VacPrep™

Sample Preparation System

micromeritics®

Operator Manual

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Apr 2021
(Rev B)

TRADEMARKS

Micromeritics is a registered trademark of Micromeritics Instrument Corporation.
VacPrep is a trademark of Micromeritics Instrument Corporation.

Copyright

The software described in this manual is furnished under a license agreement and may be used or copied only in accordance with the terms of the agreement.

WARRANTY

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

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

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

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

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

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

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

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

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

CORPORATE PROFILE

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

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ABOUT THIS MANUAL

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

NOTE — Notes contain important information applicable to the topic.

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

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

GENERAL SAFETY

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

Use caution in the areas where this symbol is displayed on the instrument — such as near the heating stations. These surfaces may be hot and could cause serious burns. Use the gloves supplied in the accessories kit.

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

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

- Wear the appropriate personal protective equipment when operating this instrument — such as eye protection, lab coat, protective gloves, etc.
- When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument. The VacPrep weighs approximately 10 kg (22 lb).
- Always pay attention to the safety instructions provided on each label affixed to the instrument and do not alter or remove the labels. When inspecting the instrument, ensure that the safety labels have not become worn or damaged.
- Proper maintenance is critical to personnel safety and smooth instrument operation and performance. Instruments require regular maintenance to help promote safety, provide an optimum end test result, and to prevent costly down time. Failure to practice proper maintenance procedures can lead to unsafe conditions and shorten the life of the instrument.
- Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

INTENDED USE

The VacPrep 061 degasser prepares samples for adsorption analysis. Moisture and other contaminants are removed by heating the sample.The degasser has six heating stations for degassing samples and six cooling stations. The required degassing temperature is set and checked at the temperature controller. Control valves provide a constant indication of the valve state. A built-in vacuum gauge provides a constant indication of the system vacuum.

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

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

TRAINING

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

ENVIRONMENTALLY FRIENDLY USE PERIOD

Hazardous Substances Table

Part Name	Hazardous Substances					
	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Metal Parts	o	o	o	o	o	o
Power Entry	o	o	o	o	o	o
Temperature Controller	o	o	o	o	o	o
Electronic Components	x	o	o	o	o	o
External Controls	x	o	o	o	o	o

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

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

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

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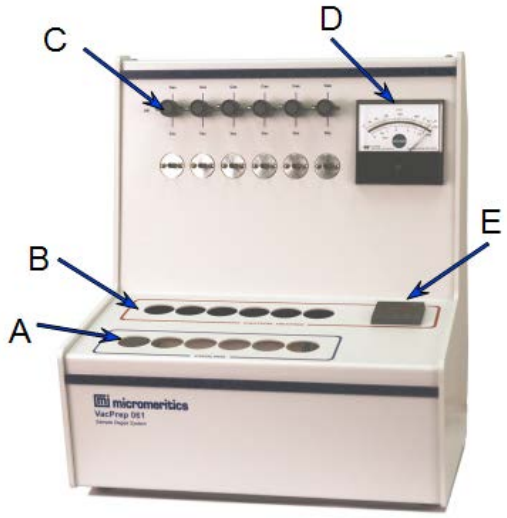
Table of Contents

1 ABOUT THE VACPREP

Use caution in the areas where this symbol is displayed on the instrument — such as near the heating stations. These surfaces may be hot and could cause serious burns. Use the gloves supplied in the accessories kit.

FRONT PANEL

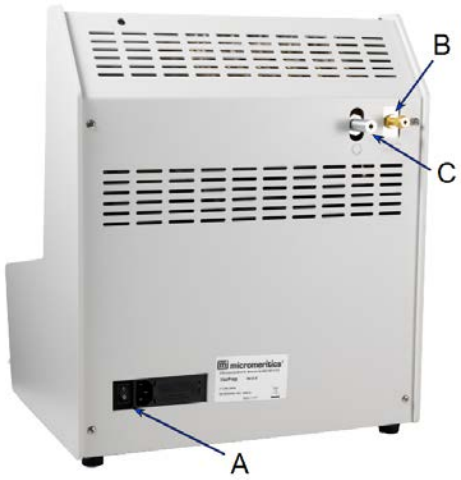


A. Cooling stations
B. Heating stations
C. VAC/GAS control knobs
D. Vacuum gauge
E. Temperature controller

VacPrep Front Panel

Component	Description
Cooling Stations	Six stations to cool the sample.
Heating Stations	Six stations to heat the sample.
Temperature Controller	Displays the set point and the current temperature of the heating stations.
VAC/GAS control knobs	Each heating station has a control knob that selects the vacuum or gas for the selected station.
Vacuum Gauge	Displays the level of vacuum available. The scale is labeled in millitorr (1 millitorr = 1 µmHg) and millibar.

BACK PANEL



A. Power switch
B. Gas port
C. Vacuum port

VacPrep Back Panel

Component	Description
Gas Port	Use to connect the gas supply to the degasser.
Power Switch	Use to power ON or power OFF the degasser.
Vacuum port	Use to connect the external vacuum pump to the degasser.

SPECIFICATIONS FOR VACPREP

Sample Tubes

Up to 3.0 cm (1 3/16 in.) OD bulbs with 1/4, 3/8, 1/2 in., 9 mm, or 12 mm OD stems

Heating Block

Temperature	10 ºC above ambient to 400 ºC
Accuracy	within ± 10 °C

Electrical

Voltage	100/120/230/240VAC ±10%
Power	250 VA maximum
Frequency	50/60 Hz

Environment

Temperature	10 to 35 ºC operating; 0 to 50 ºC non-operating
Humidity	45 to 90% relative, non-condensing
Indoor or Outdoor Use	Indoor only Altitude: 2000 m max Pollution degree of the intended environment: 2

Gases

Helium, nitrogen, argon, or any other pure gas that does not adsorb at room temperature.

Flow rate

Flowing Gas Preparation	Up to 50 cm^3/min.

Vacuum System

A vacuum source achieving better than 20 µmHg at the instrument inlet and having a device to reduce oil vapor backstreaming is recommended. The vacuum system must also have an anti-suckback valve to prevent oil from flowing into the VacPrep should there be a power failure.

Sample Capacity

6 heating stations and 6 cooling stations

Physical

Height	43 cm (17 in.)
Width	36 cm (14.5 in.)
Depth	30 cm (12 in.)
Weight	10 kg (22 lbs)

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

2 INSTALLATION

The following are required for safe installation and prep station operation:

- The installation location must be in an area that is easily accessible from all sides for servicing and operating.
- The power cable should be connected only to a suitable wall outlet.
- Gas cylinders should be placed within 6 ft of the degasser. The same gas cylinder can be used for both the prep station and the instrument by using a tee fitting on the gas supply line.
- The degasser uses up to 50cm^3/min of inert gas and does not require special ventilation, however ensure that the installation location provides adequate ventilation space near the back of the instrument.
- Use of fuses other than those specified may cause damage to the equipment. See ***Select Input Power on page 2 - 5***.
- Only inert gasses (helium, nitrogen or argon) should be used for sample preparation. Inert gasses in the quantities used by the degasser do not pose a health hazard to users.

UNPACKING AND INSPECTION

When the equipment is received, unpack and inspect the contents of the shipping container(s). Use the packing list to verify that all products, accessories, software (if applicable), and documentation are received intact and in the correct quantity. The shipping container(s) and contents should be inspected within a few days of receipt in the event damage or loss has occurred. Sort through all packing material before declaring missing equipment or parts.

Micromeritics recommends saving all shipping containers until installation of the equipment is complete. All shipping containers where equipment is to be declared as damaged or lost must be examined by the claims investigator prior to completion of the inspection report.

SHIPPING DAMAGE

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 damage or loss of equipment is discovered during shipment, report the condition to the carrier immediately. Insurance claims **MUST** be made with the freight carrier, **NOT** Micromeritics.

