micromeritics®

Vapor Adsorption Option

User's Guide

202-42854-01
Sept 2014

Alconox is a registered trademark of the Alconox Company.

WARRANTY

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

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

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

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

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

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

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

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

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

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

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

4356 Communications Drive • Norcross, GA 30096-2901 • Fax (770) 662-3636

Domestic Sales - (770) 662-3636
International Sales - (770) 662-3660

Domestic Repair Service - (770) 662-3666
Customer Service - (770) 662-3636

Form No. 008-42104-00
Rev. 12/95

TABLE OF CONTENTS

VAPOR ADSORPTION OPTION

If using the ASAP 2020 Plus, refer to the ASAP 2020 Plus Operator Manual (part number 202-42800-01) for information on using this option.

The Vapor Adsorption Option permits the adsorption of vapors from liquids that normally are liquid at or above room temperature. Allows analyses of samples at temperatures up to 40 ºC.

Description

The Vapor Adsorption option consists of a vapor enclosure, heated manifold cover, temperature controller, and vapor source tube.

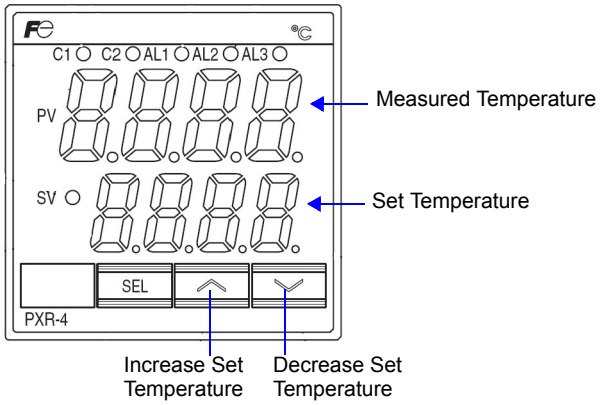
Temperature Controller

Vapor Enclosure

The vapor enclosure houses the vapor tube. Its temperature is regulated by the temperature controller. The door to the vapor enclosure should remain closed while the instrument is in operation.

Temperature Controller

The front panel of the temperature controller has two displays, one for the vapor source and one for the manifold; they are labelled accordingly.



Upon installation, the temperature of the vapor source is set at 40 ºC (for analysis temperatures up to 35 ºC). The analysis manifold is set between 55 and 60 ºC to achieve a 45 - 47 ºC manifold temperature (displayed on the instrument schematic).

Analysis Manifold

Do not adjust the temperature of the manifold. The manifold temperature has been set and calibrated to 45 ºC and is suitable for any type of analysis. If the manifold temperature is changed, recalibration by a Micromeritics service representative is required.

Do not adjust the temperature of the manifold.

The manifold temperature displayed on the schematic will differ from the temperature displayed on the temperature controller. A difference of 10 ºC to 15 ºC is typical. The schematic reading reflects the true manifold temperature and is the one used in calculations.

Vapor Source

For analysis temperatures up to 35 ºC, the recommended temperature for the vapor source is 40 ºC. For analysis temperatures over 35 ºC, use the Up and Down keys to adjust the temperature of the vapor source to a value that is midway between the manifold temperature and the analysis temperature. For example, if you intend to perform analyses at 40 ºC and the manifold temperature displayed on the instrument schematic is 45 ºC, adjust the vapor enclosure temperature to 42.5 ºC.

Vapor Tube

The vapor tube contains the vapor source and is installed inside the vapor enclosure.

Vapor Source

Always use high-purity, certified liquids as vapor sources.

It is very important to use high-purity, properly degassed liquids as vapor sources. When using water, a common vapor source, be sure it is distilled or deionized. Use extra care to ensure proper degassing.

The level of the liquid in the vapor tube should be at 25 to 50% of the tube, approximately 3 to 5 mL. If the level falls below 25%, add more vapor liquid and degas thoroughly.

