micromeritics®

ASAP 2020 Chemi

Accelerated Surface Area and Porosimetry System

Operator's Manual

Rev E

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

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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.

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1. GENERAL INFORMATION

Manual Conventions

This manual uses the following conventions:

	Indicates important information pertinent to the subject matter.
	Provides information that helps you to prevent actions that may cause personal injury.
	Provides information that helps you to prevent actions that may damage the analyzer.
Blue words	Indicate a link to additional information about the subject matter.

Equipment Description

The ASAP 2020 Chemi System consists of an analyzer and a computer, which enables you to enter analysis and report options.

The ASAP 2020 Chemi System analyzer contains two sample preparation ports and one analysis port. In-line cold traps are located between the vacuum pump and the manifold in both the analysis and the degas systems.

The analysis furnace resides on an elevator. A removable shield to enclose the furnace is also included for safety purposes.

Gas Requirements

Analyses performed by the ASAP 2020 Chemi System analyzer require compressed gases. Gas bottles or an outlet from a central source should be located as near as possible to the analyzer.

Appropriate regulators that have been leak-checked and specially cleaned are required. Pressure relief valves should be set to no more than 30 psig (200 kPag). Gas regulators are available from Micromeritics.

All gases should be of a purity of 99.999% or better.

Analysis Software

The ASAP 2020 Chemi analysis program is designed to operate in a Windows environment, providing a user-friendly interface for performing analyses and generating reports.

The ASAP 2020 Chemi System software monitors and controls the analyzer. It enables you to perform automatic analyses with just a few keystrokes, and collects and reports analysis data. You can choose from a variety of reports, which can be printed automatically after an analysis or stored and printed later.

The report system included in the software allows you to manipulate and customize reports in a variety of ways. Refer to **Onscreen Reports**, page **7-9** for details on the options available for reports. You can also zoom in on portions of the graphs or shift the axes to examine fine details. Scalable graphs can be copied to the clipboard and pasted into other applications.

To make it easier for you to obtain information, an electronic copy of the operator's manual is included on the Help menu. This enables you to access information in just a couple of mouse clicks.

Specifications

The ASAP 2020 Chemi System has been designed and tested to meet the specifications provided below.

Characteristic	Specification
PRESSURE MEASUREMENT	
Range:	0 to 950 mmHg
Resolution:	
1000-mmHg Transducer	0.001 mmHg (Analysis system) 1 mmHg (Degas system)
10-mmHg Transducer*	0.00001 mmHg
1-mmHg Transducer** (optional for high stability)	0.000001 mmHg
Accuracy (Analysis system only):	
Includes nonlinearity, hysteresis, and nonrepeatability. Transducer manufacturer's specifications.	
1000-mmHg Range	Within 0.15% of reading
10-mmHg Range*	Within 0.15% of reading
1-mmHg Range**	Within 0.12% of reading
VACUUM SYSTEM	
Vacuum Pump:	Mechanical, two-stage, for analysis; optional for degas. Ultimate vacuum 5 x 10^{-3} mmHg. Dry pumps available for systems equipped with optional High Vacuum pump.
High Vacuum Pump (if installed):	Less than 3.8 x 10^{-9} mmHg Ultimate vacuum measured by pump manufacturer according to Pneurop Standard 5608.
Vacuum Pump Exhaust:	**Analysis**: a 12.5-mm (1/2-in.) fitting is provided for the analysis pump to allow connection to a fume hood. **Degas**: An oil mist filter is provided.
* High Vacuum systems **Micropore systems	

Characteristic	Specification
MANIFOLD TEMPERATURE TRANSDUCER	
Type:	Platinum resistance device (RTD)
Accuracy:	± 0.02 °C (by keyboard entry)
DEGAS SYSTEM	
Temperature Range:	Ambient to 450 °C
Selection:	1 °C increments
Accuracy:	Within ± 10 °C of set point at thermocouple
Backfill Gas:	User-selectable, typically helium or nitrogen
SYSTEM CAPACITY	
Sample Preparation:	2 degas ports (optional)
Analysis:	1 sample port
Total Operating Capacity:	Up to two analysis units can be controlled independently by one computer
SAMPLE TEMPERATURE CONTROL SYSTEM	
Temperature Range:	10° above ambient to 1100 °C
Rate Selection:	Heat ramp rate controllable from 0.1 to 100 °C per minute to 600 °C, 20 °C per minute to 800 °C, 10 °C per minute to 1000 °C, 5 °C per minute to 1100 °C.
Temperature Selection:	1 °C increments
Accuracy:	± 1 °C of reading
Stability:	± 0.1 °C for 10 °C above ambient to 50 °C; ± 0.6 °C above 50 °C
SAMPLE TUBE SIZE	
Flowthru™ sample tube stems are 1/2-in. (12.5-mm) OD with exhaust path for flow-through preparation. Standard flowthru tubes are quartz for use up to 1100 °C.	

Characteristic	Specification
ENVIRONMENT	
Temperature:	10 to 30 °C storing or shipping; -10 to 55 °c storing or shipping
Humidity	20 to 80% relative, noncondensing
ELECTRICAL	
Voltage:	100, 115, 230 VAC ±10%
Frequency:	50/60 Hz
Power:	700 VA, operating
GASES	
Recommended:	Helium for free-space measurement and flowing preparation. Oxygen and hydrogen for flowing preparation. Hydrogen, carbon monoxide, and others for analysis.
PHYSICAL	
Height	99 cm (39 in.)
Width:	85 cm (33.5 in.)
Depth:	61 cm (24 in.)
Weight:	115 kg (250 lbs)
COMPUTER	
Minimum requirements:	Pentium 333 MHz computer (or equivalent) CD-ROM drive 128 megabytes of main memory 1-gigabyte hard drive SVGA monitor (800 x 600 minimum resolution) Windows® 7, Windows XP Professional, and Windows 2000

2. INSTALLATION

This chapter contains instructions for the following:

- Unpacking and inspecting the equipment
- Installing the analyzer
- Installing the analysis program

Initially, your ASAP 2020 Chemi analyzer will be installed and verified for operation by an authorized Micromeritics service representative or a representative of a Micromeritics distributor. If your analyzer is moved to a different location in your laboratory, use the instructions provided in this chapter for reinstallation.

Unpacking and Inspecting the Equipment

When you receive 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.

Save the shipping cartons if equipment or parts have been damaged or lost. The inspector or claim investigator must examine the cartons prior to completion of the inspection report.

Lifting the Analyzer

The ASAP 2020 Chemi analyzer weighs 115 kg (250 lbs) and requires the use of two people to lift it from its shipping carton. One person should not attempt to lift the analyzer. With one person on each side of the analyzer, lift it upright from its shipping carton. Place the analyzer on a table top of sufficient space.

Use proper lifting techniques to prevent back injury.

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, use the following procedure:

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. Referencing the sales order or purchase order, identify 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.

Setting Up the Analyzer

The ASAP 2020 Chemisorption System should be installed correctly and tested to ensure it is operating properly before actual analyses are attempted. Setup and checkout procedures are described in this section.

Installing the Vacuum Pumps

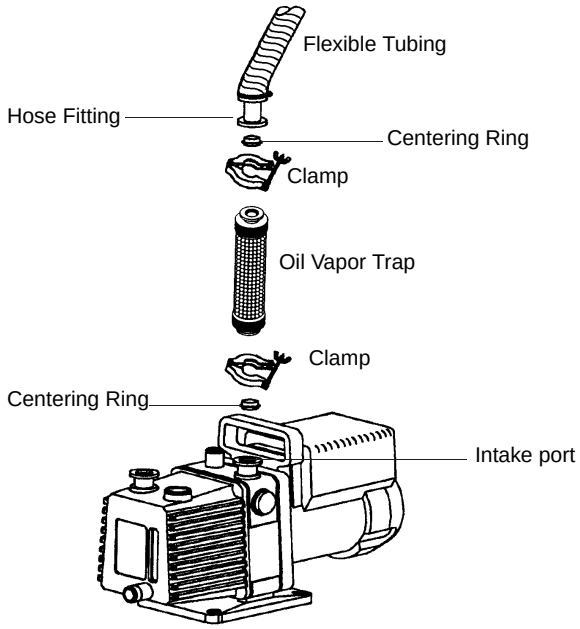
Two vacuum pumps are required for operating the ASAP 2020 analyzer; one for degas operations and one for analysis. A recessed cavity is provided on the rear side of the analyzer for placement of the vacuum pumps. The analysis pump (facing the rear of the analyzer) is on the left side and the degas pump on the right side.

Two types of vacuum pumps are available, the oil-based vacuum pump and the dry vacuum pump. Typically the dry vacuum pump is used when a mass spectrometer is attached to the analyzer and for chemisorption analyses.

Oil-Based Systems

1. Remove the vacuum pump from its shipping carton.
2. Prepare the alumina and oil vapor trap (refer to **Replacing the Alumina in the Oil Vapor Trap**, page **9-11**), then install the trap onto the intake port of the vacuum pump.

   Oil vapor traps reduce the amount of oil vapor that collects in the hoses leading to the instrument.
3. Add fluid to the vacuum pump (refer to **Inspecting and Changing Vacuum Pump Oil**, page **9-8**).
4. Install the vacuum pump exhaust filter (refer to **Replacing the Vacuum Pump Exhaust Filter**, page **9-6**).
5. Place the vacuum pump onto its drip tray and slide into the right side of the vacuum pump cavity. Be sure the power cord is facing outward; do not connect the power cord to a power source at this time.
6. Connect the flexible tubing from the analyzer to the connector on the topside of the pump. The following illustration shows orientation of the vacuum pump and its components attached to the flexible tubing of the analyzer.



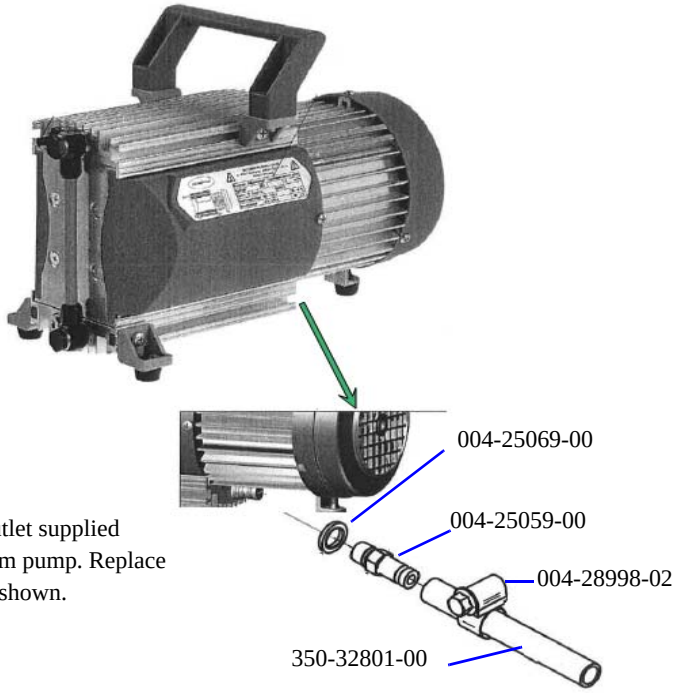
Dry Systems

1. Remove the vacuum pump from its shipping carton.

2. Slide the vacuum pump into the left side of the vacuum pump cavity. Be sure the power cord is facing outward; do not connect the cord to a power source at this time.

3. Connect the flexible tubing from the analyzer to the connector on the topside of the pump.

The gases used are diluted substantially upon being released from the vacuum pumps. However, it may be desirable in some locations to provide a fume hood for added protection from hazardous gases and vapors released into the work area. For piping to the fume hood, use the supplied fittings and tubing to connect to the vacuum pump. Reference the following diagram.



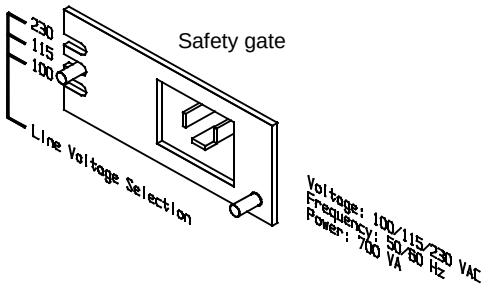
Remove outlet supplied with vacuum pump. Replace with items shown.

Verifying Line Voltage Selection

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

Verify the line voltage as follows:

1. Slide the safety gate to the right.



2. Slide the voltage selection switch to the voltage position to suit the available power supply.

3. Slide the safety gate to the left.

Selecting the Computer Power Input

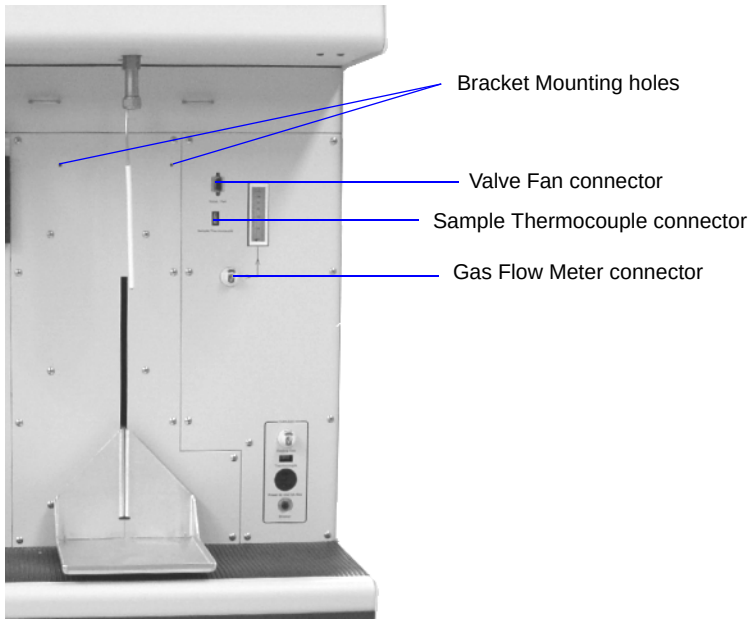
The power input selection on the computer must be set to match the input power source. The computer operates with either 100-120 VAC or 200-240 VAC at 50 or 60 Hz. Refer to the instruction manual supplied with your computer for instructions on selecting power input.

Do not connect the computer power cord to a power source until the proper voltage selection is made. Doing so could result in electrical shock and/or damage to the computer.

Installing the Chemisorption Assembly and Furnace

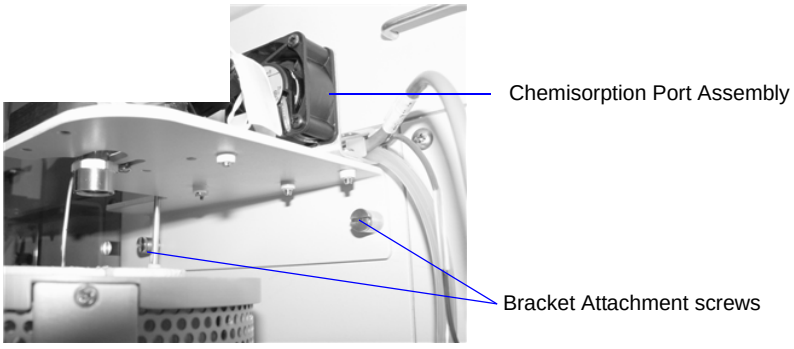
The Chemi Port assembly consist of the following components:

- An exhaust solenoid valve with an O-ring fitting for the sample tube
- A cooling fan to prevent the valve and O-rings from overheating
- A sample temperature thermocouple

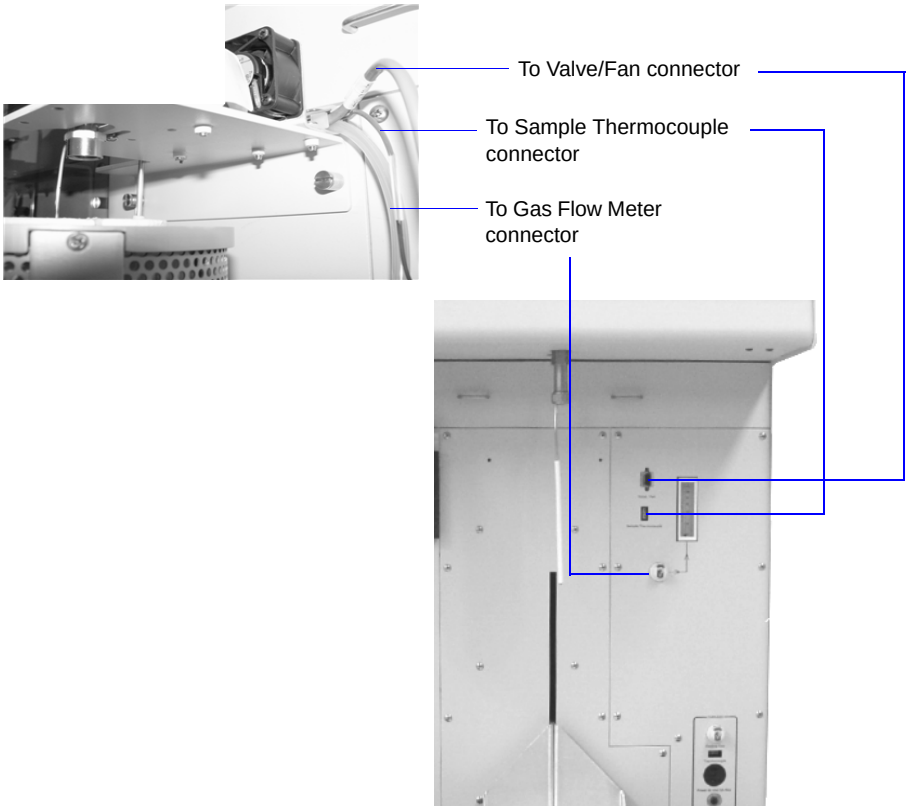


1. Locate the two bracket mounting holes on the elevator panel.

2. Insert the screws on the bracket into the mounting holes. Then turn the screws clockwise; tighten with a flat-blade screwdriver.



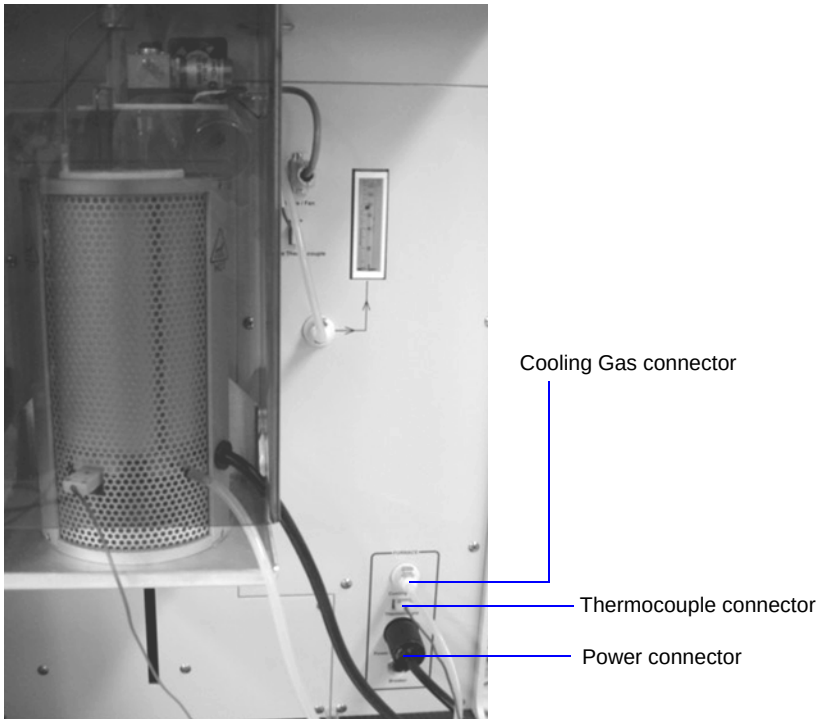
3. Attach the fan cable to the **Valve/Fan** connector.
4. Attach the sample thermocouple cable to the **Sample Thermocouple** connector.
5. Attach the gas flow tubing to the **Gas Flow Meter** connector.