- Keep all software, manuals, and accessories with the equipment.
- Report any shipping damage immediately to the carrier and follow their directions.
- Report missing or wrong parts to Micromeritics, in addition to any shipping damage, only after filing a claim with the carrier.
- Micromeritics will NOT file a claim for shipping damage.
- Do not discard shipping boxes and containers until installation is complete. If space is available, it is recommended that shipping containers be saved for future use in the event of return to factory for repair.

EQUIPMENT RETURNS

Micromeritics strives to ensure that all items arrive safely and in working order. If it is necessary to return equipment (damaged either during shipment or while in use) to Micromeritics for repair or replacement, follow these procedures:

1. Tag or identify the defective equipment, noting the defect and circumstances under which the defect is observed.
2. Refer to the sales or purchase order and provide the date the equipment was received.
3. Call Micromeritics for a Return Material Authorization number.
4. Pack the equipment in the original shipping container, if possible. If the original container is unavailable, for a nominal fee, Micromeritics can provide another container for shipping.

GUIDELINES FOR CONNECTING GASES

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

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

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

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

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

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

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

CONNECT GAS TO ANALYZER

Regulator Pressure Settings

Analyzer	Gauge should indicate
VacPrep	15 psig (103 kPag)

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

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

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

1. Loosen, then remove the plug from the gas port on the back of the degasser
2. Insert the gas line into the port and hand-tighten the connector nut. Use a 7/16 in. (11 mm) wrench to tighten the nut until very snug.
3. Connect all the remaining gases on available ports.

REMOVE STABILIZING TUBES

The degasser ships with stabilizing tubes in heating stations 1 and 6 to prevent the heater block from shifting during shipment. Ensure these tubes are removed before powering on the degasser.

SELECT INPUT POWER

Do not connect the equipment to the power source until the proper voltage selection has been made. Doing so could result in electrical shock and/or damage the assembly.

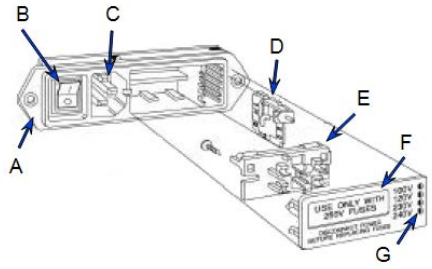
All instruments leave the factory set for 120VAC and with the line fuse removed. The correct setting of the universal power entrance must be checked and the appropriate fuse(s) installed before the instrument can be operated. The degasser is designed to operate with universal line voltage of 100/230VAC ±10, 50/60 Hz through a standard wall receptacle. Voltage selection and fusing are made at the power connector located on the rear panel of the unit.

- The power cord should be disconnected from the instrument before removing the cover from the input power connector. Failure to disconnect the power cord could result in electrical shock.
- The input power connector can be used with either a single-fuse arrangement (100/120VAC) or a double-fuse arrangement (230/240VAC). Use the appropriate fuse(s) for the input power source. The fuses must be identical in type and rating to that specified. Use of other fuses could result in electrical shock and/or damage to the instrument.

Fuses may be obtained from Micromeritics or a vendor of your choice as long as they meet the requirements specified in this section.

Power Source	Required Fuses
100/120VAC	Single fuse, 3AG (Slo-Blo) 250V, 2.5A, 6.35mm x 31.75mm. Single pole input.
230/240VAC	Double fuse, Time-lag (Slo-Blo), 250V, 1.25A, 5mm x 20mm. Double pole input.

1. Ensure the instrument is powered off and the power cord is disconnected from the analyzer or power source.
2. Check the voltage setting on the cover of the power entrance.



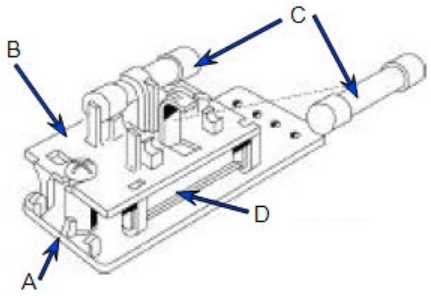
A. Housing
B. Power switch
C. Power connector
D. Voltage selector card
E. Fuse holder
F. Fuse cover
G. Fuse setting indicator

CAUTION

When opening the fuse cover, do not pry in the middle of the cover near the hinges. Doing so may break the hinge.

For European fuse arrangement:

1. Remove the fuse block:
   a. Open the cover to the right of the power entrance by inserting a small screwdriver in the left side of the power module, then gently pry the cover open.
   b. Loosen the Phillips screw two turns.
   c. Remove the fuse block by sliding it up, then away from the Phillips screw and lifting the block from the pedestal.

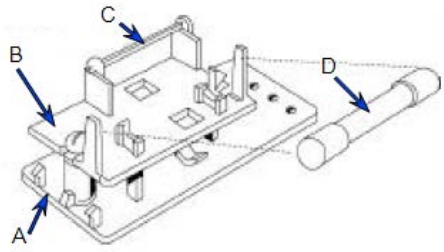


A. Fuse cover
B. Fuse block
C. Fuses
D. Jumper

2. Insert the fuses. Two European fuses are required although a dummy fuse may be used in the lower holder.
3. Set the voltage. See ***Select the Voltage on page 2 - 8*** then continue with these steps.
4. Invert the fuse block and slide the block back onto the Phillips screw and pedestal.
5. Tighten the Phillips screw and and replace the cover. The fuse(s) that go into the housing first are the active set.

For North American fuse arrangement:

1. Remove the fuse block:
   a. Open the cover to the right of the power entrance by inserting a small screwdriver in the left side of the power module, then gently pry the cover open.
   b. Loosen the Phillips screw two turns.
   c. Remove the fuse block by sliding it up, then away from the Phillips screw and lifting the block from the pedestal.

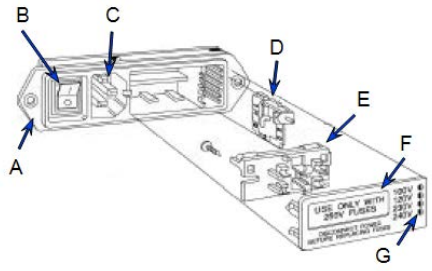


A. Fuse cover
B. Fuse block
C. Jumper
D. Fuse

2. Insert the fuse.
3. Set the voltage. See ***Select the Voltage on the next page*** then continue with these steps.
4. Slide the block back onto the Phillips screw and pedestal.
5. Tighten the Phillips screw and replace the cover.

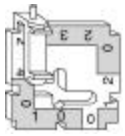
SELECT THE VOLTAGE

1. Pull the voltage selector card straight out of the power connector housing. If necessary, use needle-nose pliers to grasp the card.

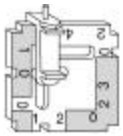


A. Housing
B. Power switch
C. Power connector
D. Voltage selector card
E. Fuse holder
F. Fuse cover
G. Fuse setting indicator

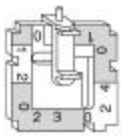
2. Set the voltage:
   a. Position the selector card so that the required voltage is readable at the bottom and the selector pin is pointing up.



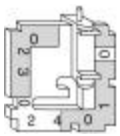
100V



120V



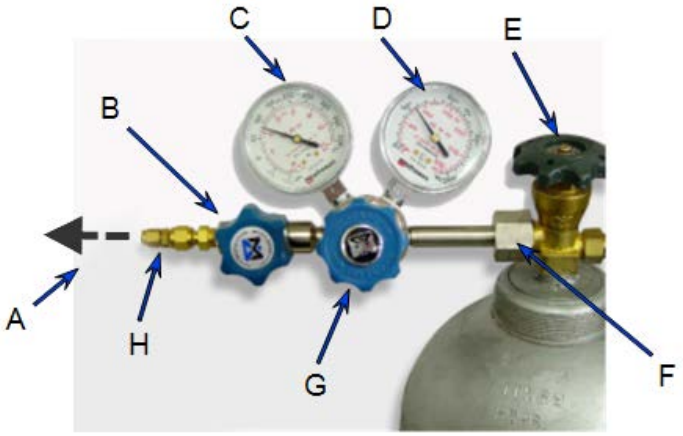
230V



240V

3. Insert voltage selector card into the housing. The printed side of the card must face toward IEC connector and the edge displaying the voltage goes in first.
4. Replace the fuse cover.
5. Verify that the indicator pin shows the required voltage.