Cleaning the Vapor Source Tube

A clean vapor source tube is as important as high-purity vapor sources. You should clean the tube when:

- changing vapor sources
- the vapor source becomes discolored

Clean the vapor tube as follows:

1. Turn on a drying oven and set the oven temperature to 110 ºC.
2. Check the bowl of the ultrasonic cleaning unit to make sure it is clean.
3. Using five grams of Alconox® (or other suitable laboratory detergent) per 500 mL of warm water, fill the reservoir of the ultrasonic unit with enough water to cover the vapor tube. Make sure the detergent is dissolved before placing the vapor tube into the water. If too much detergent is used, it may be difficult to rinse from the tube.
4. Fill the vapor tube with warm water and place it in the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.
5. Remove the vapor tube from the reservoir.
6. Clean the interior of the tube with a sample tube brush.
7. Rinse the vapor tube with hot water, then with isopropyl alcohol.

If isopropyl alcohol is not available, deionized water may be used to rinse the tube.

8. Stand the vapor tube on a sample tube rack and place the rack in the preheated drying oven for two hours.
9. Remove the tube from the oven and allow to cool.

Degassing the Vapor Tube

Vapor source liquids must be degassed thoroughly to remove air (and other dissolved gases) to achieve desirable results.

It is very important to degas vapor liquids on a weekly basis to maintain purity.

Degas vapor liquids:

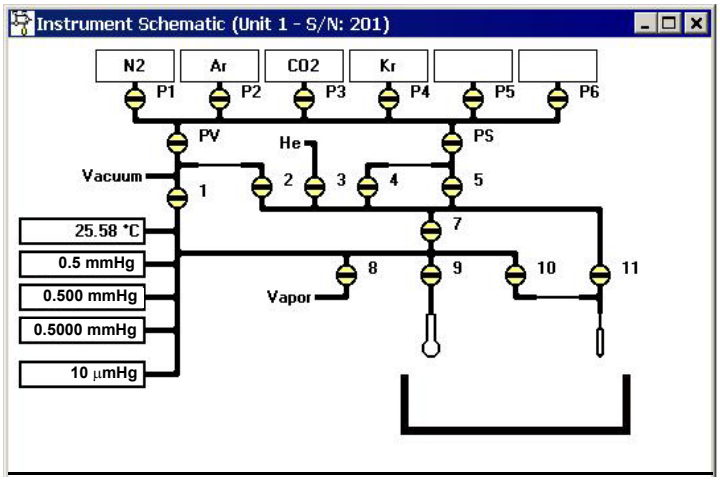
- on a regular, weekly basis
- when adding fresh vapor liquid
- when changing vapor liquids

The vapor tube may be degassed by using the vacuum method or the freezing method. The vacuum method is generally sufficient.

Vacuum Method

1. Be sure the cold trap Dewar has been removed.

1. Display the instrument schematic and enable manual control. Be sure all valves are closed (as shown here).



You will be using Valves 1 (vacuum), 7 (isolation), and 8 (vapor).

Do not open Valves 1 and 8 concurrently. Doing so may cause damage to the instrument.

2. Open Valves 1 and 7; wait until the vacuum gauge reading is 10 µmHg (approximately 5 min).

3. Close Valve 1 and open Valve 8; wait approximately 2 min.

4. Close Valve 8 and open Valve 1; wait approximately 2 min.

5. Repeat Steps (3) and (4) six times so that the vapor source will be degassed thoroughly (or until the 1000-mmHg transducer reads between 40 and 45 mmHg for water at 35 ºC in the vapor enclosure).

The pressure in the manifold will vary if you are using a vapor other than water at 35 ºC. Refer to a Saturation Pressure vs. Temperature table to determine the appropriate pressure for the vapor you are using.

Freezing Method

The vacuum method is typically sufficient for degassing water. However, the freezing method may be used when maximum purity is required.

It is very important to degas vapor source liquids on a weekly basis to maintain purity.

1. Place a Dewar of LN_2 on the instrument cold trap and another Dewar of LN_2 about the vapor tube using the supplied Dewar and stand.

2. Open Valve 8 and evacuate until a vacuum of 10 μmHg is achieved.

3. After 30 minutes, remove the LN_2 from the vapor tube and close Valve 8.

4. When the water in the vapor tube warms nearly to ambient temperature, open Valve 8 and allow evacuation to proceed for 10 minutes.

