)} \right]^{③\ 0.333}$$

The Halsey* equation has three editable parameters; a prompt is displayed for each. The range and default for each parameter is as follows:

Parameter	Range	Default
1	0.0001 to 9999.00	3.540
2	0.0001 to 9999.00	5.000
3	0.01 to 1.00	0.333

*Halsey, G., *J. Chem. Phys 16*, 931-932 (1948)

```
2390    Vx.xx    SN123    a
Report Options
Report t-method?
No
```

(continued)

Magee-STSA

$$t = \overset{①}{0.88}\left(\frac{P}{Po}\right)^{\overset{②}{2}} + \overset{②}{6.45}\left(\frac{P}{Po}\right) + \overset{③}{2.98}$$

The Magee-STSA* equation has three editable parameters; a prompt is displayed for each. The range and default for each parameter is as follows:

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

*Magee, Ricky, Columbian Chemicals Company (personal communications).

```
2390    Vx.xx    SN123    a
t-method
Minimum thickness:
3.5 A
```

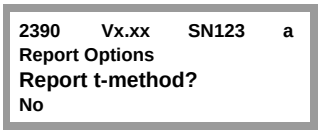
Enter the minimum thickness for points to be used in t-method calculations and press **ENTER**.

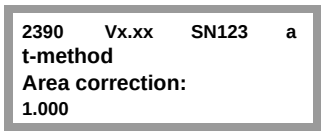
Range: *0.0 to 99.99 Angstroms*
Default: *3.5 A*

```
2390    Vx.xx    SN123    a
t-method
Maximum thickness:
5.000 A
```

Enter the maximum thickness for points to be used in t-method calculations and press **ENTER**.

Range: ***Minimum thickness*** *to 99.99 Angstroms*
Default: *5.0 A*





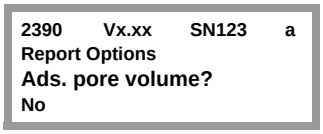
(continued)

Enter the surface area correction factor to be used in t-method calculations and press **ENTER**.

Johnson* recommends a surface area correction factor of F = 0.975 for oxide-type catalysts to get the BET area and external surface area to agree in the absence of zeolite.

Range: *0.100 to 1.000*
Default: *1.000*

*Marvin F.L. Johnson, "Estimation of the Zeolite Content of a Catalyst from Nitrogen Adsorption Isotherms," *Journal of Catalysis 52*, 425-431 (1978).

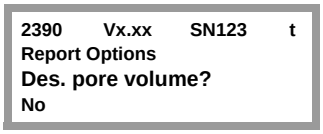


Enables you to have the pore volume calculated from the highest point of the adsorption isotherm.

Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: *No, Yes, Pass/Fail*

If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.



Displays only for the Gemini II 2390*t*.

Enables you to have the pore volume calculated from the highest point of the desorption isotherm.

Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: *No, Yes, Pass/Fail*

If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.

```
2390 Vx.xx SN123 a
Report Options
Report BJH?
No
```

BJH reporting assumes nitrogen is the adsorptive. Select **Yes** or **No** and press **ENTER**.

If you select **Yes**, the following prompts are displayed:

```
2390 Vx.xx SN123 a
BJH
Minimum diameter:
17.0 A
```

Enter the minimum diameter and press **ENTER**.

Range: 1.0 to 99998.9 Angstroms
Default: 17.0 A

```
2390 Vx.xx SN123 a
BJH
Maximum diameter:
3000.0 A
```

Enter the maximum diameter and press **ENTER**.

Range: 1.1 to 99999.0 Angstroms
Default: 3000.0 A

```
2390 Vx.xx SN123 a
BJH
Report adsorption?
No
```

Choices: No, Yes, Pass/Fail

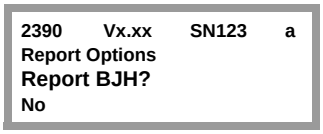
If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.

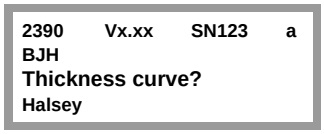
```
2390 Vx.xx SN123 t
BJH
Report desorption?
No
```

Displays only for the Gemini II 2390t

Choices: No, Yes, Pass/Fail

If you choose **Pass/Fail**, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.



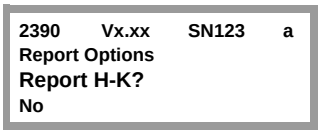


(continued)

Select the type of thickness curve.

Choices: *Halsey, Harkins and Jura, Magee-STSA*
Default: *Halsey*

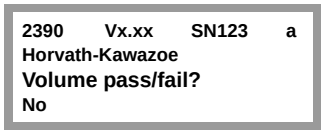
Refer to **t-method report** on page **5-20** for an explanation of the thickness curves.

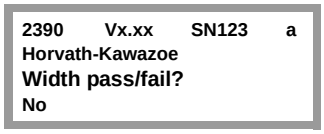


Press **CHOICE** to select one of the three options and press **ENTER.**

Choices: *No, Yes, Pass/Fail*

If you select **Pass/Fail**, you can choose to specify ranges for volume and width; the following prompts display:





Each prompt has a **Yes**/**No** response. If you choose **Yes** at any of the **Pass**/**Fail** prompts, two prompts enabling you to specify a range are displayed. Enter appropriate ranges; press **ENTER** after each entry.

After specifying **Pass**/**Fail** criteria, the prompts that display for the **Yes** option are shown (see **Yes** response) next.

```
2390    Vx.xx    SN123    a
Report Options
Report H-K?
No
```

(continued)

If you choose **Yes** for the H-K report, the following prompts are displayed.

```
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
```

These two prompts are displayed in sequence so that you can specify a pressure range for this report.

Range: 0.0001 to 1.0 P/Po (both prompts)

Select **Yes** or **No**, then press **ENTER**.

```
2390    Vx.xx    SN123    a
Horvath-Kawazoe
Pore geometry:
Slit
```

Choices: Slit, Cylinder, Sphere

Press **CHOICE** to select the shape which best represents the physical geometry of the micropores in your sample material. Press **ENTER**.

If **Slit** or **Cylinder** is selected, the following displays.

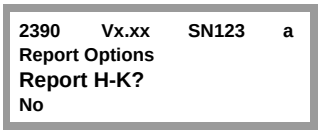
```
2390    Vx.xx    SN123    a
Horvath-Kawazoe
Interaction param.:
3.49e-43 erg cm4
```

Does not display for Sphere.

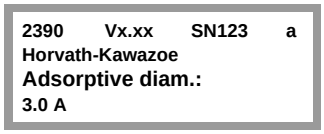
Range: 1.00e-46 to 9.99e-40 erg cm^4
Default: 3.49e-43 erg cm^4

Input into this field is slightly different from that of other fields. You can edit the significand and the exponent. For example, the prompt shown above shows 3.49 as the significand. Enter 2.34 to replace 3.49; the next keystroke automatically replaces the exponent.

Enter a value for the interaction parameter to be used in report calculations and press **ENTER**.



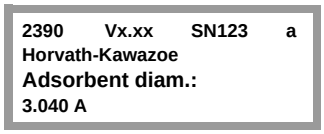
(continued)



This is the diameter of the adsorptive molecule.

Range: *0.0 to 99.999 Angstroms*
Default: *3.0 A (N2)*

Edit or accept the default, then press **ENTER**.

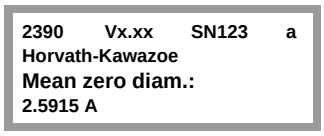


This is the diameter of the sample adsorbate atom.

Range: *0.0 to 99.999 Angstroms*
Default: *3.04 (Zeolite)*

Edit or accept the default, then press **ENTER**.

If you selected **Slit** for **Pore geometry**, this prompt is displayed.



Does not display for Cylinder or Sphere.

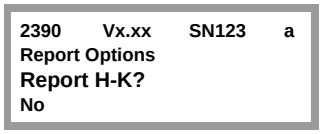
The mean zero diameter (s) is the gas-solid nuclear separation at zero interaction energy,

$$\sigma = \frac{Z_S + Z_A}{2}$$

where Z_S is the sample equilibrium diameter at zero interaction energy and Z_A the adsorptive zero interaction energy diameter.

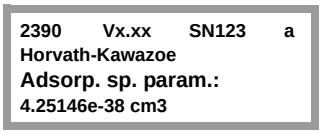
Range: *0.0 to 99.9999 Angstroms*
Default: *2.5915 A*

Enter the appropriate value and press **ENTER**.



(continued)

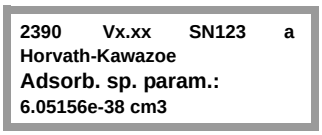
If you selected **Sphere** for **Pore geometry**, these two prompts are displayed.



Does not display for Slit or Cylinder.

Range: *1.0e-40 to 9.99999e-37 cm3*
Default: *4.25146e-38 cm4*

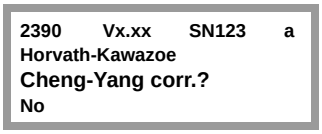
This is the adsorptive spherical parameter; enter the appropriate value (refer to **Spherical Parameters**, page **C-27** for additional information) and press **ENTER**.



Does not display for Slit or Cylinder.

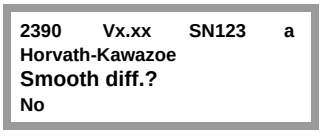
Range: *1.0e-40 to 9.99999e-37 cm3*
Default: *6.05156e-38 cm3*

This is the adsorbent spherical parameter; enter the appropriate value (refer to **Spherical Parameters**, page **C-27** for additional information) and press **ENTER**.



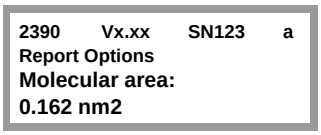
If you choose **Yes**, the Cheng/Yang correction is applied to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation (refer to **Horvath-Kawazoe**, page **C-19** for calculations).

Select **Yes** or **No**, then press **ENTER**.



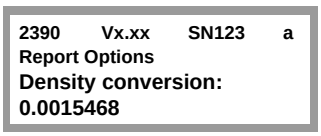
This option enables you to smooth the differential calculation, eliminating variations in the differential computation caused by noise in the input data.

Select **Yes** or **No**, then press **ENTER**.



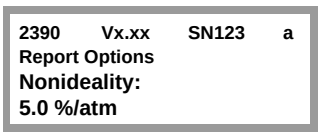
Enter the molecular cross-sectional area of the adsorptive gas and press **ENTER**.

Range: 0.001 to 1.000 nm^2



Enter the adsorptive gas-to-liquid density conversion factor and press **ENTER**.

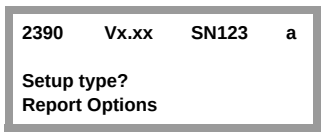
Range: 0.0000001 to 1.0000000



Enter the nonideality correction factor for the adsorptive gas and press **ENTER**.

Range: 0.0 to 99.9

Nonideality is calculated when a free-space value (other than **none**) is selected.

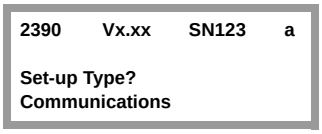


Displays again; you may:

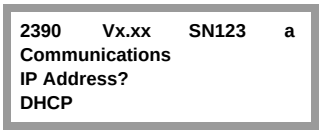
- Press **SAVE** to save the information you entered and return to the **Reload** prompt.
- Press **ENTER** to review or edit report options.
- Select a different setup type.
- Press **Alt + CLEAR** to cancel any changes, delete all input, and return to the **Reload** prompt.

Communications

Communications enables you to specify communications criteria. The parameters specified for these prompts apply to all Setup groups.



Press **CHOICE** until **Communications** is displayed, then press **ENTER**.

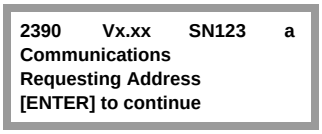


This prompt enables you to choose the manner in which your IP address is assigned. With an IP address, you can access a web browser to view analysis results. Typically, after an analysis (or calibration) is started, you cannot review results from the previous operation. However, results from five analyses are stored in the controller. A web browser enables you to view them. (Refer to **Using a Web Browser**, page **4-23** for step-by-step instructions.) An IP address must exist for this operation. You can have an IP address detected and assigned automatically, or you can specify one.

Choices: DHCP, Specify

Choose **DHCP** to have an IP address assigned automatically, or **Specify** to specify an address.

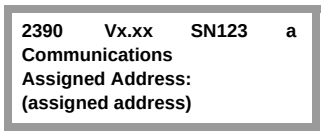
Press **CHOICE** until the desired option is displayed, then press **ENTER**.



Does not display for Specify

Displays when you choose **DHCP** for the first time.

Press **ENTER**; the system automatically tries to assign an IP address. If the system is able to determine an address, the following prompt containing the assigned address is displayed.



```
2390 Vx.xx SN123 a
Communications
Requesting Address
[ENTER} to continue
```

(continued)

If an address cannot be determined, this prompt is displayed.

```
2390 Vx.xx SN123 a
Communications
Requesting Address
[ESC] to cancel.
```

Press **Alt + CLEAR** to cancel the operation and return to the **Reload** prompt. Contact your IT Department, or choose the **Specify** option and enter the appropriate information.

```
2390 Vx.xx SN123 a
Communications
IP Address:
(user-entered)
```

Does not display for DHCP

Displays when you choose **Specify**, enabling you to enter an appropriate IP address.

This is a numerical field in the following format: nnn.nnn.nnn.nnn

Enter the address (including periods) and press **ENTER**.

```
2390 Vx.xx SN123 a
Communications
Subnet Mask:
(user-entered)
```

Does not display for DHCP

Displays this prompt so that you can enter the subnet mask number.

This field is also numerical in the same format as the IP address.

Enter the subnet mask number and press **ENTER**.

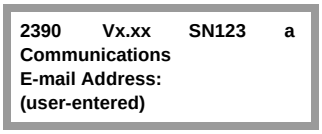
```
2390 Vx.xx SN123 a
Communications
Gateway:
(user-entered)
```

Does not display for DHCP

The Gateway address is used for communicating outside of your 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. For example, nnn.nnn.nnn.1

Contact your IT Department if you have questions.

Enter the gateway address and press **ENTER**.