Attaching the Furnace

Attach the furnace as follows:

1. Place and center the furnace on the elevator.
2. Insert the furnace power cable into the Power connector; twist to lock into place.
3. Connect the furnace thermocouple plug to the Thermocouple connector.
4. Connect the cooling gas tube to the Cooling Gas connector.



5. A supply of dry, clean, house/compressed air should be available to attach to the furnace for cooling. The air pressure should be well regulated and adjustable at pressures less than 20 psig at the outlet.

   Make sure the supply gas is set at 5 psig (35 kPag). If the pressure is below 3 psig (20 kPag) or above 10 psig (70 kPag), the furnace controller may not be able to control the stability of ramping, or may be unable to cool the furnace within a reasonable amount of time.

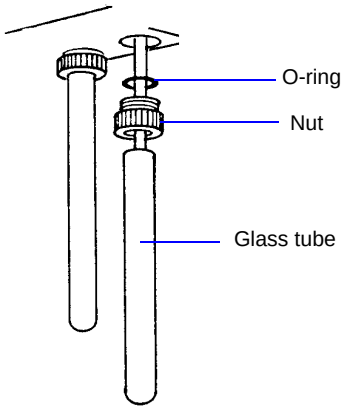
6. Connect a hose from the house/compressed air supply to the furnace cooling gas inlet located on the side panel of the analyzer.

Do not allow the pressure to exceed 20 psig (140 kPag). Pressures higher than 20 psig may cause the internal tubing fittings to fail.

Installing the Cold Trap Tubes

The glass cold trap tubes are supplied with the accessories. Install them as follows:

1. Loosen the nut and O-ring.
2. Slide the glass tube up around the metal tube and secure it with the O-ring and nut.
3. Repeat for the second cold trap port.



Connecting Cables and Power Cords

All cables must be connected securely to their respective connectors for proper operation of the analyzer and its peripheral equipment.

Refer to the instruction manuals supplied with the computer, video monitor, and printer for their respective voltage requirements.

1. Connect the keyboard cable, the monitor cable, and the mouse cable into their respective connectors on the rear panel of the computer. Refer to the manual provided with your computer if you are unsure of connector locations.

Some monitors and printers shipped from the United States must be connected to a 100-120 VAC power source. Connecting this equipment to a 200-240 VAC power source could result in electrical shock and/or damage to the equipment.

2. Plug one end of the printer cable into the connector on the rear panel of the printer. Plug the other end of the printer cable into the connector labeled **LPT1** (may be labeled **PARALLEL**) on the rear panel of the computer.
3. Plug one end of the instrument communications cable into the connector labeled **RS232** on the right side of the analyzer. Plug the other end into the communications port on the computer.

4. Insert one end of the analyzer power cord into the input power connector on the right side of the analyzer and the other end into an appropriate power source.

5. Plug all other power cords, including the vacuum pumps, into an appropriate power source.

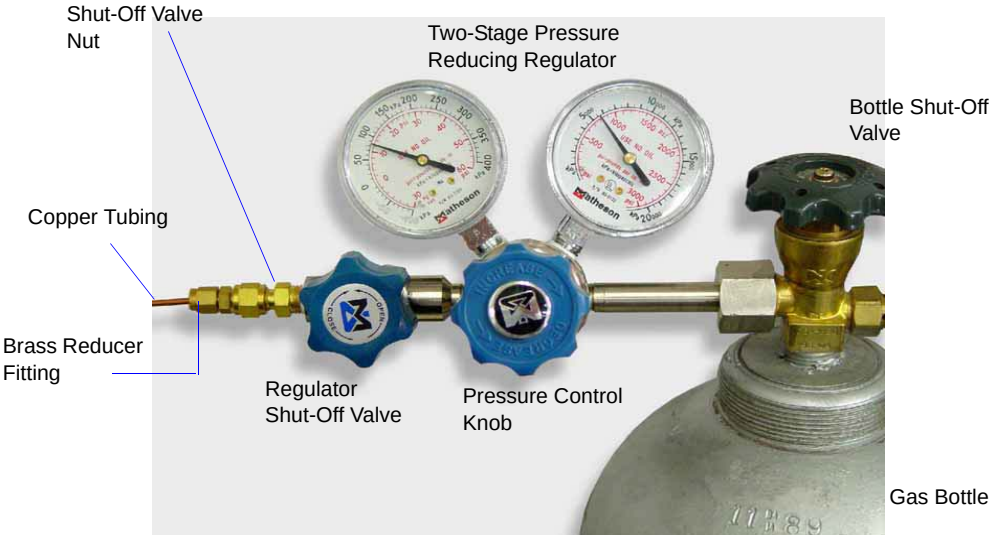
Connecting the Gas Supply and Tubing

Delivery tubes for connecting the gases used with the ASAP 2020 Chemi system are supplied with the instrument. If the gases you are using are incompatible with brass and copper, substitute compatible tubing and fittings. Stainless-steel tubes with fittings are available from Micromeritics. A regulator is required for each gas bottle connected to the analyzer. Appropriate regulators are available from Micromeritics. Refer to Ordering Information, page **10-1** for part numbers.

The quality of the gas regulators used with the ASAP 2020 Chemi System must be comparable to that used for semiconductor manufacturing and gas chromatography. Regulators must avoid or minimize exposing the gas to lubricants and elastomers. Lubrication and elastomers from low-quality regulators may contaminate the gas stream and cause problems such as catalyst poisoning. Low-quality regulators must be flushed with the gas to purge them of contaminants such as air, moisture, and lubricants. These regulators may be unsuitable for evacuation. High quality regulators which match local gas connections and meet safety regulations are usually available from chromatography gas suppliers and semiconductor gas suppliers. Regulators available from Micromeritics can be evacuated thoroughly to remove contamination.

Connecting a Regulator to the Gas Bottle

1. 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 valve.

3. Tighten the regulator shut-off valve.

Do not overtighten the fittings. Doing so could collapse the brass fitting and cause a leak.

4. Attach the copper delivery tubing to the regulator or 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 high 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.

If you are using hazardous gases, ensure that the gas supply equipment is adequately vented to prevent purging the regulator and tubing into the lab atmosphere when performing the next step.

   e. Turn each pressure regulator control knob clockwise until the outlet pressure gauge indicates 15 psig (103 kPag). Open each regulator shut-off valve by turning it counterclockwise briefly. Then close each valve.

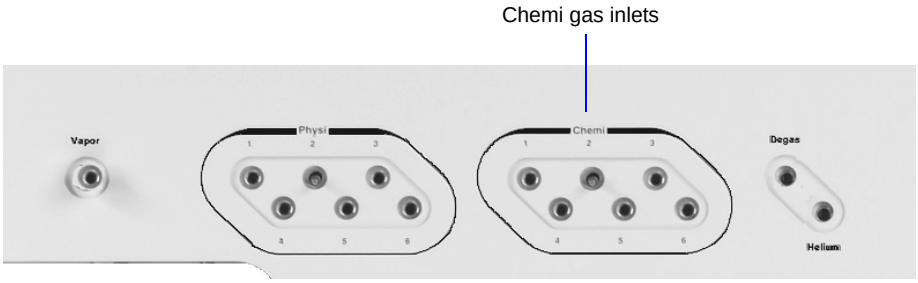
      The gases used for flowing preparation (usually hydrogen and oxygen) may require a pressure other than 15 psig. Start with 10 psig and monitor the gas flow rotameter during the flowing preparation to determine the adequate flow rate. Refer to Appendix H for the procedure on converting the required flow rate to standard cubic centimeters per minute (SCCM) of air. SCCM units are shown on the rotameter.

   f. Make sure the gas bottle valve is completely closed.
6. Repeat steps 2 through 5 for each gas bottle to be attached to the analyzer.
7. Proceed to the next section to attach the other end(s) of the copper delivery tubing to the analyzer.

Connecting the Gas Delivery Tubing to the Analyzer

The ASAP 2020 Chemi analyzer allows for connection of up to six chemisorption gases for chemi operations, up to six physisorption gases for physi operations, one degas backfill gas, and Helium for free-space measurements. Nitrogen or helium (or other suitable gas) can be used as the degas backfill gas.

Gas inlet connections are located on the right side panel of the analyzer labeled 1 through 6 for the Chemisorption gases, Degas for the backfill gas, and Helium.



A typical hook-up for chemisorption gases is as follows:

1	Hydrogen
2	Oxygen
3	Carbon monoxide
4	As desired
5	As desired
6	As desired
Degas	Nitrogen
Helium	Helium

Attach the other end of the copper tubing (from the regulator) to the appropriate gas port on the back of the analyzer.

Be sure to specify which gas is attached to the ports you are using (refer to Specifying Gas Ports, page 2-30).

Turning On the System

1. Place the ON/OFF switches for the computer, monitor, and any peripheral devices in the ON position.

2. Place the analyzer power switch in the ON position; verify that the green LED on the front panel is illuminated.

Turning Off the System

Always make sure you exit the program before turning off the computer. Failure to do so could result in loss of data.

1. Select **File > Exit** or **Close** from the System menu.

   If you exit the ASAP 2020 Chemi System program with analyses in progress, the analyses continue and data are collected. Reports that are queued under the Print Manager will print. If, however, a power failure occurs and a UPS is not attached, the data collected after exiting the ASAP 2020 Chemi System program are lost.

2. Place the computer, monitor, and printer ON/OFF switches in the OFF position.

3. Place the analyzer power switch in the OFF position.

Installing the Analysis Program

The ASAP 2020 Chemi program is also available as a standalone option so that you can install it on a computer other than the one controlling the analyzer. This allows you to create or edit sample and parameter files, as well as generate reports on completed sample files. Review the Micromeritics PROGRAM License Agreement for restrictions on the use of additional copies.

Power Management features should be disabled so that the Micromeritics application can communicate properly with the instrument during operation. These features can be disabled in the computer Setup configuration; through Windows; or through a utility supplied by the computer manufacturer.

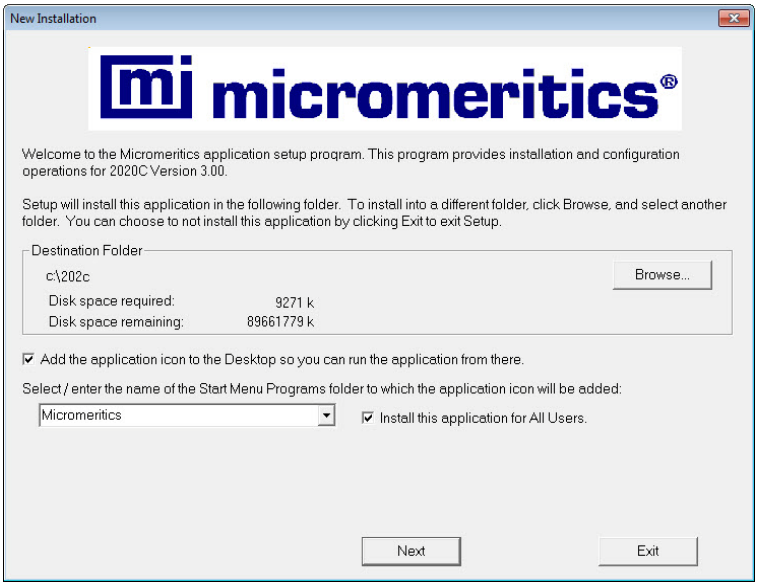
Initial Installation

The ASAP 2020 Chemi System program is supplied on a CD. Perform the following steps to install the program:

1. Turn on the analyzer.
2. Insert the program CD into your CD-ROM drive.
3. Select **Start** from the **Status** bar, then **Run** from the **Start** menu.
4. Enter the name of the drive designator, followed by setup; for example,

   e:setup

5. Click **OK**; the New Installation dialog is displayed.



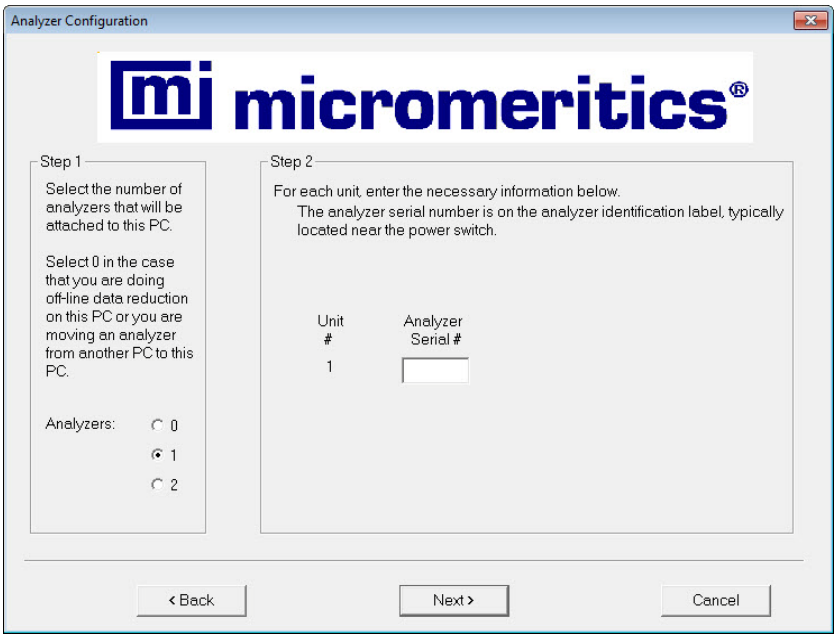
You may cancel the installation at any time by selecting Exit. If you do so, you must start the installation program from the beginning to install the analysis program.

The **Destination Folder** group box displays the amount of current disk space, the amount of disk space required for the analysis program, and the directory into which the application will be installed. Click **Browse** to install the application into a different directory.

6. If you want to run the application from the desktop, select the checkbox just below the **Destination Folder** group box to add an icon.

7. The ASAP 2020 Chemi icon is added to the Micromeritics folder by default. If you prefer a different folder, enter or select one from the drop-down list.

8. Click **Next**; the Analyzer Configuration dialog is displayed.



9. In the **Step 1** group box, click the radio button for the number of analyzers to be attached to this computer. If you are attaching multiple analyzers, it may be necessary to add additional serial ports to your computer.

Choose 0 (zero) if you are installing this program for data manipulation on a computer other than the one controlling the analyzer, or if this is the destination computer in a Move operation.

10. In the **Step 2** group box, enter the serial number(s) for the analyzer(s) you are attaching to this computer.

11. Click **Next**; the Calibration File Installation dialog is displayed. Read the information in the dialog and proceed accordingly.

If you selected 0 as the number of instruments to install, the Calibration dialog is not displayed.

12. After the calibration files are installed, the Installation Complete dialog containing the **Readme** file is displayed.

13. Use the scroll bar if you want to read the contents of the file, then click **Finish** to close the dialog.

14. Remove the Setup CD and store in a safe place. The original Setup CD contains the calibration files specific to your instrument. Upgrade CDs do not contain calibration files. Therefore, it is important that you maintain your original Setup CD in a secure location in the event calibration files need to be reinstalled.

Using the Setup Program for Other Functions

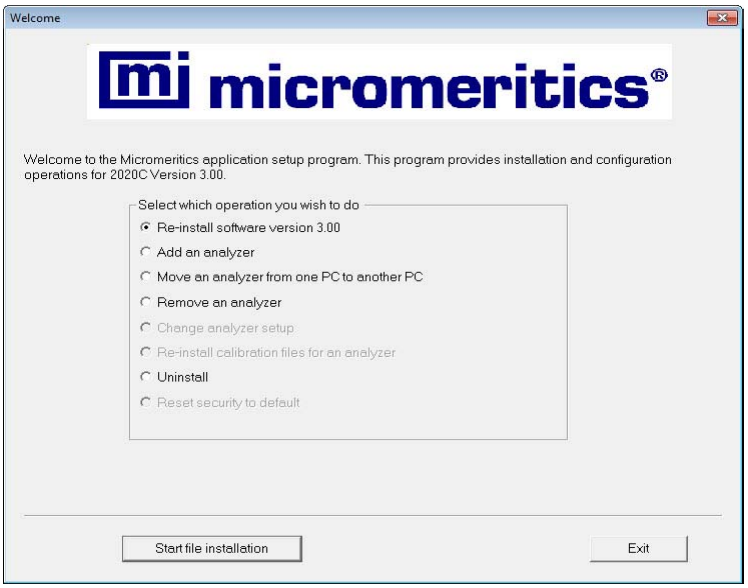
After initial installation of the ASAP 2020 Chemi analysis program, the application setup program can be used to:

- Upgrade software, page **2-20**
- Add an analyzer, page **2-21**
- Move an analyzer from one computer to another, page **2-22**
- Remove an analyzer from the computer, page **2-25**
- Change the analyzer setup, page **2-26**
- Reinstall calibration files, page **2-27**
- Uninstall the analysis program, page **2-28**

To start the application setup program:

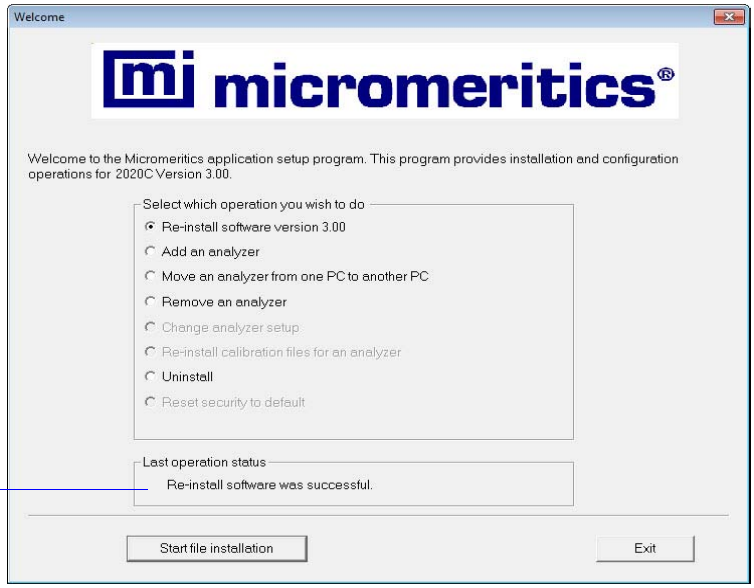
1. Ensure that the analysis program is not operating.
2. Insert the CD into your CD-ROM drive.
3. Select **Start** from the Windows Task bar.
4. Select **Run** from the **Start** menu.
5. Enter the drive designator of the CD-ROM drive, followed by setup. For example: **e:setup**.

   Alternatively, you can click **Browse**, navigate to your CD-ROM drive, and select **setup.exe**.
6. Click **OK**; the setup Welcome screen showing the options available is displayed.



7. Select the operation to perform. Procedures for performing each operation are located in subsequent sections.

   After the requested operation is completed, the setup Welcome screen is again displayed. A confirmation message indicating completion of the operation is shown in the lower section of the dialog.



Indicates status of last operation performed.

8. After you have completed all operations, click **Exit** to close the dialog.

Installing Subsequent Software Versions

When you install a software upgrade, the system installs all of the application files and any status files that do not already exist on the computer. Existing analyzer status files are not affected and default and data files are not overwritten. There are three types of subsequent installation; the software version controlled by the setup program is:

- a later version than the version installed on the computer
- the same version as the version installed on the computer
- an earlier version than the version installed on the computer

The setup program automatically detects which type of installation applies and customizes the selection in the Setup dialog accordingly.