REAR PANEL CONNECTIONS



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

1. Insert one end of the power cord into the input power connector at the rear of the degasser and the other end into the appropriate power source.
2. Attach an appropriate regulator to the gas cylinder. Leave the gas cylinder shut-off valve closed until instructed otherwise.
3. If the regulator has a 1/8 in. outlet, proceed to Step 4. If the regulator has a 1/4 in. outlet, attach the reducer fitting to the outlet of the regulator shut-off valve.
4. Tighten the regulator shut-off valve nut.

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

5. Attach the copper tubing to the brass reducer fitting.
6. Attach the other end of the copper tubing to the fitting on the back of the degasser.

A. Gas port (connect to gas cylinder with gas fitting and copper tubing)
B. Vacuum port

7. Open the gas cylinder shut-off valve.
8. Use the regulator pressure control knob to adjust the pressure. The following *Flow Rate* table shows pressures and their resulting flow rates (tested with helium and nitrogen). If unsure of which flow rate to use, start with about 10 cm^3/min. Experience with samples will help determine whether a change is required.

Gas Flow Rate for the VacPrep

Flow cm^3/min	Gauge Pressure	
	psi	kPa
10	3	21
15	5	35

A flow rate of 10-15 cm^3/min is the maximum flow rate that would generally be used in normal operation.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

9. Close the gas cylinder shut-off valve. Check the pressure. If it decreases, gas is probably leaking at the fitting. Tighten the fitting. Then check the pressure again.
10. Open the regulator shut-off valve. The pressure should stabilize after about five minutes. If it does not, gas is probably leaking at the brass reducer fitting or at the gas port on the rear panel of the degasser. Tighten the fittings. Then check the pressure again.
11. Open the gas cylinder shut-off valve.
12. Attach an adequate length of vacuum pump tubing (that will fit over the 1/2 in. degasser connector) to a vacuum pump and to the vacuum port on the back of the degasser. Use hose clamps to keep the hose in place. An appropriate vacuum pump system that includes hose clamps and tubing is available from Micromeritics. Cut the tubing to the length needed. Follow the installation instructions provided with the pump.

CONNECT THE POWER CABLE

1. Connect a power cord from the wall outlet to the power connector on the back of the instrument.
2. Connect the other end of the power cord to the wall power source.

Do not connect the equipment to the power source until the proper voltage selection has been made. Doing so could result in electrical shock and/or damage the assembly.

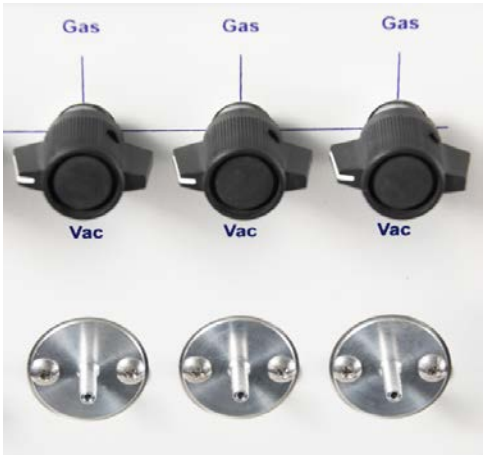
Use of a power cord or power supply not provided with the instrument could cause personal injury or damage to the equipment. If a replacement is needed, contact your Micromeritics Service Representative. Detachable power supply cords with an inadequate rating could cause significant instrument damage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

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

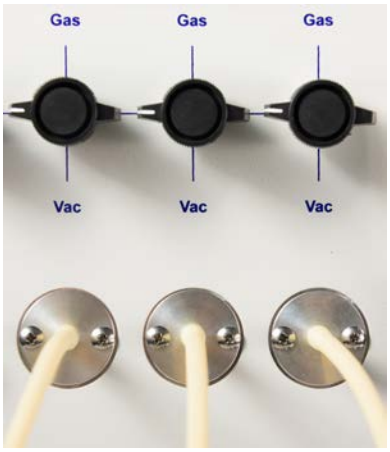
Do not remove or disable the grounding prong on the instrument power cord.

ATTACH FLEXIBLE TUBING

Six pieces of flexible tubing are included in the accessories kit. Attach one piece of tubing to the barbed vacuum/gas fitting at each degas station on the front panel.



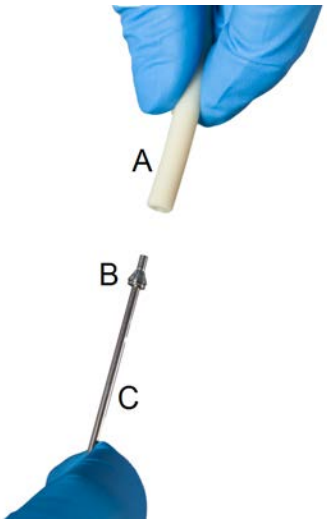
Barbed fitting



Flexible tubing installed

ATTACH GAS DELIVERY TUBES

Six stainless steel gas delivery tubes are included in the accessories kit. Attach a gas delivery tube to the flexible tubing at any degas station that will be used for flowing gas preparation. Attach the tube by pressing the flexible tubing completely over the ferrule at the end of the gas delivery tube.



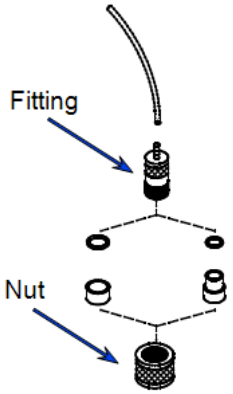
A. Flexible tubing
B. Ferrule
C. Gas delivery tube

ATTACH VACUUM FITTINGS FOR VACUUM PREPARATION

If the sample fluidizes easily and you are using a Flow Control Valve, install it at this time using the instructions provided with the valve.

Six vacuum fittings with knurled retaining nuts, ferrules, and O-rings, are included in the accessories kit. Attach a fitting to the flexible tubing at any degas station that will be used for vacuum or vacuum-backfill sample preparation. Press the flexible tubing completely over the barbed nipple on the end of the fitting. Loosely attach the knurled retaining nut, ferrule, and O-ring.

1/2 in. or 12 mm O-ring and ferrule (for 1/2 in. or 12 mm sample tubes)



3/8 in. or 9 mm O-ring and ferrule (for 3/8 in. or 9 mm sample tubes)

VACUUM GAUGE CALIBRATION

Create a Sorption Pump on the next page

The vacuum gauge on the VacPrep was calibrated and tested at the factory using a 115 V, 60 Hz electrical supply. When powered by other voltages and frequencies, the gauge must be recalibrated.

1. Power ON the degasser.
2. Turn all VAC/GAS control knobs to the OFF position.
3. Power ON the attached vacuum pump and allow the pump to run for at least two hours.
4. Insert the small, flat-blade screwdriver (included in the accessories kit) through the opening on the back of the degasser and into the slot on the adjustment screw.

To confirm the accuracy of the vacuum pump gauge, attach an independent gauge to the end of the vacuum pump hose. Most good quality vacuum pumps can achieve a vacuum of less than 10 millitorr (10 µmHg). A better vacuum may be obtained for calibration purposes by using a sorption pump.

Outside view - no need to remove cover

Inside view

5. Turn the adjustment screw to obtain a reading on the vacuum gauge that matches the pressure level achieved by the vacuum pump.