5. Close Valve 8.

Vapor Analysis

Important things to remember for vapor analyses:

- Remove the cold trap Dewar to prevent vapor from freezing in the cold trap.
- Thoroughly degas the vapor source; refer to **Degassing the Vapor Tube,** page **4**.
- Use the metal jacket on the sample tube when performing analyses at temperatures above ambient; refer to **Using a Metal Jacket on the Sample Tube,** page **13**.
- Close the door on the vapor enclosure.

Specifying Analysis Parameters

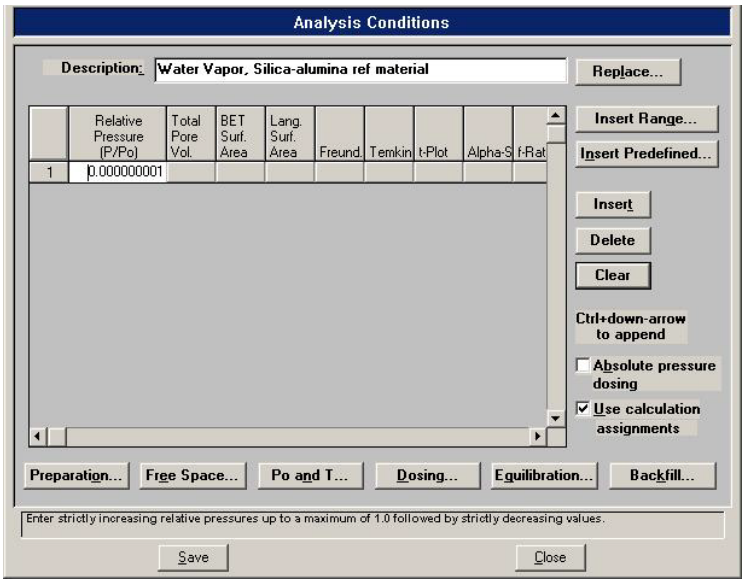
Create standalone parameter files for analysis conditions and adsorptive properties for the materials you typically analyze. Then you can use the **Replace** pushbutton to import file values, making it quick and easy to create your sample file.

The example shown here is for silica-alumina reference material having a relatively large pore volume and analyzed at 25 ºC with water vapor derived from a 35 °C source.

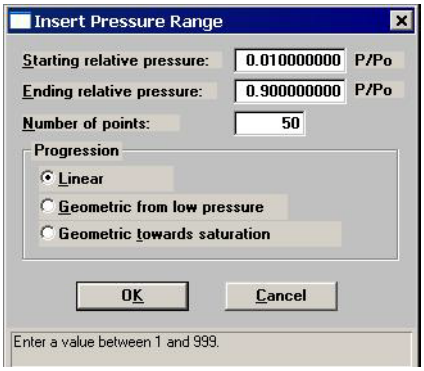
The values used in this example are appropriate for silica-alumina reference material; they will be different for other materials.

Analysis Conditions

1. Select **File > Open > Analysis Conditions** from the menu bar; the Open Analysis Conditions dialog is displayed.
2. Enter an appropriate name, click **OK**, then **Yes** to create the file. The Analysis Conditions dialog is displayed. If the pressure table is populated, click **Clear** to clear the table.