1. Start the Setup program. Choose the software option; remember, only the applicable option will display; it will be one of the following:

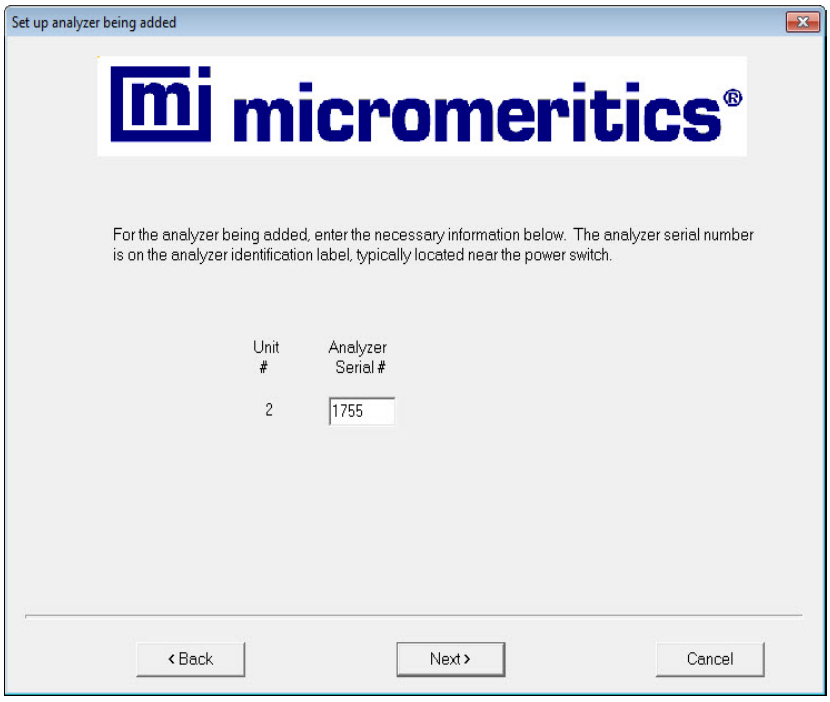
   - Upgrade software to version (number) from version (number)
   - Reinstall software version (number)
   - Downgrade software to version (number) from version (number)

2. Click **Start File Installation**; the application installs the software and redisplays the setup Welcome dialog. Click **Exit** to close the dialog.

Adding an Analyzer

Add an analyzer to the existing application as follows:

1. Start the Setup program. Select **Add an analyzer**, then click **Next**; the Setup analyzer dialog is displayed:



2. Enter the serial number of the analyzer being added, then the communications port to which it is to be connected.

3. Click **Next**; the Calibration Installation dialog is displayed.

4. Select the location of the calibration source files. If the calibration files are located in a directory other than the one displayed, click **Browse** to select the directory. Click **Finish**; a media change dialog is displayed.

5. Click **OK** to install the calibration files. A dialog containing the **Readme** file is displayed after the calibration files are installed.

6. Click **Exit** to close the dialog.

Moving an Analyzer from one PC to another PC

You can move an analyzer, along with its status and calibration files, from one computer (Source PC) to another computer (Destination PC).

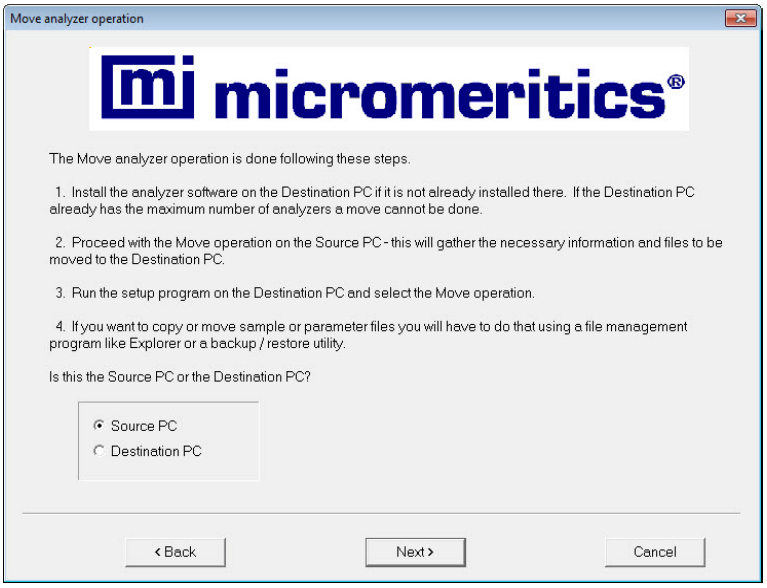
This operation does not move sample or parameter files. To move these files, use a file management program such as Explorer or a backup/restore utility.

1. Install the analysis program on the destination computer (refer to **Installing the Analysis Program**, page **2-15**). Be sure to select **0** as the number of instruments; all related instrument information will be transferred in the Move operation.

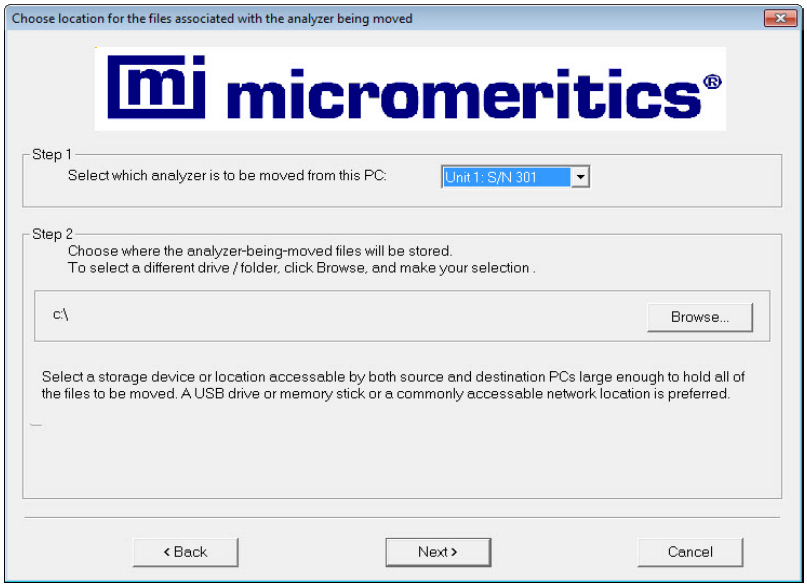
   If the analysis program is already installed on the destination computer, proceed to Step 2.

2. Start the application setup program on the source computer. Refer to **Using the Setup Program for Other Functions**, page **2-18**.

3. In the Setup dialog, select **Move an analyzer** from one PC to another PC, then click **Next**; the Move analyzer operation dialog is displayed.



4. Select **Source PC**, then click **Next**; the following dialog is displayed.



5. In the **Step 1** group box, select the analyzer that is to be moved.

6. In the **Step 2** group box, choose a location in which the moved files will be stored. If possible, choose a floppy drive or a shared network drive. If this is not possible, select a local folder. After the files are placed there, use a folder transfer utility to copy this folder from the Source PC to the Destination PC.

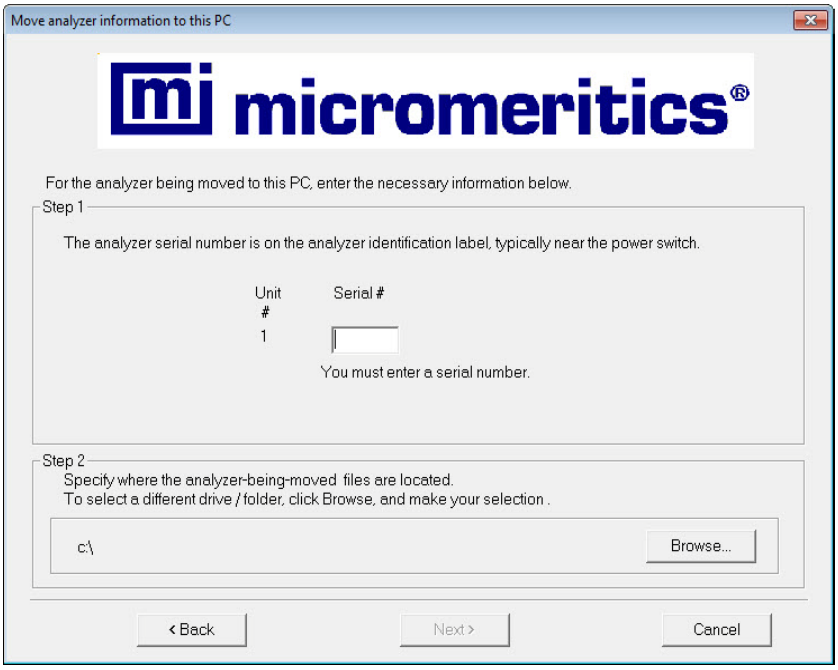
   Calibration files are not included in the moved files. These files must be copied and moved separately to the destination computer. The calibration files are located in a subdirectory (folder) of the Hardware directory. The subdirectory is named 202-(serial number).

7. Click **Finish**; the files are moved and the setup Welcome screen is displayed.

8. Start the application setup program on the destination computer.

9. In the Setup dialog, select **Move an analyzer from one PC to another PC**; the Move analyzer operation dialog is displayed (shown on previous page).

10. Select **Destination PC**, then click **Next**; the following dialog is displayed.



11. In the **Step 1** group box, enter the serial number of the analyzer being moved and the communications port to which it will be attached.

12. In the **Step 2** group box, click **Browse** and choose the location of the moved files.

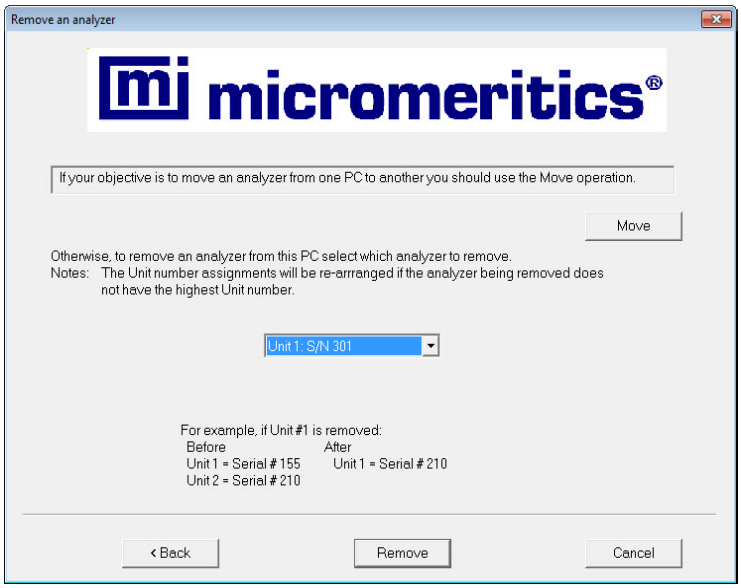
13. Click **Finish**; the files are moved and the setup Welcome dialog is displayed.

Removing an Analyzer

You can remove an analyzer from the computer as follows. When you remove an analyzer, the status files are removed as well.

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions**, page **2-18**.

2. From the Setup dialog, select **Remove an analyzer**, then click **Next**; the Remove an analyzer dialog is displayed.



3. From the drop-down list, choose the serial number of the analyzer you want to remove.

4. Click **Remove**; the analyzer is removed and the Welcome screen is again displayed.

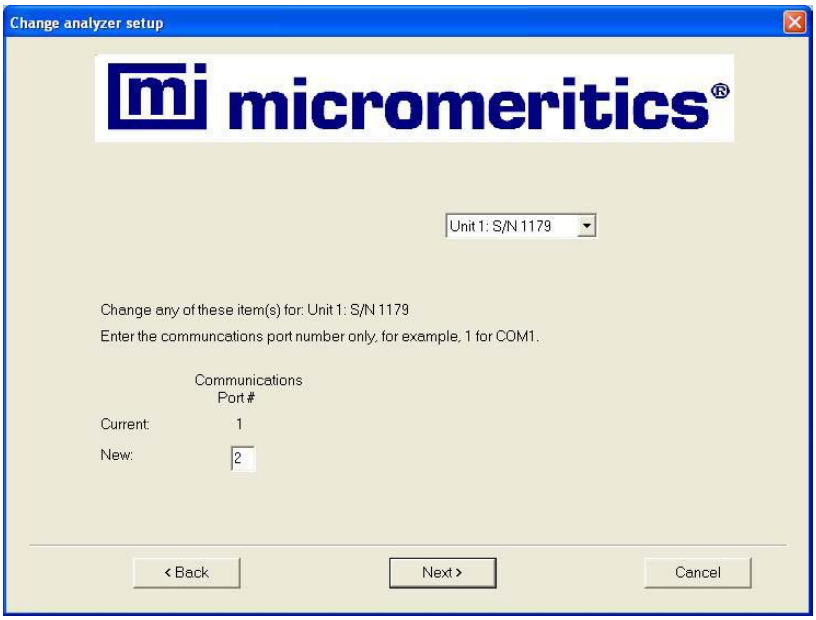
5. Click **Exit** to close the dialog.

Changing an Analyzer Setup

Change the analyzer setup as follows:

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions**, page **2-18**.

2. From the Setup dialog, select **Change analyzer setup**, then click **Next**; the Change analyzer setup dialog is displayed.



3. From the drop-down list, choose the analyzer you want to change.

4. Enter the new port number in the space provided.

5. Click **Finish**; the change is completed and the Welcome dialog is again displayed.

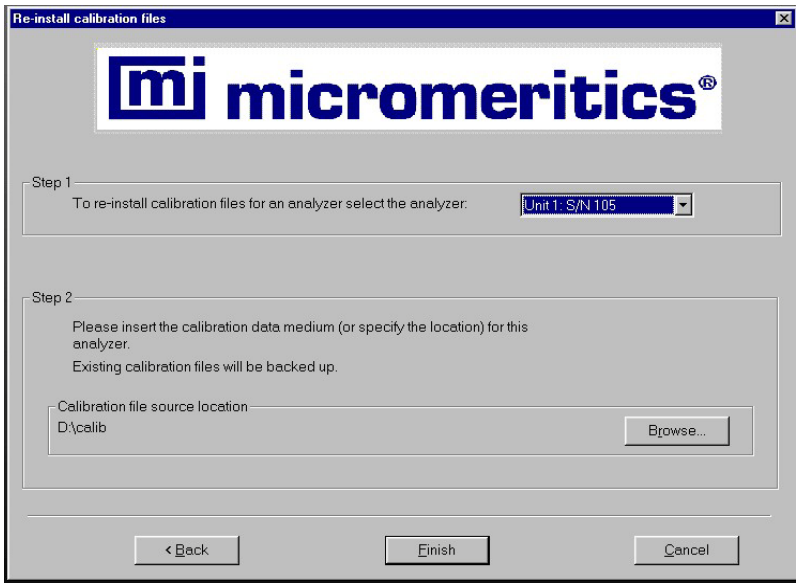
6. Click **Exit** to close the dialog.

Reinstalling the Calibration Files

You can reinstall the files containing an analyzer's factory calibration data as follows:

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions**, page **2-18**.

2. From the Setup dialog, select **Re-install calibration files for an analyzer**, then click **Next**; the Re-install calibration files dialog is displayed.



3. Select the analyzer whose calibration files to reinstall from the drop-down list. If you have only one analyzer installed, a drop-down list is not included.

4. Insert the CD containing the calibration files; ensure that the CD drive is displayed as the calibration file source location. If not, click **Browse** and choose the CD drive.

5. Click **Finish**; the calibration files are reinstalled and the Welcome dialog is again displayed.

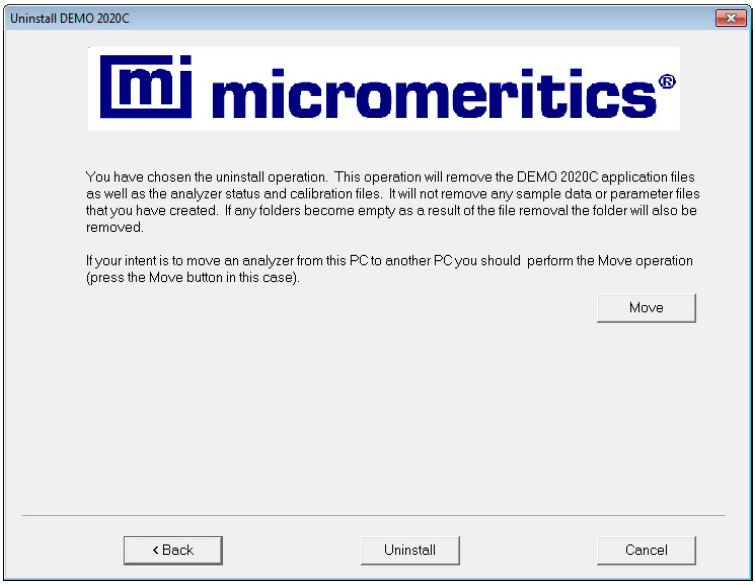
6. Click **Exit** to close the dialog.

Uninstalling the Analysis Program

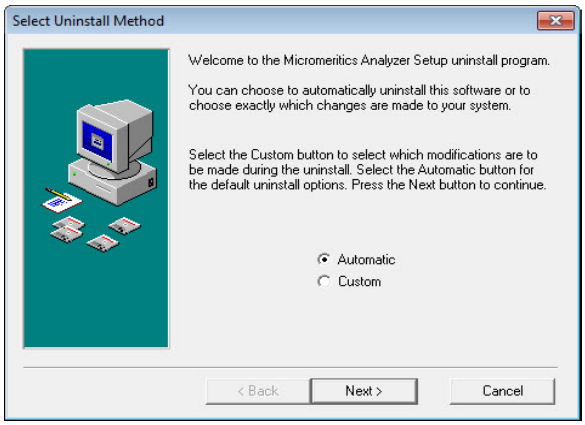
You can remove the ASAP 2020 Chemi analysis program as follows. When you perform this operation, the application removes the analysis program, status files, analyzer setup files, and resulting empty directories. It does not remove data files.

1. Start the Setup program. Refer to **Using the Setup Program for Other Functions**, page **2-18**.

2. From the Setup dialog, select **Uninstall**, then click **Next**; the Uninstall dialog is displayed.



3. Click **Uninstall**; the Select Uninstall Method dialog is displayed.



4. Choose one of the following:

   - **Automatic**: click **Next**; the system uninstalls the analysis program automatically and the setup Welcome dialog redisplays.

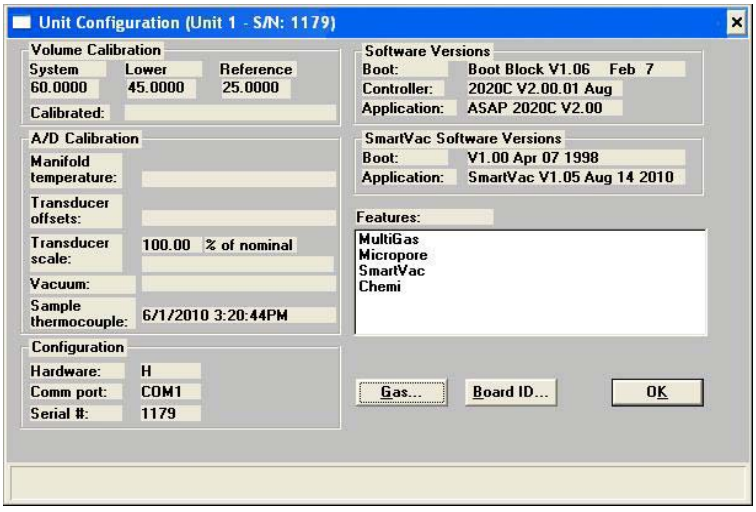
   - **Custom**: click **Next**; a series of dialogs is displayed, allowing you to choose the files to uninstall. After all files are selected and uninstalled, the setup Welcome dialog redisplays.

5. Click **Exit** to close the Welcome dialog.

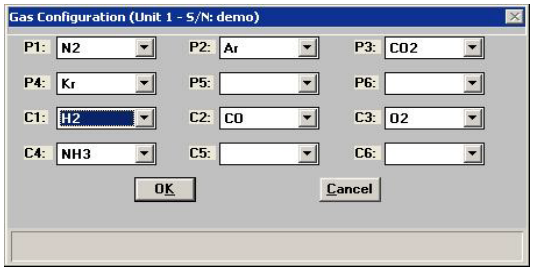
Specifying Gas Ports

After all gases have been attached to the analyzer, perform the following steps to specify gas ports:

1. Select **Unit > Unit Configuration**; the Unit Configuration dialog is displayed.



2. Click **Gas**; the Gas Configuration dialog is displayed.



3. Click on the down-arrow at each field for the ports to which gases are attached and choose the appropriate gas. The ports preceded with a **P** are for physisorption gases and the ones preceded with a **C** are for chemisorption gases.

