micromeritics®

FlowSorb III 2305/2310

for determining
Single Point and Multipoint Surface Area

Operator's Manual

231-42801-01
March 2012

TABLE OF CONTENTS

1. GENERAL INFORMATION

2. INSTALLATION

3. OPERATION

4. TROUBLESHOOTING AND MAINTENANCE

5. ORDERING INFORMATION

APPENDICES

CHAPTER 1

GENERAL INFORMATION

- Introduction
- Conventions
- Intended Use and Precautions
- Description
- Principle of Operation
- Basic Construction
- Components and Controls
- Specifications

GENERAL INFORMATION

Introduction

This manual describes the installation and operation of the FlowSorb III; its contents are organized as follows:

Chapter 1 **General Information**. Provides a general description of the FlowSorb III, its features, safety precautions, and specifications.

Chapter 2 **Installation**. Describes how to unpack, inspect, and install the FlowSorb III.

Chapter 3 **Operation**. Provides instructions on operating the FlowSorb III.

Chapter 4 **Troubleshooting**. Provides troubleshooting information.

Chapter 5 **Ordering Information**. Provides ordering information for accessories and components available for the FlowSorb III.

Appendix A **Theory**. Provides a discussion on the theory of the FlowSorb III.

Appendix B **Other Gases and Compositions**. Provides information on recommended gas compositions.

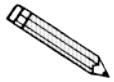
Appendix C **Specialty Gas Source**. Contains the addresses of recommended gas sources.

Appendix D **Sample Tubes**. Provides illustrations and a discussion of the sample tubes to be used with the FlowSorb III.

Index Provides quick access to a subject matter.

Conventions

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



Notes contain pertinent information with reference to the subject matter.

Cautions contain information to help you prevent actions which could damage the instrument.

Warnings contain information to help you prevent actions which could cause personal injury.

Intended Use and Precautions

Intended Use

The FlowSorb III is designed to measure rapidly the surface area on the molecular level of stable, granulated, and powdered materials. Surface area measurement can be accomplished either by a simplified, single-point procedure or by the more conventional multipoint BET technique (see Appendix A). The FlowSorb III 2310 is automatic and requires no operator intervention other than preparing, installing, and removing the sample. The FlowSorb III 2305 is manually operated.

Both models measure with diminishing effectiveness the physical properties of materials which have appreciable vapor pressures, i.e., materials which slowly evaporate or sublimate. Deposition of recondensed vapors within the instrument will eventually lead to unsatisfactory performance.

Precautions

Read all of the precautions contained in this section and observe them at all times when operating the FlowSorb III.

General

- Certain solids when heated give off corrosive vapors. Such vapors can damage instrument components. The degassing feature of the FlowSorb is intended primarily for the removal of adsorbed water vapor and non-condensible gases acquired through normal atmospheric exposure. Cleanup of unusually contaminated samples should be accomplished prior to installation on the instrument. The DeSorb 2300A is available for this use as well as for routinely speeding sample throughput.

- The gas composition detectors are sensitive to the presence of oxygen when turned on. The FlowSorb must be purged of oxygen by flowing an inert gas, or mixture of inert gases, through it for at least five minutes before power is applied. This precaution applies especially when the instrument is initially installed and after it has been stored for a period of time. Should the inert gas supply be inadvertently depleted or somehow disconnected while the FlowSorb is in operation, the power should immediately be turned off and the system purged of oxygen before reapplying power.

- Unplug the power cord before removing the rear panel to gain access to internal components. Connections carrying potentials as great as 240 V can be encountered.

- While liquid nitrogen is the most commonly employed coolant, it is possible to use liquid argon and a variety of slush baths involving organic solvents. These latter require special precautions. Be sure that no hot or burning combustible material comes near either liquid argon or flammable solvents. A lighted cigarette poses a particular hazard in both cases.

- Do not invert a sample holder once powdered sample is placed in a sample tube and the tube is attached to the holder. Particles can lodge in the quick-disconnect seals causing them subsequently to malfunction. Retention of particles in the mechanism also produces a sample weight error. Should this happen, thoroughly clean the holder and reweigh the sample before proceeding.

- Do not detach from the FlowSorb a sample holder that contains a sample at, or near, liquid nitrogen temperature and having gas still adsorbed on the sample. The sample holder seals automatically upon removal and, upon warming, previously adsorbed gas may be released, raising the pressure in the holder to a dangerous level.

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.

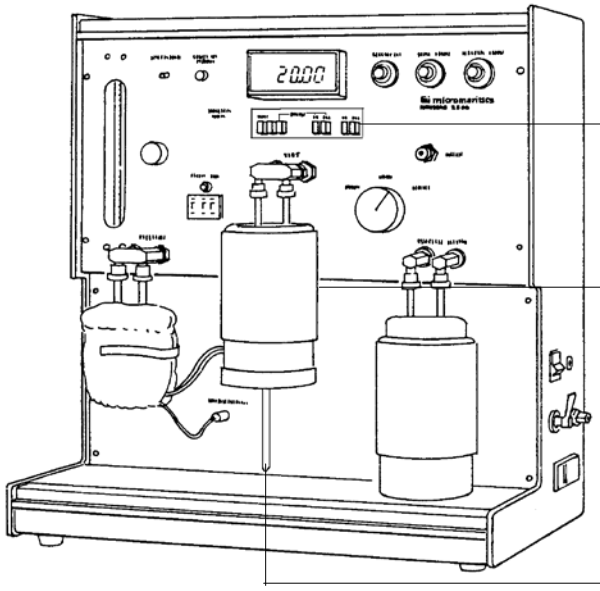
We recommend the following be observed when handling dewars containing liquefied gases:

- 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 dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some type of protective coating.
- Do not remove the mesh covering from the Dewar flask. This covering is in place to minimize the risk of flying particles if the Dewar is accidentally knocked over or dropped and broken.
- Do not handle heavy objects above the 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.

Description

The FlowSorb III is available in two models: the FlowSorb III 2305 and the FlowSorb III 2310. The FlowSorb III provides single-point and multipoint analyses, as well as the capability of reporting desorption isotherms. Adsorption isotherms can also be reported if using the FlowSorb 2305. Samples from 0.01 m^2/g to greater than 1,000 m^2/g are easily accommodated with a reproducibility of better than 0.5%. Both models are shown in the following sections with the differing features called out. All other features are the same on both models.

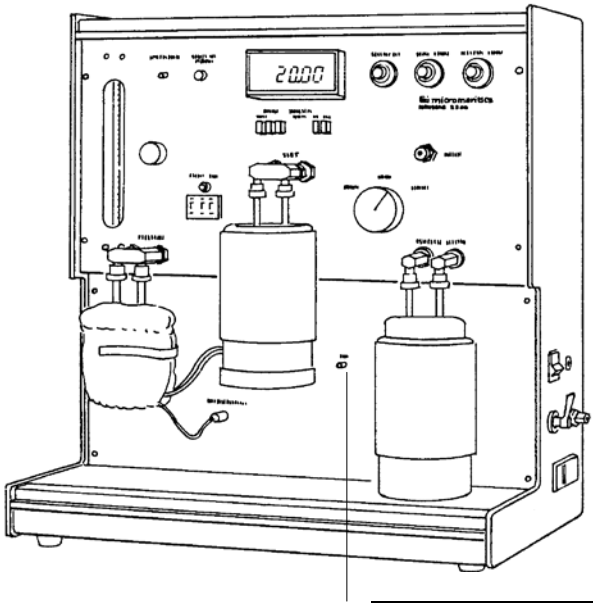
FlowSorb III 2310



The FlowSorb III 2310 allows you to measure surface area from desorption measurements. This model is automatic and requires no operator intervention after the Dewar is placed on the Dewar tray and the **Start** push button is depressed. The Dewar rises automatically about the sample and analysis begins. After adsorption is completed, the Dewar lowers and a fan turns on to warm the sample to room temperature, allowing desorption to proceed to completion.

FlowSorb III 2305

The FlowSorb III 2305 is manually operated and does not include Start and Reset buttons, a warming fan, or an elevator.



The FlowSorb III 2305 reports adsorption and desorption measurements. This model requires the operator to place the Dewar on the Dewar tray and move it up about the sample for analysis to begin. After adsorption is complete, the operator is required to remove the Dewar and warm the sample to room temperature, after which the desorption measurement is reported.

Principle of Operation

The surface area of granulated and powdered solids or porous materials is measured with the FlowSorb III by determining the quantity of a gas that adsorbs as a single layer of molecules, a so-called monomolecular layer, on a sample. This adsorption is done at or near the boiling point of the adsorbate gas. Under specific conditions, the area covered by each gas molecule is known within relatively narrow limits. The area of the sample is thus calculable directly from the number of adsorbed molecules, which is derived from the gas quantity at the prescribed conditions, and the area occupied by each.

For a nitrogen and helium mixture of 30 volume percent nitrogen, conditions most favorable for the formation of a monolayer of adsorbed nitrogen are established at atmospheric pressure and the temperature of liquid nitrogen. Parameters defining the conditions at which the monolayer forms are incorporated in the multipoint analysis result by the data treatment. Atmospheric pressure and ice water temperature may establish appropriate conditions for a n-butane and helium mixture. Other gases at other conditions are also usable.

In general, a surface area result obtained by the multipoint method using nitrogen as the adsorbate is somewhat more reliable than a single point one, and nitrogen gas is preferable over other gases. Unless special circumstances dictate, nitrogen is recommended.

Refer to Appendix A for additional information on FlowSorb theory.

Basic Construction

Functionally, the FlowSorbs III 2305 and 2310 are identical. The FlowSorb III 2305 requires the operator to submerge the sample in cryogenic liquid to induce adsorption, then to remove the cryogen and warm the sample for desorption. This operation is automatic when using the Flowsorb III 2310.

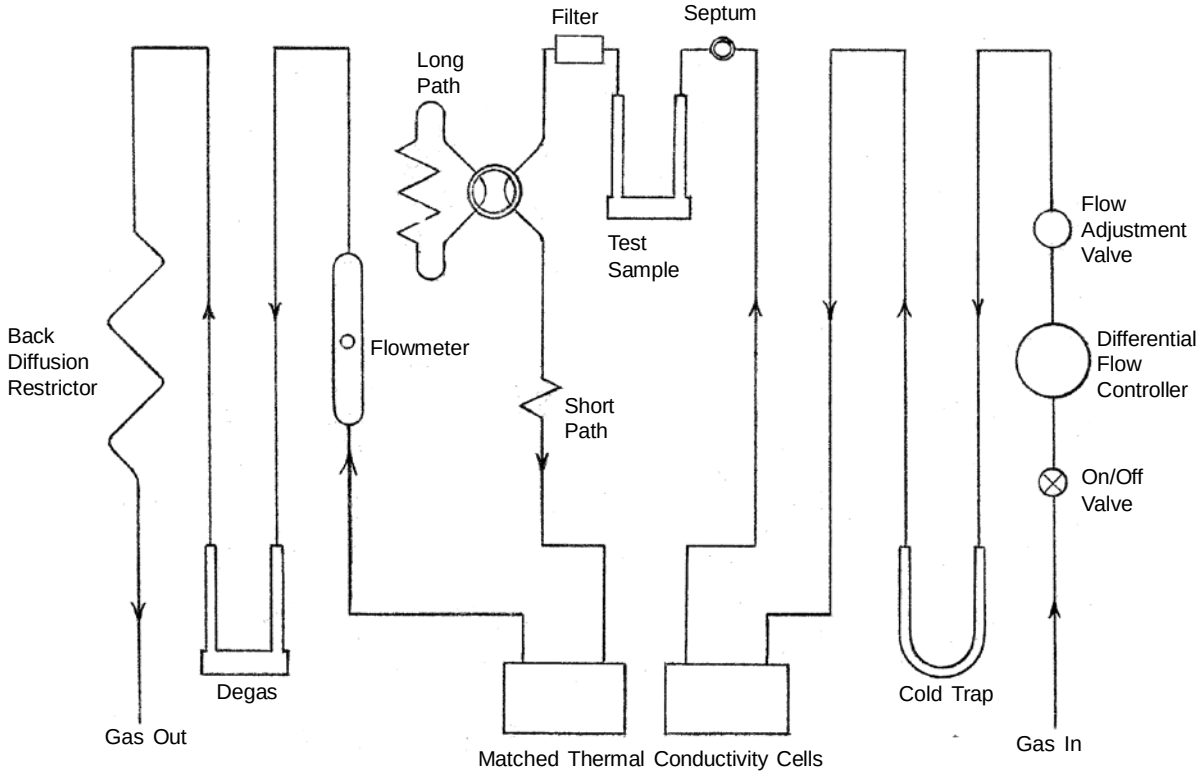


Figure 1-1. Schematic Diagram

Mechanically, the FlowSorb III consists of: (1) components to turn on and off gas flow (the ON/Off Valve), regulate the flow (the Flow Adjustment Valve), direct the flow (the Path Switch), and indicate the flow rate (the Flowmeter); (2) attachment points at which the gas passes through a freeze-out trap to remove traces of moisture that may be present in the gas (**Cold Trap**), points at which the sample is exposed to heated gas to free it of extraneous gases and vapors (**Degas**), and points at which to perform the test (**Test**); (3) a septum by means of which a known amount of gas is introduced for calibration purposes; (4) a filter to prevent powder carryover into critical parts; (5) matched thermal conductivity cells operating at a fixed temperature of 42 ±1 ^{o}C (107.6 ±1.8 ^{o}F) for gas concentration detection; (6) a valve arrangement by which gas entry into one of the cells can be delayed more than normally; (7) an automatic elevator on the FlowSorb III 2310 and a Dewar flask support platform on the FlowSorb 2305; and (8) an air blower in the FlowSorb III 2310 to warm the sample automatically to room temperature.