CREATE A SORPTION PUMP

The VacPrep vacuum can be improved by creating a sorption pump using a sample tube and a microporous material with a high surface area.

1. Partially fill a sample tube with material of a high surface area, such as aquarium charcoal, other forms of activated carbon, or zeolite. Avoid using fine powders.
2. Attach the sample tube to one of the degas stations.
3. Set the temperature controller to approximately 150 ºC.
4. Turn the appropriate VAC/GAS control knob to VAC and allow the sample tube to be evacuated for approximately one hour or until the vacuum gauge reading falls below 50 millitorr (50 µmHg).
5. Leave the VAC/GAS control knob in the VAC position and move the sample tube to a cooling station.
6. After the sample tube has cooled sufficiently, immerse the lower two thirds of the tube into a liquid nitrogen bath.
7. The resulting sorption pump will reduce the pressure in the VacPrep to less than 1 millitorr (1 µmHg).
8. The vacuum gauge may be recalibrated with the sorption pump attached.
9. Turn the VAC/GAS control knob to GAS for about 30 seconds, then to OFF before removing the sorption pump from the fitting.

For sample tubes having a capacity greater than 6 cm^3 and for tubes containing high surface area materials, a longer backfill time will be required.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

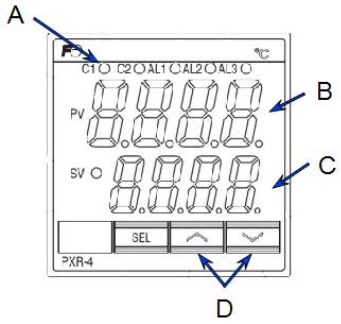
3 SAMPLE PREPARATION

Heating stations, sample tubes, and heating station adapters may be very hot.

- Wear the proper gloves and hold the sample tube by the upper part of the stem.
- If the adapter has a handle, hold the adapter by the handle.
- Do not place fingers inside the heating stations. The stations may be very hot.
- Do not place adapters near any combustible material or touch them with fingers.
- Do not place a hot adapter on a unprotected surface such as a countertop. The adapter may burn the surface.
- Failure to observe these precautions may result in severe burns.
- The vacuum fitting for the sample tube may be hot enough to cause burns after the sample has been heated. Wear the cotton gloves supplied in the accessories kit and handle the fitting carefully to avoid burns.

CONTROL THE TEMPERATURE OF THE HEATING STATIONS

Use the temperature controller to specify the temperature of the heating stations and display the current temperature.



A. Heating station power indicator
B. Current temperature
C. Set Point temperature
D. Change Set Point temperature

The controller always displays the current temperature (PV = Process Value) and the set point (SV = Set Value). Press the up or down buttons until the preferred temperature set point is displayed. The keys may be held down to rapidly increase or decrease the setting.

The set point is retained when the degasser is powered off then back on.

CLEAN AND LABEL SAMPLE TUBES

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

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

Materials required for cleaning	
■ Acetone or isopropyl alcohol ■ Analytical balance and weighing support ■ Nitrogen or helium ■ Fume hood ■ Detergent (such as Alconox) ■ Drying oven ■ Forceps ■ Insulated gloves ■ Pipe cleaners ■ Rubber gloves or clean, lint-free cloth	■ Pipe cleaners ■ Rubber gloves or clean, lint-free cloth ■ Safety glasses ■ Sample tubes ■ Sample tube brush ■ Sample tube rack ■ Sample tube stopper ■ Funnel ■ Ultrasonic cleaning unit ■ Waste container ■ Safety glasses

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

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

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

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

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

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

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

DETERMINE THE SAMPLE MASS

Sample Data Worksheet for Gas Adsorption on page B - 1

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

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

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

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

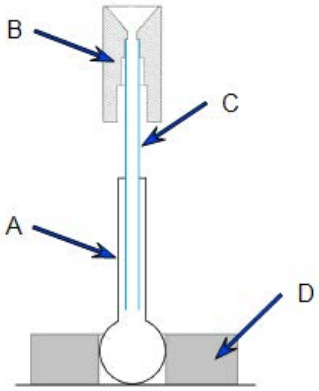
Analysis results are expressed in units of surface area per gram of sample; therefore, it is important to know the true sample mass.

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

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

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

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



A. Sample tube
B. Funnel
C. Straw
D. Weighing support

TIP: Use a drinking straw tube (approx. 6mm [1/4in.] diameter) that fits inside the sample tube to keep the inside wall of the sample tube clean.

Balance

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

HEATING STATION ADAPTERS

Heating stations, sample tubes, and heating station adapters may be very hot.

- Wear the proper gloves and hold the sample tube by the upper part of the stem.
- If the adapter has a handle, hold the adapter by the handle.
- Do not place fingers inside the heating stations. The stations may be very hot.
- Do not place adapters near any combustible material or touch them with fingers.
- Do not place a hot adapter on a unprotected surface such as a countertop. The adapter may burn the surface.
- Failure to observe these precautions may result in severe burns.

Heating station adapters are included in the accessories kit for use with sample tubes. There are three models of heating station adapters. Use the appropriate model as shown in the *Heating Station Adapters* table. Additional adapters can be ordered from Micromeritics.

The adapters used with Gemini sample tubes may remain in the stations until a different type of sample tube is needed.

The adapters used with the TriStar and ASAP Series sample tubes must be removed when the sample tube is removed. The adapter may be stored in the heating station or in a cooling rack.

Adapters constitute an isothermal shield and help to maintain uniform temperature throughout the sample bed. Sample temperature, however, depends on several factors:

- The thermal conductivity of the sample material
- The quantity of sample material
- The rate of gas flow through the sample bed

All materials do not heat identically. The actual sample temperature can be expected to be within ±10 ºC of the current temperature (PV) displayed on the temperature controller. If an exact sample temperature is critical to the application, measure the sample bed with a temperature probe and adjust the set point temperature accordingly.

Heating Station Adapters

Analyzer Model	Sample Tubes	Adapter Model
TriStar Flex		
	1/4, 3/8, or 1/2 in. stem, 1.2 in. (30 mm) bulb	Part number: 060-25850-00
Gemini (bulb tube)		
	3/8 in. stem, 3/4 in. (19 mm) bulb	Part number: 065-25811-00
Gemini (straight-wall tube)		
	3/8 in. stem	Part number: 065-25812-00

Heating Station Adapters (continued)

Analyzer Model	Sample Tubes	Adapter Model
TriStar Flex		
	1/4, 3/8, 1/2 in. 9mm, 12mm stem	Part number: 060-25850-00

TRISTAR AND ASAP SERIES SAMPLE TUBES

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

Use caution in the areas where this symbol is displayed on the instrument — such as near the heating stations. These surfaces may be hot and could cause serious burns. Use the gloves supplied in the accessories kit.

Insert Adapters

1. Place the adapter over the sample tube.
2. Hold both the adapter handle and sample tube stem when placing the sample tube and adapter in the heating station.

   Alternately, insert the sample tube in the heating station, then place the adapter over the sample tube in the station.

Remove Adapters

Use caution when moving sample tubes and heating station adapters to the cooling station.

1. Hold the stem of the sample tube and the adapter handle and remove the adapter and sample tube.

2. Place the adapter and sample tube in a cooling station.

   After the sample tube has been installed on the analyzer, place the adapter back into the heating station for storage.

GEMINI SAMPLE TUBES

Use caution in the areas where this symbol is displayed on the instrument — such as near the heating stations. These surfaces may be hot and could cause serious burns. Use the gloves supplied in the accessories kit.

Insert Adapters

Use an adapter insertion tool to insert an adapter into the heating station before inserting the sample tube. An adapter may be inserted into the heating station with either end in the upward position.

To insert the adapter, insert the tool into the adapter so that the pronged tip of the adapter tool fits into one of the small holes in the side of the adapter. Slide the adapter into the heating station and remove the adapter.