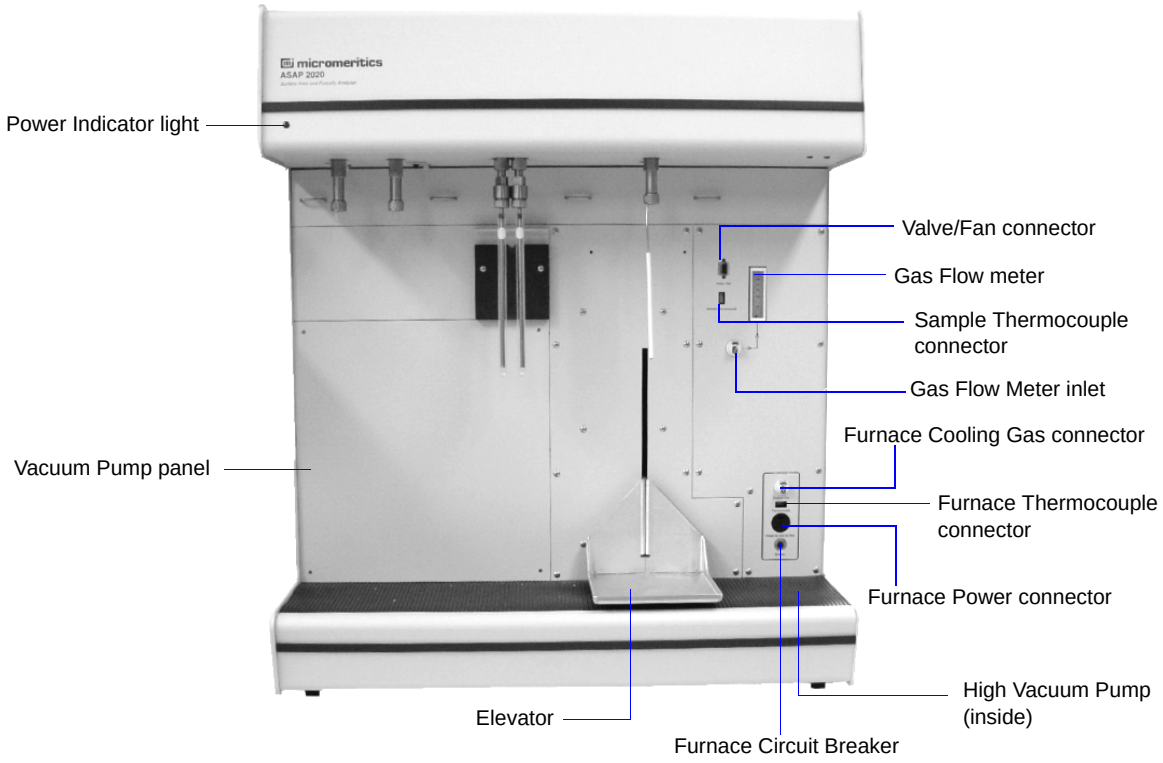
4. Click **Exit** to close the dialog, and then again to close the Unit Configuration dialog.

3. USER INTERFACE

This chapter contains information to familiarize you with the hardware/software of the ASAP 2020 Chemi system.

Controls, Indicators, and Connectors

Front Panel



Power indicator light — Illuminates when power is applied to the analyzer.

Valve/Fan connector — Connects the sample exhaust valve (X), as well as the cooling fan. The cooling fan prevents overheating which could cause damage to the valve and/or O-rings; it is controlled by a thermostat and operates only when excessive heating occurs.

Elevator — For placement of the furnace.

Vacuum pump panel

Allows access to the vacuum pumps. Remove this panel when you need to service the pumps.

You do not have to remove the panel to inspect the condition of the oil or the oil level (oil-sealed forepump). To accomplish either of these tasks, remove (or fold over to the right) the protective rubber mat from the work surface and lift off the metal cover; this will expose the vacuum pump sight window.

Vacuum pump sight window

The vacuum pump sight windows enable you to inspect the oil levels in the degas and analysis pumps.

Gas flow meter

Indicates the flow rate of gas passing through the sample tube. It is calibrated in cc/min STP for air; however, the flow rate indicated varies depending on the gas. Appendix H includes a chart of conversion factors for the flow rates of various gases.

Sample Thermocouple connector

Allows connection of the sample thermocouple.

Gas flow meter inlet

Connects by a valve to the exhaust port of the sample tube to allow verification of the gas flow during the preparation steps.

Furnace Cooling Gas

Allows connection of a compressed air supply for cooling the furnace.

Furnace thermocouple connector

Allows connection of the furnace thermocouple.

Furnace power connector

Allows connection of the furnace power cord.

Furnace circuit breaker

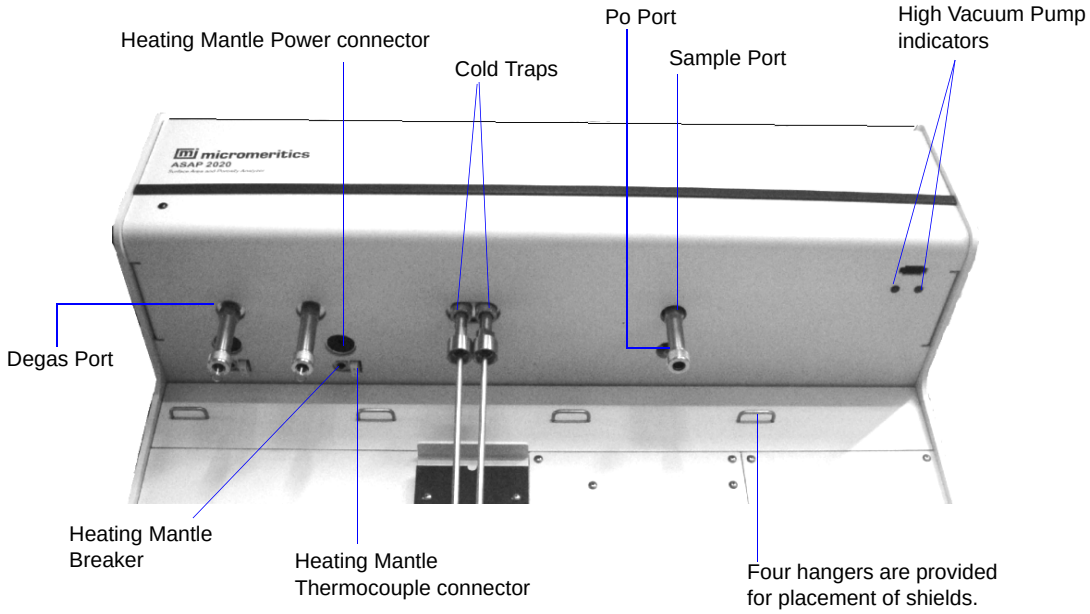
Protects the furnace in the event of a fault in the wiring. If the circuit breaker trips (pops out), call your Micromeritics service representative.

High vacuum pump

A high vacuum pump is used when dry evacuation methods are required. Connections, as well as an on/off (breaker) switch, are located on the front right side of the analyzer beneath the work surface. Remove (or fold over to the left) the rubber mat from the work surface and lift off the metal cover.

Indicator lights (located on the underside of the upper extension of the front panel) illuminate when the high vacuum pump is operating.

The following components are located on the underside of the upper extension of the front panel.



Degas ports

Allow you to degas up to two samples. Each degassing port has connections for a heating mantle.

Heating mantle thermocouple

Allows connection of a heating mantle thermocouple.

Heating mantle power connector

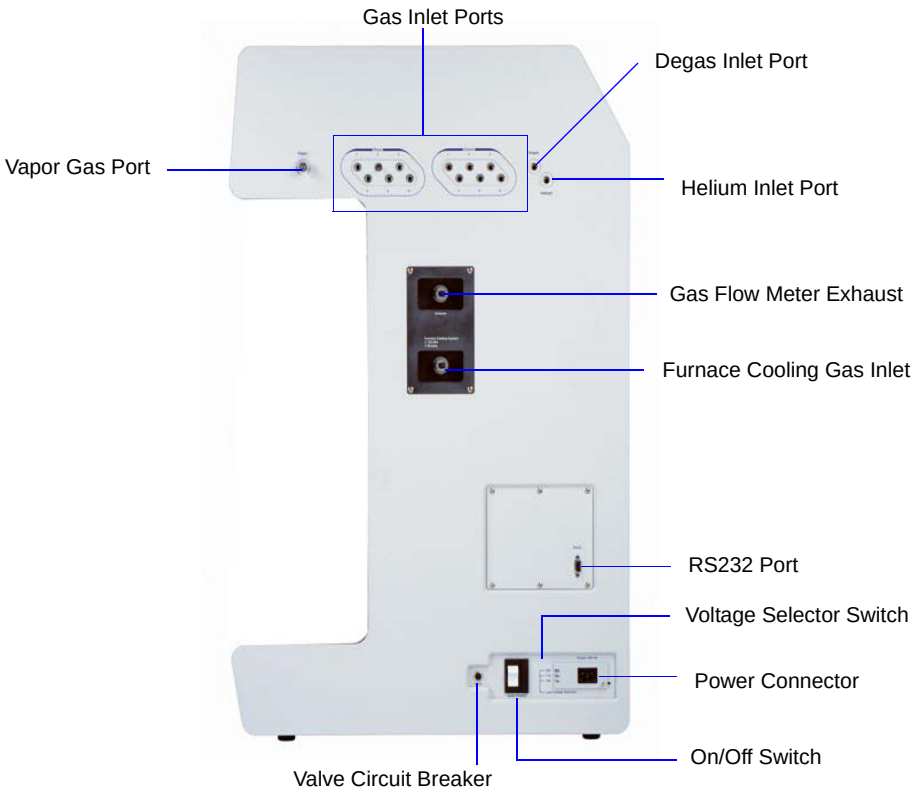
Allows connection of the heating mantle power cord.

Heating mantle breaker

Protects the circuitry for the heating mantle in the event of a failure. If the circuit breaker trips (pops out), call your Micromeritics service representative.

Cold Traps	Two cold traps are provided; one for the degassing operation and one for analysis.
High vacuum pump indicators	Illuminate when the high vacuum pump(s) is (are) operating at normal speed. The left indicator is for degas operations and the right one for analysis.
Sample port	For installing the sample tube containing the material to analyze.
Po port	For installing a Po (saturation pressure) tube when performing physisorption analyses. A Po tube is not used for chemisorption analyses.

Side Panel



Vapor gas port

For attaching the water vapor option, or connecting a vapor gas. Refer to Chapter 10 for ordering information on the water vapor option.

Gas inlet ports

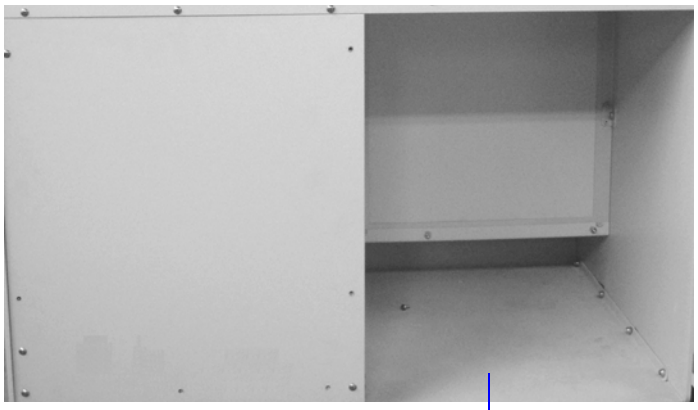
Allows connection of gases to the analyzer. The inlet ports are grouped in two sets of six. The group on the left is for physisorption operation and the group on the right for chemisorption operation.

The chemisorption manifold is constructed of stainless steel and, therefore, allows use of mildly corrosive gases. The physisorption manifold is constructed of aluminum; do not attach corrosive gases to the physisorption ports.

Do not connect corrosive gases to the physisorption ports; doing so may cause damage to the block and gas inlet manifold.

Degas inlet port	Allows connection of the backfill gas used for degassing.
Helium inlet port	Allows connection of Helium used for measuring the free space.
Furnace cooling gas inlet	Admits the air supply for cooling the furnace.
Gas flow meter exhaust	Allows connection to a fume hood for safe venting of preparation gases.
RS232 port	For connecting the analyzer to a computer.
Valve circuit breaker	Protects the circuitry for the valves in the event of a failure. If the circuit breaker trips (pops out), call your Micromeritics service representative.
On/Off switch	For turning the analyzer on and off. This switch also serves as the main breaker for the analyzer; it switches off automatically in the event of an electrical fault.
Power connector	For connecting the analyzer to the electrical supply.
Voltage selector switch	For setting the analyzer to the correct incoming AC line voltage.

Rear Panel



Vacuum pump recess Provides for placement of the vacuum pumps. Vacuum pumps can be serviced from the front of the instrument by removing the vacuum pump panel located on the lower left side of the front panel (see Front Panel described previously).

Using the Software

Keyboard Shortcuts

Keyboard shortcuts can be used to activate some menu commands. Keyboard shortcuts (when applicable) are listed to the right of the menu item.

Certain menus or functions can also be accessed using the Alt key plus the underlined letter in the menu command. For example, to access the File menu, press Alt + F, then press the underlined letter on the submenu. For example, Alt + F opens the File menu, then press O to access the File Selector for opening files.

Key(s)	Function
F1	Access electronic copy of operator's manual
F2	Open a sample information file
	Clear the field of existing date (Select Dates dialog)
F3	Open an analysis conditions file
	Insert the current date (Select Dates dialog)
F4	Display a calendar from which to choose a date (Select Dates dialog)
F5	Open a report options file
F6	Tile open windows
F7	Cascade open windows
F8	Start report
F9	Close all open reports
F10	Generate a Heat of Adsorption report
Alt + F4	Exit the ASAP 2020 Chemi program
Shift + F2	List statistics for sample information files
Shift + F3	List statistics for analysis conditions files
Shift + F5	List statistics report options files
Shift + F9	Access shortcut menu of (1) selected component on instrument schematic, or (2) onscreen reports

File Status and Description

Status	Description
All	All sample information files in the specified directory and within the specified range of dates
Analyzing	Sample information files that are currently being used with an analysis
Complete	Sample information files that have been used with an analysis
Entered	Sample information files that contain manually entered data
No Analysis	Sample information files that have not been used in a degassing operation or an analysis
Prepared	Sample information files that have been used in an automatic degassing operation and are ready for analysis
Preparing	Sample information files that are currently being prepared for analysis

File Name Conventions

For sample information files, a default file name (the next available sequence number) and a default extension are displayed when the Files list box is opened. For analysis conditions and report options, only a default extension is displayed with an asterisk (*.ANC, for example). The following table shows the file name extensions for the AutoChem 2950 HP analysis program.

File Type	Extension
Sample Information	SMP
Degas Conditions	DEG
Analysis Conditions	ANC
Report Options	RPO
Export to disk (ASCII)	ISO
Report to disk	RPT
List to disk	LST
The following types are available for reports saved from the Report window:	
Report	REP
Spreadsheet	XLS
ASCII	TXT

Menu Structure

All functions for the analyzer are accessed from the main menu bar. Brief descriptions of each menu are provided below; refer to the chapter given in parentheses for a detailed description of the options contained on that menu.

File	Enables you to maintain system files.
Unit [n]	Enables you to start an analysis, manually control the system, and perform calibrations. This menu is not shown if the 2020 Chemi program is being used for offline data manipulation.
Reports	Enables you to generate and close reports. Also provides sample copies of reports.
Options	Enables you to select data presentation formats, and enter system default values.
Windows	Enables you to arrange the windows on your screen. It also displays the names of all open files.
Help	Displays Help information.

4. OPERATING PROCEDURES

This chapter contains general instructions for ASAP 2020 Chemi operating procedures. It does not contain detailed descriptions of the dialogs used to perform these procedures. Refer to Chapters 5 through 8 for dialog descriptions. Use the index or bookmark panel of the operator's manual (accessed from the Help menu) to assist you in locating the appropriate dialog.

Specifying Sample File Defaults

The ASAP 2020 Chemi System automatically generates sample information file names and assigns values to them based on the defaults specified in Sample Defaults on the Options menu. This feature makes it easy for you to apply the same conditions to many samples.

The system is shipped with a complete set of default values for sample information files as well as parameter files. **Appendix E**, **Default Files and System Files** beginning on page **E-1** lists the names of default files and a brief description of each.

You can edit the system default values to create a default sample information file which meets your laboratory's requirements. The defaults you specify are the ones you see when you create a new sample or parameter file. Therefore, it is best to specify (or enter) parameters that you plan to use most frequently. For example; specify defaults for your most commonly analyzed sample material. You can always edit parameters in the sample file when it is created.

You can specify sample defaults in the Basic or Advanced format.

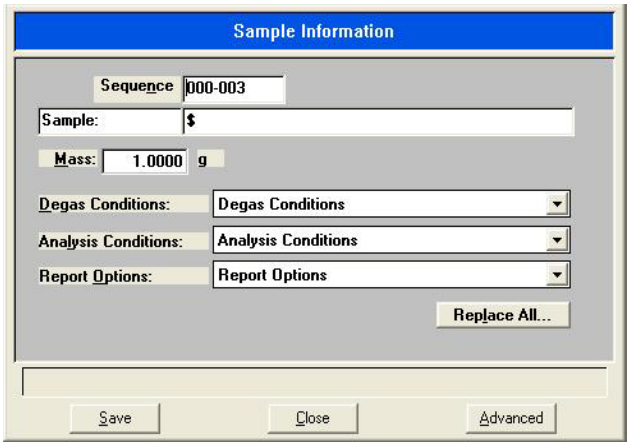
Basic Format

Perform the following steps to define defaults for a sample information file in the Basic format.

Select Options > Option Presentation and ensure that Basic is selected as the format.

Refer to **Basic**, page **8-6** for a detailed description of the fields on this dialog.

1. Select **Options** > **Sample Defaults**; the default Basic Sample Information dialog is displayed.



2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File >Open > Sample information.**

3. In the field on the right of the **Sample** line, enter a format for the sample identification. Be sure to include the **$** symbol to have the sample file number included as part of the identification.

You also can edit the word Sample. For example, you may prefer to use *Material* or *Test*.

4. Enter a default value in the **Mass** field. An approximate value is sufficient; a more accurate sample mass can be entered in the sample file.

5. Select the down arrow to the right of the following fields to choose a default parameter file:

   - Degas Conditions
   - Analysis Conditions
   - Report Options

6. Click **Save**, then **Close**.

Advanced Format

The Advanced Sample Defaults dialog resembles a set of index cards. You can move from one set of parameters to another by clicking the parameter tab or by using **Next** and **Prev**. The values you specify in the parameter portions of the sample file (Degas Conditions, Analysis Conditions, and Report Options) are saved as the defaults for newly created parameter files.

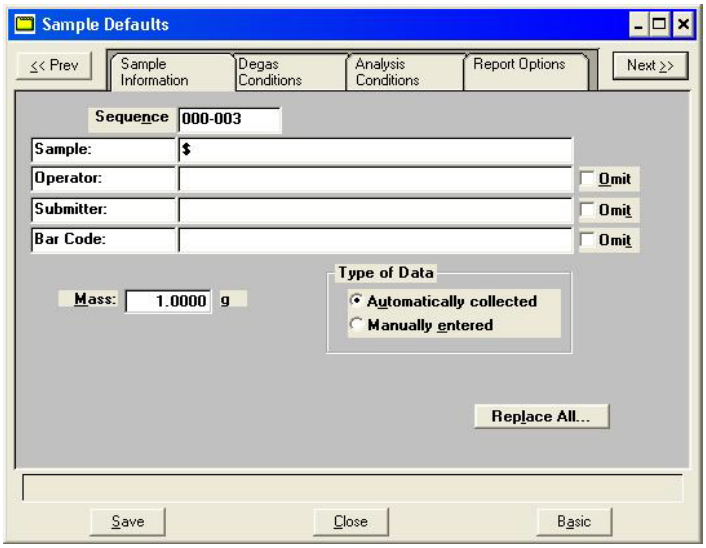
For example, after specifying defaults for a sample file in the Advanced format:

- Select **File > Open > Sample information**; **Yes** to create the file; and the defaults you specify display for all parameters.
- Select **File > Open > Analysis Conditions**, enter a name, then **Yes** to create the file, and the defaults you specify in the Analysis Conditions portion of the Advanced Sample Defaults dialog display in the fields.

Select Options > Option Presentation and ensure that Advanced is selected as the format.