Electrical and electronic components include: (1) an on/off power switch, universal power entrance, and fuse block; (2) a power supply; (3) a thermal conductivity sensor and amplifier circuit; (4) a conductivity cell output linearization circuit; (5) a heating mantle temperature setting and feedback control system; (6) an indicator with appropriate selection switches for displaying the measured surface area by the single-point technique and the adsorbed gas volume for multipoint testing, the sample degassing temperature, and the thermally detected signal; (7) adjustments for initial calibration; and (8) on the FlowSorb III 2310 only, start and reset controls.

Components and Controls

This section provides a brief description of the use, manipulation, and function of major components and controls of the FlowSorb.

Sample Tube

Glass tubes in a variety of generally U-shaped configurations are used to hold the sample. Pertinent details about these tubes are given in Appendix D. The two stems of a tube are inserted into tightenable connectors on the sample tube holder. The tightenable connectors seal by means of compressed O-rings. This assembly is then installed in the **Degas** or **Test** position on the front panel. Chapter 2 provides instructions on how to install a sample tube.

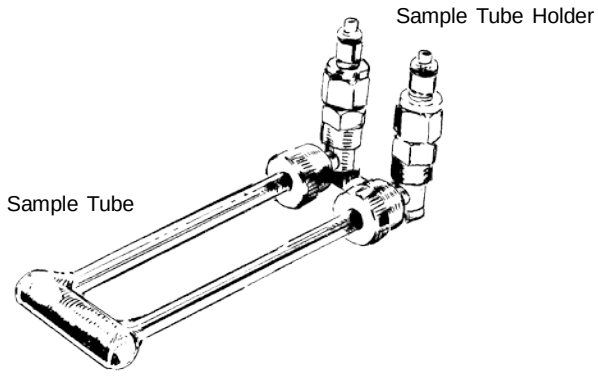


Figure 1-2. Sample Tube and Holder

Heating Mantle

A heating mantle is installed on a sample tube only when in the **Degas** position. Its purpose is to maintain the specified temperature (specified using the **Temp Set** dials). The mantle is slid upward about the sample tube and held in place with a flared U-clamp. Refer to Chapter 2 for instructions on properly installing a heating mantle.

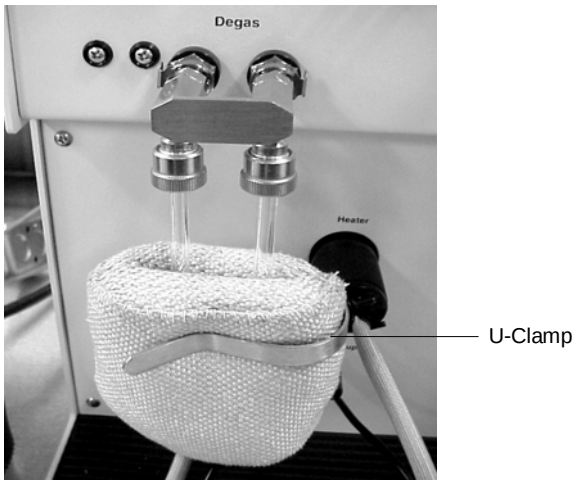


Figure 1-3. Mantle Installation

Dewar Support

A Dewar of cooling liquid around the sample tube is required to initiate gas adsorption.

- **FlowSorb III 2310**: The Dewar tray is directly below the sample analysis port near the base of the instrument platform. The Dewar is placed on the tray. The tray rises automatically when the analysis is started, inserting the sample tube into the cooling liquid within the Dewar. The tray lowers for removal of the Dewar automatically after adsorption data are collected. No operator involvement is required.

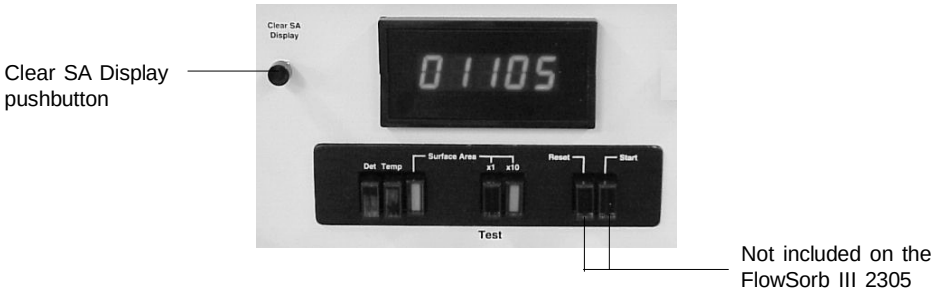
- **FlowSorb III 2305**: The Dewar is supported by a hinged platform. The platform hangs down when not in use. The Dewar is manually brought up around the sample and the platform swung upward where it locks into place. The Dewar is removed by holding the Dewar with one hand and pressing the **Release** button to the right of the platform. This causes the platform to drop out of the way, allowing you to remove the Dewar.

When the platform is raised, the instrument automatically collects adsorption data. When it is down, desorption (or evaporation) data are collected. There is no need, nor other means provided, to select between adsorption and desorption data.

Instrument Display

A single, multifunction display meter in the upper center of the front panel serves to display the following (selected by pushbuttons below the display):

- surface area for single-point analyses
- adsorbed gas volume for multipoint analyses
- degas temperature during degassing
- detector signal



The signal chosen for display can be shifted at any time in any sequence without detriment to the other signals. Surface area information and gas volume data, whether displayed or not, are retained after a test is completed until the **Clear SA Display** button to the left of the meter is depressed.

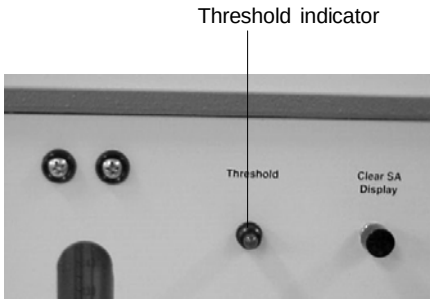
Surface area and adsorbed gas volume are displayed in terms of the quantity of sample contained in the sample tube. This means the displayed number must be divided by the sample weight to convert it to specific surface area (m^2/g) or specific volume (cm^3/g).

The detector signal is calibrated to indicate the instantaneous difference in percent nitrogen between the reference mixture and that currently in the detector. The **x1** scale indicates differences up to 100% while the **x10** scale indicates a maximum difference of 10%.

The degas temperature is displayed as degrees Celsius.

The FlowSorb III 2310 has two additional pushbuttons below the display meter. The **Start** push button is depressed after the degassed sample has been transferred to the **Test** position for analysis. The **Reset** button is used to abort an analysis; the analysis stops and the instrument returns to initial conditions.

Threshold



The **Threshold** indicator lamp to the left of the display meter flashes when the accumulated adsorption or desorption signal is greater than the threshold level. The more rapid the flashing, the greater the magnitude of the signal. This lamp is designed to begin indicating accumulation of signals as follows:

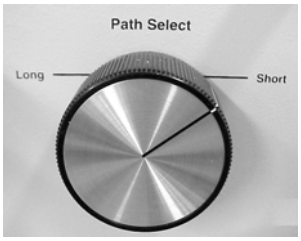
- **x1 scale**: between +0.06% and +0.07% nitrogen. It stops indicating when the signal falls between +0.03% and +0.04% nitrogen.
- **x10 scale**: between +0.010% and +0.011% nitrogen. It stops indicating when the signal falls between +0.008% and +0.009% nitrogen.

Cold Trap

A **Cold Trap** is provided for the removal of vaporous impurities in the analysis gas stream, especially water vapor. These type impurities tend to be present in small amounts in commercial gases and are detrimental to surface area evaluation.

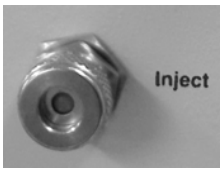
A Dewar with a segmented stopper and a longer-than-normal U-tube are provided for this use. The U-tube is attached by means of the same tightenable connectors as on sample tubes. The stopper is designed to encase snugly the U-tube stems. The stopper is segmented so that the Dewar can be initially filled and refilled in place by removing only the front half of the two-part stopper.

The Dewar is normally filled with liquid nitrogen when making surface area tests although other coolant baths can be used with special gases.



Two gas flow paths, labeled **Short** and **Long**, are provided downstream of the sample **Test** position. The purpose of both is, upon removal of the liquid nitrogen, to delay the arrival at the detector of the nitrogen-rich gas (created by sample desorption) long enough for the flow rate to return to normal. The **Short** path is usually sufficient for surface area testing. Greater surface area materials may give up too much gas over too long a time for a short path and, therefore, the **Long** path should be used.

Septum



FlowSorb calibration is accomplished by means of a septum through which known volumes of gas are injected with a precision syringe. Any septum will leak after some number of penetrations. The supplied syringe needles have side-entry ports which greatly extend this life. Nevertheless, this septum will require replacement periodically. Refer to Chapter 4 for instructions on replacing the septum.

The knurled nut retaining the septum should be finger-tightened. Insufficient or excessive tightening may cause septum leakage.

Other

The controls not covered here are fully described in connection with the operation or calibration procedures.

Specifications

The FlowSorb III has been designed and tested to meet the specifications provided below.

Feature	Specification
SAMPLE PARAMETERS	
Surface Area	
Minimum	0.1 m^2
Maximum	280 m^2
Specific Surface Area	
Minimum:	Approximately 0.01 m^2/g
Maximum:	Limited only by weighing accuracy of smallest sample
Sample Tube Volume:	1 cm^3, standard; other sizes available
Throughput:	
Single Point:	Typically 5 per hour
Multipoint:	Typically 1/2 per hour
Degassing Temperature:	35-350 °C
ACCURACY/REPRODUCIBILITY	
Low Specific Surfaces:	Typically better than ± 3% (single point method) and ± 2% (multipoint method), both within ± 0.5% reproducibility
Moderate and High Specific Surfaces:	Typically better than ± 2% (single point method) and ± 1.5% (multipoint method), both within ± 0.5% reproducibility
ELECTRICAL	
Voltage:	100, 120, 220 or 240 VAC ± 10%
Operating Current:	1.25 A (100/120 VAC) 0.75 A (220/240 VAC)
Frequency:	50/60 Hz

Feature	Specification
SUPPLIES	
Gas:	Mixtures, with helium, of nitrogen, argon, krypton, carbon dioxide, ethane, n-butane, and other non-corrosive gases. A mixture of 30% N_2 and 70% He is recommended for single-point analyses. Mixtures of He and approximately 5, 12, 18, and 24% N_2 are suggested for multipoint use.
Coolant:	Liquid nitrogen or argon, solvent slush baths, ice water as appropriate for adsorbate
EXPOSED MATERIALS	
Sample Tube:	Borosilicate glass, usable to 400 °C
Internal Components:	Type 316 stainless steel, chrome plated brass, copper, Buna-N
ENVIRONMENT	
Temperature:	15-32 °C (59-90 °F) operating; 0-50 °C (32-122 °F) storing and shipping
Humidity:	20-80% relative (non-condensing)
CABINET	
Dimensions:	46.5W x 53H x 30.5D cm (18.3W x 20.9H x 12D in.)
Weight:	
FlowSorb 2305	18 kg (40 lbs)
FlowSorb 2310	20 kg (44 lbs)

CHAPTER 2

INSTALLATION

- Unpacking and Inspecting the Equipment
- Selecting the Location
- Selecting the Input Power
- Connecting the Gas Supply
- Installing and Removing a Sample Tube
- Installing a Heating Mantle
- Using a Cold Bath

INSTALLATION

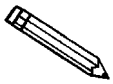
This chapter describes how to

- unpack and inspect the equipment
- select an appropriate location for the FlowSorb
- install the FlowSorb

Unpacking and Inspecting the Equipment

The FlowSorb III instrument and its accessories should be visually inspected as soon as unpacked to ensure that all items have been received and none has sustained physical damage.

When you unpack the shipping cartons, carefully compare the packing list with the equipment actually received, while checking for equipment damaged during shipment. Be sure to sift through all packing materials before declaring equipment missing.



It is very important to save the shipping cartons when equipment is to be declared as damaged or lost. The inspector (or claim investigator) must examine the cartons prior to completion of the inspection report.

Equipment Damage or Loss During Shipment

When equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The carrier, not the shipper, is responsible for all damage or loss. In the event of equipment damage or loss during shipment, contact the carrier of the equipment immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, equipment is received which is not in working condition. When it is necessary to return equipment (damaged either during shipment or while in use) to Micromeritics for repair or replacement, the following procedure should be followed:

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 identify the defective equipment, noting the defect and circumstances, if any, under which the defect is observed.