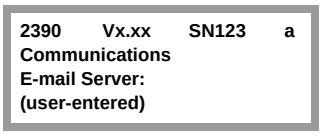


When connected to a network via the ethernet connection on the rear panel of the analyzer, you can have analysis results e-mailed automatically at their completion. This prompt enables you to enter the e-mail address to which results are to be sent.

If you are not using DHCP and the e-mail address is outside the local network, be sure to specify a gateway address (see previous prompt).

To use this option, you should have a keyboard connected to one of the USB connectors.

Enter the e-mail address and press **ENTER**.

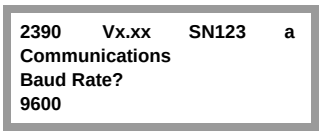


This prompt enables you to enter the numerical address of the SMTP server (the computer that is going to deliver the results). If this address is unknown, contact your IT Department.

Your IT Department may need to configure the server to accept e-mail from the Gemini. For proper configuration, the following information may be required:

- IP address for the Gemini
- Sender line: **Micromeritics_2390**

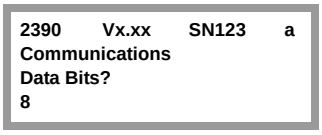
Press **ENTER**.



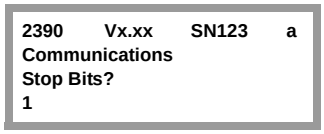
Baud rate specifies the rate at which data are transmitted via the RS232 port.

Choices:	*9600*	*600*
	110	*1200*
	150	*4800*
	300	*19200*

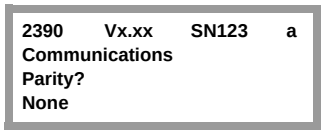
Press **CHOICE** until the desired value is displayed, then press **ENTER**.



Press **CHOICE** to select **8** or **7**, then press **ENTER**.

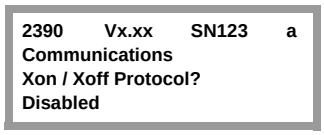


Press **CHOICE** to select **1** or **2**, then press **ENTER**.



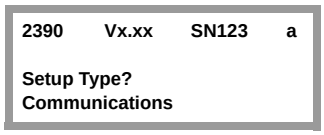
Press **CHOICE** until the desired option is displayed, then press **ENTER**.

Choices: None, Even, Odd



This prompt enables you to use the Xon/Xoff protocol for transmitting data.

Press **CHOICE** to select **Disabled** or **Enabled**, then press **ENTER**.

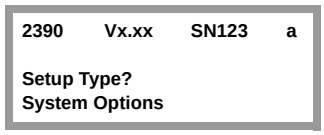


Displays again; you may:

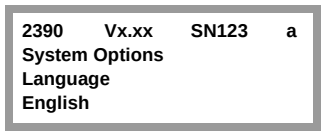
- Press **SAVE** to save the information you entered and return to the **Reload** prompt.
- Press **ENTER** to review or edit communication parameters.
- Select a different setup type.
- Press **Alt + CLEAR** to cancel any changes, delete all input, and return to the **Reload** prompt.

System Options

The following prompts are displayed for **System Options**. The parameters specified for most of these prompts apply to all Setup groups. The parameters specific to a Setup group are marked accordingly.



Press **CHOICE** until **System Options** is displayed, then press **ENTER**.



This prompt enables you to choose the operating language.

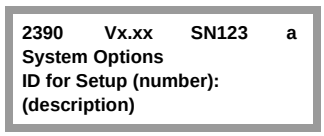
Choices: English, Deutsch, Francais, Espanol, Italiano

After you select a language, the change is effective immediately; you do not have to press **SAVE**. For example, the next prompt displays in Italian if you choose **Italiano**.

However, any error messages that may be in the message queue remain in English (or the current selected language); they are not translated. Error messages that occur after you select the language will be shown in that language.

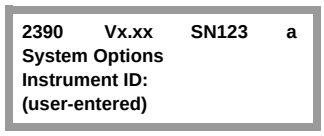
Keypad overlays containing translations for the alternate functions printed above the keys are available for each language (refer to **Ordering Information**, beginning on page 7-1).

Choose the desired language, then press **ENTER.**



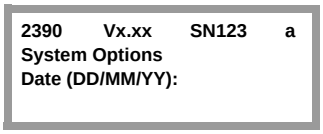
Enter the ID for the Setup Group, then press **ENTER**. Press **.** (decimal) from the Gemini keypad to enter a slash.

This prompt is specific to a Setup group.



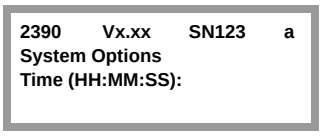
The instrument ID is a unique identifier of the analyzer. It can contain 1 to 20 numbers or dashes. (If you have several analyzers, you can use this field to identify each analyzer on printed or transmitted reports.)

Enter an instrument ID and press **ENTER**. Press **.** (decimal) from the Gemini keypad to enter a dash.



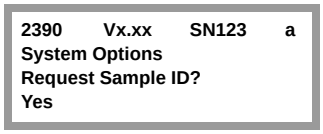
Enter the current date (day/month/year) using the decimal key for a slash (/) and press **ENTER**.

Range: *Day:* *1 to 31*
Month: *1 to 12*
Year: *00 to 99*



Enter the current time (HH:MM:SS) using the decimal key for a colon (:) and press **ENTER**.

Range: *Hours:* *0 to 23*
Minutes: *0 to 59*
Seconds: *0 to 59*

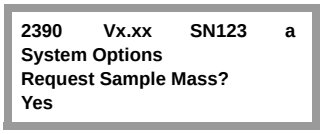


The sample ID is a unique identifier of the sample. It can contain 1 to 20 numbers or dashes. Using sample IDs can help you keep track of data from various analyses.

Press **CHOICE** to select **Yes** or **No** and press **ENTER**.

If you select **No**, this prompt does not appear when you use the **Analyze** and **Review** functions.

This prompt is specific to a Setup group.



Prompts can be shown in the **Analyze** function for entering the sample mass, or for entering criteria to have it calculated automatically.

Choices: *Yes, Tube and tube+sample, No*

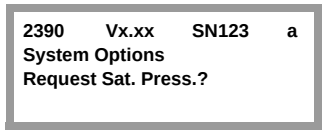
Yes displays a prompt enabling you to enter the sample mass.

Tube and tube+sample displays two prompts enabling you to enter 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.

Press **CHOICE** to select the desired option, then press **ENTER**.

This prompt is specific to a Setup group.



Press **CHOICE** to choose the type of saturation pressure, then press **ENTER**.

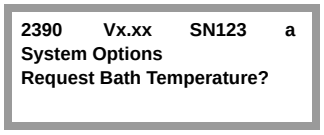
Choices: Entered, Previously measured, During analysis (not available for the 2390a)

During analysis: the saturation pressure is measured for each data point taken; applicable only for the 2390*p* and 2390*t*.

Previously measured: the saturation pressure from the last data point of the previous analysis is used.

Entered: a prompt is displayed when the analysis is started allowing you to enter a saturation pressure.

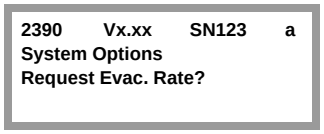
This prompt is specific to a Setup group.



Press **CHOICE** to select **Yes** or **No** and press **ENTER**.

If you select **No**, this prompt does not appear when you use the **Analyze** and **Review** functions.

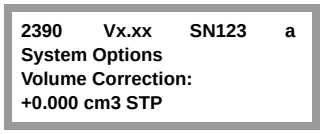
This prompt is specific to a Setup group.



Press **CHOICE** to select **Yes** or **No** and press **ENTER**.

If you select **No**, this prompt does not appear when you use the **Analyze** and **Review** functions.

This prompt is specific to a Setup group.



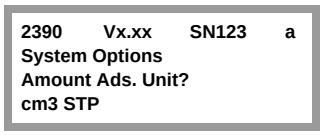
Enter the difference in gas capacity between the balance volume and the sample tube, using **CHOICE** to change the plus or minus sign if necessary, then press **ENTER**.

Range: -99.990 to +99.990

The volume correction is only used when calculated free space is selected. It accounts for any difference in volume between the balance tube and the empty sample tube, and may be determined by the following procedure:

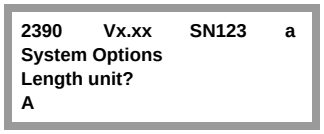
Perform a liquid nitrogen analysis using two empty sample tubes of the same size. Select **Measure** for free-space option (Analysis conditions, page **5-11**).

After the analysis, record reported free-space difference and use it for these two tubes only for future calculated runs.



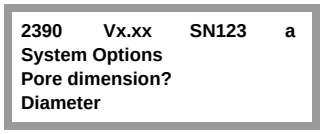
Choices: cm3 STP, mmol

Press **CHOICE** until the desired unit is displayed, then press **ENTER**.



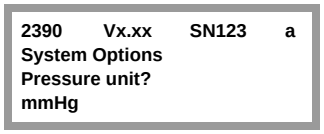
Choices: A (Angstrom), nm (nanometer)

Press **CHOICE** until the desired unit is displayed, then press **ENTER**.



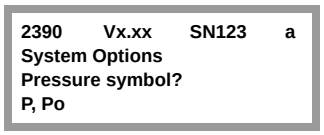
Choices: Diameter, Width, Radius

Press **CHOICE** until the desired unit is displayed, then press **ENTER**.



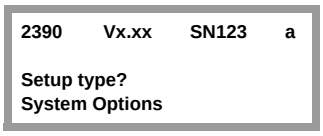
Choices: mmHg, kPa, mbar

Press **CHOICE** until the desired unit is displayed, then press **ENTER**.



Choices: P, Po or p, p^o

Press **CHOICE** until the desired unit is displayed, then press **ENTER**.

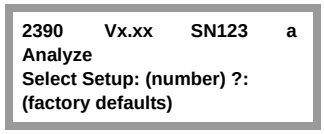


Displays again; you may:

- Press **SAVE** to save the information you entered and return to the **Reload** prompt.
- Press **ENTER** to review or edit parameters for system options.
- Select a different setup type.
- Press **Alt + CLEAR** to cancel any changes, delete all input, and return to the **Reload** prompt.

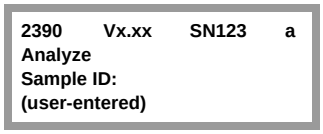
Analyze

This command enables you to perform an analysis; press **Alt** + **4** on the keypad.



Enter the number of the Setup group you wish to use for the current analysis or press **CHOICE** until the desired number is displayed, then press **ENTER**.

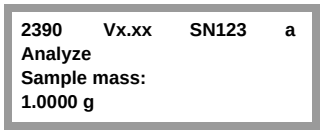
Choices: 0 thru 9



Displays if **Request Sample ID** is set to **Yes**. (Refer to **System Options**, page 5-34).

Enter the sample ID and press **ENTER**. Press **.** (decimal) to insert a dash, if required.

Range: 1 to 20 numbers and dashes

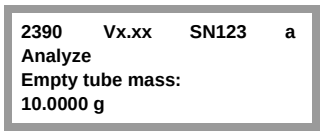


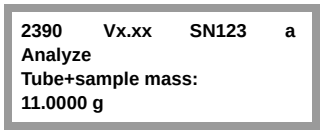
Displays if **Request Sample Mass** is set to **Yes**. (Refer to **System Options**, page 5-34.)

Enter the sample mass and press **ENTER**.

Range: 0.0010 to 999.9990 g
Default: 1.000

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 your analytical balance to transfer the mass; most balances use the **ENTER** button (refer to the manufacturer's manual for information on transferring the value).

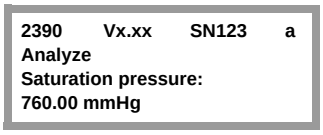




These two prompts display in sequence when **Request Sample Mass** is set to **Tube and tube+sample**. (Refer to **System Options**, page 5-34.)

Enter the mass of the empty tube in the first prompt, press **ENTER**, then enter the mass of the Tube plus sample in the field of the second prompt. Press **ENTER**.

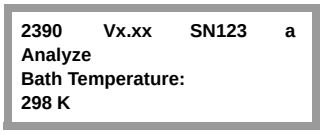
Range: 0.0010 to 999.9990 g
Default: 1.000



Displays if **Saturation Pressure** is set to **Entered**. (Refer to **System Options**, page 5-35.)

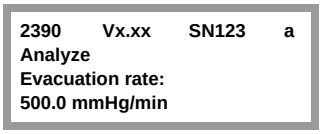
If the saturation pressure was measured using Gemini's Po function, the measured value is displayed here. If not, enter the saturation pressure and press **ENTER**.

Range: *500.00 to 900.00 mmHg*
Default: *Measured value, 760 00 mmHg or previous entry*



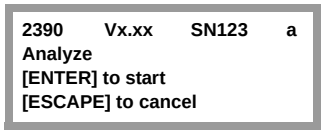
Enter the bath temperature and press **ENTER**.

Range: *0 to 999.999 Kelvin*



Enter the evacuation rate and press **ENTER**.

Range: *1.0 to 1000.0 mmHg/min*
Default: *500.0 mmHg/min or previous entry*



Choose one of the following:

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

  The analysis begins and operational status messages are continually displayed during analysis.

- Press **Alt + CLEAR** to cancel the analysis.

- Press **SAVE** to store the data you entered and return to the **Reload** prompt.

QuickStart

QuickStart enables you to perform a series of analyses using the same Setup number. Like the Analyze function, the prompts you see with QuickStart are based on the Setup parameters specified for the Setup number you use. Refer to **Analyze**, page **5-37** for an explanation of the prompts that may be displayed using the QuickStart method.

Press **Alt + 9** to access the QuickStart function.

The parameters for the last Setup group you used will be applied to the QuickStart analyses.

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

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

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

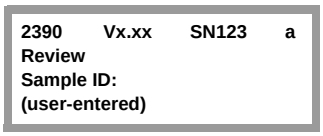
```
2390      Vx.xx      SN123      a
QuickStart
[ENTER] to Start
[ESCAPE] to Cancel
```

Press **ENTER** to start the analysis.

After the first analysis is completed, you are returned to the **Reload** prompt. Press **Alt + 9** to begin another QuickStart analysis.

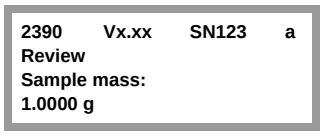
Review

The review function enables you to review and edit the results of the last analysis; press **Alt** + **5** on the keypad.