Refer to **Advanced**, page **8-9** for a detailed description of the fields on this dialog.

1. Select **Options** > **Sample Defaults**, the default Advanced Sample Defaults dialog is displayed.



2. In the **Sequence** field, specify a default string for the sample file number. This is the number that appears in the **File name** field when you select **File > Open > Sample information**.

3. In the right-side field of the **Sample** line, enter a format for the sample's identification. Be sure to include the $ symbol if you want the sample file number (Sequence) included as part of the identification.

4. Edit the **Operator** and **Submitter** lines. Or have them omitted entirely by selecting **Omit**.

5. If bar code information is not applicable, select **Omit** to omit this field from the sample information dialog. Or, if you prefer to use this line for some other type of information, edit the label.

6. Enter a default value in the **Mass** field. An approximate value is sufficient; a more accurate mass can be entered at the time of analysis.

7. Choose to have data collected automatically or if you plan to enter the data. You can change this option at the time of analysis.

8. Click the Analysis Conditions tab. Choose the analysis conditions appropriate for your most commonly analyzed material

It is not necessary to click Save on each dialog. Clicking Save on any dialog after all defaults have been specified is sufficient. All dialog defaults will be saved.

9. Click the Degas Conditions tab. Specify degassing criteria.

10. Click the Report Options tab. Choose reports, using **Edit** to specify details; then click **Save**.

11. Click **Close** to close the dialog.

Creating Sample Information Files

A sample information file must be assigned to every sample that is analyzed. When you create a sample file, you can accept the default values specified using Sample Defaults, or you can edit them. You can create a sample information file using the Basic, Restricted, or Advanced format.

Sample information files reside in a directory. The more files in a directory, the longer it may take to access a file. Therefore, it is a good practice to limit the number of files in a directory to approximately two hundred. You may create additional directories as needed. Refer to the operating system manual for more information.

Basic and Restricted Formats

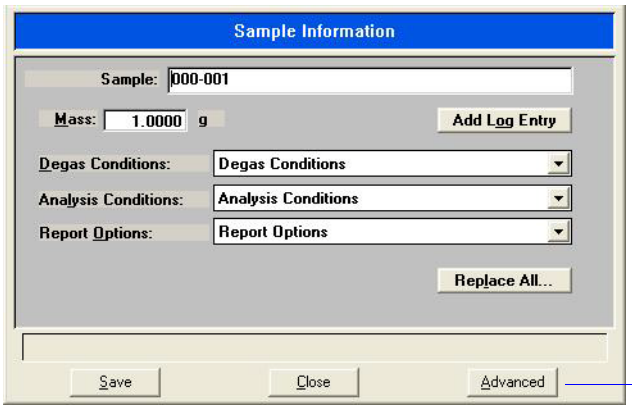
Sample information files are created in the Basic and Restricted formats using predefined parameter files.

Refer to **Basic Format**, page **5-8** for a description of the fields associated with creating a Basic or Restricted sample file.

1. Select **File > Open > Sample Information**; the Open Sample Information File dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Sample Information dialog is displayed.



4. Accept the default identification or change it to an appropriate one. The dialog above shows the sample file number because the dollar ($) symbol was used in Sample Defaults.

5. Enter the sample's mass (if different from the default value) in the **Mass** field.

6. Using the down arrows to the right of each parameter field, select an appropriate file (or accept the defaults). You can review or edit the contents of these files by switching to the Advanced format.

You cannot switch to the Advanced format if you are using the Restricted format.

7. Click **Save**, then **Close**.

Advanced Format

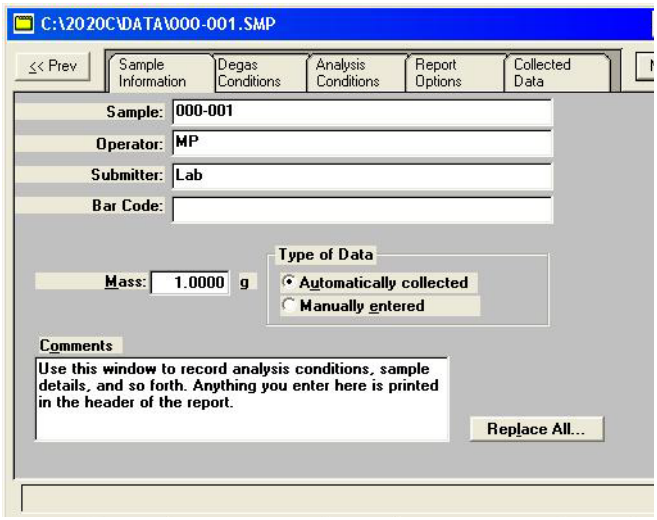
The Advanced format allows you to customize the parameters of a sample information file.

Refer to **Advanced Format**, page **5-6** for a description of the fields associated with creating a sample information file using the Advanced format.

1. Select **File > Open > Sample Information**; the Open Sample Information File dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Sample Information dialog is displayed.



The defaults that appear in the fields are the ones specified in Sample Defaults.

If you selected Omit beside the Operator and/or submitter fields when you specified Sample defaults, these fields will not display.

4. Accept the default identification in the **Sample** field or change it to an appropriate one. The above dialog shows the sample file number because the dollar (**$**) symbol was used in sample defaults.

If a sample information file already exists containing the values to use in this file, you can click Replace All to copy those values into this one. You can still edit the values after they are loaded.

5. Edit the **Operator** and/or **Submitter** fields as needed.

6. Enter bar code information (if used). This field will also accept input from a bar code reader (connected to a USB port on the computer).

7. Enter the sample's mass (if different from the default value) in the **Mass** field.

8. Choose whether you want data automatically collected by the system or whether to enter the data.

Use the Comments window to record specifics of the analysis or its conditions. Anything you enter in this window is displayed in the report header.

9. Click **Save** to save the information you entered.

10. The steps for completing the remaining parameters of the sample information file are explained in subsequent sections:

    - Degas Conditions, page **4-8**
    - Analysis Conditions, page **4-9**
    - Report Options, page **4-11**

    Simply click on the tabs to open its associated dialog.

Defining Parameter Files

The following file types can exist as part of the sample information file or as an individual parameter file:

- Degas conditions
- Analysis conditions
- Report options

Having these files exist independently allows you to use them over and over again.

Several predefined parameter files are included with the ASAP 2020 Chemi program. Although these files may come close to the needs of your laboratory, to define additional ones. Or you can use a predefined file as a starting point. This can be done by creating a new file, then clicking **Replace** and choosing the file containing the values to use. You can edit the values after they have been copied into the new file. The file from which the values were copied remains intact and ready for the next use.

To have parameter files display in the drop-down lists on the Basic and Restricted Sample Information dialogs, be sure to save them to the directory specified as the Parameter Files Directory (refer to **Parameter Files Directory**, page **8-16**).

Degas Conditions

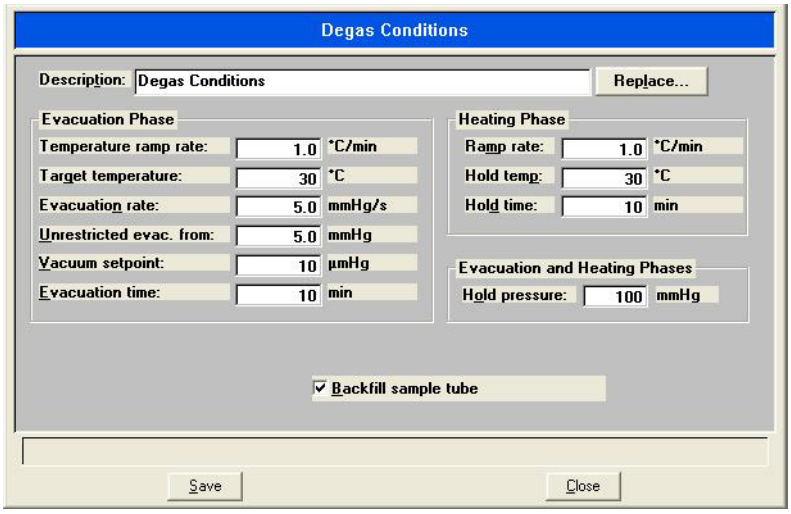
Degas Conditions files contain degassing information for sample preparation. These files are applicable only if your are using the SmartVac for automatic degassing.

Refer to **Degas Conditions**, page **5-11** for a description of the fields on this dialog.

1. Select **File > Open > Degas Conditions**; the Open Degas Conditions File dialog is displayed.

2. Enter a name in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Degas Conditions dialog is displayed.



4. Enter a description in the **Description** field. Use an intuitive description, one that will be easily recognized when needed.

5. Specify conditions for the evacuation and heating phases of the degassing operation.

6. Click **Save**, then **Close**.

Analysis Conditions

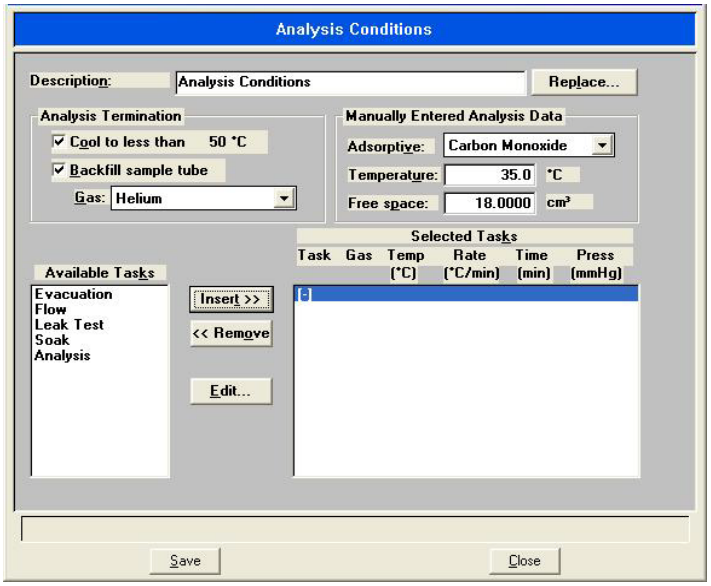
Analysis conditions specify the data used to guide an analysis. An analysis conditions file may be assigned a unique name, and you can direct any sample to be analyzed according to the conditions in any existing analysis conditions file.

Refer to **Analysis Conditions**, page **5-13** for a description of the fields on this dialog.

1. Select **File > Open > Analysis Conditions**; the Open Analysis Conditions File dialog is displayed.

2. Enter a name in the **File name** field, then click **OK**.

3. Click **Yes** to create the file; the Analysis Conditions dialog is displayed.



4. Enter a description in the **Description** field. Use an intuitive description, one that will help you identify the type of sample you plan to analyze using these analysis conditions.

5. Choose one:

If.....	Then....
you plan to use this file for an analysis in which data are to be collected automatically:	In the **Analysis Termination** group box, choose post-analysis conditions for the sample.
	In the **Available Tasks** window, choose and define tasks as required.
you plan to use this file for manually entered data:	In the **Manually Entered Analysis Data** group box, enter the conditions.

6. Click **Save**, then **Close**.

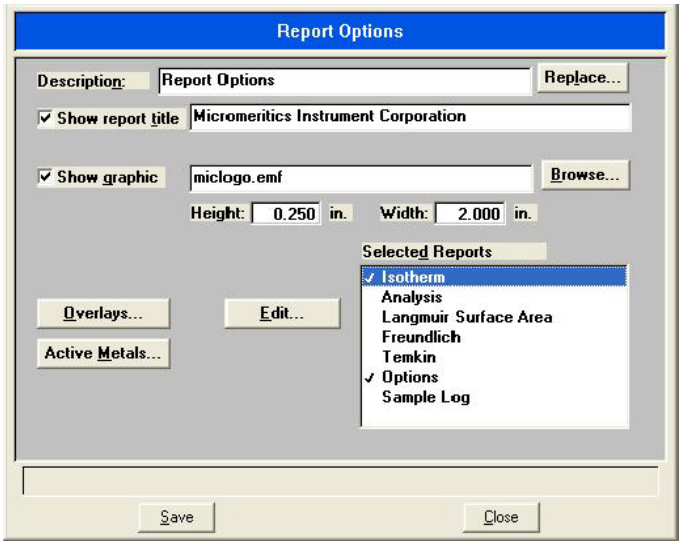
Report Options

Report options specify the types of reports generated from an analysis or manually entered data. They also help you customize details of reports such as axis scale, axis range, and column headings.

Tailor report options files to accommodate the requirements of your analyses. For example, generate a single-page report initially to verify particular characteristics of the sample; then create a more sophisticated report to present details. You can use report options files to generate reports at any time during or after an analysis. However, a report generated during an analysis only includes data collected up to the time of the report.

Refer to **Report Options**, page **5-26** for a description of the fields on this dialog.

1. Select **File > Open > Report options**; the Open Report Options File dialog is displayed.
2. Enter a name in the **File name** field, then click **Save**.
3. Click **Yes** to create the file; the Report Options dialog is displayed.



4. Enter a description in the **Description** field. Use an intuitive description, one that will be easily recognized when needed.
5. Select **Show report title** and enter the title to appear at the top of the report. Or deselect this option if you prefer not to have a report title.

If your company logo exists as a bitmap or enhanced metafile, you can have it display in the report header by selecting Show graphic. Then click Browse to select the file. Use the Height and Width fields to specify the size.

6. To choose the files. Then be sure to access the graph options dialog and choose **Overlay samples**.

7. The reports that may be generated are listed in the window adjacent to the **Edit** button. Select reports by double-clicking on the report. A report is selected when it is preceded by a check mark. Reports are deselected in the same manner.

   You can edit some reports by highlighting the report and clicking **Edit**.

8. Click **Save**, then **Close**.

Preparing Samples

Care should be taken in preparing samples for analysis. The following guidelines help ensure accurate, reproducible results.

Cleaning and Labeling Sample Tubes

Sample tubes must be clean and dry before samples are added and weighed. The following procedures are recommended. Refer to the following table for a list of materials needed to clean and weigh samples properly.

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

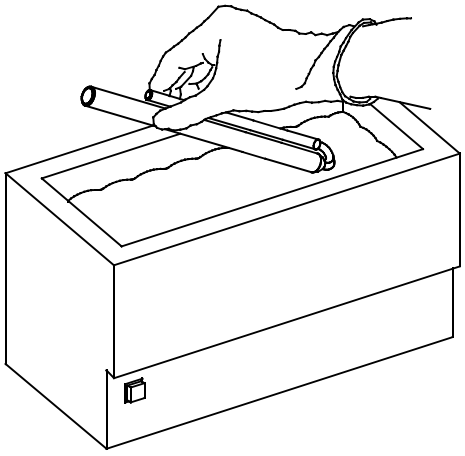
Supplied by Micromeritics	Supplied by User
Sample tube	Drying oven
Quartz wool	Ultrasonic cleaning unit
Sample tube brush	Alconox Rubber gloves or lint-free cloth
Stopper and cap for sample tube	Acetone or isopropyl alcohol
Sample tube rack	Safety glasses
Sample weighing support	Forceps
Cloth gloves	Insulating gloves
Reference material	Waste container
Funnel	Balance
Sample data worksheet (copied from Appendix A of this manual)	Pipe cleaners

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

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

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

4. Fill the sample tubes with warm water and place them in the reservoir of the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit for approximately fifteen minutes.



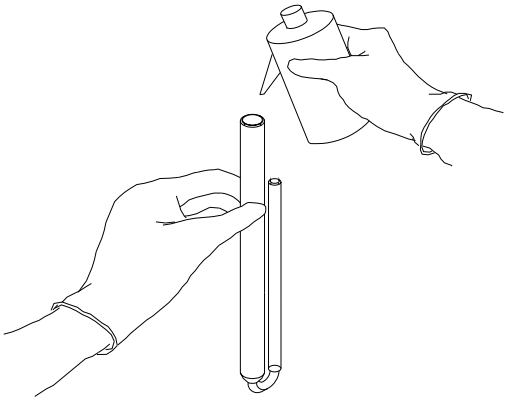
5. Using rubber gloves, remove the sample tubes from the reservoir.

6. Clean the interior of the sample tubes with the brush supplied with the ASAP 2020 Chemi System.

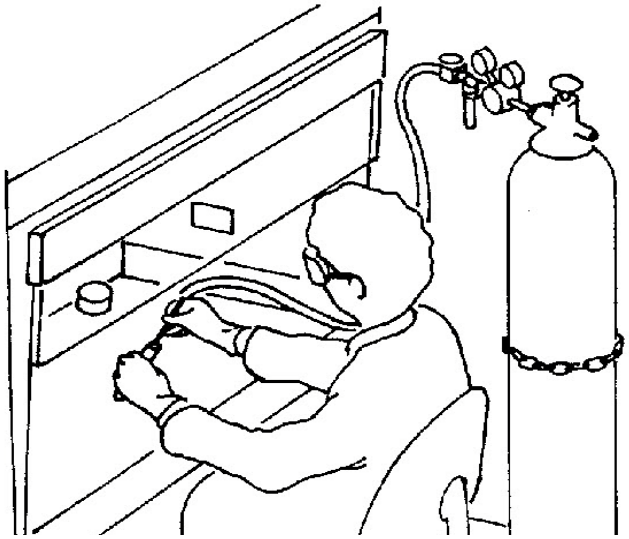
7. Rinse the sample tubes thoroughly with hot water. Then rinse them with isopropyl alcohol or acetone using a waste container to collect used solvent.

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

Avoid touching clean sample tubes with your bare fingers. Skin oils weaken the quartz tubes when the tubes are heated.



8. Using dry, compressed nitrogen or air, dry the interior of the sample tubes under a vent hood. Use a tubing extension long enough and small enough in diameter to fit inside the tubes.



9. Stand the sample tubes on the sample tube rack. Bake for two hours.

10. Remove the sample tubes from the oven and allow them to cool.

11. Using either rubber gloves or a lint-free cloth (but not bare hands), insert a piece of quartz wool into the sample tube. Push it gently to the bottom using a clean, dry stirring rod or piece of wire. Do not pack the wool, or you will inhibit gas flow.

After loading a powder or sample made of fine particles into the sample tube, be prepared to insert another piece of quartz wool into the tube on top of the sample. This prevents particles from being drawn up into the instrument and contaminating the manifold.

12. Wipe the rubber stopper and cap with a lint-free cloth.

13. Label the sample tube, stopper, and cap for identification.

14. Fill the tube with the backfill gas. Use a tubing extension long enough and small enough in diameter to fit inside the tube. Insert the stoppers (and additional quartz wool, if required) quickly to avoid releasing the gas.

To obtain the accurate mass of a degassed sample, the gas in both the empty sample tube and in the tube with the sample must be the same.

After the analysis, the sample tube contains the analysis gas or, if free space was measured, helium. If you want to backfill the sample tube following the analysis, avoid mixing gases and creating a potentially dangerous situation. Insert an evacuation task after the analysis task.

Determining the Sample's Mass

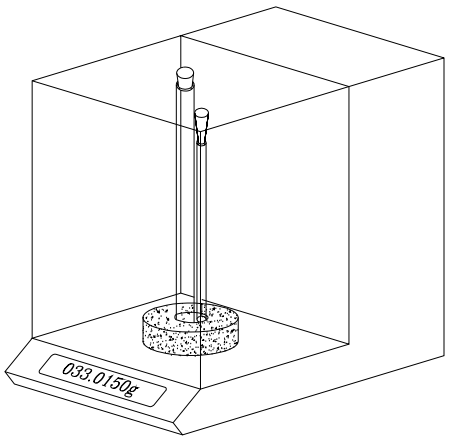
Analysis results are expressed in units of area per gram of sample (m^2/g) or cubic centimeters per gram of sample (cm^3/g); therefore, the true weight of the sample must be known. Carefully weigh each sample tube set and sample as described below.

1. Write the **Sample Tube** number on the Sample Data Worksheet. (A Sample Data Worksheet, which you may copy, is included in Appendix A, page **A-3**.)

2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).

3. Push a piece of quartz wool down inside the sample tube. If you are analyzing a powder or sample made of fine particles, put a second piece of quartz wool just inside the sample tube. Put the stopper and the cap on the sample tube.

4. Place the sample tube set (sample tube, stopper, cap, and quartz wool) on the sample support. Record the stabilized weight on the Sample Data Worksheet as **Mass of empty sample tube**. Remove the sample weighing support and sample tube set from the balance.