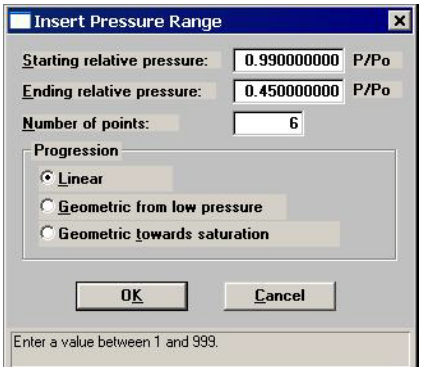


3. Click **Insert Range**.



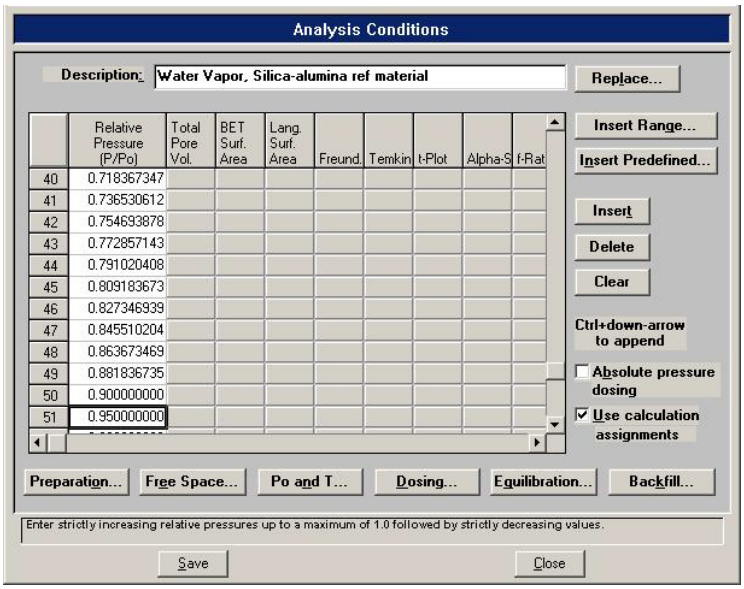
Enter 0.01 for the starting pressure, 0.9 for the ending pressure, and 50 for the number of points; then click **OK**.

4. Click **Insert Range** again.



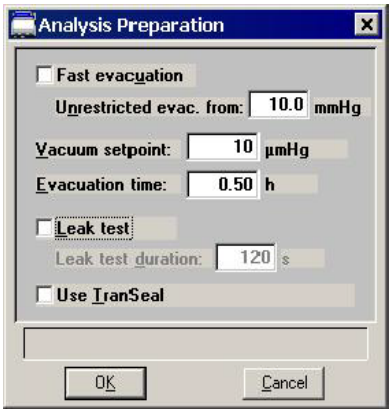
Enter 0.99 for the starting pressure, 0.45 for the ending pressure, and 6 for the number of points; then click **OK**.

5. Select pressure point number **51** in the pressure table, then click **Insert** to insert a new row.

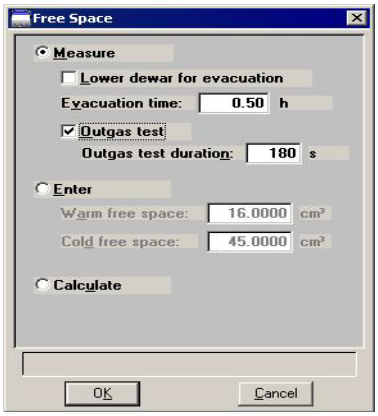


Enter 0.95; the table should have a total of 57 points.

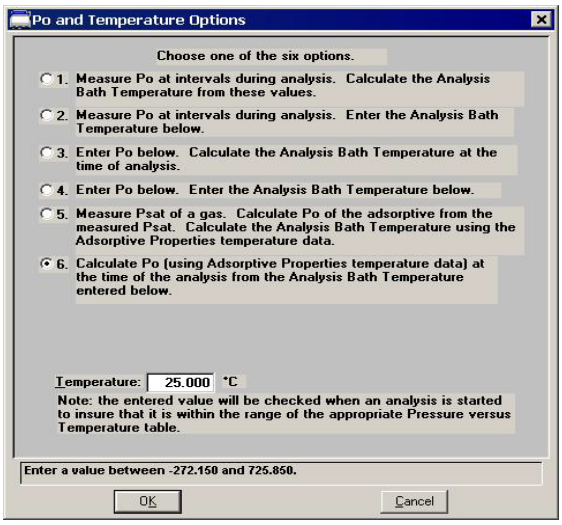
6. Click **Preparation**; specify parameters as shown here.