Heating Station Sample Tube Adapter Insertion / Removal Tool

Heating Station Sample Tube Adapter

A. Adapter insertion tool
B. Sample tube adapter

Remove Adapters

Adapters for the Gemini may remain in the heating station when not in use. If removing the adapter, use the adapter tool.

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4 DEGAS THE SAMPLE

Heating stations, sample tubes, and heating station adapters may be very hot.

- Wear the proper gloves and hold the sample tube by the upper part of the stem.
- If the adapter has a handle, hold the adapter by the handle.
- Do not place fingers inside the heating stations. The stations may be very hot.
- Do not place adapters near any combustible material or touch them with fingers.
- Do not place a hot adapter on a unprotected surface such as a countertop. The adapter may burn the surface.
- Failure to observe these precautions may result in severe burns.
- The vacuum fitting for the sample tube may be hot enough to cause burns after the sample has been heated. Wear the cotton gloves supplied in the accessories kit and handle the fitting carefully to avoid burns.

There are three methods of sample preparation:

- **Vacuum preparation.** The sample is heated as a vacuum is applied to the sample tube to remove moisture and other contaminants. This method should be used only with materials which are not likely to fluidize.
- **Vacuum-backfill preparation.** The sample is heated as a vacuum is alternately applied and then flows gas into the sample tube to remove moisture and other contaminants. The alternating process may be repeated several times. The instrument is designed for low-pressure backfilling only.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

- **Flowing gas preparation.** The sample is heated as the operator flows gas into the sample tube to remove moisture and other contaminants.

Samples that contain a high level of moisture could benefit from a higher flow rate than 5 psi regulator pressure when using the flowing gas method. A flow rate up to 50 cm^3/min (approximately 16 psi regulator pressure) may be used to aid in purging moisture from the sample when using flowing gas preparation only.

VACUUM PREPARATION

The vacuum preparation method should be used only with materials which are not likely to fluidize.

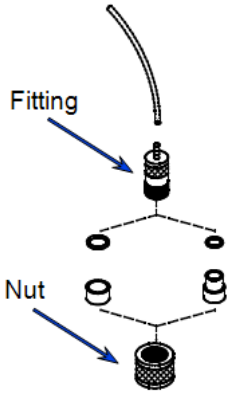
CAUTION

Some fine-powdered samples such as controlled pore glass, fluid-cracking catalysts, and carbon may be difficult to prepare by vacuum preparation. Such materials tend to fluidize and float up the sample tube toward the vacuum source. Micromeritics recommends that such samples be first prepared by flowing gas preparation and, if necessary, then be subjected to vacuum preparation.

If sample has been accidentally sucked into the instrument, see *Clean and Replace Vacuum Fittings on page 5 - 13*.

1. Turn all VAC/GAS control knobs to the OFF position. Ensure that the vacuum pump is powered ON and that gas is being supplied to the degasser.
2. Attach a vacuum fitting to each piece of flexible tubing at a degas station to be used for sample preparation.

1/2 in. or 12 mm O-ring and ferrule (for 1/2 in. or 12 mm sample tubes)



3/8 in. or 9 mm O-ring and ferrule (for 3/8 in. or 9 mm sample tubes)

3. Power on the VacPrep.
4. Set the temperature controller using the temperature set point.
5. Remove the stopper from the sample tube and attach the sample tube to the vacuum fitting.
    a. Loosen the knurled retaining nut.
    b. Insert the sample tube fully into the fitting. There will be a slight resistance as the tube passes through the O-ring.
    c. Turn the knurled retaining nut until it is finger-tight.

CAUTION

Tighten the fitting retaining nut only finger-tight. Over-tightening may break the sample tube.

6. Hold the sample tube outside of the heating station to perform the next three steps. This position allows makes it easier to observe the sample as the analyzer begins to evacuate the tube and to stop evacuation if sample material appears likely to be pulled out of the tube.
7. Turn all VAC/GAS control knobs to the OFF position.

To help prevent sample material from being pulled into the instrument, slowly turn the VAC/GAS control knob to the VAC position.

8. Turn the VAC/GAS control knob for the sample to be degassed to the VAC position and observe the sample. If the analyzer begins to pull sample material out of the tube, immediately turn the VAC/GAS control knob to the OFF position.
9. When the vacuum gauge reads 100 millitorr (100 µmHg) or less, turn the VAC/GAS control knobs for any previously evacuated stations to the VAC position.
10. Using an appropriate heating station adapter, insert the sample tube into the heating station.

11. Allow the sample(s) to degas for the required length of time. One to two hours is typical for many samples at temperatures above 200 ºC. To determine the optimum time, select the highest safe temperature for the sample, then degas it for various intervals, determining the surface area after each interval.

For example, degas the sample for 15 minutes, then measure the surface area. Repeat this process until the surface area no longer increases. The length of time required to reach this point is the minimum amount of time required to degas the sample.

12. After the sample is sufficiently degassed, remove the sample tube from the heating station. Leave the VAC/GAS control knob in the VAC position.

CAUTION

Keep the sample tube attached to the vacuum fitting while cooling to prevent contamination from the ambient atmosphere.

13. Place the sample tube, with the vacuum fitting still in place, in a cooling station.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

14. When the sample has sufficiently cooled, turn the VAC/GAS control knob to the OFF position and then to the GAS position.
15. Allow the gas to flow for approximately 30 seconds; then turn the VAC/GAS control knob to the OFF position.
16. Loosen the knurled retaining nut and slide the sample tube out of the vacuum fitting. Quickly insert a stopper into the sample tube.
17. Place the vacuum fitting in a cooling station until the next sample is attached.
18. Reweigh the sample tube with stopper and sample to ensure that the correct sample weight is recorded.

CAUTION

The weight recorded at this step may be compared to the weight recorded before preparation to determine if adequate degassing has occurred.

ALTERNATING VACUUM BACKFILL PREPARATION

Alternating vacuum-backfill sample preparation utilizes a combination of vacuum and gas backfilling techniques to remove contaminants and prepare samples for analysis. The VacPrep is designed for low-pressure backfilling only.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

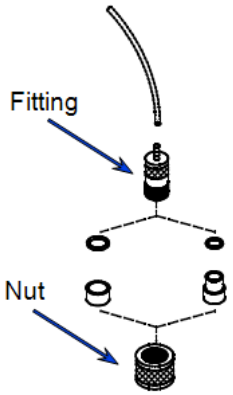
CAUTION

Some fine-powdered samples such as controlled pore glass, fluid-cracking catalysts, and carbon may be difficult to prepare by vacuum preparation. Such materials tend to fluidize and float up the sample tube toward the vacuum source. Micromeritics recommends that such samples be first prepared by flowing gas preparation and, if necessary, then be subjected to vacuum preparation.

If sample has been accidentally sucked into the instrument, see *Clean and Replace Vacuum Fittings on page 5 - 13*.

1. Turn all VAC/GAS control knobs to the OFF position. Ensure that the vacuum pump is powered ON and that gas is being supplied to the degasser.
2. Attach a vacuum fitting to each piece of flexible tubing at a degas station to be used for sample preparation.

1/2 in. or 12 mm O-ring and ferrule (for 1/2 in. or 12 mm sample tubes)



3/8 in. or 9 mm O-ring and ferrule (for 3/8 in. or 9 mm sample tubes)

3. Power on the VacPrep.
4. Set the temperature controller using the temperature set point.
5. Remove the stopper from the sample tube and attach the sample tube to the vacuum fitting.
   a. Loosen the knurled retaining nut.
   b. Insert the sample tube fully into the fitting. There will be a slight resistance as the tube passes through the O-ring.

c. Turn the knurled retaining nut until it is finger-tight.

Tighten the fitting retaining nut only finger-tight. Over-tightening may break the sample tube.