5. If you do not want the sample's mass before degas or analysis, skip to step 7.

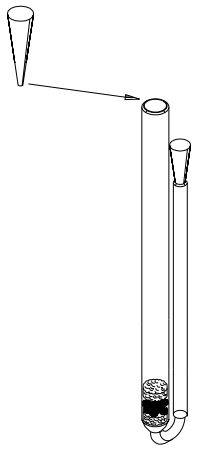
   Place a sample container on the balance. Tare the balance and allow it to stabilize at zero (0).

Do not touch the sample (reference material) with bare hands while performing the next step. Oil from your fingers could affect the accuracy of results.

6. Slowly add the sample to the sample container.

7. Remove the large rubber stopper from the sample tube. If you are analyzing a powder or sample made of fine particles, also remove the top portion of quartz wool from the tube.

8. Using a funnel, pour sample from the container into the sample tube on top of the quartz wool remaining in the tube. If some sample clings to the inside of the sample tube above the last 3 in. (7.5 cm) of the tube, remove it using a pipe cleaner or lint-free wipe.



9. Insert the top portion of quartz wool (if used) into the tube and press it down.

10. Insert the large rubber stopper.

11. Weigh the sample tube set containing the sample and record its mass on the Sample Data Worksheet as **Mass of sample tube plus sample (Before Degas)**.

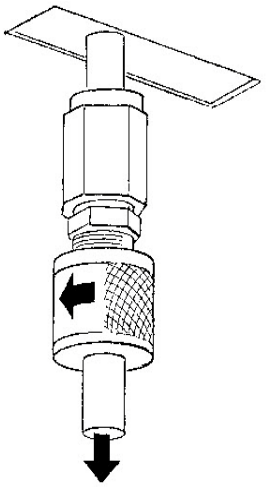
12. Subtract the **Mass of empty sample tube** from the **Mass of sample tube plus sample**; record this value **Mass of sample (Before degas)**.

Degassing the Sample

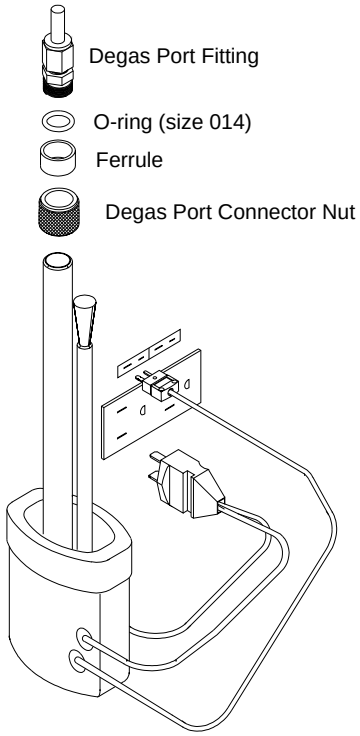
Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean before an analysis is performed. The sample is heated and placed under vacuum to remove moisture and other contaminants. This process is called degassing the sample. Degassing the sample is easy and virtually automatic if you have the SmartVac installed on your ASAP 2020 Chemi analyzer.

The ASAP 2020 Chemisorption System performs all degassing and preparation steps with the sample loaded on the analysis port. You may degas a heavily contaminated sample on the degas ports prior to loading the sample on the analysis port; the analysis port can then be more productively used for flowing or static preparation, evacuation, or analysis. The following procedure describes degassing a sample on the degas ports.

1. While holding the degas port plug, remove the connector nut and plug from the degas port by turning the connector nut counterclockwise.



2. Place the degas port connector nut, ferrule, and O-ring onto the sample tube set as shown in the following figure.



3. Remove the large rubber stopper from the sample tube and attach the sample tube set to the degas port. Be sure to push the sample tube straight in to a full stop.

Mounting the sample tube at an angle might break the tube and cause injury.

Secure the sample tube in place by sliding the connector nut, ferrule, and O-ring up to the degas port and turning the connector nut clockwise. Tighten the nut securely by hand.

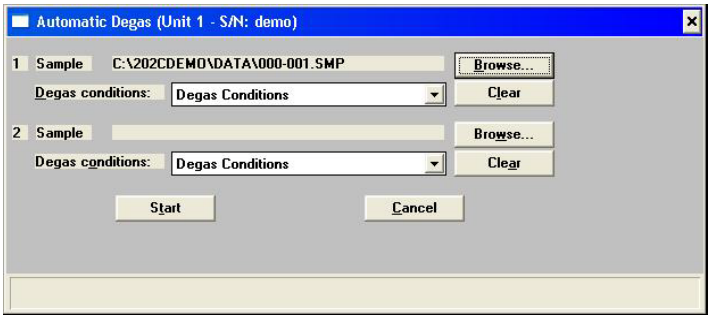
Never use a tool to tighten the nut; doing so may cause the sample tube to break.

4. Make sure the exhaust tube of the sample tube has been capped.

5. Place a heating mantle over the end of the sample tube and secure the mantle in place with a mantle clip.

6. Insert the heating mantle thermocouple plug into the appropriate connector. Then insert the heating mantle power plug into its appropriate connector. Make sure both plugs are completely inserted.

7. Select **Unit > Degas > Start Degas**; the Start Degas dialog is displayed.



8. Click **Browse** to the right of the Sample field to choose your degas file.

   Repeat this step if you are degassing two samples.

9. Click **Start** to begin the degassing operation.

Observe the status bar of the degassing operation to determine when degassing is complete.

10. After degassing has completed, transfer the sample tube to the analysis port to start the analysis (next section).

Transferring the Degassed Sample to the Analysis Port

The sample tube must be removed from the degas port, weighed, and then placed onto the analysis port for analysis.

If the sample tube is not mounted to the analysis port immediately, either leave it on the degas port or remove it and insert the rubber stopper and cap into the sample tube openings.

1. Allow the sample tube to cool to room temperature (approximately 15 minutes).

Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube, heating mantle, or heating mantle clip could result in burns.

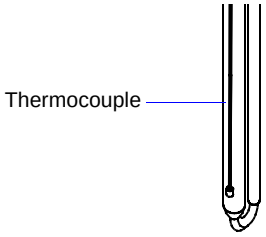
2. Holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. Insert a stopper immediately. Remove the connector nut, ferrule, and O-ring from the sample tube stem.

3. Weigh the sample tube set. Enter the value on the Sample Data Worksheet as **Mass of sample tube plus sample (After Degas)**.

4. Subtract the **Mass of empty sample tube (Before Degas)** from the **Mass of sample tube plus sample (After Degas)** to determine the mass of the sample. Record this value as the **Mass of sample (After Degas)**.

5. Remove the stopper and cap and attach the sample tube to the analysis port, pushing it fully up. Secure it in place by screwing the connector nuts onto the analysis port connectors. Tighten the connector nuts by hand.

6. Insert the sample temperature thermocouple into the holder on the sample tube.



7. Place the furnace onto the elevator.

8. Slide the insulator disk onto the sample tube. The insulator disk surrounds the sample tube when the furnace is raised, preventing heat loss and improving thermal stability.

9. Select **Unit > Show Instrument Schematic** to show the instrument schematic; if already displayed, proceed to the next step.

10. Select **Unit > Enable Manual Control**.

11. Select the furnace icon on the schematic; right-click and choose **Raise**. You may stop the elevator at any intermediate height by pressing the **Spacebar**.

    Be sure the insulating disk seats properly on the top of the furnace.

Installing the Cold Trap 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:

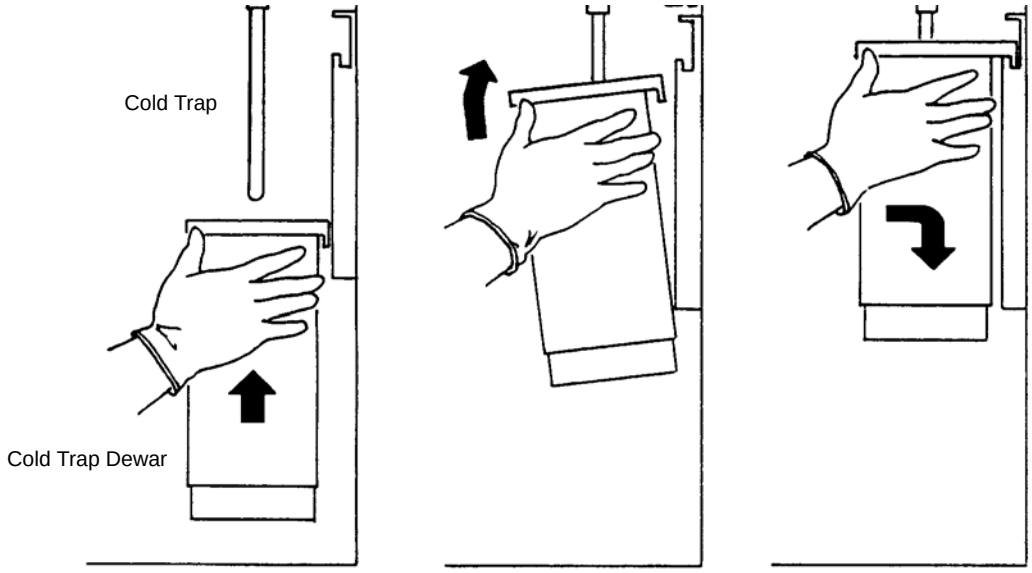
- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.
- When pouring liquefied gases from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the liquefied gas slowly to prevent splashing, 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 form of protective coating.
- Do not remove the mesh covering from the Dewar flask.
- 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.

Cold Trap Dewar

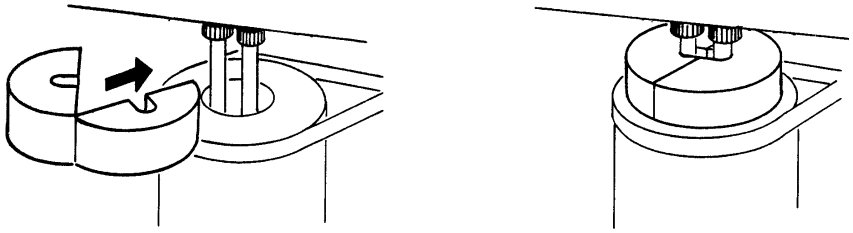
Cryogens can cause frostbite injury. Wear safety glasses and insulating gloves when handling cryogens.

1. Fill the cold trap Dewar with a cryogen such as liquid nitrogen to about 5 cm (2 in.) from the top.

2. Hang the cold trap Dewar around the cold trap port as shown below.



3. Place the insulator/stopper over the Dewar opening as shown below.



Performing an Analysis

After the sample has been transferred to an analysis port and the elevator and furnace fully raised, you may begin automatic chemisorption preparation and analysis. The ASAP 2020 Chemi allows you to perform single analyses or sequenced analyses.

You cannot perform an analysis while calibration is in progress or if all calibration operations have not been performed.

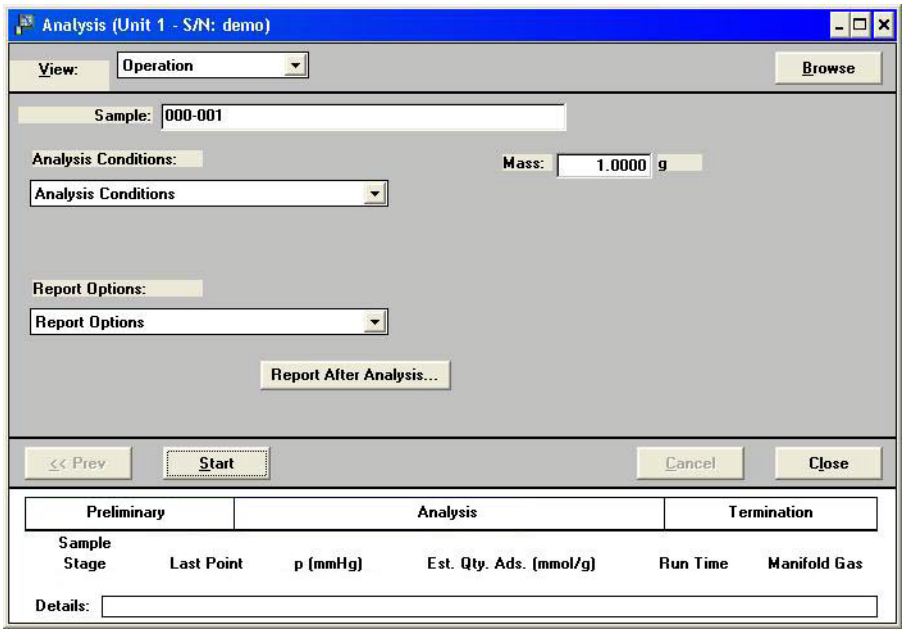
Single Analysis

Make sure the furnace is raised to its uppermost position. If it is not completely raised, hazardous conditions can result.

1. Select **Unit > Sample Analysis**; the Analysis dialog is displayed with the Start Analysis dialog positioned on top.

2. Choose the sample file to use with the current analysis and click **OK**; the Analysis dialog containing the parameters of the selected file is displayed.

You can also accept the next sequenced number and create a new file. If you choose this method, however, you can only use the predefined files from the drop-down lists.



3. Verify the sample's mass; edit the value if required.

4. Click **Start** to begin the analysis.

   A graph will appear in the window when data begin to collect. After the analysis is complete, you can click **Start** on the final analysis window to begin another analysis.

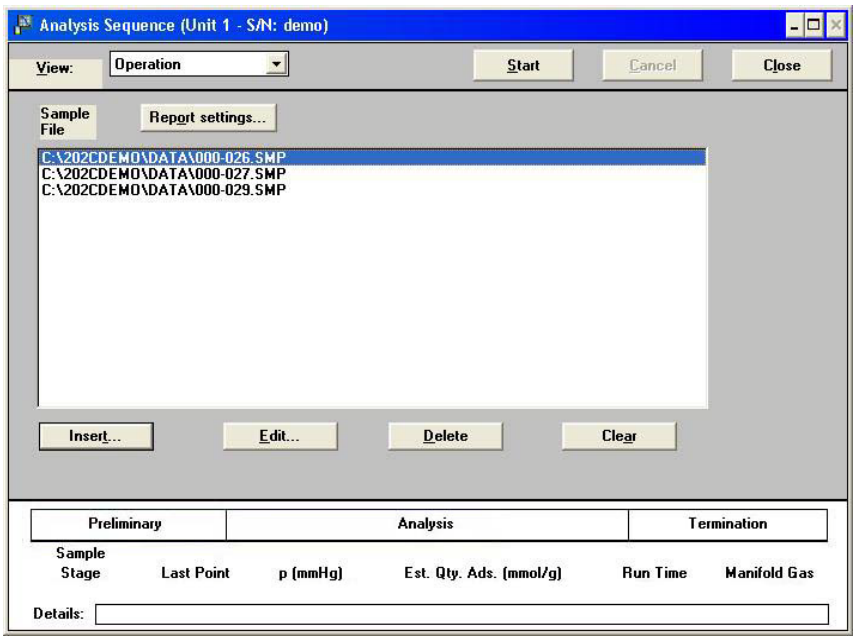
Sequenced Analyses

Performing several single analyses on the same sample can be tedious. The Analysis Sequence option on the Unit menu allows you to program the software to perform a sequence of analyses automatically.

Make sure you have created the sample information files required for the analyses before beginning this procedure. Refer to Creating Sample Information Files, page 4-5 for step-by-step instructions on creating a sample file.

Make sure the furnace is raised to its uppermost position. If it is not all the way up, hazardous conditions can result.

1. Select **Unit > Analysis Sequence** to display the Analysis Sequence dialog.



2. Click **Insert** to display the Select Sequence File dialog.

3. Choose a file from the **Files** list window, then click **OK**; the Analysis Sequence dialog appears again with the name of the sample information file displayed in the Sample File list box.

   Be sure to select files that have the **No analysis** (files that have not been used to perform an analysis) status for sequencing.

   Repeat this step for as many analyses as required.

4. To generate reports automatically after each analysis, click **Report Settings** and select **Report after analysis**, then specify the report **Destination**.

5. Click **Start** to begin the first analysis in the sequence. As soon as the analysis is started, the file name is removed from the sequence.

If a calibration is in progress, you must wait for the calibration to complete before you can start the sequence.

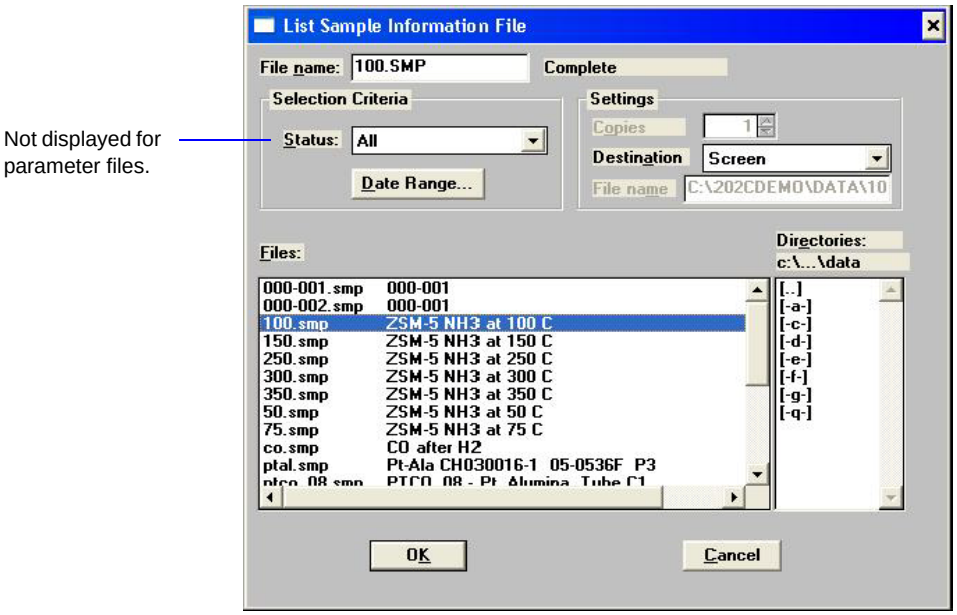
Listing File Statistics

You can generate a list of the following information on one or more files:

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status (sample information files only)

Perform the following steps to generate a list:

1. Select **File** > **List** > **(file type)**; a dialog similar to the one shown below is displayed.



2. From the **Files** list window, choose the file(s). Tto include all files in the list, leave all files *deselected*.

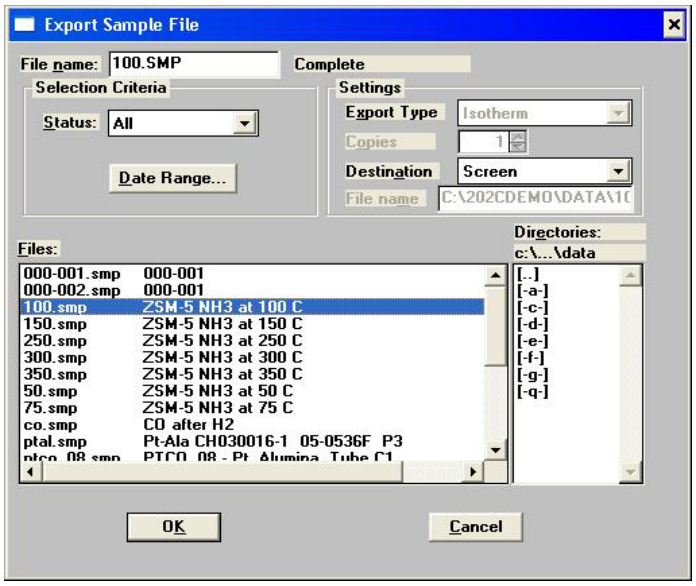
3. At the **Destination** field, click on the down arrow and choose a destination for the list output. If you choose **File** as the destination, enter a name in the **File name** field (below the **Destination** field).

4. Click **OK**, a list for the requested file(s) is sent to the specified destination.

Exporting Isotherm Data

The Export option on the File menu allows you to copy the isotherm data in the sample information file and reformat it in ASCII text. If saved to a File, the data can be imported into applications, such as spreadsheets. Refer to **Appendix D**, **Format of Exported Data**, for record descriptions.