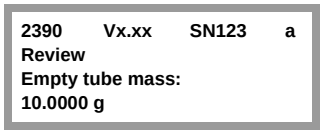
Displays when **Request Sample ID** is set to **Yes**. (Refer to **System Options**, page 5-34.)

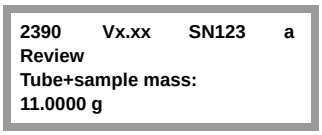
The sample ID entered in the **Analyze** function is displayed. Enter a new ID if desired and press **ENTER**.



Displays when **Request Sample Mass** is set to **Yes**. (Refer to **System Options**, page 5-34.)

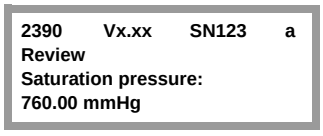
The value you entered for the sample mass in the **Analyze** function is displayed. Enter a new mass if desired and press **ENTER**.





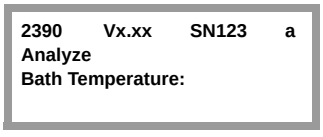
These two prompts display in sequence when **Request Sample Mass** is set to **Tube and tube+sample**. (Refer to **System Options**, page 5-34.)

Displays the values you entered for these prompts in the **Analyze** function. Press **ENTER**.

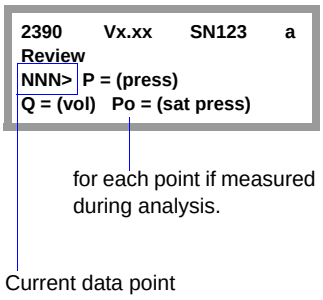


Displays if **Saturation Pressure** is set to **Previously measured** or **Entered**. (Refer to **System Options**, page 5-35.)

The last measured saturation pressure entered in the **Analyze** function is displayed. Enter a new saturation pressure if desired and press **ENTER**.



Displays the temperature of your cryogen bath; press **ENTER**.

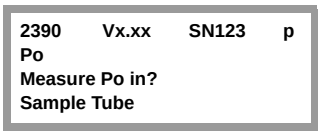


Displays for all collected data points.

- Press **CHOICE** to include unmarked points in the calculations or to remove marked points from use in the calculations.
- Press **ENTER** to advance to the next data point.
- Press **SAVE** to store the data and return to the **Reload** prompt.
- Press **Alt** + **CLEAR** to cancel any changes, delete all input, and return to the **Reload** prompt.

Po

The Po command allows you to measure the saturation pressure (Po); press **Alt** + **.** (decimal) on the keypad.



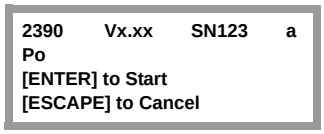
Does not display for the 2390a.

Displays when you have the **2390*p*** or **2390*t*.**

Choices: Sample Tube, Po Tube

If you choose **Sample Tube**, be sure that empty sample tubes of the same size are installed on the analysis port and balance port. You do not have to install tubes on both ports if you choose **Po tube**.

Press **CHOICE** to select the preferred method for measuring the saturation pressure, then press **ENTER** to begin.



This prompt displays when you have the **2390*a***.

Before beginning, be sure that empty sample tubes of the same size are installed on the analysis port and balance port.

Press **ENTER** to begin measuring the saturation pressure.

Status messages display while the saturation pressure is being measured and when complete, the **Reload** prompt is displayed. The saturation pressure is stored in memory and used in report calculations.

Print

Press **Alt** + **6** on the keypad to print a report from the last analysis. A printed report contains more information than the report generated to the keypad display, and is easier to read. It also provides a hard copy of data results. You can print a report to a **Printer** or through a serial transmission line. Reports are also sent to the Display, regardless of the specified destination. See **Report Options**, page **5-16** for information on specifying a destination for report output.

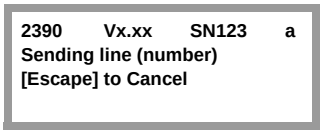
Reports are generated after analysis and remain available for viewing or printing until another analysis is performed. When you perform an analysis, reports from the previous analysis are deleted and, therefore, no longer available to print using the **Print** funtion.

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. Refer to page **Communications**, page **5-29** for additional information on this feature and to **Using a Web Browser**, page **4-23** for step-by-step instructions and an example of a report.

Transmit

The Gemini Analyzer 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 popular spreadsheet and data manipulation programs.

Press **Alt + 3** on the keypad to transmit data from the last analysis; the following prompt is displayed:



Indicates the data is being transmitted.If you wish to cancel the transmission, press **Alt** + **Clear** (**Escape**).

Refer to Appendix D, page **D-1** for the format of transmitted data and Appendix F, page **F-1** for details on RS-232 operation.

Diagnostics

The diagnostic prompts enable you to:

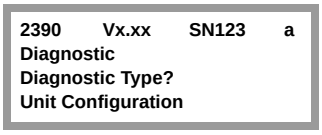
- View analyzer and calibration statistics
- Clean and verify the gas line for the adsorptive gas
- Clean and verify the gas line for helium
- Check the system for leaks (service request only)
- Verify that the analyzer is operating properly (service request only)
- Zero transducers to stored offsets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

After a test has completed, a report is generated automatically to the destination(s) specified in Report Options (refer to page **5-16** for information on report destinations). Results for Diagnostic reports can also be viewed using a web browser (refer to **Using a Web Browser**, page **4-23**). It is not recommended that you transmit diagnostic reports via serial line communication. Diagnostic results in this form produce values only and will require interpretation by your Service representative.

You can press **Alt** + **CLEAR** to cancel and return to the **Reload** prompt at any time.

Press **Alt + 7** to access Diagnostics.



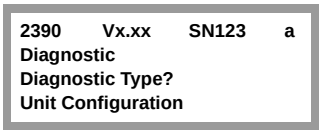
Press **CHOICE** until the desired test is displayed, then press **ENTER**.

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

These options are explained in subsequent sections.

Unit Configuration

This series of prompts enables you to view instrument and calibration statistics. You cannot edit any of the information contained in the prompts.



Press **ENTER** at each prompt to navigate through the prompts. Prompts are in the following order:

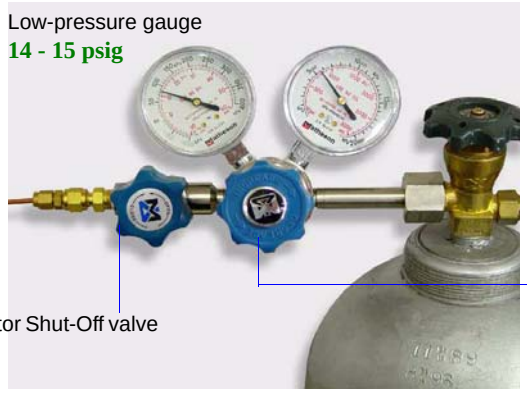
IP Address
Controller Boot
Last Measured Po
Sample Offset
Sample Slope
Adsorbed Offset
Adsorbed Slope
Balance Offset
Balance Servo
Sample Servo Low
Sample Servo High
Po Offset
Po Slope

Adsorptive Line Test

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

This test should be performed every time the adsorptive gas bottle is replaced. Follow the instructions provided in **Connecting Gases**, page **2-3**.

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




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

Press **CHOICE** until **Adsorp. Line Test** is displayed, then press **ENTER**.

A series of prompts of the following types is displayed. Each time a prompt displays, the analyzer beeps to get your attention.

- **Informative**; for example, advising how long the test will take or how long before you will be required to open or close a valve.

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

  Read the message and press **ENTER** to proceed.

- **Operator action**; for example, you may be asked to open a gas bottle valve.

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

  Perform the task first, then press **ENTER** to proceed. Do not press **ENTER** before completing the task.

When the test is finished, the following prompt displays:

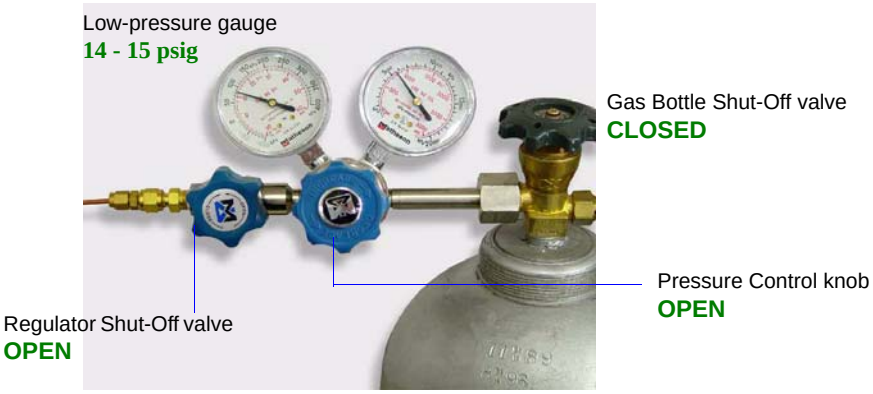
```
2390 Vx.xx SN123 a
Diagnostic Passed

[ENTER] to continue.
```

Indicates that the test has passed or failed. If **Failed** is displayed, tighten connections for the adsorptive gas and repeat the test.

Press **ENTER** to return to the **Reload** prompt.

Helium Line Test

This test helps to clean and verify the helium gas supply; first from the instrument to the regulator shut-off valve, then from the instrument to the gas bottle shut-off valve. Before beginning, confirm that the state for valves and the low-pressure gauge reading are as shown here.




```
2390 Vx.xx SN123 a
Diagnostic
Diagnostic Type?
Helium Line Test
```

Press **CHOICE** until **Helium Line Test** is displayed, then press **ENTER**.

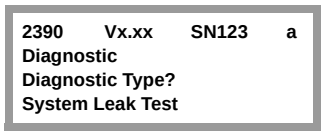
The remaining prompts for the Helium Line Test are the same as those for the Adsorptive Line Test (refer to **Adsorptive Line Test**, page **5-46** for a description of the types of prompts that display).

System Leak Test

This test checks for atmospheric leaks among instrument components and typically is performed only when requested by your service representative.

This test requires operator attention; the instrument will beep when attention is required.

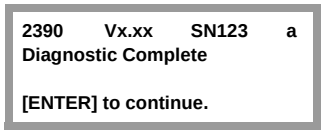
Before beginning this test, be sure that sample tubes or plugs are installed on the sample and balance ports.



Press **CHOICE** until **System Leak Test** is displayed, then press **ENTER**.

A series of informational and action prompts displays (refer to **Adsorptive Line Test**, page **5-46** for a description of the types of prompts that display). Observe the information or perform the task and press **ENTER**.

When the test is finished, the following prompt displays



Press **ENTER** to return to the **Reload** prompt.

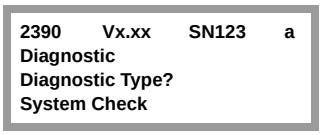
System Check

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, be sure that:

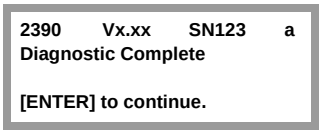
- sample tubes or plugs are installed on the sample and balance ports
- the helium gas inlet is plugged or if helium is attached, the regulator shut-off valve is closed
- the adsorptive gas in attached to its inlet port
- the vacuum pump is installed



Press **CHOICE** until **System Check** is displayed, then press **ENTER**.

A series of informational and action prompts displays (refer to **Adsorptive Line Test**, page **5-46** for a description of the types of prompts that display). Observe the information or perform the task and press **ENTER**.

When the test is finished, the following prompt displays



Press **ENTER** to return to the **Reload** prompt.

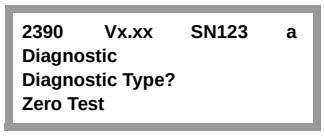
Zero Test

This test compares and zeroes transducers to stored offsets.

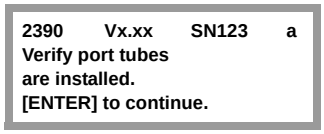
This test should be performed only when requested by your service representative.

This test does not require operator attention.

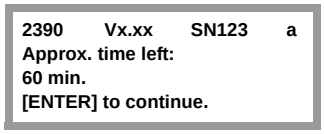
Before beginning this test, be sure that clean, empty sample tubes are installed on the sample and balance ports



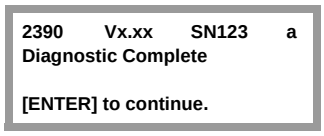
Press **CHOICE** until **Zero Test** is displayed, then press **ENTER**.



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



This prompt informs you how long the test will take to complete. This test takes 60 minutes; no operator attention is required. Press **ENTER**.



The test is complete, press **ENTER** to return to the **Reload** prompt.

Manual

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 your service representative.

Do not use this mode of operation without direction from your 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.

A brief description of the functions are shown below. Following these descriptions is an annotated example of the display window when in manual mode.

Press **Alt** + **1** to enter manual mode.

RAW/TRUE Displays the **raw** A/D readings for the sample, balance, and Po transducers or quantity adsorbed transducer, or the **true** pressure and volume readings.

B/S

Disables/enables the balance servo.

- A **B** in the display window indicates that the balance servo and sample servo are enabled.
- An **S** indicates that only the sample servo is enabled; the balance servo is disabled.

For this command, you must press the **Alt** key before pressing **8**.

SERVO

The commands above the **4**, **5**, and **6** keys relate to the servo valves.

ON/OFF (above 4)

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.

UP / DOWN ARROWS

↑ ↓ (above 5)

Press ↑ ↓ to change the direction of the servo; ">" displays when in dose mode and "<" when in evacuate mode.

TARGET (above 6)

Displays the following prompt, allowing you to edit the sample servo target pressure.

```
2390      Vx.xx      SN123      a
P=760.000                V– –R– –
Target Pressure:
(current target pressure)
```

.

The fourth line defaults to the current target pressure. Press **Clear** to clear the field of its entry, or simply begin to enter the new value. Use **Alt + .** (period) to erase the previous keystroke.

SAVE

Exit manual mode and return to the **Reload** prompt.

VALVES

The commands above the 1, 2, and 3 keys operate system valves.

Po / VACUUM (above 1) Opens and closes the Po or vacuum valve.

- Press **1** for the vacuum valve
- Press **Alt + 1** for the Po valve (not applicable for Gemini 2390*a*)

HELIUM (above 2) Opens and closes the helium valve.