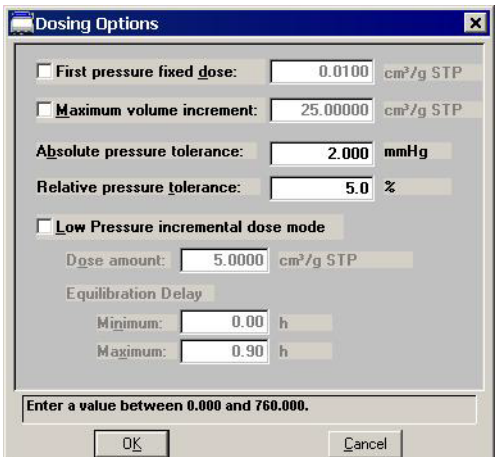
7. Click **Free Space**; enter the parameters as shown here.



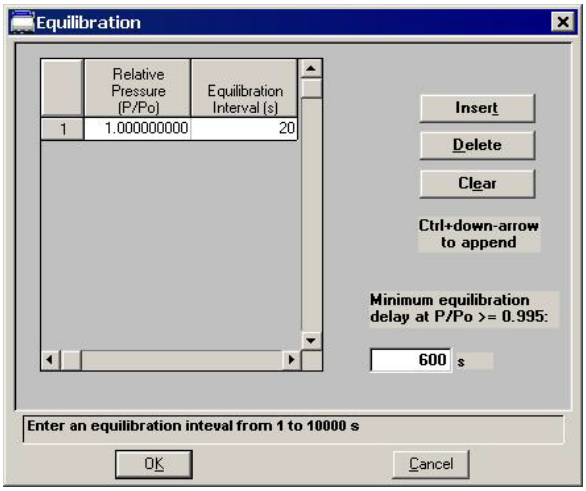
8. Click **Po and T**; select Option **6** and enter **25** in the **Temperature (ºC)** field.



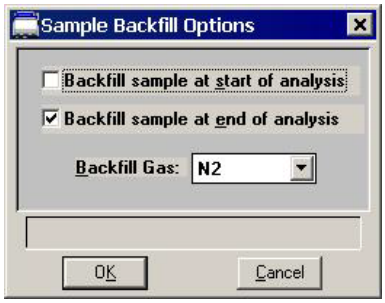
9. Click **Dosing**; specify parameters as shown.



10. Click **Equilibration**; specify parameters as shown.



11. Click **Backfill**; disable (deselect) backfill options.



The sample must be evacuated before analysis when you disable Backfill sample at start of analysis.

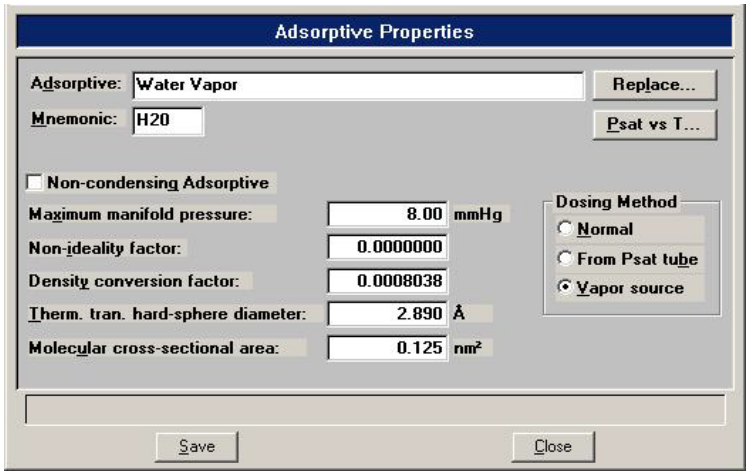
When you deselect **Backfill sample at start of analysis**, you receive a message indicating that disabling this option may damage the instrument. When dosing water vapor, there is no risk of generating high pressures; therefore, it is safe to deselect this option. Click **Yes**, then **OK** to close the dialog.