1. Select **File > Export**; the Export Sample File dialog is displayed.



2. From the **Files** list window, select the file(s) to export. To choose multiple files, hold down **Ctrl** while making your selections.

3. Choose an output destination. If you choose **File** as the destination, the **File name** field (below the **Destination** field) is enabled so that you may enter a name (or you can accept the default). If you have selected multiple files, each file is exported as its file name with the extension .**EXP**.

4. Click **OK**; the file(s) is (are) exported to the specified destination.

Generating Overlays

Use graph overlays to compare graphically the results for multiple samples. You may overlay the results of up to eight samples on top of a previously selected sample.

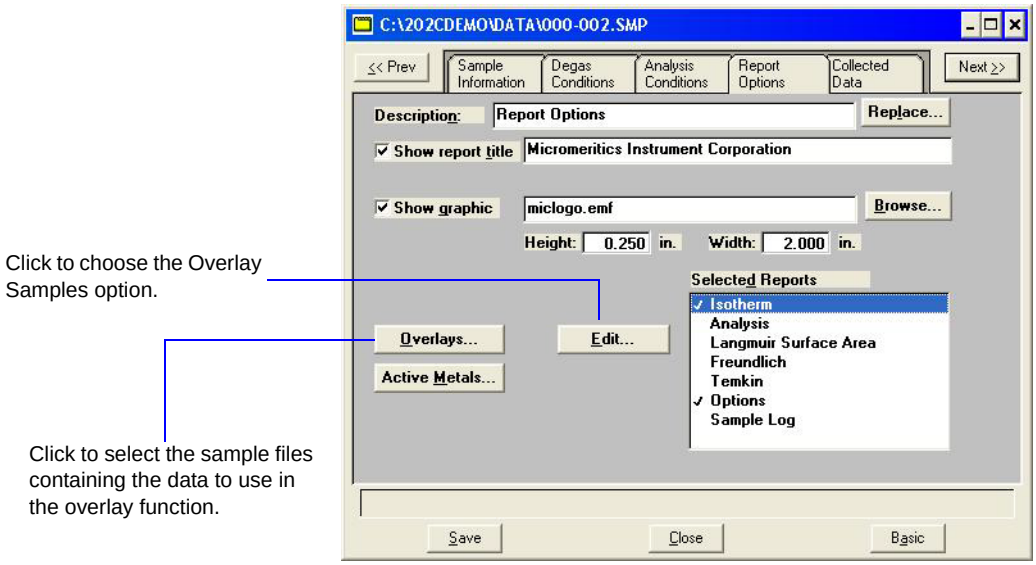
You cannot use the overlay function when using the Basic format; you must switch to the Advanced format to perform this operation.

Perform the following steps to overlay graphs:

1. Select **File > Open > Sample Information**; the Open Sample Information File dialog is displayed.

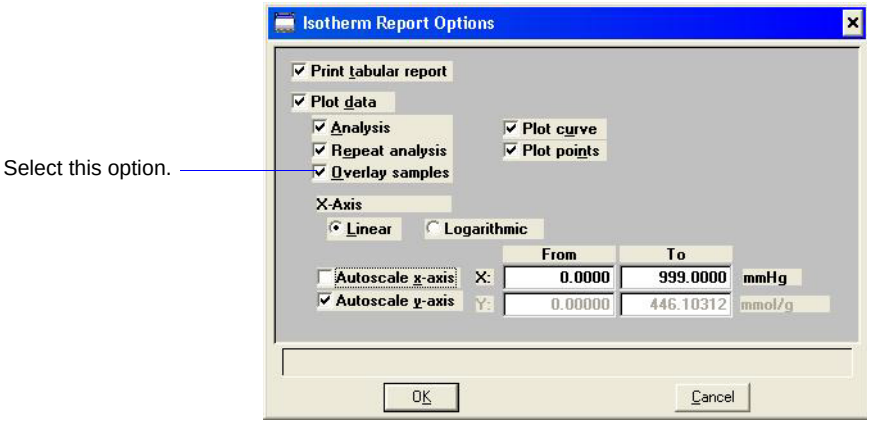
2. Select a sample on which you to overlay graphs of other samples, then click **OK**; the Sample Information dialog is displayed.

3. Select the Report Options tab to display the Report Options dialog.



4. Make your selections.

5. Select the graph to overlay, then click **Edit**; a report options dialog for that graph is displayed.



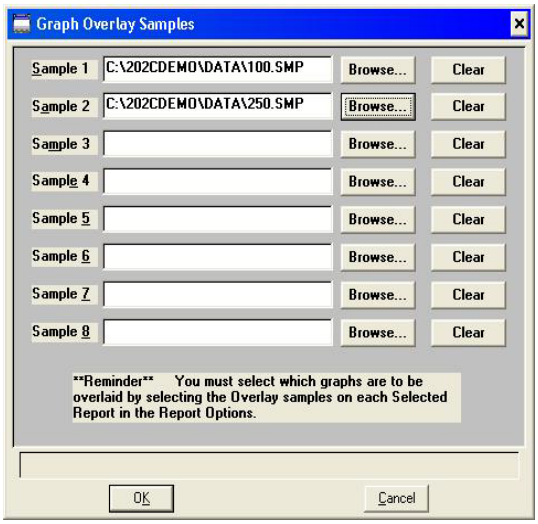
6. Select **Overlay Samples**; do any other editing.

7. Click **OK** to return to the Report Options dialog.

8. Click **Overlays**; the Graph Overlay Samples dialog is displayed.

9. Click **Browse** to the right of the **Sample [n]** field; the Plot Overlay Sample Selection dialog is displayed.

10. Select a sample, then click **OK**. You can choose up to eight samples in this manner.



11. After choosing the number of samples, click **OK** to return to the Report Options dialog.

12. Select **Save** to save the changes you have made. Even if you don't save, all of the options regarding overlays and anything else are available as your reports are generated.

13. Select **Reports > Start Report** to display the Start Report screen; the name of your primary file is shown in the **File name** field.

14. Select a destination for report output.

15. Click **OK** the reports containing the overlays you requested are printed to the requested destination.

5. FILE MENU

This chapter describes the file maintenance options used to create, edit, print, list, and export sample and parameter files.

Description

Listed below are brief descriptions of the commands on the File menu. Detailed descriptions follow this section.

Open — Allows you to create a new sample or parameter file, or open an existing one.

Save — Saves the file in the active window. P

Save As — Allows you to save the file in the active window under a new name. It can also be used to save a subset of a sample file as a parameter set.

Close — Closes the file in the active window.

Close All — Closes all open files.

Print — Prints the contents of one or more files.

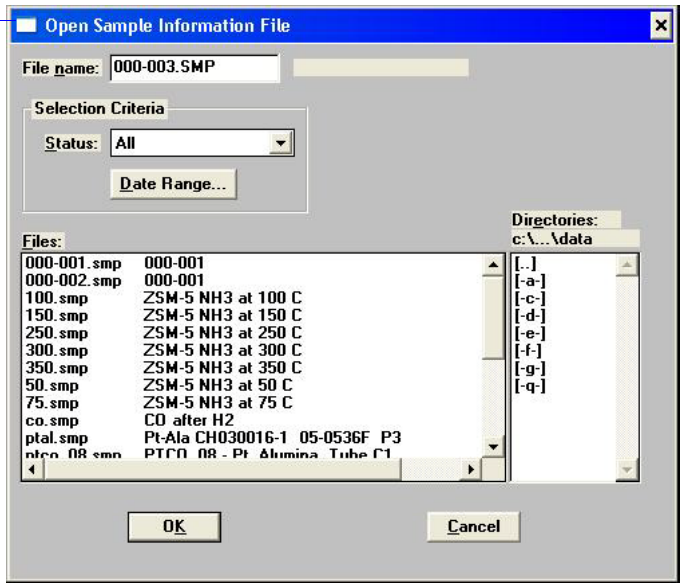
List	Generates a list of certain information for specified sample or parameter files.
Export	Exports isotherm data from a sample information file in ASCII format..
Exit	Exits the ASAP 2020 Chemi program.

Open

Open enables you to create a new sample or parameter file, or to edit an existing one

Select the file type; a dialog similar to the following is displayed:

Title bar displays the type of file you are opening or creating; in this example, a sample information file.



File name

For **sample information** files, this field contains the next sequenced file name (as specified in sample defaults) generated by the system. If this is a new file, you can use the name displayed or enter a different one.

For **parameter files**, the name displayed includes the wild card (*) and the default extension as follows:

*.DEG for degas conditions
*.ANC for analysis conditions
*.RPO for report options

If you are creating a new file, enter a name in the **File name** field.

If you are opening an existing file, select the name from the Files window, then click **OK**.

Date Range

Click this button to display files created within a certain range of dates.

Status

Drop-down list of the types of sample files you can display in the **Files** window. All files of the type you choose, within the range of dates, and within the current directory are displayed. This option does not display for parameter files.

Refer to , page **3-9** for a description of the file types.

Directories

Displays the current directory (folder). You can navigate to a different directory by clicking in the **Directories** list window or by entering the directory in the **File name** field.

The more files in a directory, the longer it may take to access a file. Therefore, it is a good practice to limit the number of files in a directory to approximately 200.

Sample Information

Sample information files contain data that guide the analysis. They consist of:

- sample information
- degas conditions
- analysis conditions
- report options
- collected (after analysis) or manually entered data

Parts of the sample information file can also exist as standalone parameter files. Having these files exist independently allows you to use them as many times as needed. For example, if you typically use the same analysis conditions for many of your analyses, you can create an analysis conditions file containing the conditions. Then when you create your sample file, select that file for your analysis conditions; the values will be copied into the sample file. After it becomes part of the new sample file, you can edit it in any way needed. The file from which the values were copied remains intact and ready for the next use.

Sample information files reside in a folder, more commonly referred to as a directory. The more files in a directory, the longer it may take to access a file. Therefore, it is a good practice to limit the number of files in a directory to approximately 200. You may create additional directories as needed. Refer to the operating system manual for more information.

Sample information files can be created and presented in the Advanced, Basic, or Restricted format.

- **Advanced**
  Presents all parts of the sample information file in a tabbed dialog. Each tab opens its associated dialog, allowing you to edit conditions.

- **Basic**
  Presents all parts of the sample information file as a single dialog. This format allows you to create quickly a sample information file using predefined parameter files. You can also switch to the Advanced format.

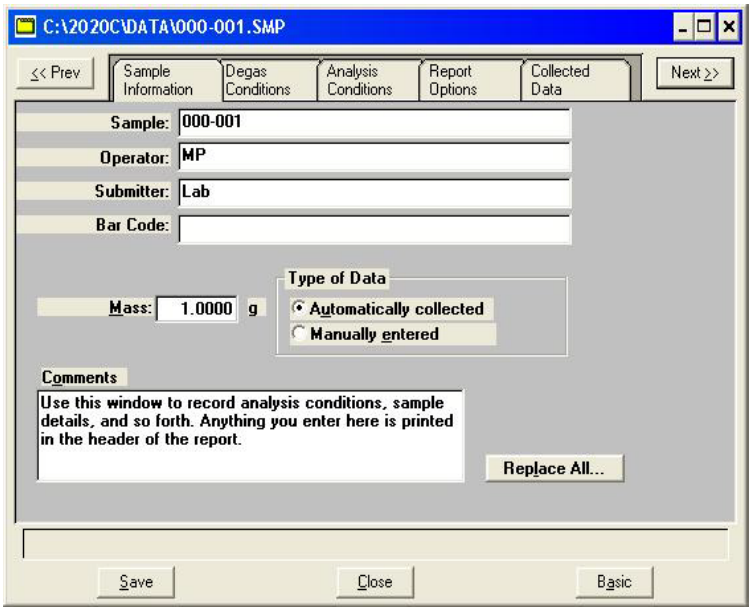
- **Restricted**
  Identical to the Basic format except that you cannot switch to the Advanced format for editing. Certain functions also are disabled.

Advanced Format

The Advanced format of a sample information file displays all parts of the sample file in a tabbed dialog. Quick-access tabs enable you to move quickly and easily among the parameters; or you can use **Prev** and **Next**.

Your selection of the type of data (**Automatically collected** or **Manually entered**) on the Sample Information dialog determines whether the Collected Data or Entered Data tab appears. In the following example, the tab displays Collected Data because **Automatically collected** is selected as the type of data.

Refer to **Advanced Format**, page **4-6** for step-by-step instructions for creating an Advanced sample information file.



The prompts for the **Sample**, **Operator**, **Submitter**, and **Bar Code** fields may be customized by selecting **Options > Sample Defaults** (refer to **Sample Defaults**, page **8-6**).

Sample

Contains the description of the current sample file.

If this is a new file, this field contains the next sequenced file description based on the format specified in **Sample Defaults**.

You can enter a new description or edit the existing one.

Operator
Submitter

Displays the operator and submitter names of the current sample file.

If this is a new file, these fields contain the names specified in **Sample Defaults**.

You can enter a different name or edit the current one.

Bar Code

This field enables you to enter bar code information. If bar code information is not used, you can use this field to enter additional information about the sample; for example, to enter the lot number of your sample.

This field, as well as the **Operator** and **Submitter** fields, will accept data from a bar code reader.

Mass

Enter the sample's mass. If you are using the Sample Data Worksheet, enter the value recorded as **Mass of Sample After Degas**.

The value in this field is used in report calculations; therefore, it should be exact. To ensure accuracy, reweigh the sample after analysis. If the value is not equal (or very close) to the one entered for **Mass of Sample After Degas**, you may want to change the value to **Mass of Sample After Analysis**.

If you use a backfill gas that is different from the analysis gas, the sample's mass after degas will not be equal to its mass after analysis. For example; if you use nitrogen to backfill after degassing and helium after the analysis, the sample's mass can vary as much as 0.02 gram.

Type of Data

Displays the type of data for the current sample file.If this is a new file, choose **Automatically collected** to have data collected automatically, or **Manually entered** if you plan to enter data collected from another source.

Comments

Allows you to enter pertinent information about the sample or analysis. The information you enter in this window is displayed in the report header.

Replace All — Use this button to replace the values of all parameters of the current file with those from an existing one. You can edit the values after they have been copied into the current file; editing the current files will not affect the file from which they were copied.

Save — Saves all parameters of the current file; the dialog remains open.

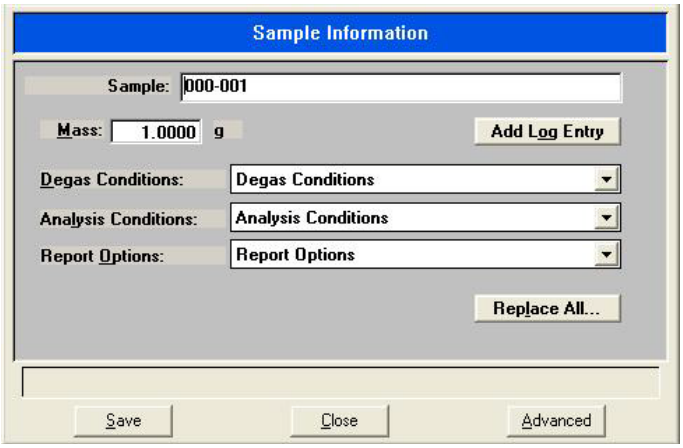
Close — Closes the dialog. If the dialog contains changes that have not been saved, you will be prompted to save them before the dialog closes.

Basic — Switches the sample editor to the Basic format.

Basic Format

The Basic format displays all parts (parameters) of the file on a single dialog. With this format, you can quickly create a sample information file using predefined parameter files. You can also switch to the Advanced format if a specific parameter requires editing.

Refer to **Basic and Restricted Formats**, page **4-5** for step-by-step instructions for creating a sample file in the Basic or Restricted format.



If you are creating a new file, this dialog displays the defaults you specified in Sample Defaults. Chapter 8 explains how to establish sample defaults.

Sample

Contains the description of the current sample file.

If this is a new file, this field contains the next sequenced file description based on the format specified in Sample Defaults. The above dialog shows the sample file number because the dollar ($) symbol was included when sample defaults were specified.

You can enter a new description or edit the existing one.

Mass

Allows you to enter the sample's mass. If you are using the Sample Data worksheet, this is the value recorded for **Mass of Sample After Degas**.

Degas Conditions
Analysis Conditions
Report Options

Each parameter field contains the description of the current parameter file.

If this is a new sample file, these fields contain the descriptions of the files chosen as the defaults.

Click on the down arrow to the right of each field to choose a different file. File parameters can be viewed or edited by switching to the Advanced format.

Replace all

Use this button to replace all parameters of the current file with those copied from an existing one. A dialog is displayed allowing you to choose the file.

Save

Saves all parameters of the current file; the dialog remains open.

Close

Closes the dialog.

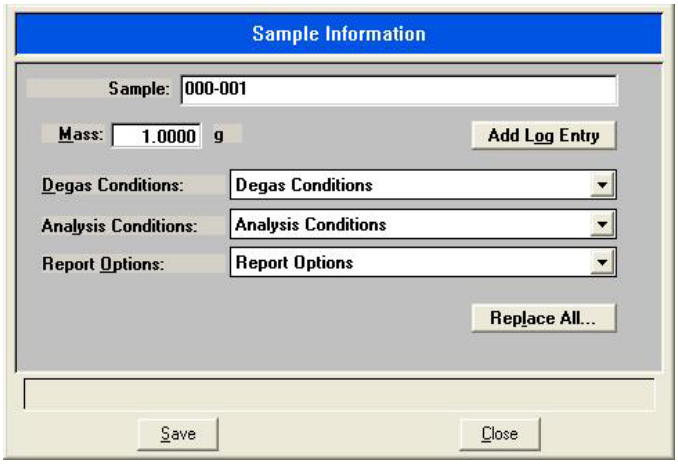
Advanced

Switches the sample editor to the Advanced format, allowing you to view or edit parameters of the current file.

Restricted Format

The Restricted format is used when analysis parameters must remain constant. For example, in the pharmaceutical industry where consistency and accuracy are crucial. A password is required to enter and exit this format. Refer to **Restricted**, page **8-5** for additional information on the Restricted format.

When you open an existing sample information file or create a new one using the Restricted format, all parts of the sample file are displayed in the same manner as the Basic format. Some menu functions, however, are disabled and you cannot switch to the Advanced format to edit file parameters.

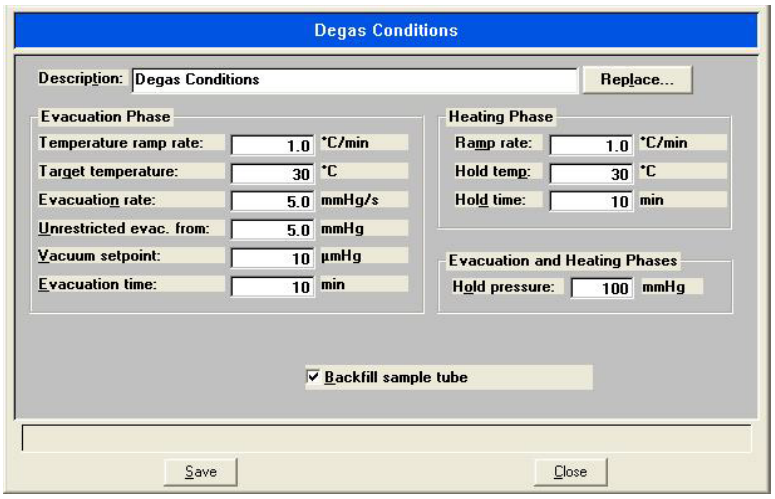


This dialog is identical to the Basic dialog except that it does not allow you to switch to the Advanced format.

Degas Conditions

This dialog is used for specifying degas conditions and is applicable only if your ASAP 2020 Chemi is equipped with the SmartVac degas option.

Refer to **Degas Conditions**, page **4-8** for step-by-step instructions on creating a degas conditions file.



Description — Contains the description of the current Degas Conditions file.

If this is a new file, this field contains the name (description) you specified as the default. You can enter a new description or add to the existing one.

Replace — Use this button to replace the current degas conditions with those from another file. The Open Degas Conditions File dialog is displayed, allowing you to choose the file.