NITROGEN (above 3) Opens and closes the nitrogen valve.

RESERVOIRS Opens and closes the balance and sample reservoir valve in unison.

PORTS Opens and closes the balance and sample port valves in unison.

ELEVATOR

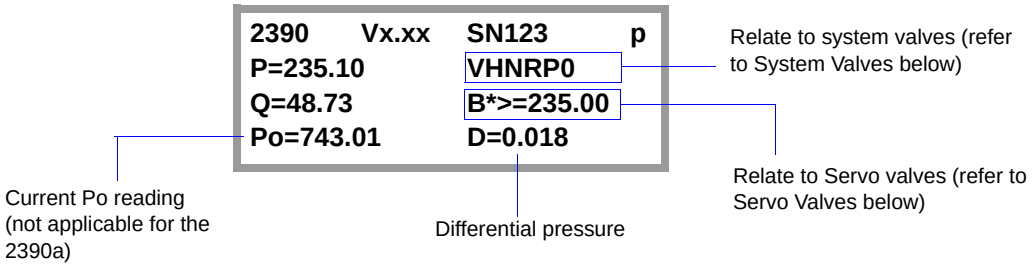
UP / DOWN ARROWS

↑ ↓

Press ↑ (Up arrow) to raise the elevator. Press the key again to stop the elevator.

Press ↓ (Down arrow) to lower the elevator. Press the key again to stop the elevator.

When performing the manual control functions, pertinent information is displayed in the keypad window. The following example shows the type of information that displays; you may be requested to provide this information by your service representative.



System valves:

V = Vacuum

H = Helium

N = Nitrogen

R = Reservoir

P = Port

0 = Po valve (not applicable for Gemini 2390*a*)

When the actual character displays, the valve is open; for example, the display window shown above indicates that all valves are open. If the valve is closed, a dash (–) displays in the valve position; for example, **V– – RP0** indicates that the helium and nitrogen valves are closed and all others are open.

Servo valves:

B = Servo valves; an S displays in this position if the balance servo is disabled

* = valves are on

> = servo direction

234.56 = target pressure

6. TROUBLESHOOTING AND MAINTENANCE

This chapter includes:

- common operational problems and their solutions
- preventive maintenance schedule
- maintenance procedures

If further assistance is needed after following the procedures in this chapter, contact a Micromeritics Service Representative.

Troubleshooting

Operating problems encountered with the analyzer are usually easily corrected. Typical problems and the steps required to correct them are described in the following table.

Table 6-1. Common Operational Problems

What Happened	Why	What To Do
Unit does not work when power switch is turned on.	Power cord not fully inserted at one end or the other.	Insert power plug firmly into power source and analyzer power connector.
	No power at outlet.	Plug in lamp or small appliance to test outlet. If there is no power, contact electrician.
	Plug prongs bent so that contact not made at outlet.	Gently move power plug at outlet while watching display. If display comes on, have electrician replace outlet or plug.
	Power cord damaged.	Have electrician check cord using a test meter. Replace the cord if defective.
	Loose internal connection or broken wire.	Call a Micromeritics Service representative for repair or replacement information.

Table 6-1 Common Operational Problems *(continued)*

What Happened	Why	What To Do
Specified pressure not reached.	Sample or balance tube not properly attached.	Reattach sample and balance tubes, making sure both are securely attached to ports. Replace O-rings if they are defective.
	Leak in gas line(s).	Perform the Adsorptive and Helium Line tests to determine if a leak exists. Refer to **Diagnostics**, page **5-44** for details on these tests.
Nitrogen or helium drained from tank, or depleted in a short period of time.	Leak(s) in the gas line connection.	Perform the Adsorptive (or Helium) Line test to determine the location of the leak. Refer to **Diagnostics**, page **5-44** for details on these tests.
Expected results are not within range.	Sample improperly degassed.	Verify that the degassing temperature and degas time were set properly.
	Gas bottle(s) may be almost empty, causing gas impurity.	Replace gas bottle. Refer to **Connecting Gases**, page **2-3**.
	Undetermined	Do the following: 1. Perform a blank analysis (see **Performing a Blank Analysis for Diagnostic Purposes**, page **6-7**); print the results. 2. Perform a reference material analysis (see **Performing a Reference Analysis**, page **2-20**); print the results. 3. Contact your Micromeritics service representative.

Table 6-1 Common Operational Problems *(continued)*

What Happened	Why	What To Do
Unable to reach a satisfactory vacuum.	Vacuum pump oil level is low or needs to be changed.	Inspect the oil to see if it is low or needs changing. Refer to **Inspecting the Oil**, page **6-10**
	Vacuum pump is leaking.	Locate source of leak and have repaired, or replace pump.
	Centering ring has become too flat and unable to hold a vacuum..	Check the centering ring at the pump intake port; replace if necessary (refer to page **6-15**).
		Check the centering ring at the top of the oil vapor trap; replace if necessary (refer to page **6-16**).
Results will not print as requested.	Printer is not compatible with the Gemini analyzer.	Refer to Appendix E, page **E-1** to ensure your printer meets the criteria necessary for use with the analyzer.

Preventive Maintenance

The table below lists the preventive maintenance procedures you should complete to keep your analyzer operating at peak performance. Instructions for each procedure follow the table. Micromeritics also recommends that you have preventive maintenance procedures and calibration performed by one of our service representatives every 12 months.

Maintenance Required	Frequency
Clean the outside of the analyzer, page **6-5**	As required or every 6 months
Clean the analysis Dewar, page **6-5**	Weekly
Replace port frit and O-ring, page **6-6**	Every 30 days
Inspect vacuum pump oil, page **6-10**	Every 3 months
Change vacuum pump oil, page **6-10**	As required
Replace alumina in oil vapor trap, page **6-13**	As required
Replace vacuum pump exhaust filter, page **6-18**	As required or every 12 months
Blank tube analysis, page **6-7**	As required

Cleaning the Analyzer

The exterior casing of the analyzer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution. Do not use any type of abrasive cleaner.

Cleaning the Analysis Dewar

When handling glass Dewars, be sure to observe the Dewar precautions outlined in Preparing the Analysis Dewar, page 4-15.

Ice and suspended frost particles may accumulate in the bottom of an analysis Dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tubes and the bottom of the Dewar, causing the Dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tubes. 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 or balance tube causing breakage.

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

1. Be sure the elevator is at its lowest position.

2. Carefully remove the Dewar from the elevator, avoiding contact with the sample and balance tubes.

3. Pour the liquid nitrogen from the Dewar into an appropriate cryogenic container.

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

4. Wait approximately 10 minutes (allowing the Dewar to warm), then rinse the Dewar with *warm* (not hot) water to melt any remaining ice accumulation; dry thoroughly.

Do not rinse an iced Dewar with hot water; this could cause the Dewar to implode.

Recovering from a Power Failure

The analyzer saves entered and collected data in case of power failure. Setup 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 started again in order to produce complete results.

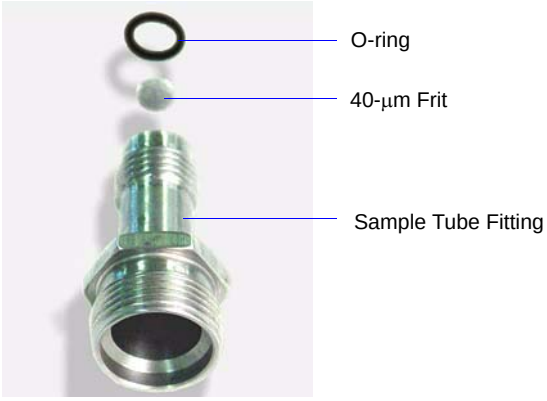
Replacing Analysis Port Frit and O-Ring

A 40-µm frit is located in the analysis port of the analyzer. If the frit is contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated frit on the analysis port may be indicated by a leak or by a free-space reading much lower than normal. The balance port contains a 2-µm frit and typically does not need replacing. In the event replacement is necessary for the balance port, follow the same procedure listed here using the 2-µm frit.

Be sure to use a 40-µm frit in the sample port. The instrument will not operate properly if a 2-µm frit (used in the balance port) is installed in the sample port.

A contaminated frit should be replaced as follows:

1. Remove the sample tube fitting from the analysis port using a wrench. Pry out the frit and O-ring.



To avoid outgassing problems, the frit and O-ring should be clean and should not be touched with bare hands.

2. Replace the frit and O-ring. Carefully reassemble the sample tube fitting and tighten by hand. Then tighten with a wrench to prevent leaks.

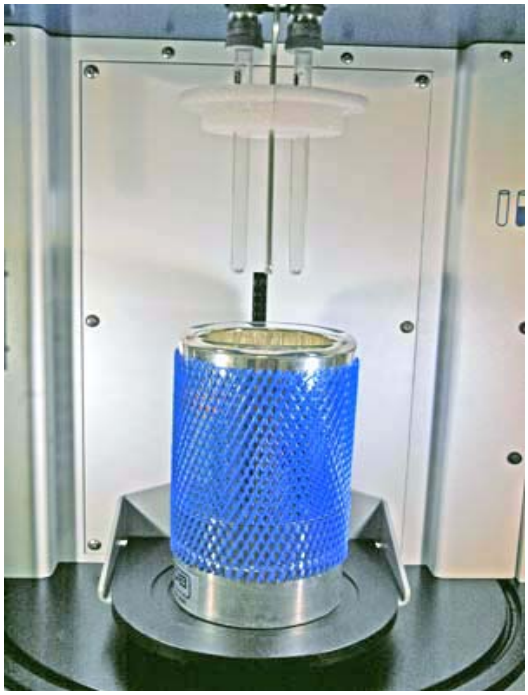
Performing a Blank Analysis for Diagnostic Purposes

A simple means of pinpointing operational problems in the Gemini is performing a blank analysis. Leaks, as well as electronic problems will result in an unsatisfactory blank analysis. Perform this test before you begin detailed troubleshooting. The data produced during the blank analysis can be very helpful in identifying the cause of a problem. For this reason, we recommend that you perform a blank analysis (described below) before contacting your Service Representative. Helium is required for performing a blank analysis since the free space needs to be measured. If helium is unavailable, contact your 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. Be sure that 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 2390*t*; place the Dewar directly on the elevator.

3. Press **Alt** + **2**; the **Select Setup** prompt is displayed.

4. Press **CHOICE** until an Unused Setup number is displayed, or choose an existing Setup.

5. Press **ENTER**; the **Select Action** prompt is displayed.

6. Press **CHOICE** until **Edit Setup** is displayed, then press **ENTER**; the **Setup type** prompt is displayed.

7. Press **CHOICE** to:

   a. Access **Analysis conditions**; enter the following parameters. Press **ENTER** after each prompt to access the next one.

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

   b. Press **CHOICE** to access **System options**. Press **ENTER** until **Request Sat. Press.?** is displayed; select the following:

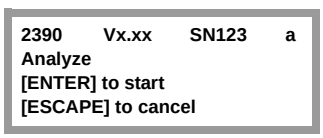
Parameter	Entry
Request Sat. Press.?	Entered

   c. Press **SAVE** to save the information you entered and return to the **Reload** prompt.

8. Press **Alt** + **4**; enter the following parameters. Press **ENTER** after each prompt to access the next one.

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

   When the following prompt is displayed, press **ENTER** to begin the analysis.



9. Review the analysis results for acceptability. They should be within the tolerances shown in the following table. If these results are not achieved, call your Service Representative.

P/Po	Tolerance in cm^3
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

Inspecting and Changing Vacuum Pump Oil

The oil in the vacuum pump should be changed every three months, when the efficiency of the vacuum pump declines (requiring increased time to reach vacuum levels), or if it becomes discolored. The oil is easily inspected to determine if a change is necessary.

These instructions are for maintenance of a vacuum pump purchased from Micromeritics. If you did not purchase your pump from Micromeritics, instructions may vary. Refer to the documentation provided with your vacuum pump if needed.

Inspecting the Oil

View the vacuum pump oil through the oil-level window. The oil level should be midway between the indicators on the oil-level window. Oil in good condition is clean, clear or light in color, and transparent.

- Change the oil if it has darkened
- Add oil if it is below the midway level

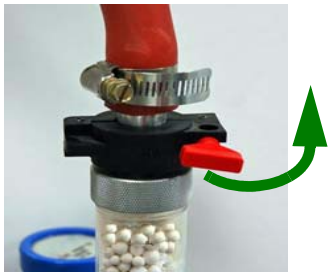
Changing or Adding Oil

Always drain the vacuum pump while the pump is warm and disconnected from the power source.

Use oil supplied by Micromeritics, or refer to the vacuum pump manual for other acceptable oils.

1. Unplug the vacuum pump from the power source.

2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.



3. Grasp the handle on top of the vacuum pump and place it on a work table.

4. Drain the used oil:

If you are adding oil, skip this step and continue with Step 5.

a. Place a waste container under the drain spout.

b. Remove the plug from the drain spout; allow the oil to drain into the waste container.

c. Replace the drain plug.

5. Remove the plug from the oil-fill port.

6. Slowly add oil to the port until the level is midway between the indicator lines in the oil-level window.

Do not allow oil to rise above the midway position. Doing so may cause oil to splash into the oil filter and contaminate it.

7. Insert the oil-fill plug and turn clockwise to tighten.

8. Check the alumina in the oil vapor trap. If most of the pellets are no longer white, replace the alumina in the oil vapor trap before reattaching the vacuum pump. Refer to **Replacing the Alumina in the Oil Vapor Trap**, page **6-13** for instructions.

9. Reconnect the vacuum pump hose.

10. Reconnect the power cord to the power source.

11. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

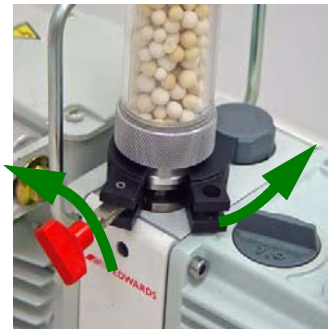
Replacing the Alumina in the Oil Vapor Trap

The activated alumina in the oil vapor trap becomes saturated during use. The alumina should be inspected periodically and replaced when most of the alumina pellets are no longer white.

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire or an explosion.

1. Disconnect the vacuum pump from the analyzer and place it on a work table (refer to **Changing or Adding Oil**, page **6-10**, steps 1, 2, and 3 for instructions).

2. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Swing the clamp open and remove the trap.



3. Remove one end fitting from the trap body; dispose of the used alumina in an appropriate manner.

4. Wash the trap body with a detergent-based soap. Rinse with water, then with isopropyl or ethyl alcohol. Set the trap aside and allow to dry thoroughly.

Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.

5. Prepare fresh alumina as follows:

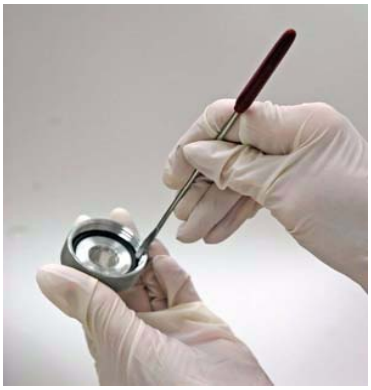
   a. Preheat a drying oven to 300 °C.

   b. Pour approximately 180 grams of fresh alumina into a glass or metal container (approximately 250 mL if a graduated beaker is used). Place the container in the oven.

   c. Bake the alumina for two hours.

   d. Remove the baked alumina from the oven and allow it to cool until lukewarm. A desiccator may be used to speed the cooling process.

6. Using a small spatula, gently pry the O-ring from the end fittings of each end of the trap body.



7. Inspect the O-rings.

   - If dusty, clean with a lint-free tissue.
   - If damaged, replace with a new O-ring.

8. Screw one of the end fittings onto the trap body.

9. Be sure the trap body is dry and the alumina is lukewarm; pour the alumina pellets into the trap until they are just below the threads of the trap body.

10. Screw the other end fitting back onto the trap and tighten securely by hand.

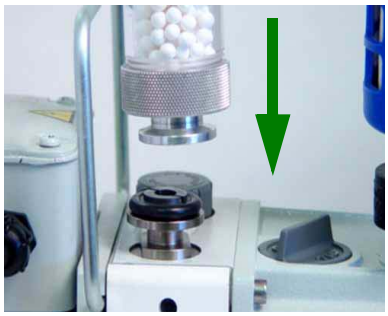
11. Lightly tap both ends of the trap body on the work surface. This will remove any remaining dust from the pellets.

12. Inspect the centering ring before placing it back on the intake port. If it appears to have flattened, replace it. A flattened centering ring can cause vacuum leaks.

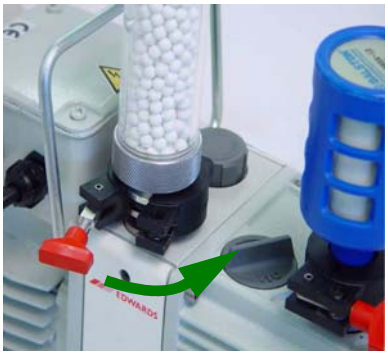
    There are two types of centering rings, use the one with the smaller opening at the intake port

Centering Ring

13. Place the trap on the centering ring.



14. Open the clamp and place it around the flange of the intake port and the flange of the trap. Swing the clamp fastening screw toward the intake port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.



15. Reconnect the hose from the analyzer to the oil vapor trap.

    a. Inspect the centering ring before placing it on the trap body. If it appears flattened, replace it with a new one. A flattened centering ring can cause vacuum leaks.

       Use this type of centering ring for the trap body.

    b. Place the clamp around the flange of the vacuum pump hose and vapor trap.

c. Swing the clamp fastening screw around until it fits into the slot on the other half of the clamp. Tighten the wing nut securely by hand.

16. Plug the pump power cord into the power source.

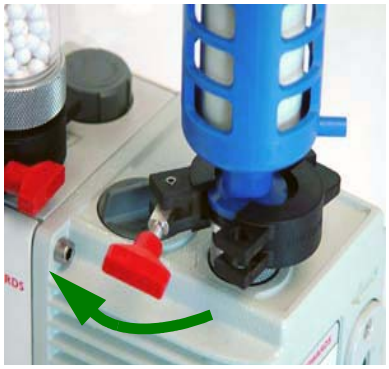
17. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the vacuum hose and oil vapor trap, and to produce efficient vacuum operations.

Changing the Vacuum Pump Exhaust Filter

The gases used by the Gemini are exhausted by the vacuum pump. An exhaust filter is installed on the exhaust port of oil-filled pumps. The filter minimizes the release of oil vapor and should be replaced when it becomes so saturated with oil that it is ineffective (refer to **Ordering Information** beginning on page **7-1**).

Exhaust filters are used to minimize the release of oil vapors. The gases are diluted substantially upon release from the vacuum pump. However, it may be desirable in some locations to provide a fume hood for protection from hazardous gases and vapors released into the work area.

1. Loosen the wing nut of the clamp at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.



2. Remove and discard the exhaust filter; do not remove the centering ring.
3. Make sure the centering ring is in place on the exhaust port.
4. Place the new filter on the centering ring.
5. Open the clamp and place it around the flange of the exhaust port and the flange of the exhaust filter. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

7. ORDERING INFORMATION

The Gemini components and accessories can be ordered using one of the following methods:

- Call our Customer Service Department at (770) 662-3636
- Access our web site at www.micromeritics.com
- Contact your local sales representative

When ordering, please use the information provided in this chapter to place your order:

Part Number	Item and Description
060-00023-00	FlowPrep 060, degasses up to six samples at up to 400 ºC with flowing gas (Requires gas regulator)
061-00023-00	VacPrep 061, degasses up to six samples at up to 400 ºC; uses flowing gas or evacuation by vacuum (evacuation requires a vacuum pump and a regulator)
065-00023-00	SmartPrep 065, Windows interface allowing programmable ramp and soak rates; degasses up to six samples with flowing gas
021-00000-00	Liquid Nitrogen Transfer System, Model 021
003-63801-01	Cable, ethernet straight-thru
300-25861-20	Dewar, 2.75 L, glass; for Gemini 2390*t*
239-31802-00	Dewar cover; for 2.75 L Dewar
004-61064-00	Dewar, 600 mL, glass; for Gemini 2390*a* and 2390*p*
236-31802-01	Dewar cover, right side; for 600 mL Dewar
236-31802-02	Dewar cover, left side; for 600 mL Dewar
239-33601-00	Dewar, 2.2 L, stainless steel; includes cover (Gemini 2390*a* and 2390*p*)
239-33602-00	Dewar, 4.0 L, stainless steel; includes cover (for Gemini 2390*t*)
236-61703-00	Sample tube, straight-wall; 3/8-in. (9.6 mm) OD x 6.1 in. (15.5-cm) long OD
236-61702-00	Sample tube, bulb; 3/8-in (0.95-cm) OD x 6.1-in. (15.5-cm) long, 3/4-in. (1.91-cm) OD bulb
238-61703-00	Sample tube, straight-wall; 3/8-in (9.6-mm) OD x 7.9 (18.0 cm) in. long (for 2390*t*)
004-32004-00	Sample tube stopper
240-25853-00	Sample tube funnel, polypropylene
236-61704-00	Filler Rod, 1/4 in. (7 mm) x 14.61 cm (5.75 in.)
236-61705-00	Filler Rod, 1/4 in. (7 mm) x 10.16 cm (4 in.)

Part Number	Item and Description
004-27046-00	Frit, for sample tube, 40 µm
004-27024-00	Frit, for balance tube, 2 µm
280-32800-00	Weighing support
240-14855-00	Sample tube rack
004-54611-00	Sample tube brush, 8-mm diameter, for straight-wall sample tube
004-54612-00	Sample tube brush, 25-mm diameter, for bulb sample tube
004-25619-03	Sample tube ferrule, 3/8 in.
004-25022-00	Sample port O-ring, size 012, 3/8-in ID
004-54618-00	Tool, for removing sample port O-ring
062-00000-11	Vacuum pump with built-in anti-suckback valve, 100/120 VAC; includes hose kit
062-00000-23	Vacuum pump with built-in, anti-suckback valve, 220/240 VAC; includes hose kit
062-00003-00	Vacuum pump, hybrid (oil-free), 220/240 VAC; includes vacuum hose, fittings, and user's guide
062-33002-00	Oil vapor trap, for vacuum pump
004-16830-00	Activated alumina, 500 grams; for oil vapor trap
004-25652-00	O-ring, for oil vapor trap (end fitting)
004-25653-00	Centering ring, NW 16/10; for vacuum pump intake port
004-25630-00	Centering ring, NW 16; for all other locations
004-16003-01	Vacuum pump oil, 1 liter
004-27040-00	Filter, for vacuum pump exhaust
004-62230-58	Gas Regulator, CGA 580, 30 psig, for helium or nitrogen
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure Relief Kit (recommended for both gas supplies); prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
290-25846-00	Gas inlet line, 1/8 in. x 6 ft long, copper
290-25846-01	Gas inlet line, 1/8 in. x 16 ft long, copper
239-42701-01	Keypad overlay, French
239-42701-02	Keypad overlay, German
239-42701-03	Keypad overlay, Spanish
239-42701-04	Keypad overlay, Italian
004-16833-00	Reference material, carbon black, ~ 30.6 m^2/g, 10 g

Part Number	Item and Description
004-16818-00	Reference material, glass, SA ~ 5.8 m^2/g, 10 g
004-16816-00	Reference material, Alumina, ~ 0.25 m^2/g, 15 g
004-16821-00	Reference material, silica alumina, ~ 215 m^2/g, 10 g
004-61065-00	Glass beads, 3 mm diameter, 90 grams
239-42801-00	Operator's Manual
236-33005-00	PrepSeal Accessory Kit; for degassing and transferring air-sensitive samples to an analysis port without contamination
239-33002-00	Gemini VII Windows software package; allows operation of the Gemini analyzer in a Windows environment, includes software and operator's manual

A. FORMS

This appendix contains the following form:

- Sample Data Worksheet

Copy and use this form as needed.

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Sample Data Worksheet

Use this worksheet to record the values necessary to calculate the sample mass.

Sample Tube Identification:_____

Sample Mass

NOTE: Record all values in grams.

	Before Degas	After Degas	After Analysis
A. Mass for empty sample tube set:	_____		
B. Sample tube set plus sample mass:	_____	_____	_____
C. Sample mass (B - A):	_____	_____	_____

Degas Information

Degas apparatus: _____

Temperature (ºC): _____

Time (hours): _____

Actual time started: _____

Actual time finished: _____

Degas notes: _____

_____

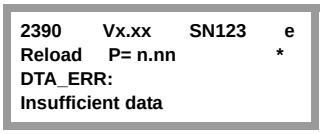
_____

_____

_____

B. ERROR MESSAGES

Error messages display in the third and fourth lines of the keypad window at the conclusion of an operation; an asterisk also displays on the second line.



Press **CHOICE** to navigate through the messages; report data are displayed after messages. Press **CLEAR** to delete the message. If you don't understand the message, look it up in this appendix for a cause and a resolution before deleting. Messages that are not cleared remain in the queue until the next analysis is performed; at that time, any remaining messages are cleared automatically.

Error messages are listed in alphabetical order. When you see NN/ZZ in a message listed below, **NN** = number of cycles completed and **ZZ** = number of cycles requested.

ANLS_ERR:
Dosing timeout

Cause: The adsorbate (or helium) gas supply was depleted by the instrument during analysis or termination.

Action: Make sure the adsorbate gas supply cylinder is not empty. Verify that the inlet valve on the gas supply cylinder is open. Verify that the gas pressure regulator setting is between 15 and 18 psig (103.4 and 124.1 kPa).

ANLS_ERR:
Elevator timeout

Cause: Elevator did not reach its limit in the expected amount of time.

Action A: Ensure there are not obstructions in the path of the elevator.

Action B: Contact your Micromeritics service representative.

ANLS_ERR:
Evacuation timeout

Cause: The analyzer failed to reach a pressure level of 10 mmHg within the specified evacuation time plus three minutes; or, the analyzer failed to reach a pressure level of 5 mmHg within an additional three minutes.

Action: Reattach the sample and balance tubes, making sure both are securely in the fitting. Verify that the vacuum pump is operating correctly. If these actions do not correct the problem, degas the sample again and repeat the analysis. If the analyzer continues to display this message, call a Micromeritics Service Representative.

ANLS_ERR: Operator
cancelled operation

Cause: Operator cancelled the current operation.

Action: Restart the operation when appropriate.

ANLS_ERR:
Po fill timeout

Cause A: Adequate pressure in the Po tube could not be reached.

Action A: Confirm that the gas supply is sufficient.

Cause B: The Dewar does not contain enough cryogen to take accurate readings in the Po tube.

Action B: Refill the Dewar with liquid nitrogen and restart the analysis.

ANLS_ERR:
Pressure overrange

Cause: The sample pressure transducer detected a pressure measurement that is too high because the liquid nitrogen level in the Dewar is too low or a valve is leaking.

Action: Add liquid nitrogen to the Dewar. If this does not correct the problem, contact the appropriate service personnel.

ANLS_ERR:
Pressure underrange

Cause: A test of the sample pressure transducer was made during evacuation. The test showed a low reading. The analysis will continue.

Action: Call a Micromeritics Service Representative.

ANLS_ERR: Quantity
adsorbed underrange

Cause A: The analysis was canceled because the number of quantity adsorbed A/D counts was too low at the end of equilibration of free space or an analysis point.

Action A: Reduce volume of sample used.

Cause B: The analysis was canceled because the number of volume adsorbed A/D counts was too low when quantity adsorbed was recorded during a scanning run.

Action B: Reduce volume of sample used.

Cause C: Balance tube is too large.

Action C: Match size of balance tube to that of the sample tube.

ANLS_ERR: Quantity adsorbed overrange

Cause: The rate of adsorption by the sample exceeded the response capacity of the analyzer, preventing the normal refilling of the gas reservoirs.

Action: Repeat analysis with a smaller amount of sample in the tube.

DTA_ERR: Insufficient data

Cause: BET or Langmuir multipoint surface area analysis was requested but fewer than two data points were selected for surface area calculations.

Action: Return to Review mode and select more points for calculation.

DTA_ERR: No data to compute

Cause A: An automatic operation was canceled before all necessary data could be collected.

Action A: Restart the automatic operation.

Cause B: All data points have been excluded in **Review** mode.

Action B: Return to **Review** mode and include some data points.

Cause C: A report has been requested and no analysis data have been collected or previously collected data have been deleted.