12. Click **Save**, then **Close**.

Adsorptive Properties

1. Select **File > Open > Adsorptive Properties** from the menu bar; the Open Adsorptive Properties dialog is displayed.

2. Enter an appropriate name, click **OK**, then **Yes** to create the file.



If the adsorption test is solely for the purpose of discovering how water is taken up by the sample, the **Density conversion factor**, **Therm. tran. hard-sphere diameter**, and **Molecular cross-sectional area** values are of little consequence. All that is needed is the adsorption isotherm and, perhaps, the desorption isotherm.

a. **Non-ideality factor**: derived as follows,

$$NIF = \frac{\frac{1}{Z(Po, T_{analysis})} - 1}{Po}$$

where **Z** is the compressibility factor at the saturation pressure of the vapor and **T** is the analysis temperature.

b. **Density conversion factor**: derived as follows,

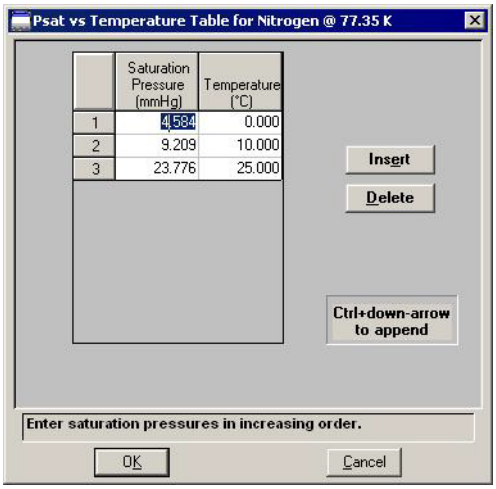
$$DCF = \frac{V_m}{22414\ cm^3/\text{mol}}$$

where V_m is the molar volume of the liquid at the analysis temperature.

c. **Thermal transpiration hard-sphere diameter**: enters into calculations when high precision requires that low-pressure measurements take into account small differences in temperature along gas passageways. The subject is thoroughly treated in Ross, Sidney and Olivier, James P., *On Physical Adsorption*, Interscience Publishers, NY, 1964.

d. **Molecular cross-sectional area**: required when, from the isotherm, the surface area of the sample material is to be calculated. The technical literature, beginning with the extensive tabulation of McClellan and Harmsberger ["Cross-sectional Areas of Molecules Adsorbed on Solid Surfaces," J. Coll. and Interface Sci. 23, 577-99 (1967)], is replete with adsorbed cross-sectional areas on various substrates at different temperatures. In the case of silica-alumina and water, these values range from 0.108 to 0.198 nm^2. A value of 0.125 nm^2 gives BET surface area results that agree well with nitrogen analyses.

3. Click **Psat vs T**; enter the values shown below.



4. Click **OK** to save the table and return to the Adsorptive Properties dialog.

5. Click **Save**, then **Close**.

Using a Metal Jacket on the Sample Tube

A two-piece metal jacket is included in the accessories kit to use when performing analyses at temperatures above ambient. The jacket helps to maintain strictly decreasing temperatures from the instrument to the sample in order to prevent condensation in the portion of the tube above the sample.

The sample may be degassed with the jacket in place.

Install the jacket on the tube before attaching the sample tube to the sample port.

1. Using latex gloves, place a retaining O-ring onto the sample tube; slide it toward the bulb.

The O-rings for the metal jacket are larger than the ones for the sample port. Do not attempt to use sample port O-rings for retaining the jacket, or vice versa.

2. Position both sides of the metal jacket on the sample tube. Slide the O-ring into the lower groove of the jacket.

3. Slide the other O-ring onto the top of the sample tube and into the upper groove of the jacket. The top of the jacket should be approximately 3 cm (1 in.) from the top of the tube to allow for installation in the sample port

4. Attach the sample tube to the sample port.

5. Gently slide the jacket upwards until it touches the sample tube connector nut.

Performing an Analysis at Ice Water Temperature

1. Create a sample file using the analysis conditions and adsorptive properties for water. (Click **Replace** to import the values from the files created in previous sections.)