3. Reference the sales order or purchase order, and provide the date that the equipment was received.

4. Notify the Micromeritics Service Department of the defect and request shipping instructions. The service department will assign a Returned Materials Authorization (RMA) number. Write the RMA number on the outside of the shipping carton.

Selecting the Location

The instrument performs best in a regulated temperature environment. It should be installed on a workbench 75 to 90 cm (30 to 36 in.) high in a location free of drafts from either a forced-air heating or cooling system. It should not be located near a window through which sunlight may periodically fall.

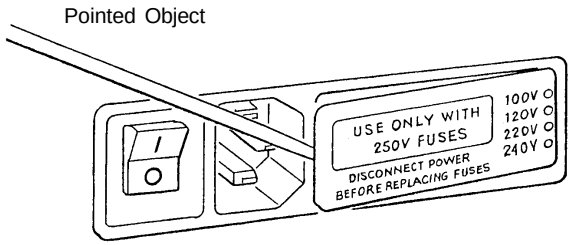
A square meter (10 ft^2) of free space to one side at least and a few centimeters to the rear of the instrument should be provided for working space. Ready access to an analytical balance capable of weighing to 1 mg and a drying oven, preferably a vacuum oven, for sample preparation is advantageous. Space near the instrument in which to mount securely the appropriate gas cylinder, or cylinders, is also required.

Selecting the Input Power

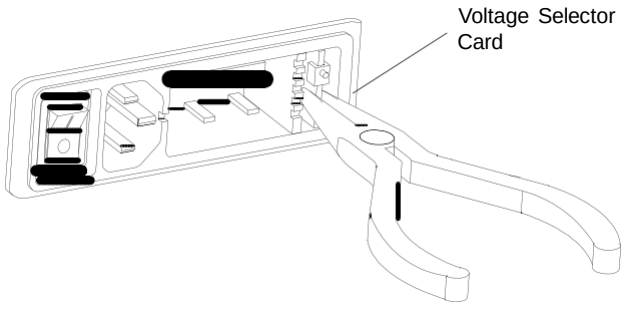
All instruments leave the factory set for 120 VAC and with the line fuse removed. The correct setting of the universal power entrance must be checked and the appropriate fuse installed before the FlowSorb can be operated. The FlowSorb is designed to operate with 100, 120, 220, or 240 VAC at 50 or 60 Hz. Voltage selection and fusing are made at the power connector, which is located on the rear panel of the unit.

The power cord should be disconnected from the FlowSorb before removing the cover from the input power connector. Failure to disconnect the power cord could result in electrical shock.

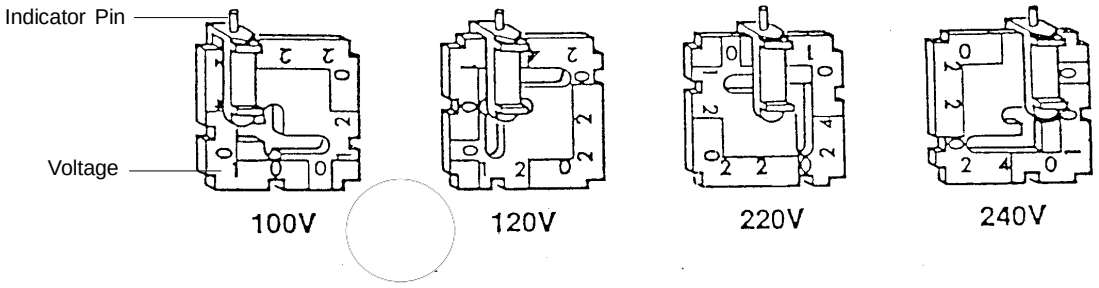
1. Make sure the power cord is disconnected from the FlowSorb.
2. Using a pointed object, remove the fuse block and cover assembly from the power connector on the side panel of the FlowSorb.



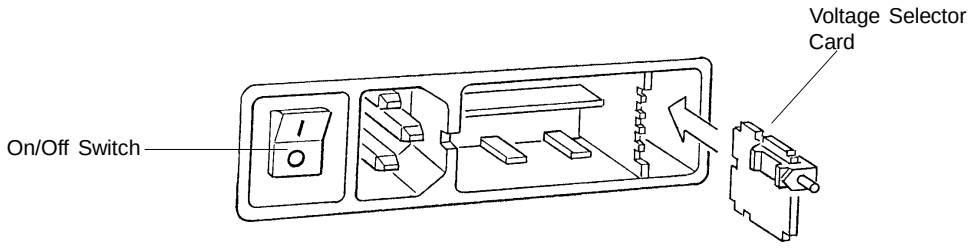
3. Pull the voltage selector card straight out of the power connector housing.



4. Orient the voltage selector card so that the desired voltage is indicated at the bottom. Orient the indicator pin so that it points upward as shown in the following illustration.

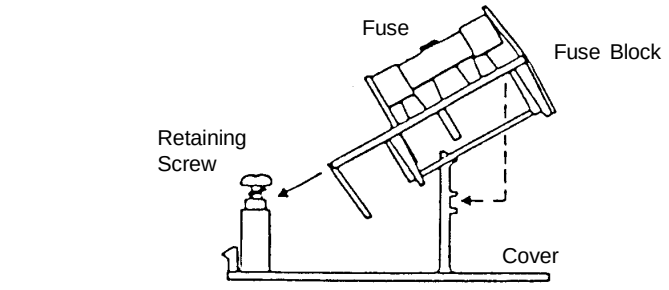


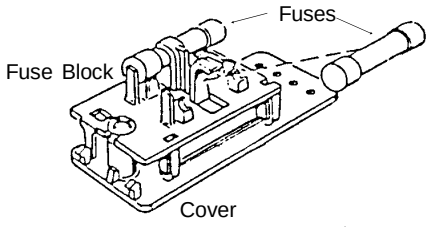
5. Insert the voltage selector card into the power connector housing with the edge containing the desired voltage first and with the printed side facing the power ON/OFF switch.



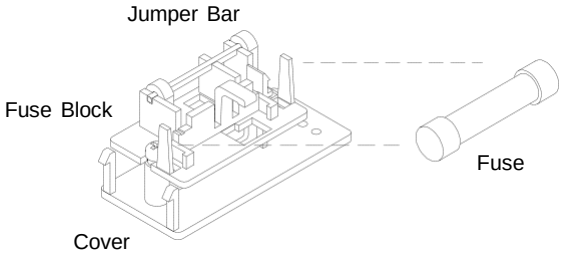
6. Fuse the power line according to local safety practices.

The double-fuse arrangement requires 20-mm fuses.





DOUBLE-FUSE ARRANGEMENT



SINGLE-FUSE ARRANGEMENT

The power cord must be disconnected from the analyzer when installing or replacing fuses. Failure to do so could result in electrical shock.

a. Observe the position of the fuse block, using the illustration shown above for reference.

- If the single-fuse arrangement is desired, position the fuse block so that the side with the single fuse slot and the jumper bar is away from the cover.

- If the double-fuse arrangement is desired, position the fuse block so that the side with the double fuse slots is away from the cover.

b. If the fuse block is positioned properly for the desired fusing, proceed to Step c.

If the fuse block is not positioned properly:

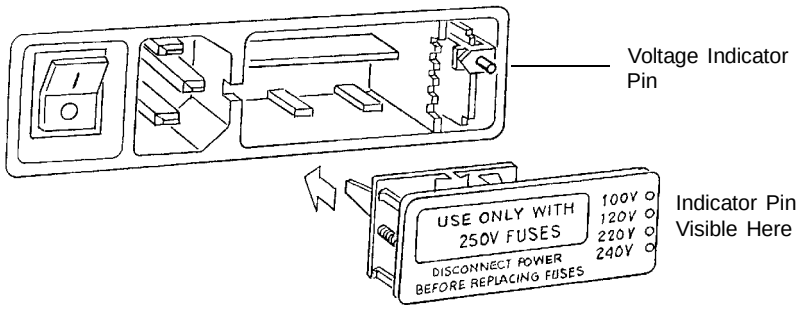
1) Remove the fuse block retaining screw.
2) Lift the fuse block from the cover.
3) Rotate the fuse block.
4) Mount the fuse block to the cover.
5) Replace the retaining screw.

The fuses used in the FlowSorb must be identical in type and rating to that specified. Use of other fuses could result in electrical shock and/or damage to the analyzer.

c. Insert appropriate fuse(s) for the input power source. Refer to the chart below for the appropriate fuse rating.

Power Source	Fuse
100-120 VAC	2.0 Amp
220-240 VAC	3.15 Amp

7. Insert fuse block and cover assembly into input power connector and snap it into place. Once the fuse block and cover assembly are in place, the position of the indicator pin shows the input power selected.



Connecting the Gas Supply

A variety of gas mixtures may be employed as described in Appendix B. The most common for single-point surface area evaluation is a mixture of 30% nitrogen and 70% helium. Several mixtures containing between approximately 5 and 24% nitrogen are required when making multipoint surface area analyses. Premixed gases are conveniently employed for single-point and multipoint surface area measurements. A Multigas Manifold is recommended for performing multipoint analyses (refer to Chapter 5 for ordering information).

Whatever the gas mixture or its source, it should be regulated to a pressure of at least 0.11 MPa (15 psig) by a reliable, leak-tested regulator. Suitable regulators are available from Micromeritics (refer to Chapter 5 for ordering information).

Some commercial regulators incorporate internally a lubricant that can migrate and contaminate downstream systems. These types must be avoided.

One regulator is required to make single-point tests. As many as four can be used to make multipoint analyses with pre-mixed gases. Micromeritics uses and recommends the use of research-grade gases. If unobtainable, the highest purity gas conveniently available will probably prove satisfactory. Since the same gas is employed in calibration as in making measurements, small proportions of such gases as argon, carbon dioxide, carbon monoxide, and methane will have an insignificant effect on results. Water vapor, however, is particularly degrading; gas having a dew point of at least -67 °C (-88 °F) should be sought.

Appendix C lists addresses worldwide of one specialty gas source as supplied by that company.

Connect the gas supply as follows:

1. Attach an appropriate regulator to the gas bottle. Leave the gas bottle shut-off valve closed until instructed otherwise.

2. If the regulator has a 1/8-in. outlet, proceed to the next step. If the regulator has a 1/4-in.outlet, attach the reducer fitting to the outlet of the regulator shut-off vlave.

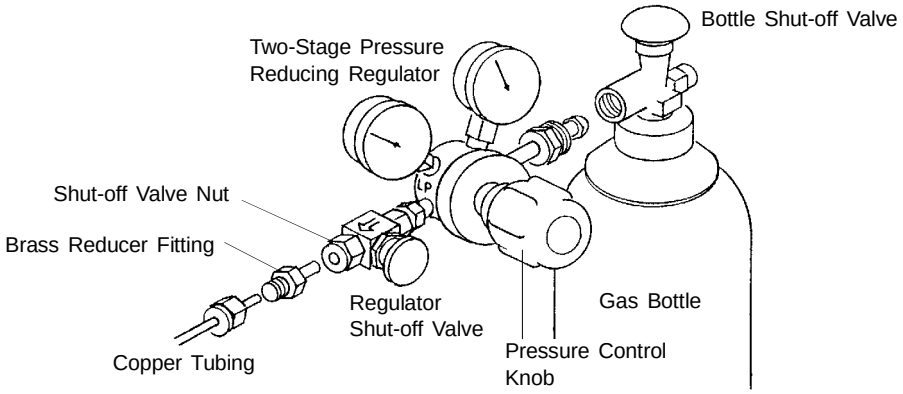


Figure 2-1. Typical Gas Pressure Regulator Assembly

3. Tighten the regulator shut-off valve nut.

Do not over-tighten the fittings. Doing so can collapse the brass fitting and cause a leak.

4. Attach the copper tubing to the regulator or to the brass reducer fitting.

5. Purge the regulator as follows:

   a. Close the regulator shut-off valve by turning it fully clockwise. b.

   Turn the pressure regulator control knob fully counterclockwise.

   c. Open the gas bottle valve by turning it counterclockwise, then close the gas bottle valve.

   d. Observe the gas bottle pressure gauge. If the pressure decreases, tighten the nut connecting the regulator to the gas bottle. If the pressure is stable, proceed to step e.

For hazardous gases, make sure the gas supply equipment is adequately vented.

   e. Turn the pressure regulator control knob clockwise until the outlet pressure gauge indicates 0.11 MPa (15 psig). Then open the regulator shut-off valve by turning it counterclockwise. This action will purge the regulator and copper tubing.

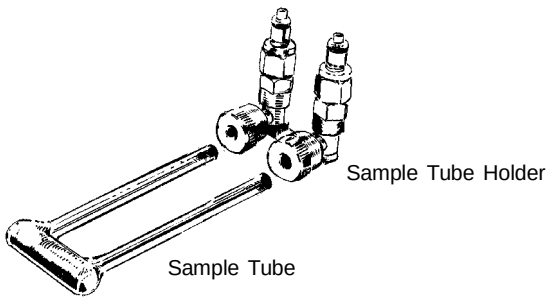
   f. Make sure the gas bottle valve is completely closed.