Action C: Perform a sample analysis to collect data.

HW_ERR:Reset to factory defaults

Cause A: The operator pressed **.** (decimal) during analysis module power-up and initialization. All previously stored data have been deleted and all parameters have been reset to factory defaults. Stored Setups are not affected.

Action A: Reset parameters if desired.

PRT_ERR: Timeout failed to respond

Cause: The printer took longer than 30 seconds to acknowledge receipt of data from the analyzer.

Action: Check to make sure the printer is properly connected to the analyzer, is turned on, has paper, and is on line.

SYS_ERR: Power Fail NN/ZZ completed

Cause: A power failure occurred and, when power was restored, the automatic operation was canceled. The third line displays the number of data points finished.

Action: Restart the automatic operation if desired.

TRN_ERR: Timeout, failed to respond

Cause: The receiving device took longer than 10 seconds to acknowledge receipt of data from the analyzer.

Action: Make sure the receiving device is properly connected to the analyzer RS-232 port and is turned on. Verify that the serial transmission parameters controlling the receiving device correspond with the data transmission parameters specified in the Setup mode.

USR_ERR:
No data to review

Cause: The operator selected the **Review** function and no analysis data have been collected, or previously collected data have been deleted.

Action: Perform an analysis to create data to review.

USR_ERR:
Out of range

Cause: Entered value is outside the range of values for this prompt.

Action: Enter a value within the acceptable range for this prompt.

USR_ERR:
Venting Po tube

Cause: Excessive pressure detected in the Po tube at the **Reload** prompt.

Action: If you continue to receive this message, contact your Micromeritics service representative.

Valve failure:
[ENTER] to retry

Cause: Valves failed to respond.

Action: If you continue to receive this message, contact your Micromeritics service representative.

C. CALCULATIONS

This appendix includes the calculations used for data reduction.

Free-Space Correction Algorithms

Free space differential compensation for the Gemini is accomplished in two steps. First, the free space correction, C_f (cm^3 STP/mmHg) is determined. Second, the correction is applied to quantities adsorbed during analysis.

Determining the Free-Space Correction

The free space correction C_f (cm^3 STP/mmHg) can be **Measured** or **Calculated**.

Measured

Evacuate the sample and balance ports, charge the reservoirs with helium, and dose to 760 mmHg. Measure the gas quantity differential, Q_h (cm^3 STP), and the absolute sample pressure, P_h (mmHg). Record the free space correction:

$$C_f = \frac{Q_h}{P_h}$$

Calculated

This method requires using a bath of liquid nitrogen temperature. It should not be selected for free-space correction if a liquid nitrogen bath is not being used.

Measure the free space correction as above, except use an empty sample tube of the same volume as that to be used during subsequent sample analyses. Calculate and record the system volume correction Q_{sys} (cm^3 STP):

$$Q_{sys} = 760\ mmHg \times \frac{Q_h}{P_h}$$

Calculate the sample quantity Q_{sam} (cm^3) from independently determined sample mass M_{sam} (g) and sample density D_{sam} (g/cm^3):

$$Q_{sam} = \frac{M_{sam}}{D_{sam}}$$

Calculate the quantity of gas displaced by the sample at liquid nitrogen temperature and standard pressure:

$$Q_{gas} = Q_{sam} \times \frac{273.13K}{77.15K}$$

Calculate the free space correction due to both the system volume correction and the gas displaced by the sample in cm^3 STP/mmHg:

$$C_f = \frac{Q_{sys} - Q_{gas}}{760\ mmHg}$$

Applying the Free-Space Correction to Quantities Adsorbed

Measure the uncorrected quantity adsorbed Q_{raw} (cm^3 STP) and the pressure at which this quantity is adsorbed P_{ads} (mmHg). Use the independently determined adsorbate nonideality correction Cn (%/atm) and the free space correction C_f (cm^3 STP/mmHg) to calculate the adsorbate quantity correction Q_{cor} (cm^3 STP) for this point:

$$Q_{cor} = C_f \times P_{ads} \times \left[1 + \left(\frac{P_{ads}(C_n)}{100\% \times \frac{760\ mmHg}{atm}}\right)\right]$$

Apply the correction to the uncorrected quantity adsorbed to obtain the corrected quantity adsorbed Q_{ads} (cm^3 STP):

$$Q_{ads} = Q_{raw} - Q_{cor}$$

BET Surface Area Calculations

For each point designated for surface area calculations, the BET[1] transformation is calculated as follows:

$$B_1 = \frac{P_{rel_1}}{(1.0 - P_{rel_1}) \times N_{ads_1}}$$

where

B_I = units of g/cm^3 STP

$Prel_I$ = relative pressure

$Nads_I$ = amount of gas adsorbed after equilibrating Ith dose (cm^3 STP)

A least-squares fit is performed on the (P_{relI} ,B_I) designated pairs where P_{relI} is the independent variable and B_I is the dependent variable. The following are calculated:

- Slope (S g/cm^3 STP)
- Y-intercept (Y_{INT} g/cm^3 STP)
- Error of the slope (S_{ERR} g/cm^3 STP)
- Error of the y-intercept (YI_{ERR} g/cm^3 STP)
- Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

BET Surface Area (m^2/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times (S + Y_{INT})}$$

where

CSA = analysis gas molecular cross-sectional area (nm^2)

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

1 Brunauer, S.; Emmett, P.H.; and Teller, E., J.; *Am. Chem. Soc. 60*, 309 (1938)

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

Error of the BET Surface Area (m^2/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}{}^2 + YI_{ERR}{}^2)^{0.5}}{Y_{INT} + S}$$

Single Point Surface Area (m^2/g):

$$S1PT = Va(1 - Pr) \times 4.35 \times \frac{CSA}{0.162}$$

where

Pr	=	pressure closest to 0,.3 of the relative pressure points designated for surface area calculations
Va	=	volume corresponding to Pr
0.162	=	nitrogen molecule cross-sectional area (nm^2)

Langmuir Surface Area

For each point designated for surface area calculations, the Langmuir[1] transformation is calculated as follows:

$$L_I = \frac{P_{rel_I}}{N_{ads_I}}$$

where L_I is in units of g/cm^3 STP

A least-squares fit is performed on the (P_{relI} ,L_I) designated pairs where P_{relI} is the independent variable and L_I is the dependent variable. The following are calculated:

- Slope (S g/cm^3 STP)
- Y-intercept (Y_{INT} g/cm^3 STP)
- Error of the slope (S_{ERR} g/cm^3 STP)
- Error of the y-intercept (YI_{ERR} g/cm^3 STP)
- Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Langmuir Surface Area (m^2/g):

$$SA_{LAN} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times S}$$

where

CSA = analysis gas molecular cross-sectional area (nm^2)

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{S}$$

Langmuir C Value:

$$C = [(Y_{INT})(\ V_M)]^{-1}$$

1 Langmuir, I., *J. Am. Chem. Soc. 38*, 2267 (1916); *J. Am. Chem. Soc. 40*, 1361 (1918); *Phys Rev. 8*, 149 (1916)

Error of the Langmuir Surface Area (m^2/g):

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

t-Method Calculations

For each point designated for t-Plot[5] calculations, the following calculations are made:

Thickness for the I^{th} point (Å):

$$t_I = HP1 \times \left[\frac{HP2}{\ln(P_{rel_I})}\right]^{HP3} \quad \text{(Halsey[1])}$$

or

$$t_I = \left[\frac{HJP1}{HJP2 - \log(P_{rel_I})}\right]^{HJP3} \quad \text{(Harkins and Jura[2])}$$

or

$$T_1 = CB1(Prel_1 \times Po)^2 + CB2(Prel_1 \times Po) + CB3 \quad \text{(Magee-STSA[3])}$$

where:

t_I	=	thickness for I^{th} point
HP1	=	Halsey parameter #1
HP2	=	Halsey parameter #2
HP3	=	Halsey parameter #3
HJP1	=	Harkins and Jura parameter #1
HJP2	=	Harkins and Jura parameter #2
HJP3	=	Harkins and Jura parameter #3
MP1	=	Magee-STSA parameter #1
MP2	=	Magee-STSA parameter #2
MP3	=	Magee-STSA parameter #3
P_{relI}	=	relative pressure for the Ith point (mmHg)

1 Halsey, G., *J. Chem. Phys. 16*, 931-937 (1948)
2 Harkins, W.C. and Jura, G., *J. Chem. Phys 11*, 431 (1943)
3 Magee, Ricky, Columbian Chemicals Company, personal communications

A least-squares analysis fit is performed on the (t_I, N_{adsI}) data pairs where t_I is the independent variable and $Nads_I$ is the dependent variable. Only the values of t_I between t_{MIN} and t_{MAX}, the minimum and maximum thickness, are used. The following are calculated:

- Slope (S cm^3/g-Å STP)
- Y-intercept (Y_{INT} cm^3/g STP)
- Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

External Surface Area (m^2/g):

$$SA_{EXT} = \frac{(S\ cm^3/g - A\ STP) \times (10^{10}\ A/m) \times (D\ cm^3\ liquid/cm^3\ STP)}{F \times (10^6\ cm^3/m^3)}$$

where

F = surface area correction (Report Options)
D = density conversion factor (cm^3 liquid/cm^3 STP)

Micropore Surface Area (m^2/g):

$$SA_{\mu P} = SA_{TOT} + SA_{EXT}$$

where SA_{TOT} is the BET surface area if a BET report was requested, or Langmuir surface area.

Micropore Volume (cm^3 liquid/g):

$$V_{\mu P} = (Y_{INT}\ cm^3/g\ STP) \times (D\ cm^3\ liquid/cm^3\ STP)$$

BJH Pore Volume and Area Distribution

For adsorption data, the relative pressure and volume adsorbed data point pairs collected during an analysis must be arranged in reverse order from which the points were collected during analysis. All calculations are performed based on a desorption model, regardless of whether adsorption or desorption data are being used.

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P_r), volume adsorbed (V_a) pairs from (Pr_1, Va_1) to (Pr_N, Va_N) where (Pr_N = 0, Va_N = 0) is assumed as a final point. Each data pair represents an interval boundary (or desorption step boundary) for intervals i=1 to i=N-1 where N = total number of (P_r, Q_a) pairs.

Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin[1] equation. After the core has evaporated, a layer of adsorbate remains on the wall of the pore. The thickness of this layer is calculated for a particular relative pressure from the thickness equation. This layer becomes thinner with successive decreases in pressure, so that the measured quantity of gas desorbed in a step is composed of a quantity equivalent to the liquid cores evaporated in that step plus the quantity desorbed from the pore walls of pores whose cores have been evaporated in that and previous steps. Barrett, Joyner, and Halenda[2] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used on the Gemini is an implementation of the BJH method.

Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

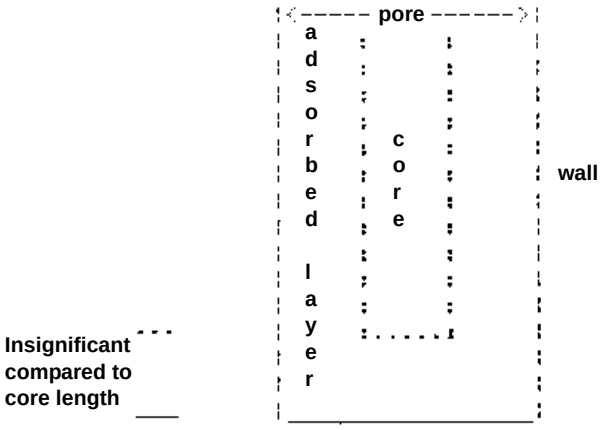
a. The ***core*** - evaporates all at once when the critical pressure for that radius is reached; the relationship between the core radius and the critical pressure is defined by the Kelvin equation.

b. The ***adsorbed layer*** - composed of adsorbed gas that is stripped off a bit at a time with each pressure step; the relationship between the thickness of the layer and the relative pressure is defined by the thickness equation.

1 Kelvin, J. (published under the name of Sir William Thomson), *Phil. Mag. 42*, 448-452 (1871).

2 Barrett, E.P.; Joyner, L.S.; and Halenda, P.P., *J. Am. Chem. Soc. 73*, 373-380 (1951).

c. The ***walls of the cylindrical pore*** - the diameter of the empty pore is required to determine the pore volume and pore area. End area is neglected.



Calculations

The quantities adsorbed (V_a) are converted to the liquid equivalent volumes (V_1, cm^3/g):

$$VI_I = (V_{a_I})(D)$$

where D is the Density Conversion Factor.

The relative pressure (Pr_1) is assumed to be close to unity so that substantially all the pores in the sample are filled.

The corresponding Kelvin core radius is calculated. Only pores smaller than this size will be included:

$$Rc_I = \frac{-A}{(1+F)[\ln(Pr_I)]}$$

where

A = adsorbate property factor
F = fraction of pores open at both ends; assumed to be zero for desorption

This radius will be adjusted for the thickness of the adsorbed layer during subsequent calculation steps.

The thickness of the remaining adsorbed layer at this relative pressure is calculated:

$$Tw_1 = HP1\left[\frac{HP2}{\ln(Pr_1)}\right]^{HP3}$$

where

HP1, HP2, and HP3 are Halsey Parameters 1, 2, and 3 (respectively) from the Halsey Thickness Equation prompt.

These calculations illustrate the use of the Halsey thickness equation. If the Harkins and Jura equation was selected, substitute the following wherever the thickness equation appears:

$$Tw_1 = \left[\frac{HJ1}{HJ2 - \log(Pr_1)}\right]^{HJ3}$$

where

HJ1, HJ2, and HJ3 are Harkins and Jura Parameters 1, 2, and 3 (respectively) from the Harkins-Jura Thickness Equation prompt.

The following calculations (a-c) are made for each relative pressure interval based on the increment of volume desorbed during that interval. The variable I refers to the interval number, that is I=1 for the first interval from Pr_1 to Pr_2, and so on. J refers to each previous interval during which new pores were found. K refers to the total number of intervals in which new pores have been found. K is also the number of lines reported on the BJH table for collected data.

a. The thickness of the adsorbed layer at the end of the interval is calculated as follows:

$$Tw_{I+1} = HP1\left[\frac{HP2}{\ln(Pr_{I+1})}\right]^{HP3}$$

(For the last pressure interval from the lowest Pr_I to zero relative pressure, Tw_{I+1} = 0.)