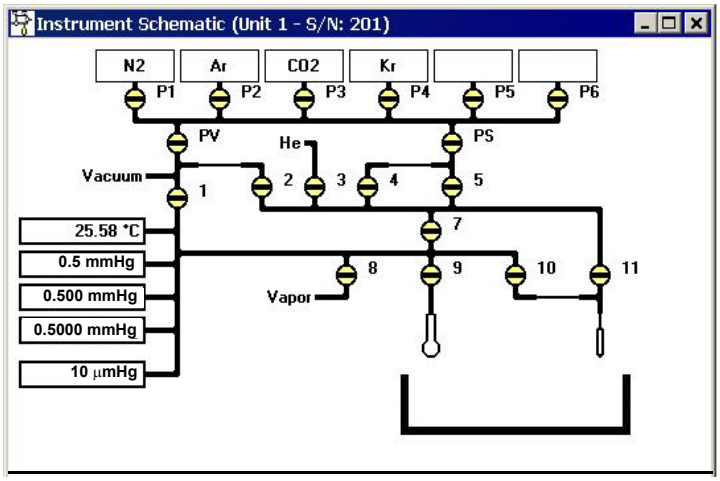
2. Degas the sample to its required conditions.

3. Prepare the ice-slush bath and place it on the elevator.

4. Transfer the sample tube from the degas station to the analysis station.

5. Manually evacuate the sample:

   a Display the instrument schematic and select **Unit [n] > Enable Manual Mode**.



   b. Be sure all valves are closed.

   c. Open Valves N2 and PS.

   d. Open Valves 5 and 7. When 760 mmHg is achieved, close Valves 5 and N2.

   e. Open Valve PV. Wait approximately 10 seconds, then close Valves PV and PS.

   f. Open Valves 9 and 2.

   g. At 10 mmHg, open Valve 1 and close Valve 7.

   h. At 10 µmHg, evacuate for 30 minutes.

6. Right-click the elevator icon on the instrument schematic screen and click **Raise** to raise the Dewar up around the sample.

7. Select **Unit [n] > Start Analysis** to start the analysis.

Shown below in Figure 1 is an example isotherm obtained using the material and analysis conditions specified in this document. A Summary Report is shown in Figure 2.

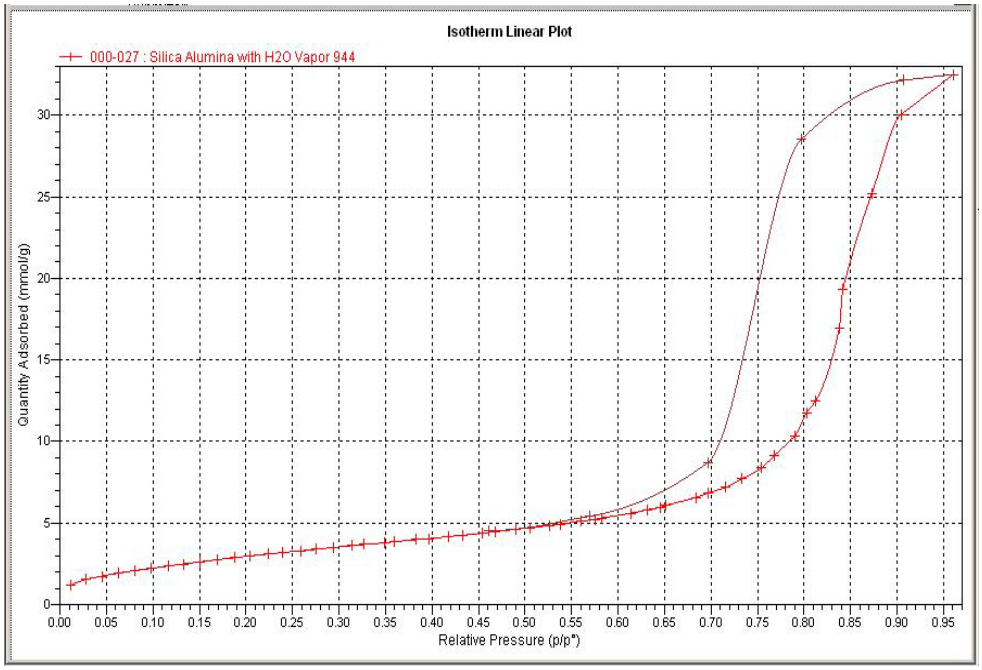


Figure 1. Isotherm for Water Vapor Adsorption on Silica-Alumina Reference Material

micromeritics®

2020 V3.02 H Unit 2 Serial #: 944 Page 1

Sample: Silica Alumina with H2O Vapor 944
Operator:
Submitter:
File: C:\2020\DATA\000-027.SMP