6. Attach the other end of the copper tubing to the gas fitting on the side of the instrument.

Safety demands that pressurized gas cylinders be securely fastened to a rigid support.

Installing and Removing a Sample Tube

Glass, U-shaped sample tubes are used to hold the sample for analysis. (Refer to Appendix D for the types of sample tubes available.) Sample tubes are installed onto a sample tube holder and the holder installed onto the connectors on the FlowSorb. Sample tube assemblies (tube and holder) are installed in the same manner in both the **Degas** and **Test** positions.



Install the sample tube assembly as follows:

1. Loosen the connector nuts on the sample tube holder.

2. Insert the stems of the sample tube fully into the sample tube holder; then tighten the connector nuts.

3. Insert the sample tube assembly into the two connectors provided at either the **Test** or **Degas** position. Push firmly until you hear it snap into place.

Remove the sample tube assembly as follows:

1. Place the thumb and forefinger on the two release tabs of the connectors on the FlowSorb.

2. Enclose the sample tube lightly with the remaining fingers and palm of your hand.

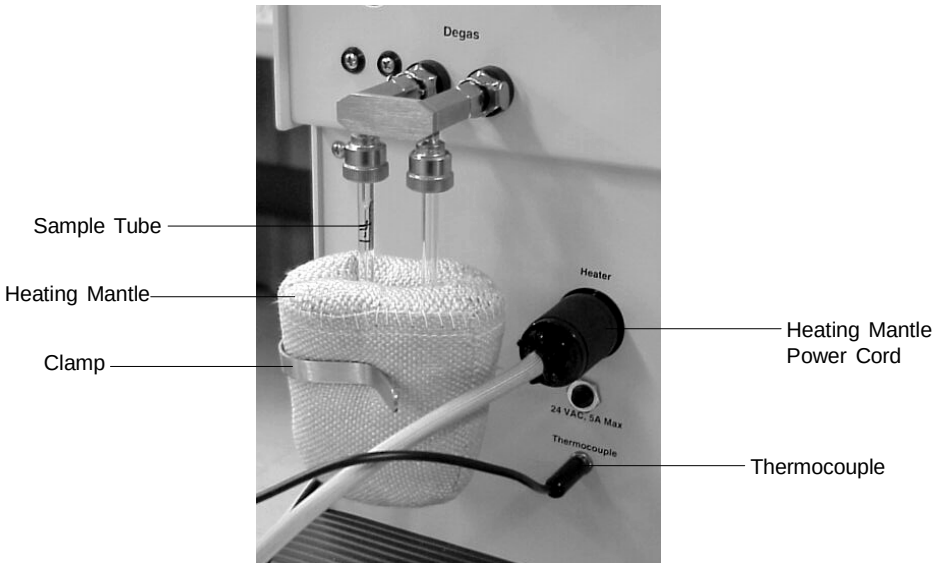
3. Squeeze together the two release tabs, then gently pull out the sample tube assembly.

Installing a Heating Mantle

A heating mantle is used on a sample tube to assist in maintaining a specified temperature. Heating mantles should be used only on samples being degassed (in the **Degas** position).

1. Slide the heating mantle upward around the sample tube.

2. Holding the mantle in place with one hand, gently push the clamp horizontally to the left (or right as shown here) about the mantle at two or three centimeters (approximately one inch) below the upper edge.



3. Plug the heating mantle power cord and the thermocouple plug into their appropriate connectors. The indicator immediately above the register alternates on and off, indicating power is being applied as needed to attain and hold the set temperature. Internal electronic circuitry limits the maximum temperature attainable to 400 °C. Attempts to exceed this limit will result in a flashing display if **Temp** is depressed.

Using a Cold Bath

Liquid argon, liquid oxygen, dry ice and acetone, ice water, and various other slush baths prepared from low melting point liquid solvents partially frozen by mixing with liquid nitrogen may be employed. Liquid nitrogen is most frequently employed. Provision for a source of supply and a suitable storage reservoir must be provided by the user.

CHAPTER 3

OPERATION

- Preparing the FlowSorb for Operation
- Degassing the Sample
- Performing a Single-Point Surface Area Measurement
- Performing a Multipoint Surface Area Measurement
- Preparing the FlowSorb for Idle Periods

OPERATION

Both FlowSorb III models permit the measurement of surface area by a single-point and multipoint determination. It is essential that you become familiar with the single-point test before attempting a multipoint analysis.

Preparing the FlowSorb for Operation

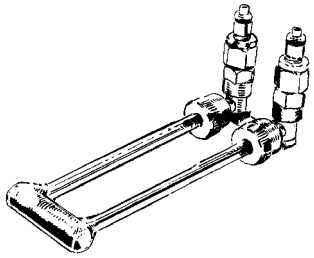
For best results, the FlowSorb should be free of air before performing analyses. It is not necessary to purge air from the system before every analysis. Purging should be performed when the FlowSorb III:

- is initially put into operation
- has been idle for several days

These instructions assume the FlowSorb has been installed as described in **Chapter 2**, **Installation**.

Remember to use caution when handling Dewars. Refer to *Precautions* in Chapter 1 for a list of recommended precautions.

1. Install an empty sample tube in a sample holder (refer to **Installing a Sample Tube** in Chapter 2). Be sure the sample tube is fully inserted and that the seals on both stems are secure.

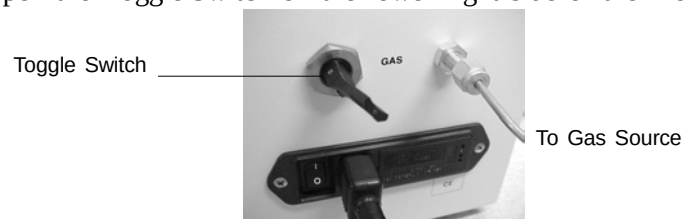


2. Install the holder onto the instrument in the **Test** position, making sure that it snaps fully into place.

3. Install an empty sample tube in a sample holder, then install the sample holder onto the instrument in the **Degas** position

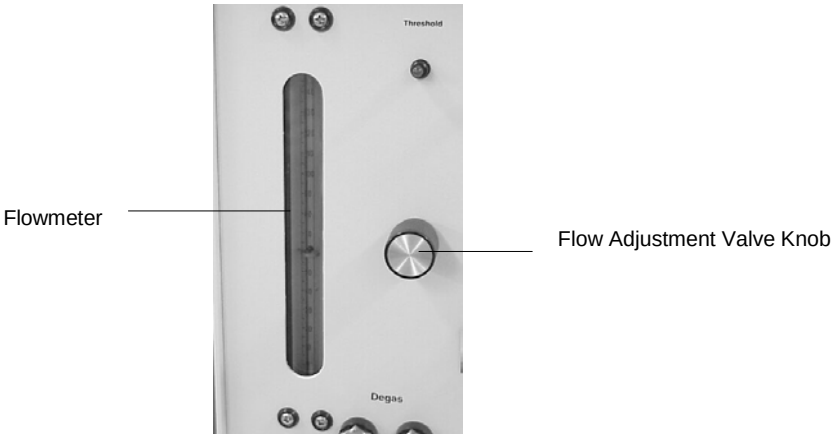
4. Install a U-tube in the **Cold Trap** position, being sure both stems are fully inserted and securely sealed.

5. Open the Toggle Switch on the lower right side of the FlowSorb to admit gas.



6. Using the Flow Adjustment Valve Knob, adjust the flow until the float is centered on or near the calibration mark on the flowmeter tube. If you are unable to accomplish this, the gas supply pressure is maladjusted. Readjust the supply pressure to approximately 0.11 MPa (15 psig) (refer to **Gas Supply** in Chapter 1).

Note that the Flow Adjustment Valve Knob should not be used to stop the gas flow. Only the Toggle Switch should be used.



7. Install a Dewar about the U-tube on the **Cold Trap**, orienting it such that the split insulating stopper can be put in place.

8. Place the rear half of the stopper on top of the Dewar.

9. Pour liquid nitrogen into the Dewar through the open front space until the liquid is within a centimeter (0.5 in.) of the lower stopper surface.

10. Place the front half of the Dewar stopper on top of the Dewar.

11. Turn the **Path** knob to the desired position to begin the purging process.

    - **Short** requires 5 to 10 minutes
    - **Long** requires 20 to 25 minutes

    Both paths should be purged.

12. After the required purging time, turn on the power switch located on the lower right side of the instrument. Allow an additional 30 minutes of purging for complete temperature equilibration and operational stability.

Sample preparation should be begun immediately so that it will be proceeding as equilibration is achieved. Adsorption measurements, likewise, need not be delayed.

Degassing the Sample

Surface areas can be measured reliably with as little as 0.1 m^2 of total sample surface or as much as 280 m^2. However, results are most accurately and quickly determined if sample quantity is adjusted to fall within 0.5 to 25 m^2 range. This usually is unlikely on the first attempt with a new material. On subsequent analyses, however, sample quantity can be optimized.

Sample weight must be established to express the final result as a specific surface area (square meters per gram) or as specific volume (cubic centimeters per gram). True weight is most reliably determined after the sample has been freed of whatever gases and vapors — especially water vapor — it may have picked up from the atmosphere. This means the weight is best established after the measurement is completed. The sample tube stems should be stoppered as soon as the tube is removed. The tube, sample, and stoppers can then be weighed on an analytical balance and the sample weight established by subtracting the weights of the tube and stoppers as determined either before or subsequent to the main weighing.

Except for the warning in **Intended Use and Precautions** in Chapter 1 against contaminated and decomposing materials that may give off corrosive or condensing vapors, sample pretreatment (or outgassing) is readily accomplished on the FlowSorb. However, samples which have been predried, perhaps in a vacuum oven, will clean up faster. Preconditioning of samples is recommended whenever possible.

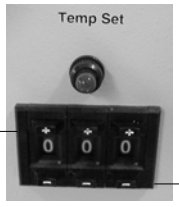
1. Pour the sample into a dry, clean sample tube, being careful to leave some free space above the sample for the unimpeded flow of gas.

2. Install the tube securely in a holder and attach the holder to the connector labeled **Degas**.

3. Place a heating mantle about the sample (refer to **Installing a Heating Mantle** in Chapter 2).

4. Plug the heating mantle power cord and thermocouple into their appropriate connectors.

5. Specify the desired temperature using the **Temp Set** dials. The actual temperature to which the sample is being subjected at any time thereafter can be read on the instrument meter by depressing the **Temp** switch just below the instrument display.



Use + to increase the setting.

Use – to decrease the setting.

Degassing Considerations

Heating to the highest temperature consistent with the thermal stability of the sample gives the most rapid degassing. The upper limit of both a glass-fiber heating mantle and a borosilicate glass sample tube is about 400 °C. Degassing at 200 to 250 °C for 15 to 20 minutes usually is adequate. Many materials degas well at 120 to 150 °C.

Repetitively adsorbing and desorbing nitrogen at the **Test** position can be employed as a degassing means for those materials that cannot tolerate elevated temperature degassing.

There is only one sure way to establish degassing requirements. A sample is adequately degassed when further treatment results in no increase in measured surface area. With unfamiliar materials, you may wish to perform a series of tests varying either time of treatment or temperature or, perhaps, both to establish degassing conditions. (For further insight on establishing degassing parameters, contact your local Micromeritics sales engineer and request Application Note No. 121, Establishing Sample Degassing Conditions for the FlowSorb.)

Sample Considerations

A portion of some very fine, fluffy powders may be carried from the sample tube by the gas passing over the sample. This problem can be corrected by one of the following:

- Reduce the quantity of sample; such a fine powder is likely to exhibit a comparatively great surface area. This increases the free space above the sample and reduces the gas velocity over it.

- Insert a bit of glass wool into the exit stem of the sample tube to confine the sample. Facing the installed sample tube, the exit stem is to the right.

- Reduce the gas flow rate; the only significant effect here is a lengthening of analysis time. Select a number on the flowmeter below the standard marked level, say 30, and adopt this as the standard mark. Note that both calibration and testing must be carried out at the same flowmeter setting. Make the test in all other respects as before.

The level of the liquid nitrogen in the **Cold Trap** during surface area measurements should be held more or less constant by replenishing it as necessary every 20 to 30 minutes, preferably *between* analyses. Avoid adding liquid nitrogen while an analysis is in progress. The small, temporary flow rate change thereby introduced may slightly alter results. Rapid and disruptive detector drift may occur if the liquid nitrogen in the cold trap is allowed to evaporate below the level of the cold trap tube.

Performing a Single-Point Surface Area Measurement

Calibration

The FlowSorb III should be calibrated at the start of each 8-hour operational period. Calibration is accomplished by injecting a precise volume of a gas mixture into the instrument through a septum using a syringe and needle. Side-port needles are supplied to prolong septum life; sharp-edge needles should be avoided. The septum requires periodic replacement, nevertheless. Replacement is required more frequently when using the larger needle of the 10 mL syringe (approximately after 20 injections) than with the smaller needle of the 1 mL syringe (approximately after 100 injections). Refer to Chapter 5 for ordering information.