For the first pressure interval, there are no previously opened pores so the volume desorbed from walls of previously opened pores is zero ($Vd_1 = 0$), and the remainder of Step a is skipped.

The change in thickness of the wall layer due to desorption from previously opened pores is calculated:

$$\Delta Tw = Tw_1 - Tw_{I+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all previously opened pores:

$$CSA_J = \pi[(Rc_j + \Delta Tw)^2 - Rc_j{}^2](10^{-16}\ cm^2/A^2)$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_I = \sum(Lp_J)(CSAa_J) \text{ for all previously opened pores}$$

where LP_J = length of previously opened pores as calculated in Step b(2).

b. The physical processes occurring for this pressure interval are determined as follows:

(1) If Vd_I is greater than the current increment of volume desorbed (Vl_I - Vl_{I+1}), desorption from walls only is occurring. Total surface of walls exposed thus far (cm^2/g) is calculated as follows:

$$SA_W = \sum\pi(LP_J)(D_{avg_J})(10^{-8}\ cm/A) \quad \text{for all previously opened pores}$$

where

D_{avgJ} = weighted average pore diameter calculated below in Step b(2).

A new layer thickness (ΔTw) that will not overcompensate for the actual volume desorbed in this interval is calculated:

$$\Delta Tw = \frac{(V1_I - V1_{I+1})(10^8\ A/cm)}{SAw_I}$$

Since no cores are evaporated in this pressure interval, no new pores are revealed. Thus no ending Kelvin radius and average pore diameter are calculated for this interval. Note that this means the report may have fewer tabulated intervals on the collected data report than experimental pressure intervals.

(2) If Vd_I is less than the volume increment desorbed during this interval $(Vl_1 - Vl_{l+1})$, the remaining volume is due to new pores with core evaporation taking place in this interval. K, the number of intervals with new pores exposed, is increased by 1. (For the interval from the lowest Pr_1 to zero relative pressure, no new pore volume is calculated and the rest of Step b is skipped.)

The volume desorbed from newly opened pores in this interval is calculated as follows:

$$Vc_I = (VI_I - VI_{I+1}) - Vd_I$$

The Kelvin radius for the end of the interval is calculated as follows:

$$Rc_{K+1} = \frac{-A}{(1+F)[\ln(Pr_{I+1})]}$$

All new pores opened in this interval are represented by one pore having a length-weighted average pore diameter and a corresponding length sufficient to account for the required volume of adsorbate. The weighted average pore diameter is calculated as follows:

$$D_{avg_K} = \frac{(2)(Rc_K + Rc_{K+1})(Rc_K)(Rc_{K+1})}{Rc_K{}^2 + Rc_{K+1}{}^2}$$

D_{avgK} is the diameter of a pore which would have a surface area that is the average of the areas for pores with radius Rc_K and Rc_{K+1}, if its length was the mean of the lengths at those radii.

The relative pressure corresponding to D_{avgK} is calculated as follows:

$$P_{avg_K} = \ln^{-1}\left[\frac{-2A}{(1+F)(D_{avg_K})}\right]$$

The thickness of the adsorbed layer at this pressure is calculated as follows:

$$Tw_{avg_K} = HP1\left[\frac{HP2}{\ln(P_{avg_K})}\right]^{HP3}$$

The decrease in thickness of the wall layer by desorption from the walls of new pores during the lower portion of the pressure interval is calculated as follows:

$$\Delta Td = Tw_{avg_K} - TW_{I+1}$$

The cross-sectional area of the newly opened pores is calculated as follows:

$$CSAc_K = \left[\frac{Davg_K}{2} + \Delta Td\right]^2 (10^{-16}\ cm^2/A^2)$$

The length of the newly opened pores is calculated as follows:

$$LP_K = \frac{Vc_I}{CSAc_K}$$

c. Pore diameters and radii are adjusted for the change in thickness of the adsorbed wall layer during this interval. If new pores were opened during this interval, the average diameter is adjusted by the change in layer thickness during the second portion of the desorption interval as follows:

$$D_{avg_{Knew}} = D_{avg_{Kold}} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as follows:

$$D_{avg_{Jnew}} = D_{avg_{Jold}} + 2(\Delta Tw)$$

(not including D_{avgK})

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as follows:

$$Rc_{Jnew} = Rc_{Jold} + \Delta Tw$$

for all except Rc_{K+1}.

Steps a to c are repeated for each pressure interval.

After the above calculations have been performed, the diameters corresponding to the ends of the intervals are calculated as follows:

$$Dp_J = 2(rc_J)$$

for all Rc_J including Rc_{K+1}.

The remaining calculations are based on $DP_{(I)}$, $D_{avg(I)}$, and $LP_{(I)}$. These calculations are only done for $D_{avg(I)}$ values that fall between the Minimum BJH diameter and the Maximum BJH diameter specified in report options.

1) Incremental Pore Volume (Vp_I, cm^3/g):

$$Vp_I = \pi(Lp_I)\left[\frac{D_{avg_I}}{2}\right]^2 10^{16}\ cm^2/A^2$$

2) Cumulative Pore Volume ($Vp_{CUM(1)}$, cm^3/g):

$$VP_{CUM_{(1)}} = \sum Vp_J\ for\ (J \le 1)$$

3) Incremental Surface Area (SA_I, m^2/g):

$$SA_I = \pi(LP_I)(10^{-2}\ m/cm)(D_{avg_I})(10^{-10}\ m/A)$$

4) Cumulative Surface Area ($SA_{CUM(I)}$, m^2/g):

$$SA_{CUM_{10}} = \sum SA_J\ for\ J \le 1$$

5) dV/dD pore volume (dV/dD_I, cm^3/g-A):

$$dV/dD_I = \frac{VP_I}{Dp_I - Dp_{I+1}}$$

6) dV/dlog(D) pore volume ($dV/dlog(D)_I$, cm^3g):

$$'Dv/d\log(D)_I = VP_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right.$$

7) dA/dD pore area (dA/dD_I, m^2/g-A):

$$dA/dD_I = \frac{SA_I}{Dp_I - Dp_{I+1}}$$

8) dA/dlog(D) pore area [$dA/dlog(D)_I$, m^2/g]:

$$dA/d\log(D)_I = SA_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right)$$

For fixed pore size tables (if selected), the following calculations are performed:

1) Average Fixed Pore Size (DF_{avgJ}, A):

$$DF_{avg_J} = \frac{Dp_{F_J} + Dp_{F_{J+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with between the Minimum BJH diameter and the Maximum BJH diameter.

2) Cumulative Pore volume (VpF_{CUMI}, cm^3/g):

$$VpF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = Dp_{J+1} and Y = VP_{CUMJ}, using an AKIMA semi-spline interpolation.

3) Incremental Pore Volume (VpF_I, cm^3/g):

$$VpF_I = VpF_{CUM_I} - VpF_{CUM_{I-1}}$$

where $VpF_{CUM0} = 0$.

4) Cumulative Surface Area (SAF_{CUMI}, m^2/g):

$$SAF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = Dp_{J+1} and Y = SA_{CUMJ}.

5) Incremental Surface Area (SAF_I, m^2/g):

$$SAF_I = SAF_{CUM_I} - SAF_{CUM_{I-1}}$$

where $SAF_{CUM0} = 0$.

6) dV/dD pore volume ($dV/dDpF_I$, cm^3/g-A):

$$dV/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = dV/dD_J.

7) dV/dlog(D) pore volume [$dV/dlog(DpF)_I$, cm^3/g]:

$$dV/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = $dV/dlog(D)_J$.

8) dA/dD pore area ($dA/dDpF_I$, m^2/g-A):

$$dA/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = dA/dD_J.

9) dA/dlog(D) pore area [$dA/dlog(DpF_I)$, m^2/g]:

$$dA/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = $dA/dlog(D)_J$.

Compendium of Variables

V_a = quantity adsorbed expressed as a volume (cm^3/g STP)
V_l = liquid equivalent volume of volume adsorbed (cm^3/g)
D = density conversion factor (cm^3/cm^3 STP)
P_r = relative pressure
D_p = pore (or core) diameter (A)
R_c = Kelvin radius (A) of core
A = adsorbate property factor
F = fraction of pores open at both ends
ΔTw = thickness of adsorbed layer desorbed during interval (A)
Tw = thickness of remaining adsorbed wall (A)

HP1, HP2, and HP3 are Halsey Parameters from the Halsey Thickness Equation prompt.

HJ1, HJ2, and HJ3 are Harkins and Jura Parameters from the Harkins and Jura Thickness Equation prompt.

V_d = volume of gas desorbed from walls of previously opened pores (cm^3/g)
D_{avg} = average pore diameter (A)
CSA_a = annular cross-sectional area of the desorbed layer (cm^2)
CSA_c = cross-sectional area of opening of newly opened pores (cm^2)
SA_w = total surface area of walls exposed (cm^2/g)
ΔTd = thickness of layer desorbed from walls of newly opened pores (A)
V_c = volume desorbed from cores of newly opened pores (cm^3/g)
L_p = length of pore (cm/g)

Single-Point Total Pore Volume

The liquid equivalent of the designated quantity adsorbed is calculated; this is the total pore volume (cm^3/g):

$$V_{TOT} = (Q_a)(D)$$

where

D = density conversion factor
Q_a = volume adsorbed

Horvath-Kawazoe

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/Po) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito/Foley)
- Sphere Pore Geometry (Cheng/Yang)

Slit Pore Geometry (original HK)

When you use the original Horvath-Kawazoe[1] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\frac{P}{Po} = \frac{K}{RT}\times\frac{IP\times 10^{32}\ JA^4/J\ cm^4}{\sigma^4\times(L-2\times d_0)}\times\left[\frac{\sigma^4}{3\times(L-d_0)^3}-\frac{\sigma^{10}}{9\times(L-d_0)^9}-\frac{\sigma^4}{3\times d_0^3}+\frac{\sigma^{10}}{9\times d_0^9}\right]$$

where:

K = Avogadro's number (6.023×10^{23})
R = gas constant (8.31441×10^7 ergs/mole K)
T = analysis bath temperature (K), from an entered or calculated value
σ = gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

where:

Z_S = sample equilibrium diameter at zero interaction energy (Å)
Z_A = zero interaction energy diameter

d_0 = $\frac{D_A + D_S(A)}{2}$

where:

D_A = molecular diameter of adsorbate (Å)
D_S = diameter of sample atom (Å)

L = pore width (nucleus to nucleus) (Å)
P = equilibrium pressure (mmHg)
Po = saturation pressure (mmHg)
IP = interaction parameter (10^{-43} ergs-cm^4)

1 Horvath, G. and Kawazoe, K., *J. Chem. Eng. Japan 16(6)*, 470 (1983).

Cylinder Pore Geometry (Saito/Foley)

When you use the Saito-Foley[1] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{3}{4}\frac{\pi K}{RT} \times \frac{IP \times 10^{32}\ JA^4/J\ cm^4}{{d_0}^4} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1-\frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} - \beta_k\left(\frac{d_0}{r_p}\right)^4\right\}\right]$$

where:

K = Avogadro's number (6.023×10^{23})
R = gas constant (8.31441×10^7 ergs/mole K)
T = analysis bath temperature (K), from an entered or calculated value
L = pore width (nucleus to nucleus) (Å)
P = equilibrium pressure (mmHg)
Po = saturation pressure (mmHg)
IP = interaction parameter (10^{-43} ergs-cm^4)

$d_0 = \frac{D_A + D_S}{2}$

where:
D_A = molecular diameter of adsorbate (Å)
D_S = diameter of sample atom (Å)

$\alpha_k = \left(\frac{-4.5-k}{k}\right)^2 \alpha_{k-1}, \alpha_0 = 1.0$

$\beta_k = \left(\frac{-1.5-k}{k}\right)^2 \beta_{k-1}, \beta_0 = 1.0$

r_p = radius of the cylindrical pore, $\frac{L}{2}$

1 Saito, A. and Foley, H.C., *AlChE Journal 37(3)*, 429 (1991).

Sphere Pore Geometry (Cheng/Yang)

When you use the Cheng/Yang[1] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{6(N_1\varepsilon^*_{12} + N_2\varepsilon^*_{22})L^3 \times 10^{32}\ J\ A^4/J\ cm^4}{RT(L-d_0)^3}$$

$$\left[-\left(\frac{d_0}{L}\right)^6\left(\frac{1}{12}T_1 + \frac{1}{8}T_2\right) + \left(\frac{d_0}{L}\right)^{12}\left(\frac{1}{90}T_3 + \frac{1}{80}T_4\right)\right]$$

where

R = gas constant (8.31441 x 10^7 ergs/mole K)

T = analysis bath temperature (K), from an entered or calculated value

$d_0 = \frac{D_A + D_S}{2}$

where:

D_A = molecular diameter of adsorbate (Å)

D_S = diameter of sample atom (Å)

L = pore width (nucleus to nucleus) (Å)

P = equilibrium pressure (mmHg)

Po = saturation pressure (mmHg)

$N_1 = 4\pi L^2 N_S$, where N_S = number of sample atoms/cm^2 at monolayer

$N_2 = 4\pi (L - d_0)^2 N_A$, where N_s = number of gas molecules/cm^2

$\varepsilon^*_{12} = \frac{A_S}{4{d_S}^6}$, where $A_S = \frac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$

$\varepsilon^*_{22} = \frac{A_A}{4{D_A}^6}$, where $A_A = \frac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$

$T_1 = \frac{1}{(1-S)^3} - \frac{1}{(1+S)^3}$

$T_2 = \frac{1}{(1+S)^2} - \frac{1}{(1-S)^2}$

1 Cheng, Linda S. and Yang, Ralph T., *Chemical Engineering Science 49(16)*, 2599-2609 (1994).

$$T_3 = \frac{1}{(1-S)^9} - \frac{1}{(1+S)^9}$$

$$T_4 = \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8}$$

where $S = \frac{L - d_0}{L}$

Cheng/Yang Correction

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometrics:

$$\ln\left(\frac{P}{Po}\right) = G(L) - \left[1 - \frac{1}{\theta}\ln\left(\frac{1}{1-\theta}\right)\right]$$

where:

- G(L) = one of the Horvath-Kawazoe equations given above
- θ = degree of void filling; θ is estimated by first computing the monolayer capacity (V_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the volume adsorbed over V_m.