Started: 11/13/2009 4:14:35PM
Completed: 11/15/2009 1:23:41PM
Report Time: 11/30/2009 9:06:26AM
Sample Mass: 0.2194 g
Cold Free Space: 27.7823 cm³
Low Pressure Dose: None

Analysis Adsorptive: VW
Analysis Bath Temp.: 25.000 °C
Thermal Correction: No
Warm Free Space: 27.7650 cm³ Measured
Equilibration Interval: 20 s
Automatic Degas: No

Comments: Need to measure free space after analysis.

Summary Report

Surface Area

Single point surface area at P/Po = 0.293583272: 238.2254 m²/g

BET Surface Area: 261.7239 m²/g

Pore Volume

Single point adsorption total pore volume of pores less than 35.270 Å width at P/Po = 0.960551270: 0.585699 cm³/g

Pore Size

Adsorption average pore width (4V/A by BET): 89.5140 Å

Figure 2. Summary Report: Water Vapor Adsorption on Silica-Alumina Reference Material

Maintenance

The only maintenance required with the Vapor Adsorption Option is replacing a blown fuse in the temperature controller.

Replacing a Fuse in the Temperature Controller

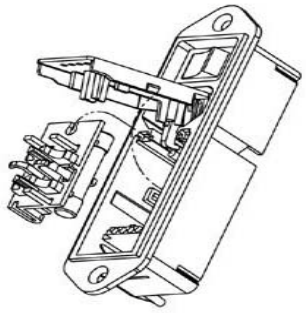
The power control module containing the fuse(s) is located on the rear panel of the temperature controller.

1. Turn off the temperature controller and disconnect the power cord. The cover of the input power connector cannot be opened when the power cord is installed.

2. Insert the tip of a small pocket screwdriver (or pointed object) into the top side of the power module, gently pry the cover open.

3. Grasp the bottom of the cover with your fingers and swing it upward; the cover is hinged and cannot be removed.

4. While holding the cover open, remove the fuse block (you may have to use needle-nose pliers to grasp the fuse block).



5. The input power connector can be used with either a single-fuse arrangement (100-120 VAC) or a double-fuse arrangement (200-240 VAC).

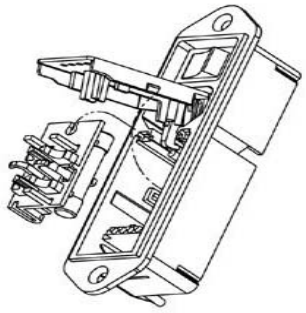
The fuses used in the temperature controller must be identical in type and rating to those specified. Use of other fuses could result in electrical shock and/or damage to the unit.

Remove the blown fuse and insert a new one (spare fuses are included in the accessories kit).

Power Source	Fuse
100-120 VAC	5 Amp, slow-blow (requires one)
200-240 VAC	3.15 Amp, 5x20 mm, slow blow (requires two)

6. Insert the fuse block into the input power connector (as shown in the following illustration).

   - If a single-fuse arrangement (100-120 VAC), the fuse block is positioned so that the side with the single-fuse slot and the jumper bar is away from the cover.
   - If a double-fuse arrangement (200-240 VAC), the fuse block is positioned so that the side with the double-fuse slots is away from the cover.



7. Snap the fuse block into place, then close the cover.

   If using the double-fuse arrangement, the fuse block will not snap into place. Simply position the fuse block in the correct orientation and close the cover; the fuse block will seat properly.

8. Reconnect the power cord and turn on the temperature controller. Allow at least four hours for the manifold temperature to stabilize.

Do not adjust the manifold temperature to speed the process. If the manifold temperature is changed, recalibration by a Micromeritics service representative is required.