A reference percentage potentiometer (small screwdriver adjustment on right panel) may occasionally need to be adjusted when replacing a gas mixture of approximately 30% N_2/70% He with another supply to account for small differences in gas composition, as well as to improve agreement of adsorption and desorption results (refer to Appendix B for details). No adjustment is required when making multipoint tests since adsorption data are not used.

As discussed in Appendix A, Theory, 1.0 cm^3 of nitrogen gas corresponds to 2.84 m^2 of sample surface in the FlowSorb when employing a mixture of 30 mole % N_2 and 70 mole % He. Liquid nitrogen is used to set the adsorbing temperature when ambient conditions are 22 °C and 760 mmHg. Procedurally the steps are identical for other gases and conditions with the exception that another constant is applicable and some electronic adjustments may be required (refer to Appendix B, Other Gases and Compositions).

Small ambient temperature deviations from 22 °C are relatively insignificant, but a calibration value differing considerably from 2.84 may be required in some localities far removed from sea-level elevations.

Perform the calibration as follows:

1. Insert empty sample tubes into two sample holders; install one at the **Degas** position and one at the **Test** position.

2. Install a U-tube at the **Cold Trap** position.

3. Install a Dewar containing liquid nitrogen about the U-tube.

4. Depress the **Det** pushbutton.

6. Select the **x1** or **x10** pushbutton; the **x1** setting is recommended for use with the **FlowSorb 2310**.

7. Turn the **Path** knob to the **Short** or **Long** position (whichever is appropriate for the sample to be analyzed subsequently).

8. **FlowSorb 2310**: Press the **Reset** button to lower the Dewar tray to its lowest postion

   FlowSorb 2305: Press the release tab to the right of the Dewar tray to allow the tray to hang down**.**

9. Fill a 1-mL precision syringe to 1 mL with nitrogen gas:

   a. Hold the needle tip immediately above the level of liquid nitrogen in the cold trap Dewar. The evaporating liquid provides an atmosphere of pure nitrogen.

   b. Flush the syringe a few times to ensure a proper fill; wipe the needle tip free of accumulated frost.

   c. Lay the syringe aside (perhaps on the rubber mat of the FlowSorb) so that the gas can equilibrate to room temperature.

10. While the syringe is equilibrating, use the FLOW knob to regulate the nitrogen-helium flow through the instrument so that the flowmeter float is centered on the marked line.

11. Use the **Coarse** and **Fine Zero** knobs to zero the instrument. Observe the display for a few minutes to establish system stability. A change of no more than 0.01 should be achieved.

    The two primary factors leading to excessive instability are:

    • instrument warm-up, which may require up to 30 minutes after initial power application, and

    • the progression of residue from a previous analysis, which can require as much as 10 minutes for elimination.

    Be sure these situations do not exist when setting the zero. Once set, the zero should not be readjusted unless a sustained change beyond ± 0.02 units occurs.

12. Depress the **Surface Area** and the **Clear SA Display** pushbuttons.

13. Hold the syringe at the rear flange so that body heat does not affect the gas volume contained by the syringe barrel.

14. Insert the needle fully into the **Inject** septum.

15. Inject the gas at a moderate rate. Withdraw the needle when the syringe is completely discharged.

    After approximately one minute (five minutes for **Long Path**), the **Threshold** light begins to flash and the indicator starts to accumulate surface area information. When the **Threshold** light ceases to flash for 15 to 20 seconds (approximately after three minutes), the accumulation may be considered complete. Completeness may be confirmed by pressing the DET. pushbutton; it should be 0.02 or less.

16. Use the **Calibrate** knob to set the surface area value (S); this is the number shown on the instrument display when **Surface Area** is depressed (refer to equation (7) of Appendix A).

    The surface area value is 2.84 when room temperature is approximately 22 °C, atmospheric pressure is near 760 mmHg, and a gas composition of 30% N_2 and 70% He is being used. The surface area value can vary considerably for conditions different from the above.

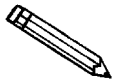
The FlowSorb III is now calibrated. Confirmation of calibration is established as deemed necessary by making repeat injections. Reproducibility should be within ± 0.02 units on the display meter.

Analysis

Materials with unknown characteristics are best tested with the multiplier set on **x1**. Only samples having less than 3.5 m^2 of surface area are ever advantageously tested with a setting of **x10** and then only when the sample is a slow desorber (perhaps due to long or tortuous passageways within the sample itself). Using the **x10** setting for excessively large surface area samples may overload the signal processing circuit and result in some error. Should this occur, a high-pitched tone will be heard above the normal clicking sound. Select the **x1** or **x10** scale and the **Short** or **Long Path** as appropriate for the sample (and as employed during calibration).

1. When adequately degassed, transfer the sample from the **Degas** position to the **Test** position.

   A small amount of air is introduced during the transfer process. Press **Det** so that the display reveals the passage through the system of this air pulse (occurring within a minute or so after sample transfer). When the display returns to within 0.02 of zero, press the **Surface Area** and the **Clear SA Display** pushbuttons to clear the display.



If desired, you may start degassing another sample after transferring the current sample to the *Test* position.

2. Prepare a Dewar of liquid nitrogen for the sample to be analyzed. The level of liquid nitrogen should be approximately 1 to 2 cm (0.4 to 0.8 in.) below the Dewar lip.

3. Check the level of the **Cold Trap** Dewar; replenish if necessary. Avoid replenishing the Dewar during analysis.

4. The steps for the remainder of the analysis depend on whether you are using a FlowSorb III 2310 or 2305; advance to the appropriate set of instructions and proceed accordingly.

FlowSorb III 2305

1. Raise the Dewar of liquid nitrogen about the sample and lift up on the Dewar tray until it locks into position; this starts the analysis.

   - After approximately one minute (five minutes for the **Long Path**) the **Threshold** light begins to flash and the instrument display shows data accumulation.

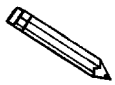
   - Ensure that the flowmeter float returns to its normal position before data accumulation begins (**Threshold** light flashing). If it does not, wait until the analysis finishes (flashing ceases) and restart the analysis using the LONG path or a lesser quantity of sample.

   - When the flashing ceases to no more than once in 15 to 20 seconds (approximately three or four per minute), or the indication with **Det** depressed has returned to 0.02 or less, adsorption is complete.

2. Record the value if desired. This value represents the sample surface area in square meters obtained from the adsorption of nitrogen gas. The adsorption peak is not as sharp and clean as the desorption one and cannot be integrated with quite the precision of the latter. However, surface areas obtained by adsorption and desorption are virtually identical for many materials when the reference potentiometer is properly adjusted (described in Appendix B). The desorption procedure is unnecessary when this is found to be the case.

3. Depress the **Clear SA Display** button to clear the display for registration of the desorption value.

4. Remove the Dewar of liquid nitrogen; allow the sample to equilibrate to room temperature so that desorption data can be calculated.



You may wish to immerse the sample tube in a beaker of room temperature water to speed up the warming process.

Special sample tubes such as those having ground glass joints must be warmed with care as they are more subject to stress development which can lead to breakage.

5. Continue the warming procedure until the flowmeter float returns to its normal level.

   • As with adsorption, the **Threshold** light flashes and the display accumulates data for several minutes.

   • When the flashing ceases to no more than once every 15 to 20 seconds (or the **Det** indicator has returned to 0.02 or less), the sample surface area is displayed.

6. Record this value along with sample descriptive information and weight. This number divided by the sample weight in grams is the sample specific surface area in square meters per gram.

FlowSorb III 2310

1. Place the Dewar on the Dewar tray.

2. Depress the **Start** button.

   • The Dewar rises about the sample and analysis begins.
   .
   • Observe the flowmeter float; ensure that it returns to its normal position *before* data accumulation begins (indicated by the flashing of the **Threshold** light). If the float does not return to its normal position, depress the **Reset** button to abort the analysis. Then repeat the test using the **Long Path** or a lesser quantity of sample.

   • The **Threshold** light begins to flash indicating data accumulation has begun. After approximately one minute (five minutes for the **Long** path), data accumulation is shown on the instrument display. The values shown will be negative numbers; do not be concerned. The surface area number derived from the subsequent desorption operation will display as a positive value.

   • The Dewar tray lowers automatically and a warming fan is activated to bring the sample to room temperature.

   • After the sample has warmed to room temperature, desorption begins (indicated by the flashing of the **Threshold** light).

   • When the flashing ceases to no more than once every 15 to 20 seconds (approximately three or four per minute), or the indication with **Det** depressed has returned to 0.02 or less, desorption is complete.

3. Record the desorption value along with other sample information. This value represents the sample surface area. The indicated number divided by the sample weight (in grams) is the sample specific surface area in square meters per gram.

Performing a Multipoint Surface Area Measurement

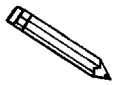
The startup and sample preparation for multipoint analysis procedures are identical to the single-point method except that a multigas manifold is needed to facilitate shifting among different gas mixtures.

Calibration

Calibration should be performed at the start of each 8-hour operational period. It is accomplished as described for single-point testing with a few exceptions.

- Repeat the steps for each of the gas mixture used.
- Determine a different number for each gas composition.

The gas flow rate may be temporarily increased, if desired, when shifting from one gas composition to another to save time. Care must be taken in doing so to prevent sample discharge from the increased flow. If you suspect this may be a problem with your sample, you can temporarily substitute an empty sample tube. After purging is complete, the flow must be restored to the operating level; i.e., the designated mark on the flowmeter and the **Det** zero confirmed.



The procedure described here presumes a Multigas Manifold is attached.

The FlowSorb must be properly flushed with an appropriate gas having a certified composition of, for example, approximately 5% N_2 and 95% helium.

1. Place the gas toggle switch on the side panel in the OPEN position, then open the appropriate valve on the manifold.

2. Fill a 1-mL precision syringe to 1 mL with nitrogen gas:

   a. Hold the needle tip immediately above the level of liquid nitrogen in the cold trap Dewar. The evaporating liquid provides an atmosphere of pure nitrogen.

   b. Flush the syringe a few times to ensure a proper fill; wipe the needle tip free of accumulated frost

   c. Lay the syringe aside (perhaps on the rubber mat of the FlowSorb) so that the gas can equilibrate to room temperature.

3. While the syringe is equilibrating, use the Flow knob to regulate the nitrogen-helium flow through the instrument so that the flowmeter float is centered on the marked line.

4. Use the **Coarse** and **Fine Zero** knobs to zero the instrument. Observe the display for a few minutes to establish system stability. A change of no more than 0.01 should be achieved.

   The two primary factors leading to excessive instability are:

   - instrument warm-up, which may require up to 30 minutes after initial power application, and
   - the progression of residue from a previous analysis, which can require as much as 10 minutes for elimination.

   Be sure these situations do not exist when setting the zero. Once set, the zero should not be readjusted unless a sustained change beyond ± 0.02 units occurs.

5. Depress the **Surface Area** and the **Clear SA Display** pushbuttons.

6. Hold the syringe at the rear flange so that body heat does not affect the gas volume contained by the syringe barrel.

7. Insert the needle fully into the **Inject** septum.

8. Inject the gas at a moderate rate. Withdraw the needle when the syringe is completely discharged.

   - After approximately one minute (five minutes for **Long** path), the **Threshold** light begins to flash and the indicator starts to accumulate surface area information.
   - When the **Threshold** light ceases to flash for 15 to 20 seconds (approximately after three minutes), the accumulation may be considered complete.
   - Completeness may be confirmed by pressing the **Det** pushbutton; it should be 0.02 or less.

9. Use the **Calibrate** knob to set the gas volume at standard conditions (refer to Appendix A). For example, if ambient temperature is 22 °C and atmospheric pressure is 740 mmHg, set the gas volume as **0.90**

   $$1.00 \times (273.2/295.2) \times (740/760) = 0.90$$

   as calculated by equation 8, Appendix A.

   Hereafter the instrument will indicate the adsorbed gas volume (STP) instead of the equivalent surface area of an adsorbent as in the single-point procedure.

10. Record the reading for subsequent use.

    Each valve on the Multigas Manifold has its own erasable tab for recording its calibration information. It is also recommended that you record the nitrogen composition of the gas bottle connected to each valve on the tab so that it is readily available when needed. Figure 3.1 shows typical data recorded as suggested here.

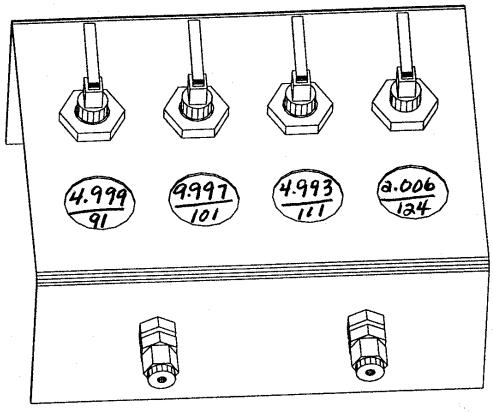


Figure 3.1. Manifold With Typical Calibration and Composition Values

11. Repeat the calibration procedure for each gas mixture and record calibration information accordingly. Wait a few minutes after shifting from one gas to another to allow the previous gas to be flushed from the instrument. The instrument display with **Det** depressed will cease registering a change as soon as the new gas has thoroughly flushed from the instrument.