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients -

$$A_S = \frac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$$

$$A_A = \frac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$$

where:

MC^2	=	kinetic energy of electron (0.8183×10^{-6} erg)
α_S	=	polarizability of sample atoms (cm^3)
α_A	=	polarizability of gas molecule (cm^3)
χ_S	=	diamagnetic susceptability of sample atom (cm^3)
χ_A	=	diamagnetic susceptability of gas molecule (cm^3)

$$IP = (N_A \times A_A) + (N_S \times A_S)$$

where:

N_A	=	number of gas molecules/cm^2 at monolayer
N_S	=	number of sample atoms/cm^2

Refer to **Interaction Parameter Components**, page **C-25** for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):
(Shell to Shell)

$$AL_I = L_I - DS$$

Cumulative Pore Volume (cm^3/g):

$$V_{CUM_I} = V_I \times D$$

where

D = density conversion factor (cm^3 liquid/cm^3 STP)

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_I} = \frac{V_{CUM_I} - V_{CUM_{I-1}}}{AL_I - AL_{I-1}}$$

Median Pore Width (Å):

$$V_{HALF} = \frac{V_{CUM_N}}{2}$$

$$D_{MED} = \exp_{10}\left[\log(D_L) + [\log(V_{HALF}) - \log(V_L)] \times \frac{\log(D_G) - \log(D_L)}{\log(V_G) - \log(V_L)}\right]$$

where

V_{CUMN} = total cumulative pore volume (V_{CUMI}) for points designated for Horvath-Kawazoe calculations
V_{HALF} = 50% of total cumulative pore volume
V_L = cumulative pore volume (V_{CUMI}) for first point less than V_{HALF}
V_G = cumulative pore volume (V_{CUMI}) for first point greater than V_{HALF}
D_L = pore width (L_I) that corresponds to V_L
D_G = pore width (L_I) that corresponds to V_G

Interaction Parameter Components

Table C-1. Interaction Parameters

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value*
Argon	87.3	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.61 5.89 3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.20 9.20 5.08
	273.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.34 9.35 5.22
	194.65	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.72 9.72 5.60
Nitrogen	77.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.84 6.53 3.49
*The interaction parameter is entered in Report Options: **Interaction parameter:** (calculated value) x 10^{-43} ergs-cm^4			

The following values were used to calculate the values in Table C-1.

Carbon-Graphite

$D_S = 3.40$
$N_S = 3.845 \times 10^{15}$
$\chi_S = 1.05 \times 10^{-29}$ (Ross/Olivier)
13.5×10^{-29} (Horvath/Kawazoe, implicit)
$\alpha_S = 1.02 \times 10^{-24}$

Zeolite

$D_S = 3.04$
$N_S = 3.75 \times 10^{15}$
$\chi_S = 1.94 \times 10^{-29}$
$\alpha_S = 0.85 \times 10^{-24}$

Nitrogen

$D_A = 3.00$
$N_A = 6.71 \times 10^{14}$
$\chi_A = 3.6 \times 10^{-29}$
$\alpha_A = 1.76 \times 10^{-24}$

Argon

$D_A = 2.95$
$N_A = 7.608 \times 10^{14}$
$\chi_A = 3.22 \times 10^{-29}$
$\alpha_A = 1.63 \times 10^{-24}$

Carbon Dioxide

D_A = 3.23
N_A = 4.567×10^{14} (25 ºC)
5.45×10^{14} (0 ºC)
7.697×10^{14} (-78 ºC)
χ_A = 5.0×10^{-29}
α_A = 2.7×10^{-24}

D_A values are from van der Waal's constant.
N_A values are from liquid densities.
χ and α values are derived from data found in Ross and Olivier[1].

The physical parameters referenced in Saito/Foley are as follows:

Aluminophosphate

D_S = 2.60
N_S = 1.48×10^{15}
χ_S = 1.3×10^{-29}
α_S = 2.5×10^{-24}

Aluminosilicate

D_S = 2.76
N_s = 1.31×10^{15}
χ_S = 1.3×10^{-29}
α_S = 2.5×10^{-24}

1 Ross and Olivier, J.P., "On Physical Adsorption," J. Wiley and Sons, New York (1964).

Spherical Parameters

The spherical parameters result from the following calculations:

$$\text{Adsorptive spherical parameter} = N_A \times \alpha_A \times \chi_A$$

$$\text{Adsorbent spherical parameter} = N_S \times \frac{\alpha_S \times \alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$$

where:

N_A = number of gas molecules/cm^2 at monolayer
N_S = number of sample atoms/cm^2
α_A = polarizability of gas molecule (cm^3)
α_S = polarizability of sample atoms (cm^3)
χ_A = diamagnetic susceptability of gas molecule (cm^3)
χ_S = diamagnetic susceptability of sample atom (cm^3)

Table C-2. Adsorptive and Adsorbent Spherical Parameters

Gas	Bath Temperature (K)	Sample Type	Spherical Parameter Calculated Value*	
			Adsorptive	Adsorbent
Argon	87.3	Carbon (Ross/Olivier value)	3.99313	4.32629
		Carbon (Horvath/Kawazoe value)	3.99313	11.00271
		Zeolite	3.99313	5.50177
Carbon Dioxide	298.15	Carbon (Ross/Olivier value)	6.16545	7.00604
		Carbon (Horvath/Kawazoe value)	6.16545	17.22493
		Zeolite	6.16545	8.79855
	273.15	Carbon (Ross/Olivier value)	7.35750	7.00604
		Carbon (Horvath/Kawazoe value)	7.35750	17.22493
		Zeolite	7.35750	8.79855
	194.65	Carbon (Ross/Olivier value)	10.39095	7.00604
		Carbon (Horvath/Kawazoe value)	10.39095	17.22493
		Zeolite	10.39095	8.79855
Nitrogen	77.15	Carbon (Ross/Olivier value)	4.25146	4.72674
		Carbon (Horvath/Kawazoe value)	4.25146	12.24482
		Zeolite	4.25146	6.05156

*The spherical parameters are entered in Report Options:

Spherical parameter: (calculated value) x 10^{-38} cm^4

D. 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. The following tables define the formats.

In all tables, units are as follows:

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

Table D-1. Report Format - Single Column

Record Number	Information Conveyed	Form
1	Software version number	21 characters
2	Instrument ID	20 characters
3	Setup 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^3 STP/760 mmHg)	1 floating point
13	Nonideality	1 floating point
14	Sample density	1 floating point
15	Saturation pressure (initial point if **During analysis** selected)	1 floating point

***Table D-1. Report Formatt - Single Column** (continued)*

Record Number	Information Conveyed	Form
16	Analysis mode 0 = equilibrate 1 = scan	1 integer
17	Equilibration time	1 floating point
18	Scan time	1 integer
	t-Method 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 Parameters:	
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-Kawazoe 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

Table D-1. Report Format - Single Column *(continued)*

Record Number	Information Conveyed	Form
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 result	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 pooint
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 **During analysis** selected)	1 floating point
66	Elapsed time in minutes*	1 integer
67	Report selections (number of points)* 0 = not selected 1 = selected for surface area 2 = selected for t-method 3 = selected for both End of transmission	1 integer

*Equals the number of points collected; for example, if five points were collected, there are five records for each field.

Table D-2. Report Format - Spreadsheet

Record Number	Information Conveyed	Form
1	Software version number	21 characters
2	Instrument ID	20 characters
3	Setup 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^3 STP/760 mmHg)	1 floating point
13	Nonideality	1 floating point
14	Sample density	1 floating point
15	Saturation pressure	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 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

Table D-2. Report Format - Spreadsheet *(continued)*

Record Number	Information Conveyed	Form
	BJH Parameters:	
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-Kawazoe 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 0 = Fail 1 = Pass or Reported-only	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 result	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

Table D-2. Report Format - Spreadsheet *(continued)*

Record Number	Information Conveyed	Form
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 pooint
61	Horvath-Kawazoe median pore width Pass/Fail result	1 integer
62	Number of points collected	1 integer
63	Space separator	
63a	Carriage return/linefeed	
63b	Carriage return/linefeed	
63c	Carriage return/linefeed	
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)	

E. 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 interface 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.

F. RS-232 OPERATION

The Gemini analyzer is a standard DTE device. The RS-232 port on the rear 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.

The pin assignment for the RS-232 port is shown in the following table; be sure your receiving device is configured to interface with these assignments. Any signals that are not listed in the table will be ignored by the analyzer.

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

The Gemini uses the DTR and DSR signals for hardware flow control. Be sure that your serial device provides these signals. For example; if attaching to a PC (also a DTE device), use a null modem cable which includes the signals designated in the above table.

If you experience 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.

G. KEYBOARD INTERFACE

A computer keyboard attached to the USB port enables you to enter alphanumeric characters at certain prompts; for example, the **Sample ID** and **Description** prompts. So that you do not have to switch back and forth between the computer keyboard and the analyzer keypad, all system commands are available through the keyboard. Tables G-1 and G-2 provide computer keyboard equivalents for the functions on the analyzer keypad.

Table G-1. Alternate Functions

Function	Key Sequence		Used To
	Keypad	Keyboard	
Analyze	**Alt** + **4**	**Ctrl + A**	Perform an analysis.
Po	**Alt** + **.** (decimal)	**Ctrl + O**	Measure the saturation pressure.
Diagnostics	**Alt** + **7**	**Ctrl + D**	Enables you to perform certain diagnostics
Escape	**Alt** + **CLEAR**	**Esc**	Discard all data entered in the current mode and return to display mode. Cancel an automatic operation in progress. Exit manual mode.
Manual	**Alt** + **1**	**Ctrl + Y**	Enable manual mode, allowing you to manually perform certain functions. Press **Alt + CLEAR** to exit manual mode and return to the **Reload** prompt.
Print	**Alt** + **6**	**Ctrl + P**	Print an analysis or calibration report. If an automatic operation is in progress, print a partial report.
QuickStart	**Alt** + **9**	**Ctrl + Q**	Begin a series of analyses.
Review	**Alt** + **5**	**Ctrl + R**	Review completed analysis data.
Set Up	**Alt** + **2**	**Ctrl + U**	Display or edit analysis parameters, report options, communication parameters, and system options.
Transmit	**Alt** + **3**	**Ctrl + T**	Transmit analysis or calibration data over the serial line. Transmit a partial report if an automatic operation is in progress.

Table G-2. Standard Key Functions

Keypad	Keyboard	Used To
CHOICE	**Ctrl + N**	Display the next message when in display mode. Display the next multiple choice item when in a command mode.
CLEAR	**Ctrl + X**	Clear a message when in display mode. Clear an entry when in a command mode.
ENTER	**Enter or Ctrl + M**	Complete an entry or begin an action
SAVE	**Ctrl + W**	Save the information you entered and return to display mode.

H. ANALYZING SAMPLES WITH 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 you are analyzing.

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

Using Glass Beads

This method typically produces the best results.

1. Place an appropriate quantity of sample in a clean sample tube.

2. Load a second sample tube of the same size with glass beads that have a total volume approximately the same as the sample volume.

   a. Determine the volume (v) of the sample in cm^3:

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

   where

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

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

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

where

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

3. Outgas the sample in the sample tube at an appropriate temperature for an appropriate amount of time.

4. Install the sample tube (containing the outgassed sample) onto the analysis port and the sample tube (containing the glass beads) onto the balance port.

5. Specify a one-point measurement (P/Po = 0.05 to 0.1) so that the initial free-space measurement can quickly be determined; then perform the measurement.

6. Using the measured free space absolute value and the following relationship, determine the mass of glass beads to remove from (or add to) the balance tube to reduce the free-space imbalance:

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

where

$2.515\ g/cm^3$ = density of glass beads
3.53 = thermal correction (no units)

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

7. Use a beaker of warm water to bring the balance tube to room temperature before removing it from the balance port of the Gemini to remove (or add) glass beads. This prevents condensation of moisture from the laboratory atmosphere onto the cold glass beads.

8. Remove the balance tube:

   - If the measured free space is negative (–), add the calculated mass of glass beads into the balance tube.

   - If the measured free space is positive (+), remove the calculated mass of glass beads from the balance tube.

9. Reinstall the balance tube onto the balance port of the Gemini analyzer.

For subsequent samples of the same material, you may simply 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 your analysis Dewar and place it on the elevator.

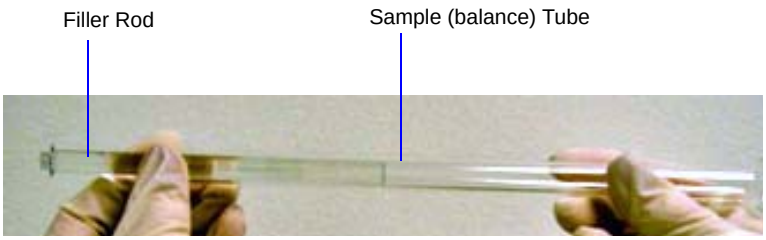
11. Close the sample compartment door and start your analysis.

Using Filler Rods

Filler rods of two different sizes are included in the accessory kit. Insert them as described below.

1. Clean your sample tube, balance tube, and filler rods; label your sample and balance tubes.

2. Hold the balance tube horizontally and carefully slide the longer filler rod into the tube.

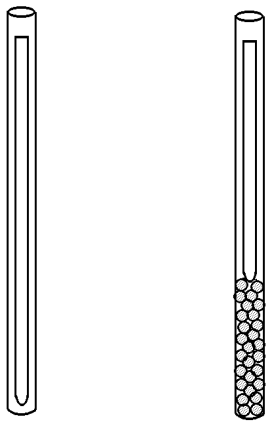


Do not hold the rod vertically and drop the rod into the tube; this may break the tube.

3. Prepare your sample and place it into the sample tube, insert the shorter filler rod into the sample tube.

   If you are using bulbous sample tubes, use the same size filler rod as used in the balance tube.

4. 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.

5. Attach the sample tube to the analysis port and the balance tube to the balance port.

6. Prepare your analysis Dewar and place it on the elevator.

7. Close the sample compartment door and start your analysis.

Be sure to select Measure for the free-space measurement.

Using Glass Beads and Filler Rods

This method may further improve results, depending on the sample material you are analyzing.

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.

   If you are using bulbous sample tubes, use the same size filler rod as used in the balance tube.

3. Attach the sample tube to the analysis port and the balance tube to the balance port.

4. Prepare your analysis Dewar and place it on the elevator.

5. Close the sample compartment door and start your analysis.

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