6. Hold the sample tube outside of the heating station to perform the next three steps. This position allows makes it easier to observe the sample as the analyzer begins to evacuate the tube and to stop evacuation if sample material appears likely to be pulled out of the tube.
7. Turn all VAC/GAS control knobs to the OFF position.

To help prevent sample material from being pulled into the instrument, slowly turn the VAC/GAS control knob to the VAC position.

8. Turn the VAC/GAS control knob for the sample to be degassed to the VAC position and observe the sample. If the analyzer begins to pull sample material out of the tube, immediately turn the VAC/GAS control knob to the OFF position.
9. Allow the sample to degas for the required length of time — approximately 10 minutes.
10. Leave the sample tube in the heating station and turn the VAC/GAS control knob to the GAS position.
11. Allow gas to flow into the sample tube for 5 to 10 seconds.
12. If preparing other samples on the degasser which are not to be evacuated, turn the corresponding VAC/GAS control knobs to the OFF position.
13. Turn the VAC/GAS control knob for the sample being prepared to the VAC position.
14. When the vacuum reading falls below 100 millitorr (100 µmHg), turn the VAC/GAS control knobs for any other samples to be evacuated back to VAC.
15. Repeat steps 9 through 14 until the sample is sufficiently degassed.
16. After the sample is sufficiently degassed, remove the sample tube from the heating station. Leave the VAC/GAS control knob in the VAC position.

Keep the sample tube attached to the vacuum fitting while cooling to prevent contamination from the ambient atmosphere.

17. Place the sample tube, with the vacuum fitting still in place, in a cooling station.
18. When the sample has sufficiently cooled, turn the VAC/GAS control knob to the OFF position and then to the GAS position.
19. Allow the gas to flow for approximately 30 seconds; then turn the VAC/GAS control knob to the OFF position.

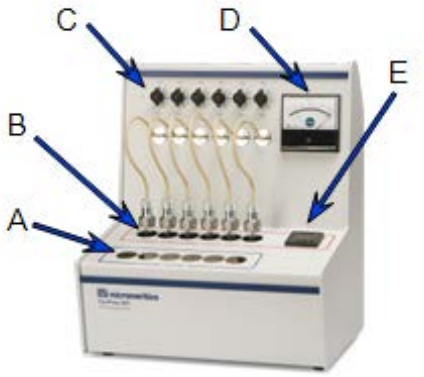
20. Loosen the knurled retaining nut and slide the sample tube out of the vacuum fitting. Quickly insert a stopper into the sample tube.
21. Place the vacuum fitting in a cooling station until the next sample is attached.
22. Reweigh the sample tube with stopper and sample to ensure that the correct sample mass is recorded.

The weight recorded at this step may be compared to the weight recorded before preparation to determine if adequate degassing has occurred.

FLOWING GAS PREPARATION

Samples that contain a high level of moisture could benefit from a higher flow rate than 5 psi regulator pressure when using the flowing gas method. A flow rate up to 50 cm^3/min (approximately 16 psi regulator pressure) may be used to aid in purging moisture from the sample when using flowing gas preparation only.

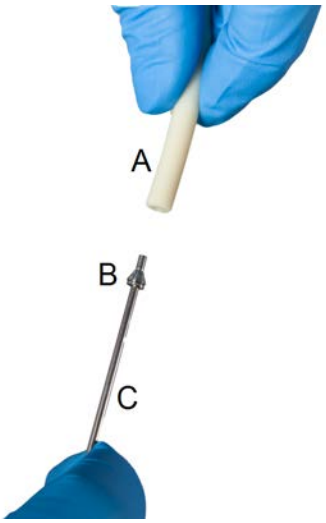
1. Power ON the degasser.
2. Set the temperature controller to the correct temperature.
3. Turn the VAC/GAS control knob on the degas station to the GAS position.



A. Cooling stations
B. Degasser stations
C. VAC/GAS control knobs
D. Vacuum gauge
E. Temperature controller

4. Wipe the gas delivery tube with a clean, lint-free cloth to remove any residue left from the previous sample.
5. Remove the stopper from the sample tube and insert the gas delivery tube into the sample tube.

Do not pierce the sample bed with the gas delivery tube. Doing so could clog the gas delivery tube. To insert the tube, tilt the sample tube nearly horizontal and tap the tube to open a passageway through the sample. Then tilt the sample tube back to a vertical position and tap the tube to settle the sample bed around the gas delivery tube.



A. Flexible tubing
B. Ferrule
C. Gas delivery tube

The end of the gas delivery tube may be hot enough to cause burns if it was stored in a heating station instead of a cooling station. Wear the cotton gloves supplied in the accessories kit and handle the gas delivery tube carefully to avoid burns.

6. Insert the stopper loosely into the sample tube. The stopper helps prevent cool moist air and dust from entering the tube, but it must be inserted loosely to allow a path for the escaping gas.

It is unnecessary to use a stopper with sample tubes having an inside diameter of only 5 mm (0.2 in.).

7. Using the appropriate heating station adapter, place the sample tube in a vacant heating station.

8. Allow the sample to degas for the required length of time. One to two hours is typical for many samples at temperatures above 200 ºC. To determine the optimum time, select the highest safe temperature for the sample, then degas it for various intervals, determining the surface area after each interval.

   For example, degas the sample for 15 minutes, then measure the surface area. Repeat this process until the surface area no longer increases. The length of time required to reach this point is the minimum amount of time required to degas the sample.

9. After the sample is sufficiently degassed, remove the sample tube from the heating station. *Do not turn off the gas flow knob.*

Keep the gas delivery tube and stopper in the sample tube while cooling to prevent contamination from the ambient atmosphere.

10. Place the sample tube, with stopper and gas delivery tube still in place, in a cooling station.

11. The sample tube should be cool enough to handle after several minutes. Remove the tube from the cooling station. Hold the stopper loosely in the tube and slowly pull out the gas delivery tube.

12. As the gas delivery tube clears the stopper, press the stopper into the sample tube to seal the tube against contamination.
13. Place the VAC/GAS control knob in the OFF position.
14. Wipe the gas delivery tube with a clean, lint-free cloth to remove any particles of sample that may have adhered to it. Then place the gas delivery tube in a cooling station until needed again.
15. Reweigh the sample tube with stopper and sample to ensure that the correct sample mass is recorded.

The weight recorded at this step may be compared to the weight recorded before preparation to determine if adequate degassing has occurred.

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5 TROUBLESHOOTING AND MAINTENANCE

The instrument requires very little maintenance to remain in top operating condition. The gas flow tubes should be wiped clean after each use and inspected weekly to ensure that they do not become clogged.

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

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

When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument. The VacPrep weighs approximately 10 kg (22 lb).

Use of a power cord or power supply not provided with the instrument could cause personal injury or damage to the equipment. If a replacement is needed, contact your Micromeritics Service Representative. Detachable power supply cords with an inadequate rating could cause significant instrument damage or physical harm.Do not add anything between the power cord and the power source that would compromise the earth ground. Do not remove or disable the grounding prong on the instrument power cord.If the equipment needs to be relocated, check with your Micromeritics service representative. The equipment must be position such that the mains supply is not obstructed and is easily accessible to disconnect the equipment from the AC main power supply.

Either no heat or insufficient heat is being supplied to the heating stations.

Cause A: Blown fuse.

Action A: Replace fuse. See ***Select Input Power on page 2 - 5***.

Cause B: A mistake was made in entering the temperature set point.

Action B: Enter the correct temperature set point on the temperature controller.

Cause C: The thermocouple or other internal component is damaged or disconnected.

Action C: Contact the appropriate service personnel.

Too much heat is being supplied to the heating stations.

Cause A: A mistake was made in entering the temperature set point.

Action A: Enter the correct temperature set point on the temperature controller.

Cause B: The thermocouple or other internal component is damaged or disconnected.

Action B: Contact the appropriate service personnel.

A newly installed fuse is blown.

Cause A: Line voltage is too high.