    The numbers will fluctuate somewhat from instrument to instrument and will vary, of course, with gas composition. Typical numbers are shown in Table 3.1 and are for illustrative purposes only.

Table 3.1. Typical Calibration Numbers

Gas Composition (% N_2)	CALIBRATE Setting
4.999	91
9.997	101
14.993	111
22.006	124
Do not use these data; they are illustrative only.	

Analysis

The objective in multipoint surface area analysis is to obtain the volume of adsorbate (nitrogen, usually) at STP taken up by the sample at a series of relative pressures and to treat these data in accordance with the BET equation (refer to Appendix A, Theory) to yield the surface area. The calibration procedure for multipoint analysis was designed to set the instrument to register adsorbed gas volumes. Now the purpose is to relate adsorbed gas volumes to conditions for a specific sample.

A multipoint analysis can be conducted as a series of single-point steps using either the FlowSorb III 2305 or 2310, progressing from lower to higher nitrogen gas concentrations. After each concentration is established in the FlowSorb, the instrument is adjusted appropriately for the new gas mixture, and the adsorbed gas volume is measured. The sample surface area is computed in accordance with the BET equation after three or four measurements are completed.

If not previously obtained, determine the sample weight when all gases have been employed and the data collected. Divide the measured adsorbed volumes by the sample weight to obtain specific adsorbed volumes. As an example, the data might be as shown in Table 3.2.

Table 3.2. Example Adsorption Data

Gas Composition (% N_2)	Specific Adsorbed Volume (cm^3/g, STP)
4.999	15.16
9.997	17.39
14.993	19.16
22.006	21.46

Now compute the sample specific surface area following the method given in **Calculating the Result**.

Calculating the Result

Appendix A gives the basic theory and pertinent equation of the multipoint calculation. Table 3.3 formalizes the procedure beginning on the left and proceeding to the right using the experimental data in Table 3.2.

Table 3.3. Example Bet Calculation

Experimental Data		(*) P/Po	1-(P/Po)	V[1-(P/Po)]	(P/Po)/V[1-(P/Po)]
Gas Composition	Specific Gas Volume (V)				
4.999	15.16	0.0490	0.9510	14.42	0.00340
9.997	17.39	0.0980	0.9020	15.69	0.00625
14.993	19.16	0.1471	0.8529	16.34	0.00900
22.006	21.46	0.2158	0.7842	16.83	0.01282
(*) $(\%N_2/100) \times 760/775 = (\%N_2/100) \times 0.981 = \%N_2 \times 0.00981$					

A plot of the sixth versus the third column is given by Figure 3.2. The slope of the plotted line is 0.0560 and the intercept is 0.0008. From equation 10 of Appendix A, the sample specific surface area (S) is thus calculated as

$$S = \frac{4.353}{0.0560 + 0.0008} = 76.6 \text{ m}^2/\text{g}$$

For purposes of comparison, a single-point specific surface area of 73.8 m^2/g was obtained for this same material using a gas composition of 30.017% N_2 and the remainder helium.

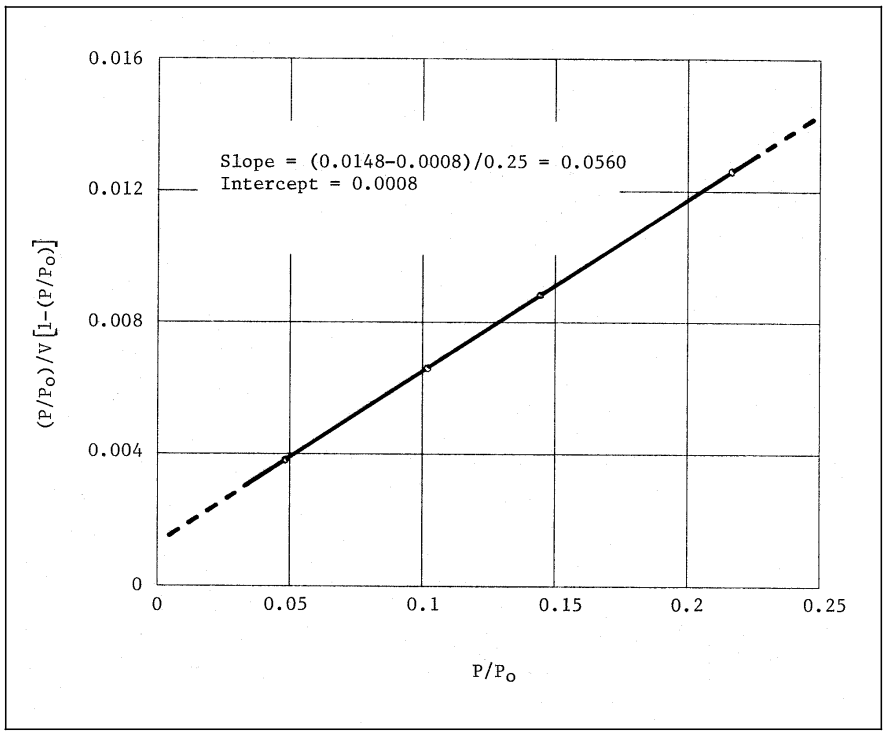


Figure 3.2. Example Plot

Preparing the FlowSorb for Idle Periods

If the FlowSorb is to be inoperative or unattended for a period long enough for the liquid nitrogen in the **Cold Trap** Dewar to evaporate (or the dry ice to evaporate from the dry ice acetone slurry), remove the Dewar and U-tube so that impurities are not released into the system. Install a clean, dry U-tube after removing the current one. If additional U-tubes are unavailable, clean the current tube and allow to dry; then reinstall at the **Cold Trap** position.

If the FlowSorb is going to be idle only for a couple of days, reduce the gas flow to approximately one-quarter of its normal value and leave power ON. The gas loss and power drain are very low and the instrument will be immediately ready for use.

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

CHAPTER 4

TROUBLESHOOTING AND MAINTENANCE

- Troubleshooting
- Replacing the Septum

TROUBLESHOOTING AND MAINTENANCE

This chapter contains:

- common problems that may occur when operating the FlowSorb III, and how you can resolve them
- instructions on replacing the septum

Contact your local Micromeritics service representative if additional assistance is required.

Troubleshooting

Detector Drift

Some detector drift when first turning on the power is normal and to be expected. This type of drift is due to the detector system coming to its regulated temperature and should disappear in approximately 30 minutes. The instrument is generally usable after 10 minutes if it has been properly purged of air.

There are several likely causes for the detector indication (when on X1) not returning to zero within ± 0.02 following a sample or calibration test with the DET. pushbutton depressed.

1	Using contaminated gas mixtures along with failure to keep the cold trap properly submerged in liquid nitrogen. A decided shift of the detector baseline occurs between the adsorption and desorption modes when the gas contains impurities and the cold trap is ineffective. Surprisingly small amounts of impurities can cause detector drift. A contaminated pressure regulator, unclean metal connecting tubing, and unclean non-metal tubing and connectors can contribute impurities to an otherwise clean gas stream. Be sure one or more of these factors is not responsible before proceeding further.
2	Using atmospheric air to perform calibrations can cause detector drift because of its water vapor content which travels slowly through the system. Using atmospheric air is discouraged and the procedure described in **Single-point Calibration** in **Chapter 3**, is recommended.
3	Using too much liquid nitrogen in the **Cold Trap** Dewar. Too much liquid nitrogen cools the metal fittings supporting the glass trap causing moisture from the atmosphere to freeze. This leads to erratic gas cooling. The liquid nitrogen level should be approximately one cm (0.5 in.) from the top of the Dewar.

4	Incompletely degassed samples cause detector drift and offset because contaminants can still be flushed from the sample at room temperature. Such drift and offset will cease once the sample is immersed in liquid nitrogen.
5	Leaks caused by a worn septum; always use a good septum. Replace the septum periodically; after approximately 100 injections when using the 1-mL syringe and approximately 20 injections when using the 10-mL syringe. Refer to **Replacing the Septum** in this chapter.
6	Leaks caused by loose plumbing. If it becomes necessary to tighten plumbing connections, be sure not to overtighten them. Overtightening can easily damage small copper tubing.
7	Leaks caused by missing or damaged sample tube O-rings. Discard cracked or chipped sample tubes to avoid leaks and avoid damaging the O-rings. Always use clean, undamaged O-rings which have a light film of vacuum grease applied.

Low Results On X10 Range

The X10 range (10 times increase in detector sensitivity) is provided when analyzing samples that produce less than a 10% enrichment for nitrogen in the gas stream. The detector signal is truncated when this 10% limit is exceeded which results in a lowered surface area value. A high-pitched audio tone is sounded if the possibility of misleading results occurs.

Erratically High Results

Failure to reset the surface area display to zero after passage of the air peak is often responsible for an apparently high result.

Air peaks or peaks due to other gases passing through the **Long** path may be forgotten due to their extended transit time and add to a subsequent surface area. You should be particularly aware of this possibility when a **Short** path adsorption followed by a **Long** path desorption is employed to save time.

Adding liquid nitrogen to the **Cold Trap** while a surface area result is accumulating can cause gas flow to decrease or even to stop momentarily. This will extend the time of gas passage and violate the assumption of constant flow rate upon which the surface area accumulation technique is based. The result is a somewhat greater surface area value than would otherwise have been achieved. The liquid nitrogen supply should only be replenished between tests or while awaiting air peak passage.

Non-Reproducible Calibrations

A damaged and leaking septum should be eliminated as the first possible cause of non-reproducible calibrations. Replace the septum with a new one if there is any doubt as to its integrity. (Refer to **Replacing the Septum** in this chapter.)

The knurled nut which retains the septum should be finger-tightened. Insufficient or excessive tightening may cause system leakage.

Check the syringe for gas tightness and freedom from obstructions in the needle. Immerse the needle tip under water and press the plunger; this should result in a steady stream of bubbles with no resistance to motion from the plunger. When performing this check, be careful not to draw water into the syringe. Allow the needle to dry thoroughly before using for calibrations.

Monitor the flow rate by observing the flowmeter float to see that it is constant. If it does not maintain a steady position, check the following:

- the gas regulator setting for its recommended 15 psig setting
- the flow control knob for smooth operation
- gas leaks about the septum, the sample tubes and holders, the gas inlet connection, and internal plumbing connections.

Also refer to **Detector Drift** in this chapter.

Surface Area Display Not Clearing to Zero

Temperature changes and aging of components may cause the surface area display to register a number slightly different from zero when it is cleared. To correct this condition:

1. Remove the instrument rear panel, being careful not to contact any of the electrical leads in the lower portion of the instrument.

Proceed with caution; electrical power must be on for this procedure. Touching a lead wire could cause electrical shock.

2. Locate the circuit board mounted beneath the top panel of the instrument and immediately above the display.

3. On the circuit board, locate R2 (the closest potentiometer to the green test point) located at the end of the circuit board and just above the edge of the display meter.

4. Hold down the display **Reset** button and using a small, flat-head screwdriver, adjust the potentiometer until the display registers zero.

Degas Temperature Fails to Function Properly

A flashing temperature display indicates that either the thermocouple is disconnected or that a temperature of 400 ^{o}C has been reached. Ensure that the thermocouple is plugged into its receptacle. If the thermocouple is plugged in, unplug the thermocouple and examine its leads for broken connections. If the thermocouple is intact, check to see if a degas temperature of 350 ^{o}C or higher has been specified; if so, specify a lower temperature. Circuitry inside the instrument protects the operator, the sample tubes, and the heating mantles by limiting the temperature to 400 ^{o}C regardless of the setting chosen by the operator.

The thermal characteristics of the heating mantles prevent temperature regulation to a precision better than approximately ± 10 ^{o}C. Normal operation is cylical with the temperature oscillating about the chosen temperature by several degrees, the initial overshoot will be the largest. The actual temperature at the sample will be somewhat lower than initially indicated and will vary less due to the moderating effects of the glass wall of the sample tube and the flowing stream of gas.

The temperature control circuitry has been designed to require about 20 minutes to reach the desired setting regardless of how high the setting may be. This controlled ramping of temperature is intended to minimize destructive changes in the sample, especially those due to the boiling off of moisture. Faster heating may be achieved by setting to a much higher temperature than desired, and then reducing the setting to the desired temperature when that temperature is reached.

Display and Indicators Fail to Illuminate

Verify that the electrical outlet is energized and power is available by plugging in another appliance. Unplug the FlowSorb and examine the fuse and the voltage selector circuit board; replace the fuse if necessary. If the problem persists, again unplug the instrument and remove the back panel. Examine all connectors to be sure that they are firmly engaged. Check for broken wires, especially in the lower areas of the instrument near the power entrance, power switch, and power transformer.

Hard-to-Release Sample Holder

O-ring, sliding seals as utilized on the connecting ends of the sample holder tend to make insertion and removal somewhat difficult if they become dry. A very small amount of stopcock grease applied to the two external O-rings will restore free movement. Only a minute quantity of grease is required; avoid application of excessive amounts.

Substantial Difference Between Adsorption and Desorption Result

Generally there will be little difference between indicated adsorption and desorption data (surface area in the case of single-point analysis and gas volume in multipoint testing). Substantial and persistent differences can be indicative of a leak somewhere in the system.

Lodged Flowmeter Float

Fine particles that escape filter entrapment, as well as accumulated vapor deposition can cause the ball float to become lodged in the glass tube of the flowmeter. Contact your local Micromeritics service representative if this occurs.

Replacing the Septum

The septum is installed at the **Inject** port and is held into place by a knurled nut.

A septum usually requires replacing after approximately 100 injections when using the 1-mL syringe and approximately 20 injections when using the 10-mL syringe. Refer to Chapter 5 for ordering information.

Replace the septum as follows:

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

CHAPTER 5

ORDERING INFORMATION

ORDERING INFORMATION

Components and accessories can be ordered by:

- contacting our Customer Service Department at 770/662-3636
- accessing our web site at http://www.micromeritics.com

When ordering, please use the information provided below.

Part Number	Description
230-62804-00	Multigas Manifold, allows attachment of multiple gases for multipoint analysis.
021-00000-00	Model 021 LN_2 Transfer System, allows easy filling of sample Dewars
023-00000-00	Desorb 2300A, provides three stations for degassing samples, enabling increased sample throughput
231-26001-00	Heating mantle, quartz fiber, for standard sample tube (450 °C)
230-25808-00	Clip, for heating mantle
004-61004-00	Dewar, 350 mL
230-31801-00	Dewar cover, for standard Dewar
230-31801-01	Dewar cover, split, for cold trap Dewar
004-32602-00	Septum, for injection port
004-61604-00	Calibration syringe, 10 mL
004-61604-01	Needle, for 10-mL syringe
004-61602-00	Calibration syringe, 1 mL
004-61602-01	Needle, for 1-mL syringe
004-25103-00	Ferrule, front
004-25104-00	Ferrule, rear
003-51131-00	Fuse, 2.0 Amp, Slow Blow
003-51198-00	Fuse, 3.15 Amp, Slow Blow
004-62230-58	Gas regulator, CGA 580 fitting, 30 psig
230-61002-00	Sample tube, cylindrical bottom, 1-cm^3 capacity
230-61003-00	Sample tube, capillary stem, 1-cm^3 capacity
230-61005-00	Sample tube, monolith
230-61001-00	U-tube, for cold trap, 0.1-cm^3 capacity
004-32004-00	Stopper, for sample tube
240-25853-00	Funnel, for pouring sample into sample tube
230-32802-00	Weighing support, for standard sample tube
230-32803-00	Weighing support, for monolithic sample tube

APPENDIX A

THEORY

THEORY

One form of the well-known BET equation* that describes the adsorption of a gas upon a solid surface is

$$(P/P_0)/V[1-(P/P_0)] = 1/(V_mC)+[(C-1)/(V_mC)]P/P_0 \quad (1)$$

where

- V = the volume (at standard temperature and pressure, STP) of gas adsorbed at pressure P
- P_0 = the saturation pressure which is the vapor pressure of liquified gas at the adsorbing temperature
- V_m = the volume of gas (STP) required to form an adsorbed monomolecular layer
- C = a constant related to the energy of adsorption

The surface area S of the sample giving the monolayer adsorbed gas volume $Vm_{(STP)}$ is then calculated from

$$S = Vm_AN/M \quad (2)$$

where

- A = Avogadro's number which expresses the number of gas molecules in a mole of gas at standard conditions,
- M = the molar volume of the gas, and
- N = the area of each adsorbed gas molecule as given in Appendix B.

Single-Point Surface Area

The constant C of equation 1 is typically a relatively large number, i.e., C>1, from which equation 1 reduces very nearly to

$$(P/P_0)/V[1-(P/P_0)] = (1/V_m)[(1/C)+(P/P_0)] \quad (3)$$

Now if $P/P_0>>1/C$, equation 3 can be further represented by

$$(P/P_0)/V[1-(P/P_0)] = (1/V_m)(P/P_0) \quad (4)$$

* Brunauer, S., Emmett, P.H., and Teller, E., *J. Am. Chem. Soc. 60*, 309A (1938).

which rearranges to

$$V_m = V[1-P/P_0)] \quad (5)$$

Another way of arriving at the same result is by recognizing that the term $1/(V_mC)$ of equation 1 is generally small. Taking it as insignificant changes the slope, and hence the value of V_m and the sample surface area as calculated by equation 2, only a small amount. Equation 1 can be rearranged with the contribution of the intercept term taken to be vanishingly small to give also

$$V_m = V[1-(P/P_0)] \quad (5)$$

Substituting equation 5 into equation 2 yields

$$S = VAN\ [1-P/P_0]/M \quad (6)$$

from which the sample surface area is readily determined once the volume V of gas adsorbed (or desorbed, which must theoretically be identical) is measured and appropriate values for the other terms are incorporated.

For nitrogen gas adsorbed from a mixture of 30 mole % nitrogen and 70 mole % helium using a liquid nitrogen bath, the values are arrived at as follows:

The volume V of gas with which the FlowSorb III is calibrated is injected at room temperature and the prevailing atmospheric pressure. This volume must thus be multiplied by the ratios 273.2/(Rm. Temp., K) x (Atm. Press., mmHg)/760 to convert it to standard conditions (0 ^oC and 760 mmHg).

Avogadro's number A is 6.023×10^{23} molecules/g mole. The molar volume M of a gas at standard conditions is 22414 cm^3/g mole.

The presently accepted value for the area N of a solid surface occupied by an adsorbed nitrogen molecule* is 16.2 x 10-20 m^2 (=16.2 $Angstroms^2$).

P is 0.3 x the atmospheric pressure in millimeters of mercury since the gas mixture is 30% nitrogen and adsorption takes place at atmospheric pressure. P_0, the saturation pressure of liquid nitrogen is typically a small amount greater than atmospheric due to thermally induced circulation, dissolved oxygen, and other factors. With fresh, relatively pure liquid nitrogen, the saturation pressure is typically about 15 mmHg greater than atmospheric pressure. It can be 40 to 50 mmHg greater if the liquid nitrogen is relatively impure. The saturation pressure should be determined by other means in the latter event.

---

* Roberts, B.F., *J. Coll. Interface Sci. 23,* 266 (1967).

The result for a 30% N_2/70% He mixture adsorbed at liquid nitrogen temperature when room temperature is 22 ºC and atmospheric pressure is 760 mmHg is the expression

$$S = V\left[\frac{273.2}{\text{Rm.Temp.}}\right]\left[\frac{\text{Atm.Press.}}{760}\right]\left[\frac{6.023\text{x}10^{23}\text{x}16.2\text{x}10^{-20}}{22.414\text{x}10^{3}}\right]\left[1-\frac{(\%N_2/100)\text{x}\text{Atm.Press.}}{\text{Sat.Press.}}\right]$$

$$= V \text{ x } \frac{273.2}{295.2} \text{ x } \frac{760}{760} \text{ x } \frac{6.023\text{x}10^{23}\text{x}16.2\text{x}10^{-20}}{22.414\text{x}10^{3}} \text{ x } 1-\frac{0.3\text{x}760}{775} = 2.84\,V \qquad (7)$$

where S is the surface area in square meters.

For calibration purposes, this means that a syringe injection of V = 1.00 cm^3 of nitrogen at 22 °C and 760 mmHg should produce an indicated surface area of 2.84 m^2.

The value of S from equation 7 changes when ambient conditions differ significantly from 22 °C and 760 mmHg, pressure changes having relatively more effect than temperature. Another value should then be calculated. For example, suppose the gas were 29.33% N_2, the laboratory were at 25 °C, atmospheric pressure were 710 mmHg, and the saturation pressure were measured to be 735 mmHg, the value, instead of being 2.84, should be 2.67.

Multipoint Surface Area

A straight line usually results between P/P_0 values from about 0.05 to 0.25 when experimental data are plotted as $(P/P_0)/V[1-(P/P_0)]$ on the ordinate against P/P_0 as the abscissa. Relative pressures within this prescribed range are typically obtained with gas compositions between about 5% and 25% N_2 with the remainder He. Equation 1 shows then that the slope and intercept of this line are, respectively, $(C-1)/V_mC$ and $1/(V_mC)$ and that both the values of V_m and C can be determined.

The FlowSorb is calibrated by injecting into it an accurately measured volume of each gas mixture at ambient conditions, calculating the volume of this gas at standard conditions, and setting the instrument to indicate thereafter adsorbed and desorbed gas volumes at standard conditions. When 1 mL of gas mixture is injected, its volume V at STP is given by

$$V = 1.00 \times \frac{273.2}{\text{Rm.Temp.}} \times \frac{\text{Atm.Press.}}{760} \qquad (8)$$

The sample specific surface area S in square meters per gram is calculated from equation 2 using appropriate constants and slope and intercept values once the plot is made. Using the constants given above, this relationship becomes

$$S = 6.023 \times 10^{23} \times 16.2 \times 10^{-20}/22414 \text{ (slope + intercept)} \qquad (9)$$

or simply

$$S = 4.353/(\text{slope + intercept}) \qquad (10)$$

APPENDIX B

OTHER GASES AND COMPOSITIONS

OTHER GASES AND COMPOSITIONS

Although nitrogen in helium (30/70) with liquid nitrogen as the cold bath provides the most frequently used conditions, there are occasions where it may be desirable to employ a different set of circumstances. For example, some investigators favor a 20/80 nitrogen-in-helium mixture when analyzing carbon blacks; carbon dioxide in helium is sometimes preferred when surveying coals; and n-butane permits analysis at ice water temperature, a convenience at some sites.

Nitrogen mixtures with helium which differ by more than ± 1% from 30 mole % nitrogen require that the instrument be calibrated to ensure linearity. This is accomplished by lowering the Dewar tray, depressing the DET. and X1 pushbuttons, and turning both the FINE and COARSE ZERO controls fully clockwise until the stops are encountered. Establish a stable flow of the differing gas mixture through the instrument and adjust the small screwdriver-adjusted potentiometer on the right panel near the gas inlet until the display indicates the percentage of nitrogen known to be contained in the mixture. Use equation 6 of Appendix A to compute a new calibration factor for the mixture and then proceed otherwise as with the 30% mixture.

The user should be aware that one-to-one surface area value correspondance with nitrogen values or among the other gases is unlikely when using these other gases. The area occupied by their molecules on solids is less investigated and apparently varies more widely depending on the nature of the solid.

The proper concentration of a gas other than nitrogen for single-point surface area measurement is selected on the basis of what concentration produces a relative pressure P/P_0 of approximately 0.3. For example, if argon as listed in Table B.1 is to be used in combination with helium, the result is

$$\text{Ar concentration} = \frac{250 \text{ mmHg} \times 30.3}{760 \text{ mmHg}} \times 100 = 9.9\%$$

since the vapor pressure of argon at liquid nitrogen temperature is 250 mmHg and the saturation pressure of liquid nitrogen is taken to be 760 mmHg for purposes of this illustration.

Table B.1 also lists basic physical property information as required in arriving at the appropriate constant in equation 6 for a number of gases at selected temperatures. By way of illustration, suppose 30 mole % n-butane mixed with 70 mole % helium were to be used at ice water temperature to measure surface area. The numerical constant in equation 7 would be

$$(273.2/295.2) \times 6.023 \times 10^{23} \times (46.9 \times 10^{-20}/22414)$$

$$\times [1-(0.3 \times 760)/1060] = 9.15$$

The FlowSorb III may require other adjustments to render it applicable under the new conditions. Contact your service representative or the factory for further information.

Table B.1. Physical Property Data

Gaseous Adsorbate	Bath Temperature (°C)	Vapor Pressure at Bath Temperature (mmHg)	Area Occupied by Adsorbed Molecule at Bath Temperature ($m^2 \times 10^{20}$)
Nitrogen	-195 (LN_2)	760	16.2 (10)
Argon	-195 (LN_2)	250	16.7 (11)
Carbon dioxide	-78 (Dry ice/acetone)	793	19.1-20.6 (11)
n-Butane	-78 (Dry ice/acetone)	30	37.5 (11)
n-Butane	0 (Ice Water)	1060	46.9 (11)

APPENDIX C

SPECIALTY GAS SOURCES

SPECIALTY GAS SOURCE

Air Products and Chemicals, INC.