Action A: Check the line voltage. See ***Select Input Power on page 2 - 5***.

Cause B: A fuse with the wrong rating was installed.

Action B: Install the appropriate fuses for the input power source. See ***Select Input Power on page 2 - 5***.

Gas seems to be leaking from the gas cylinder.

Cause A: The flow control valves were left in the OPEN position when stations were not in use.

Action A: Keep the flow control valve closed on all stations that are not being used for degassing.

Cause B: The gas flow rate is set too high.

Action B: Decrease the flow rate by turning the regulator pressure control knob clockwise.

Cause C: A flow control valve is leaking.

Action C: Contact the appropriate service personnel.

No gas flow when the flow control valve is open.

Cause A: Sample material in the gas delivery tube is blocking gas flow.

Action A: Clean the gas delivery tube and replace if necessary. See ***Gas Delivery Tubes on page 5 - 9***.

Cause B: The flexible tubing is twisted.

Action B: Remove twist and replace the flexible tubing if necessary. See ***Replace Flexible Tubing on page 5 - 10***.

Cause C: Gas pressure is set too low.

Action C: Increase the flow rate by turning the regulator pressure control knob clockwise.

Cause D: Gas cylinder is empty.

Action D: Replace the gas cylinder.

Cause E: Valve inlet tubing is detached.

Action E: Check the valve inlet tubing connections. See ***Valve Inlet Tube on page 5 - 11***.

Cause F: Flow control valve is damaged.

Action F: Contact the appropriate service personnel.

Vacuum reading too high.

Cause A: Vacuum pump is powered off.

Action A: Power on the vacuum pump.

Cause B: Vacuum tubing leak.

Action B: Replace tubing if cracked.

Cause C: Vacuum port is loose.

Action C: Remove rear panel. Gently tighten the port into the internal manifold block.

Cause D: Internal leak.

Action D: Remove rear panel. Check all ferrule connections. Tighten connections if necessary.

Cause E: Sample tube fitting is loose.

Action E: Tighten the sample tube fitting.

Cause F: Sample tube fitting O-ring is damaged.

Action F: Check O-ring and replace if necessary.

Cause G: Sample tube is broken or cracked.

Action G: Replace the sample tube.

Cause H: One of the other sample tubes has a loose fitting.

Action H: Close all valves to verify proper operation of the vacuum pump.

PARTS AND ACCESSORIES

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

SAFE SERVICING

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

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

Product specific risks that may affect service personnel:

- **Electrical**. Servicing or repair could require opening the outer panels and exposing energized electrical components.
- **Fuses.** Only use fuses rated as outlined in *Select Input Power on page 2 - 5*.
- **Heating stations**. Ensure the heating stations are cool and sample tubes have been removed from all stations. Heating stations can be very hot. Allow the heating stations to cool prior to servicing.

Protective measures for these risks:

- **Electrical**. The majority of electrical components operate at low voltage (24V or less) and pose low risk when energized. Maintenance, troubleshooting, and repairs should be performed with the instrument de-energized whenever possible, in accordance with standard electrical safety guidelines.
- **Fuses**. Use of improperly rated fuses could cause damage to the equipment.
- Sample tubes must be removed prior to repair.
- Power off and unplug the degasser from the power outlet prior to servicing.

Verification of the safe state of the instrument after repair:

- Sample tubes must be removed to prevent accidental breakage.
- Gas lines connected and pressurized to normal operating pressure with no leaks.

POWER

The is designed to operate with line voltage of 100/120/230/240VAC ±10, 50/60 Hz through a standard wall receptacle. Noise-free power of the correct voltage and frequency, with a safety earth ground, should be available through a standard wall receptacle. There should be a minimum 15A rated breaker @ 100/120VAC and a minimum 7.5A @ 240VAC.

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

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

CLEAN THE INSTRUMENT

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

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

CHECK THE GAS FLOW

Gas Delivery Tubes on page 5 - 9

Valve Inlet Tube on page 5 - 11

The gas flow through the flexible tubing and gas delivery tubes should be checked once a week to ensure that there are no obstructions or leaks in the tubing. To check the flow:

1. Place the flow control valve in the OPEN position.
2. Set the VAC/GAS control knob to GAS.
3. Insert the gas delivery tube in a beaker of water. Bubbles should appear.

   Approximately the same number of bubbles should appear in the beaker of water when moved to each station. If bubbles do not appear for a particular station, detach the suspect gas delivery tube from the flexible tubing and insert the flexible tubing in the beaker of water. If bubbles appear, the problem is a clogged gas delivery tube. If the tube cannot be unclogged, replace it with a new one. If bubbles do not appear, check the valve inlet tubing.

Gas delivery tubes and flexible tubing may be ordered in sets of six from Micromeritics.

Refer to the following options if gas flow is not present in other stations:

Gas Flow Troubleshooting

Problem	What to do...
Gas pressure is set too low	Increase the flow rate by turning the regulator pressure control knob clockwise until the correct pressure gauge reading is shown. See the following *Gas Flow Rate* chart.
Gas cylinder is empty	Replace the gas cylinder.
Valve inlet tubing is disconnected	Check the valve inlet tube connections.
Flow control valve is damaged	Contact the appropriate service personnel.

Gas Flow Rate for the VacPrep

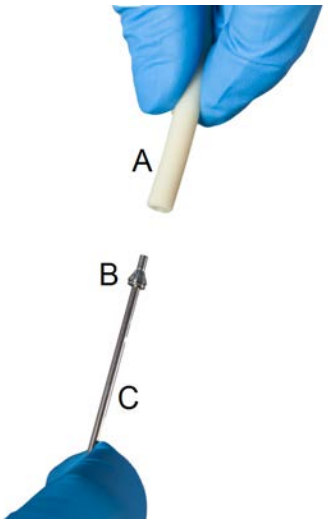
Flow cm^3/min	Gauge Pressure	
	psi	kPa
10	3	21
15	5	35

A flow rate of 10-15 cm^3/min is the maximum flow rate that would generally be used in normal operation.

Do not exceed 5 psi regulator pressure when backfilling sample tubes. If the gas pressure is greater than the recommended maximum of 5 psig (35 kPag), the sample tube could be ejected from the fitting or broken.

GAS DELIVERY TUBES

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

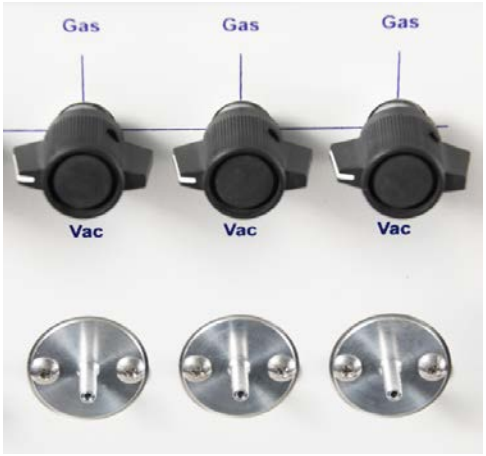


A. Flexible tubing
B. Ferrule
C. Gas delivery tube

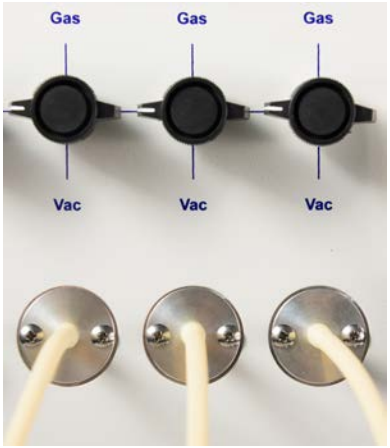
1. Remove the gas delivery tube from the flexible tubing.
2. Attach a new gas delivery tube by pressing the flexible tubing over the gas delivery tube. Ensure that the flexible tubing completely covers the ferrule at the end of the gas delivery tube.