404/451-2711 P. O. Box 80563 ATLANTA,GA.30341

DOMESTIC SPECIALTY GAS FACILITIES

TAMAQUA, PA (HOMETOWN)
Air Products and Chemicals, Inc.
Specialty Gas Department
P. 0. Box 351
R.D. 1
Tamaqua, PA 18252-0351
Phone: (717) 467-2981

CHICAGO, IL
Air Products and Chemicals, Inc.
Specialty Gas Department
12722 South Wentworth Avenue
Chicago, IL 60628-7299
Phone: (312) 785-3000

HYATTSVILLE, MD (BLADENSBURG)
Air Products and Chemicals, Inc.
Specialty Gas Department
2900 52nd Avenue
Hyattsville, MD 20781-1199
Phone: (301) 864-2345

JACKSONVILLE, FL
Air Products and Chemicals, Inc.
Specialty Gas Department
5837 W. Fifth Street
Jacksonville, FL 32205-1509
Phone: (904) 781-8450

LA PORTE, TX
Air Products and Chemicals, Inc.
Specialty Gas Department
Route 1, 10202 Strang Rd.
LaPorte, TX 77571-9271
Phone: (713) 479-5256

LONG BEACH, CA
Air Products and Chemicals, Inc.
Specialty Gas Department
23320 S. Alameda St.
Long Beach, CA 90810-1991
Phone: (213) 518-0300

MEMPHIS, TN
Air Products and Chemicals, Inc.
Sepcialty Gas Department
P. 0. Box 13268
2541 Harbor Avenue
MemphisJ TN 38113-0268
Phone: 901-947-1141)

MOUNTAIN VIEW, CA
Air Products and Chemicals, Inc.
Specialty Gas Department
465 N Hhisman Road
Mountain View, CA 94043-2112
Phone: (415) 961-4560

INTERNATIONAL

BELGIUM
Air Products S.A.
Le Souverain
Boulevard du Souverain 191-197 Box 8
1160 Bruxelles, Belgium
Phone: 660-2980
Telex: 62489

BRAZIL
Air Products Gases Industrials Ltda.
Pra & A Radialista Manoel Da Nobrega, 65
02517-Casa Verde
Sao Paulo
SP Brasil
Phone: (011) 265-0122
Telex: (011) 24007

CANADA
Air Products
2090 Steeles Avenue East
Brampton, Ontario L6T 1A7, Canada
Phone: (416) 791-2530

FRANCE
Prodair
Tour Pleyel
Centre Paris Pleyel
93521 St. Denis, Cedex 01, France
Phone: Paris (1) 809 6560/6561
Telex: 611382

GREAT BRITAIN
Air Products Limited
Hersham Place, Molesey Road
Walton-on-Thames
Surrey, KT 124RZ
Phone: 9322 49200
Telex: 917243

Air Products and Chemicals, INC.

404/451-2711 P. O. Box 80563 ATLANTA, GA. 30341

KOREA
Korea Industrial Gases Ltd.
Chung Mu Ro 2Ka 64-5, Chung Ku
Han II Bldg., Room 907
Seoul, Korea
Phone: 776-2521
Telex: K22763

MEXICO
Cryoinfra, S.A. de C.V.
Felix Guzman No. 16
53390 Nautalpan
Estado de Mexico
Phone: (905) 557-5108
Telex: 017-775921 INFRME

PUERTO RICO
Air Products of Puerto Rico
P. 0. Box W
Carretera 835, Km. 0.3
Barrio Marney
Guaynabo, Puerto Rico 00657
Phone: (809) 790-2475
Telex: 385-9434

THE NETHERLANDS
Air Products Nederland B.V.
Postbus 56
2740 AB Waddinxveen, Holland
Phone: 01828-3955
Telex: 20890

NORWAY
Gardner Cryogenics A.S.
Stavangerveien
Lura
4300 Sandnes
Norway
Phone: 04 67 81 60
Telex: 856-33363

SOUTH AFRICA
Air Products South Africa (Pty) Limited
Private Bag 2, Kempton Park 1620
Transvaal, South Africa
Phone: 36-6686
Telex: 8-2688 and 8-6379

I EST GERMANY
Air Products G.m.b.H.
Klosterstrasse 24-28
4000 Dusseldorf, West Germany
Phone: (0211) 16080
Telex: 8587306

Air Products Sales Offices

ALABAMA
Birmingham ..... (205) 87Q-5603
Dothan ..... (205) 794-5422
Mobile ..... (205) 433-6512
Muscle Shoals ..... (205) 381-0542

ARIZONA
Phoenix ..... (602) 243-2751

ARKANSAS
Ft. Smith ..... (501) 782-7219
Little Rock ..... (501) 375-1256

CALIFORNIA
Concord ..... (415) 827-1290
Long Beach (Los Angeles) ..... (213) 432-8951
Mountain View (Bay Area) ..... (415) 961-4560

COLORADO
Denver ..... (303) 321-6515

CONNECTICUT
North Haven ..... (203) 239-4201

FLORIDA
Jacksonville ..... (904) 786-2662
Miami ..... (305) 592-0709
Panama City ..... (904) 763-1672
Pensacola ..... (904) 432-1406
Tallahassee ..... (904) 222-3015
Tampa ..... (813) 621-2477

GEORGIA
Albany ..... (912) 432-0538
Atlanta ..... (404) 451-2712
Chamblee (Atlanta) ..... (404) 451-2712
Columbus ..... (404) 323-1349

ILLINOIS
Hillside (Chicago Area) ..... (312) 449-5850

INDIANA
Shelbyville (Indianapolis Area) ... (317) 398-0141
South Bend ..... (219) 288-4941

IOWA
Cedar Rapids ..... (319) 366-7371
Davenport ..... (319) 386-6142
Des Moines ..... (515) 243-8131
Fort Dodge ..... (515) 576-7159
Sioux City ..... (712) 252-4466

KENTUCKY
Louisville ..... (502) 456-6222
Paducah ..... (502) 443-4589

LOUISIANA
New Orleans ..... (504) 241-8310
Shreveport ..... (318) 869-1319

MARYLAND
Hyattsville (Washington Area) ... (301) 864-2345
Salisbury ..... (301) 742-3800

MASSACHUSETTS
Marlborough (Boston Area) ..... (617) 481-6970

MICHIGAN
Detroit ..... (313) 893-7420

MINNESOTA
Shakopee (Minneapolis-St. Paul Area)
..... (612) 445-:4610

MISSISSIPPI
Clarksdale ..... (601) 624-2547
Greenwood ..... (601) 453-5421
Jackson ..... (601) 982-5593
Tupelo ..... (601) 842-5138

MISSOURI
Kansas City ..... (913) 492-1550
Earth City ..... (314) 739-6211

NEBRASKA
Grand Island ..... (308) 382-1695
Lincolr. ..... (402) 464-3191
Omaha ..... (402) 341-5500

NEW JERSEY
Camden (Philadelphia Area) ..... (609) 365-4500
Iselin (Greater New York City) ... (201) 548-4600

NEW YORK
Latham ..... (518) 783-1745
New York City (See Iselin, New Jersey)
Rochester ..... (716) 436-7780

NORTH CAROLINA
Asheville ..... (704) 254-0958
Charlotte ..... (704) 588-2350
Greensboro ..... (919) 299-1361
Raleigh ..... (919) 828-4462

OHIO
Cleveland ..... (216) 333-2300
Columbus ..... (614) 459-0206
Middletown ..... (513) 424-1281

OKLAHOMA
Tulsa ..... (918) 492-1812

PENNSYLVANIA
Allentown ..... (215) 481-7435
Philadelphia (See Camden, New Jersey)
Creighton (Pittsburgh Area) ..... (412) 226-1511
York ..... (717) 846-1604

SOUTH CAROLINA
Charleston Heights ..... (803) 744-2228
Taylors (Greenville Area) ..... (803) 268-4085

SOUTH DAKOTA
Rapid City ..... (605) 342-4490

TENNESSEE
Chattanooga ..... (615) 265-1019
Kingsport ..... (615) 247-7193
Knoxville ..... (615) 522-5153
Memphis ..... (901) 947-1141
Nashville ..... (615) 226-4144

TEXAS
Arlington (Dallas-Fort Worth Area) (817) 265-5185
Houston ..... (713) 893-5160

VIRGINIA
Hampton ..... (804) 826-5430
Hopewell ..... (804) 796-3955
Roanoke ..... (703) 343-3683

WASHINGTON
Seattle ..... (206) 228-5515
Vancouver ..... (206) 695-4429

WEST VIRGINIA
Parkersburg ..... (304) 485-1691

WISCONSIN
Milwaukee ..... (414) 476-4370

CANADA
Brampton, Ontario ..... (416) 791-2530

PUERTO RICO
Guaynabo ..... (809) 790-2475

APPENDIX D

SAMPLE TUBES

SAMPLE TUBES

Tube 1 (see Figure D-1) primarily is used as the cold trap tube. It also can be used for sample quantities up to a bulk volume of 0.1 cm^3. Because its use increases the gas velocity across the sample, it minimizes the creation of thermal gradients. Thermal gradients cause the separation of mixed gases and are evidenced by the presence of shoulders on recorded adsorption and desorption peaks. Such shoulders give distorted results. Low specific surface materials, perhaps as low as 0.1 m^2/g, which might be thought best measured using a large quantity of sample in the more common tube, are sometimes more accurately analyzed using a much smaller sample in this tube.

Tube 2, having an internal sample space of 1.5 cm^3 and usable with a bulk sample volume of up to approximately 1 cm^3, is the primary general purpose sample holder.

Tube 3 has the same internal dimensions as Tube 2. Its outlet stem has a smaller internal diameter to increase gas velocity and turbulence and, hence, to diminish thermal gradient effects.

Tube 4 has an internal sample space of 7.0 cm^3, and is usable for sample bulk volumes up to 4.7 cm^3.

Tube 5 is designed for monolithic samples. It will accommodate cylinders up to 2 cm in diameter and 3 cm long.

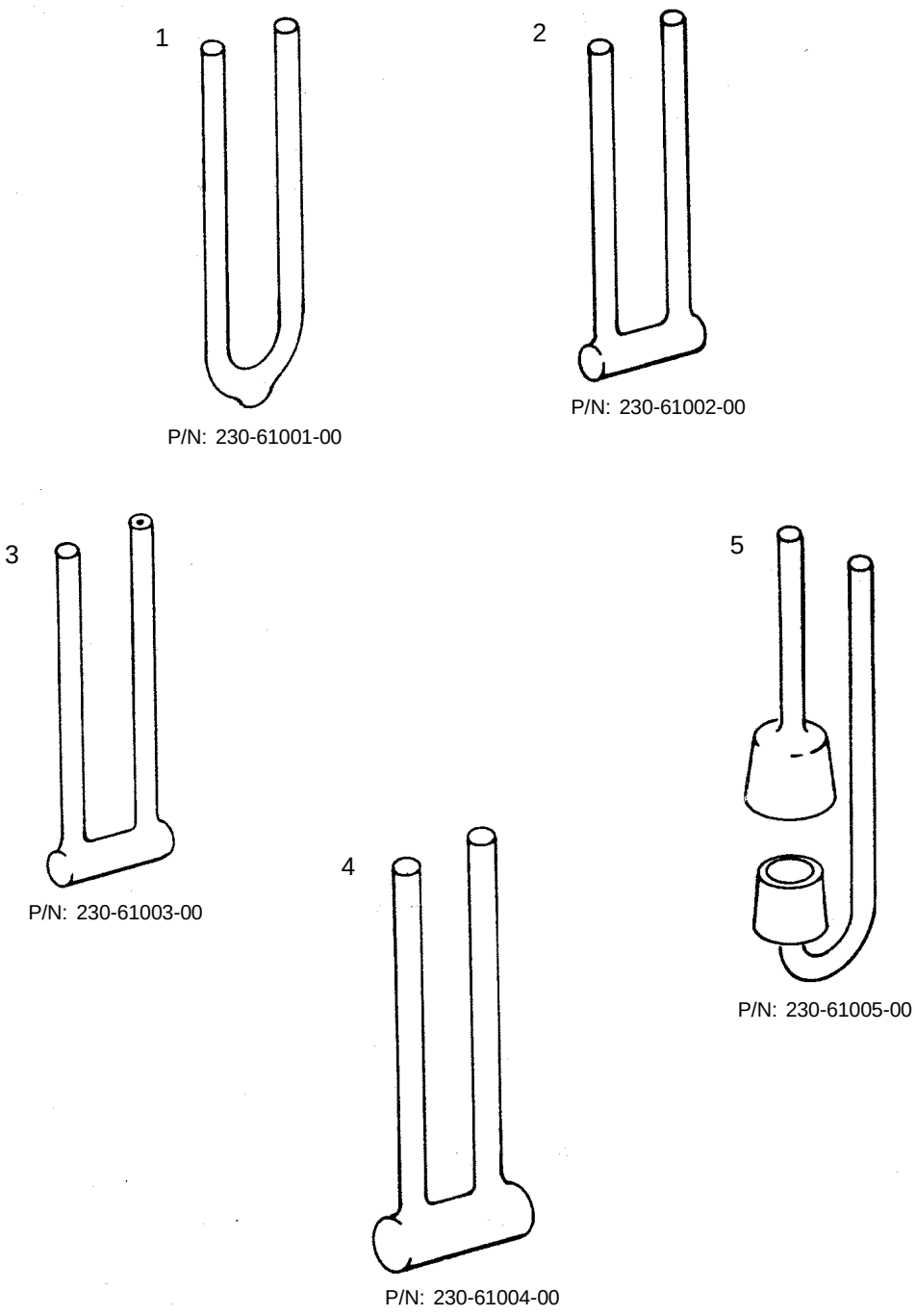


Figure D-1. Sample Tubes

INDEX

INDEX

I

L

M

N

O

P

R

S

T

U

V

W

X

Z