REPLACE FLEXIBLE TUBING

1. Remove the flexible tubing from the barbed fitting on the front panel.
2. Attach new flexible tubing.



Barbed fitting

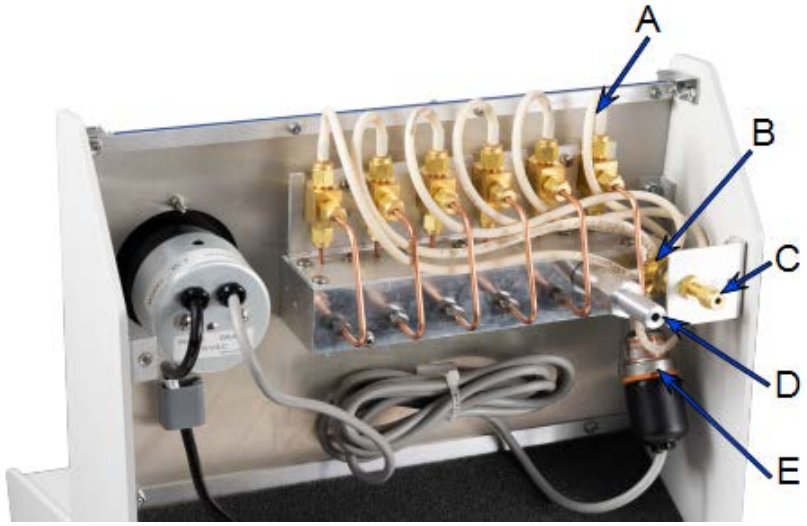


Flexible tubing installed

VALVE INLET TUBE

Flexible tubing connects the valves to the gas manifold inside the degasser. If there is no gas flow to a particular valve, the tubing could be disconnected. To check the tubing:

1. Power OFF the vacuum pump.
2. Power OFF the degasser.
3. Remove the top rear panel.
4. The tubing should be connected to the valve and to one of the spokes on the manifold. If it is disconnected at either end, connect it by pressing firmly into place. If a spoke on the manifold is loose, tighten it finger-tight plus 1/2 turn with a wrench. Do not overtighten because the gasket may become forced out of place causing the fitting to leak.

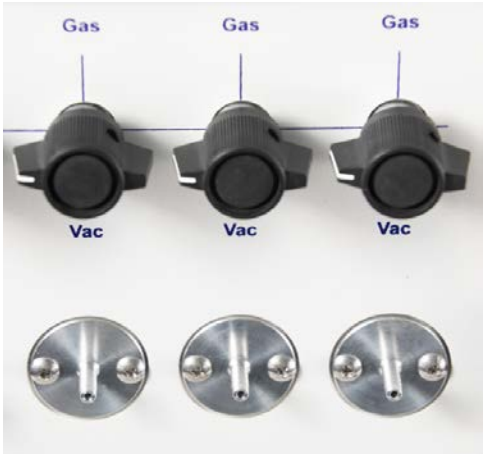


A. Flexible valve inlet tubing (one per valve)
B. Gas manifold
C. Gas port
D. Vacuum port
E. Vacuum gauge tube

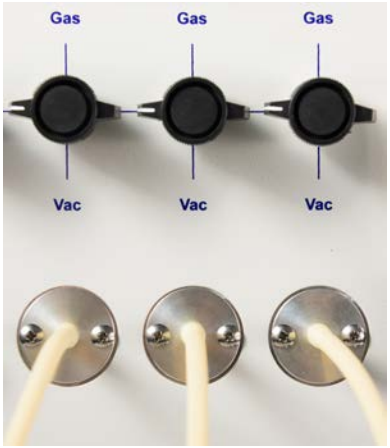
5. Replace the top rear panel.
6. Power ON the vacuum pump.

REPLACE THE VACUUM FITTINGS

1. Remove the flexible tubing from the barbed fitting on the front panel.
2. Attach new flexible tubing.



Barbed fitting



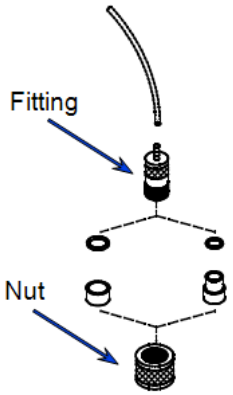
Flexible tubing installed

CLEAN AND REPLACE VACUUM FITTINGS

The interior of vacuum fittings should be wiped with a clean, lint-free cloth after each use to remove any particles of sample that may have adhered to the fitting. If vacuum fittings become clogged or damaged, they should be cleaned or replaced.

1. Remove the fittings to be cleaned or replaced from the flexible tubing.

1/2 in. or 12mm O-ring and ferrule (for 1/2 in. or 12 mm sample tubes)

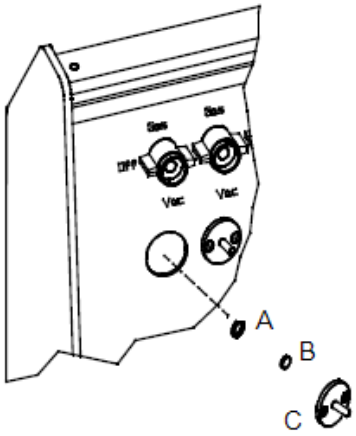


3/8 in. or 9mm O-ring and ferrule (for 3/8 in. or 9 mm sample tubes)

2. To clean the fittings, disassemble them and clean the components with isopropyl alcohol (IPA). If the fitting O-ring is cracked or broken, replace the O-ring.

Ensure adequate ventilation when using solvents for cleaning purposes.

3. Remove the two screws that hold the port nipple to the degasser. Remove the port nipple, dust frit, and O-ring. Clean all components with IPA or use an ultrasonic bath may be used. If the dust filter O-ring is cracked or broken, replace it. Reattach the dust frit, O-ring, and port nipple.



A. Dust frit
B. Dust frit O-ring
C. Port nipple

4. Attach the cleaned or replacement fittings by pressing the flexible tubing completely over the barbed nipple on the end of the fitting. Loosely attach the knurled retaining nut, ferrule, and O-ring.

PREVENTIVE MAINTENANCE

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

Maintenance Required	Frequency
Vacuum fittings	Clean after each use and replace as needed
Vacuum gauge	Calibrate when the electrical supply voltage or frequency is changed, or as needed
Gas delivery tubes	Clean after each use
Degasser exterior	Clean as needed
Flexible tubing	Check as needed for leaks
Gas flow	Check once per week

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A TEMPERATURE CONTROLLER ERROR MESSAGES

The temperature controller performs self-diagnostics when the degasser is powered ON and while operating. If an error occurs during self-diagnostics, one of the following error messages will be displayed.

LLLL

Cause: Temperature has fallen substantially below temperature scale range or the temperature sensor has failed (short circuit).

Action: Observe the display. The temperature will be displayed if it is increasing. If the temperature does not increase, the message will remain displayed. Power off the instrument and contact the appropriate service personnel.

uuuu

Cause: Temperature has risen substantially above temperature scale range or the temperature sensor has burned out (open circuit).

Action: Observe the display. The temperature will be displayed if it is decreasing. If the temperature does not decrease, the message will remain displayed. Power off the instrument and contact the appropriate service personnel.

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B SAMPLE DATA WORKSHEET FOR GAS ADSORPTION

Sample tube identification: _____

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

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

Degas Notes:

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micromeritics®

EU DECLARATION OF CONFORMITY

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

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

Hereby declares that the product:

VacPrep Sample Preparation System
Model VacPrep (061)

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

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

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

2014/35/EU (LVD)
EN 61010-1:2010 + A1:2019 - *Safety requirements for electrical equipment for measurement, control, and laboratory use — Part 1: General requirements.*

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

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

Name: John McCaffrey, Ph.D.

Title: Vice President, R & D

Signature:

Date of issue: 03/30/2021

Location: Norcross, GA USA

INDEX

T

U

V

W