Evacuation Phase group — The options in this group box allow you to specify conditions for the evacuation phase of the degassing operation.

Temperature ramp rate — Allows you to specify the rate at which the temperature is to change while advancing to the target temperature during evacuation.

Target temperature — Allows you to specify a temperature at which the sample will be held for the remainder of the evacuation.

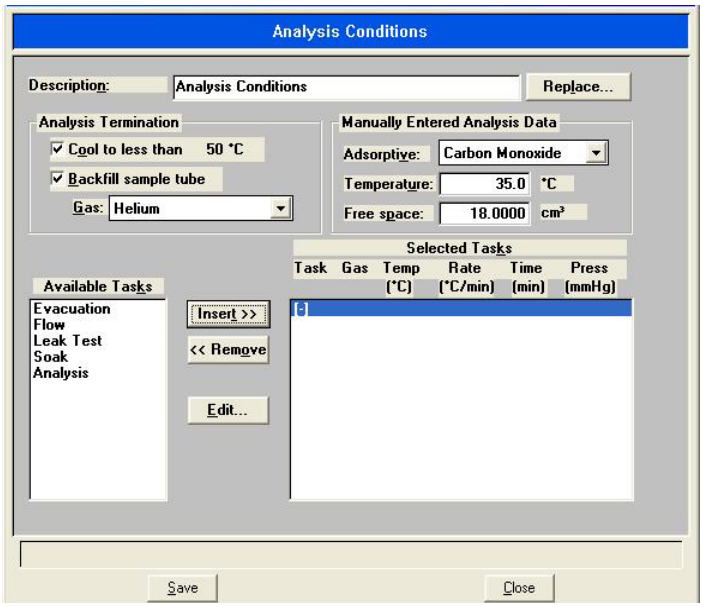
Evacuation rate	Allows you to specify an evacuation rate for initial evacuation.
Unrestricted evac. from	Allows you to specify a pressure at which unrestricted sample evacuation will begin.
Vacuum setpoint	Allows you to specify the vacuum level to be achieved before evacuation begins.
Evacuation time	Allows you to specify how long the sample is to be evacuated prior to the second state of heating.
Heating Phase group	The options in this group box allow you to specify conditions for the heating phase of the degassing operation.
Ramp rate	Allows you to specify the rate at which the temperature will change while advancing to the hold temperature.
Hold temp	Allows you to specify a temperature at which the sample is to be held during degassing.
Hold time	Allows you to specify how long the sample is to be held at the specified temperature before beginning to cool down.
Hold pressure	If during the temperature ramp the pressure exceeds the value you enter in this field, the ramp will be suspended until the pressure returns to a safe level.
Backfill sample tube	Select this option to have the sample tube backfilled. It is recommended that you backfill after degas.

Analysis Conditions

Analysis conditions specify the data, or tasks, used to guide the analysis. This section describes the analysis conditions options available on the ASAP 2020 Chemi system. An analysis conditions file may exist as an independent file or as part of the sample information file.

The types of analysis conditions files included as samples with the ASAP 2020 Chemi System are described in **Appendix E, Default Files and System Files**, page **E-1**.

Refer to **Analysis Conditions**, page **4-9** for step-by-step instructions on creating an analysis conditions file.



Description

Contains the description of the current Analysis conditions file.

If this is a new file, this field contains the identification specified in Sample Defaults. You can edit this name.

Replace

Use this button to replace the values of the current file with those of another; a dialog allowing you to choose the file is displayed. After the values are copied into the current file, you can edit them in any way.

Analysis Termination group box

The options in this group box are enabled when you choose **Automatically collected** as the type of data on the Sample Information dialog. These options enable you to specify analysis conditions for termination of the analysis.

Select **Cool to less than 50 ºC** (323 K) to cool the sample to a temperature less than 50 ºC before the analysis terminates.

Select **Backfill sample tube** to choose a backfill gas.

If you use a a backfill gas that is different from the analysis gas, the sample's mass after degas will not be equal to its mass after analysis. For example, if you use nitrogen to backfill after degassing and helium after the analysis, the sample's mass can vary as much as a 0.02 gram.

Available Tasks

This window lists the tasks available. These tasks are disabled when you choose **Manually entered** as the type of data on the Sample Information dialog.

An experiment is defined by the tasks from which it is comprised. Tasks are executed in the order in which they are sequenced. You do not have to include all the tasks in an experiment.

Choose a task by double-clicking on the task name or highlighting the task name and clicking **Insert**, the task dialog is displayed. After you define the task and click **OK**, the task name and task options appear in the **Selected Tasks** window.

The following tasks are available:

- Evacuation
- Flow
- Leak test
- Soak
- Analysis

A description of the dialog for each task is found in subsequent sections.

Selected Tasks

This window lists the tasks selected (from the **Available Tasks** window) for the system to perform.

You cannot enter data if the ASAP 2020 Chemi program is being used for offline data manipulation on a computer other than the one controlling the analyzer.

Manually Entered Analysis Data

The options in this group box are enabled when you choose **Manually entered** as the type of data on the Sample Information dialog. These options enable you to enter analysis conditions.

Adsorptive

Choose the adsorptive used in the experiment which produced the data. Gases available in this list are those specified in the Gas Table Defaults dialog (refer to **Gas Defaults**, page **8-14** for more information).

Temperature

Enter the temperature at which the analysis was performed.

Free space

Enter the free-space volume determined in the analysis.

Insert

Allows you to insert a selected Task from the Available Tasks list; displays a pertinent dialog allowing you to enter values.

Remove

Removes the selected task.

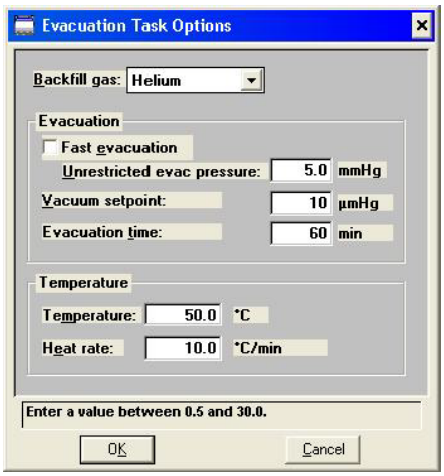
Edit

Displays a dialog pertinent to the selected task, allowing you to view or edit the values.

Evacuation Task Options

To ensure safe operation and reliable results, you should include an evacuation task under the following conditions:

- Between tasks using different gases
- Preceding a leak test
- Preceding an analysis



All fields on this dialog are disabled for sample files that have a Complete status.

Backfill gas

Allows you to choose a gas for backfilling the sample tube prior to evacuation. Make sure the gas selected is either the one used in the previous task or helium. Refer to **Gas Defaults**, page **8-14** for information on changing gases.

If you do not want to backfill the sample tube prior to evacuation, select **[-]** (none).

Evacuation

The options in this group box allow you to specify conditions during evacuation. All options are disabled during analysis or if the analysis is complete.

Fast evacuation

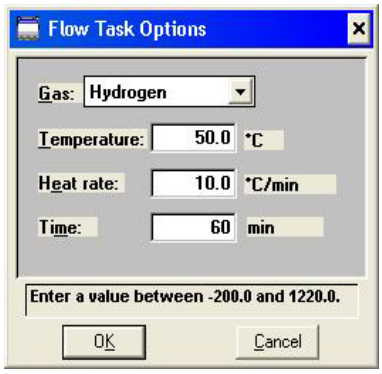
Select a fast evacuation for samples that do not fluidize or shed particles under evacuation (such as pellets).

Unrestricted evac pressure

Enabled if you do not choose **Fast evacuation**. This value represents the pressure at which unrestricted sample evacuation begins.

Vacuum setpoint	Enables you to specify the vacuum level to be achieved before evacuation begins.
Evacuation time	Enter the number of minutes for preliminary evacuation. Preliminary evacuation takes place prior to the free-space measurement.
Temperature	The options in this group box allow you to choose temperature options during evacuation. These options are disabled during analysis or if the analysis is complete. Enter the temperature during evacuation in the **Temperature** field. Enter the rate at which the temperature to change (ramp rate) in the **Heat rate** field.

Flow Task Options



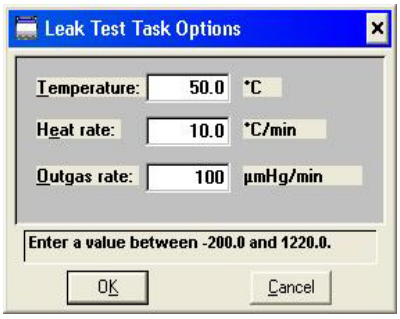
Gas — Enables you to choose a gas for the Flow task. The gases in this drop-down list are those specified in the Gas Table Defaults dialog. Refer to **Gas Defaults**, page **8-14** for information on changing or adding gases.

Temperature — Enter the temperature at which the gas is to start flowing.

Heat rate — Enter the rate at which the temperature is to change (ramp rate) while advancing to the target temperature.

Time — Enter the duration of time the sample spends at the specified temperature.

Leak Test Task Options



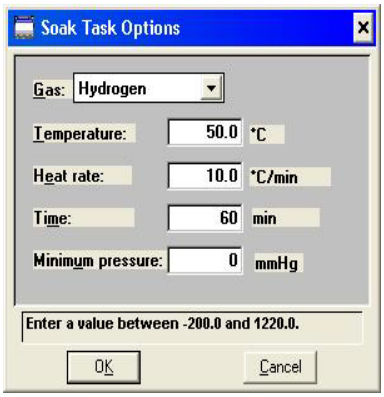
Temperature Enter the target temperature during the leak test.

Heat rate Enter the rate at which the temperature is to change (ramp rate) while advancing to the target temperature.

Outgas rate Entry in this field allows a system check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than the value in this field, the analysis proceeds but you are notified with an error message.

The results of the leak test are printed in the Options Report. Leak testing slightly increases analysis time, but it allows the program to notify you of any analyses which may contain erroneous results.

Soak Task Options



Gas — Enables you to choose a gas for the Soak task. Gases available are those specified in the Gas Table Defaults dialog. Refer to **Gas Defaults**, page **8-14** for information on changing or adding gases.

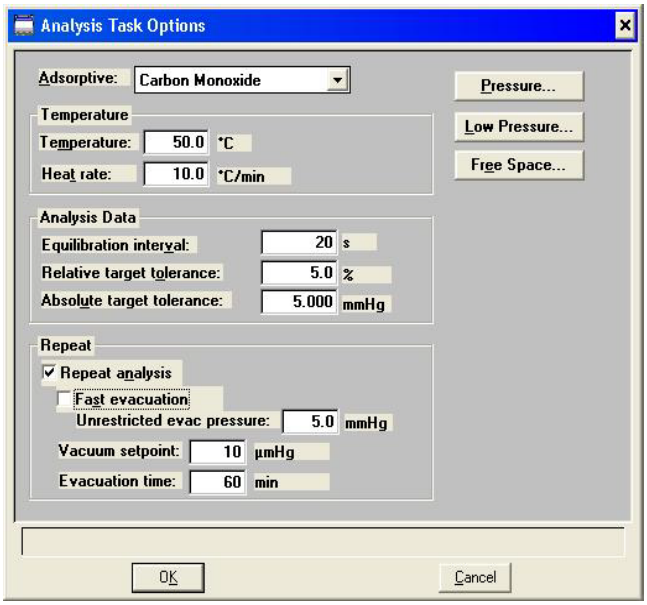
Temperature — Enter the temperature at which soaking is to begin.

Heat rate — Enter the rate at which the temperature is to change while advancing to the target temperature.

Time — Enter the duration of time the sample is to soak at the specified temperature.

Minimum pressure — Enter the minimum pressure to which the sample is to be exposed during soaking.

Analysis Task Options



All fields on this dialog, as well as on the subdialogs accessed using the buttons, are disabled for sample files that have a **Complete** status.

Adsorptive

This drop-down list allows you to select an adsorptive gas for the analysis. The gases listed are those specified in the Gas Table Defaults dialog. Refer to **Gas Defaults**, page **8-14** for information on specifying adsorptive gases.

Temperature

This group box allows you to select temperature options for the analysis. These options are disabled during analysis.

Type the analysis temperature in the **Temperature** field.

Type the rate at which the temperature changes in the **Heat rate** field.

Analysis Data

This group box enables you to specify conditions for analysis.

Equilibration interval

This is the number of seconds between successive pressure readings during equilibration. Long equilibration intervals tend to lengthen analyses but improve data integrity. Short equilibration intervals produce faster analyses but may reduce the accuracy of data.

Relative/Absolute target tolerance

The values in these fields are used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures the relative tolerance value is smaller, and at higher pressures the absolute tolerance value is smaller.

For example, with relative tolerance = 5% and absolute tolerance = 5 mmHg, the relative tolerance at 40 mmHg target pressure is 5% of 40 mmHg, or 2 mmHg; 2 mmHg is smaller than the absolute tolerance of 5 mmHg, so 2 mmHg is used. At 200 mmHg target pressure, the relative tolerance is 5% of 200 mmHg, or 10 mmHg; in this case, the absolute tolerance of 5 mmHg is smaller and is used.

In the above example, a minimum pressure of 40 - 2 = 38 mmHg must be attained to collect data for a target pressure of 40 mmHg. For a target of 200 mmHg, 200 - 5 = 195 mmHg must be attained.

Unnecessarily tight tolerances lengthen the analysis.

Repeat

The options in this group box allow you to specify conditions for a repeat analysis.

Repeat analysis

Select this option to repeat the analysis. The initial analysis measures chemisorption and physisorption activity. The repeat analysis measures only physisorption activity. The difference between the initial and repeat analysis is the chemisorption activity of the sample.

Fast evacuation

Select this option for samples that do not fluidize or shed particles under evacuation (such as pellets). The check box is disabled during analysis.

Unrestricted evac pressure

Enabled when **Fast evacuation** is deselected. This reading is the pressure at which unrestricted sample evacuation is to begin.

Vacuum setpoint

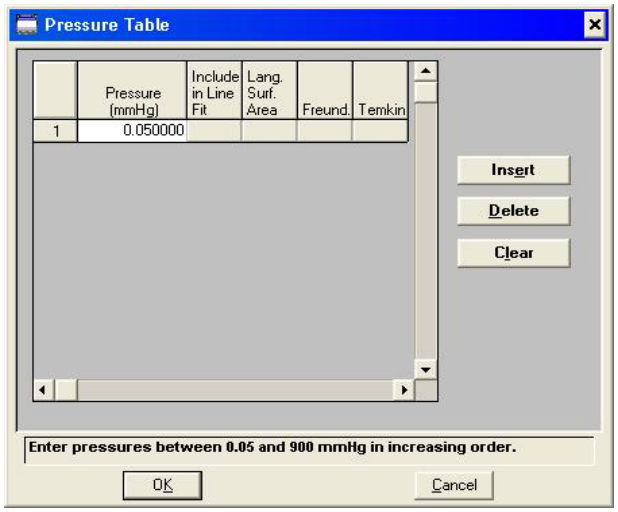
Enables you to specify the vacuum level to be achieved before evacuation begins.

Evacuation time

Enter the number of minutes for preliminary evacuation. Preliminary evacuation takes place at the start of a repeat analysis and at the start of the free-space measurement.

Pressure

Displays the Pressure Table dialog so that you may create or edit a pressure table.



A pressure table is a table of pressure points at which data are to be collected and included in a line fit plot. The pressures may span the entire range from the lowest absolute value of 0.0050 to the maximum value of 900.0000 mmHg.

Several analysis conditions files are included with the ASAP 2020 Chemisorption software and can be found in the params subdirectory. Refer to **Appendix G**, **Chemisorption Methods**, for information on the contents of the default analysis conditions files. These files (and all other parameter files) can be edited. If you want to use any of the files in your analyses, it is recommended that you save any changes to the file under a different name.

The pressure table for automatically collected data includes the pressure points for data collection and identifies which points are used in the line fit plot.

To enter a pressure point in the Pressure (mmHg) field: press **Ctrl** ↓ if you are at the top of the table, **Ctrl** ↑ if you are at the bottom of the table, or click **Insert**. The program inserts the next number based on the existing pressure point sequence. You may edit the number.

Pressure

(continued)

The pressure table must contain a sequence of strictly ascending absolute pressures. You can enter a maximum of five hundred pressure points.

To specify whether a given pressure is included in the line fit plot and difference calculations, move the pointer to the pressure point and click the mouse pointer on it or press the spacebar. An **X** in the field indicates the calculation is selected.

Insert

Inserts a row above the selected one; the cursor moves to the new line.

Delete

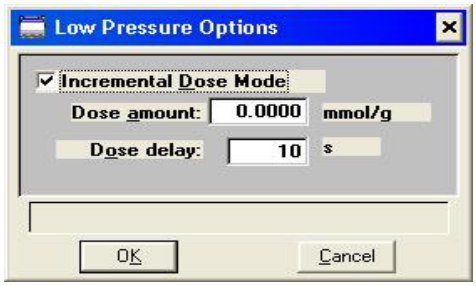
Deletes the selected row.

Clear

Removes all but one entry from the table; one entry is required. A warning message appears requesting confirmation before the table is cleared.

Low Pressure

Displays the Low Pressure Options dialog, enabling you to enter or edit low-pressure options.



Incremental Dose Mode

Select this option to dose the sample successively with a specified amount of gas until the first pressure point is reached. Because the data points recorded during Incremental Dose Mode may define most of the analysis, one point on the pressure table can be sufficient and serve as the end point for the analysis. This mode measures equilibrium points at approximately equal intervals on the volume adsorbed axis. Each dose is fully equilibrated and recorded as a data point.

Dose amount

The amount of gas added to the sample for each data point until the first point on the pressure table is reached.

Dose delay The amount of time the adsorptive remains in the manifold before being dosed onto the sample. This delay enables the gas to reach thermal equilibrium with the manifold before being dosed onto the sample.

Free Space Displays the Free Space Options dialog so that you can specify free-space measurement conditions.

Measure Select this option to have free space measured automatically by the system during analysis. The radio button is disabled during analysis. It is also disabled when analysis is complete if free space was not measured during analysis. Type the sample tube gas capacity in the Estimated free space field.

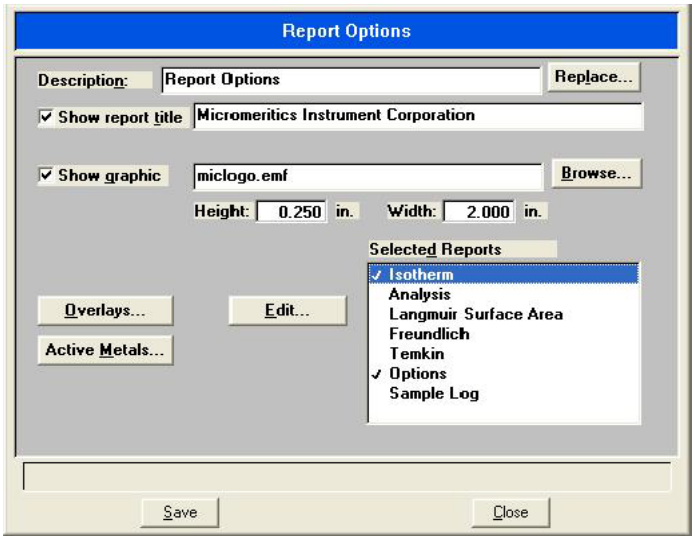
Enter Select this option to enter free space manually. The option may be necessary if the sample retains helium, and measurement of pressures below 0.1 mmHg is required. (The radio button is disabled during analysis.) Type the sample tube gas capacity in the **Free space** field.

If you change the entered free-space value after the preliminary analysis stage is 50% complete, the change does not affect the analysis.

Report Options

Report options specify the types of reports generated from an analysis or manually entered data. They also help you customize details of reports such as axis scale, axis range, and column headings. A report options file may be created as an independent file, or as part of a sample information file.

Refer to **Report Options**, page **4-11** for step-by-step instructions on creating a report options file.



Description

Contains the description of the current Report Options file.

If this is a new file, this field contains the description you specified as the default. You can enter a new description or add to the existing one .

Show report title

Select this option to have a title display on your report; the adjacent field is enabled allowing you to enter the title.

If this is a new file, the title you specified as the default is displayed. Accept the default title or enter a different one.

If you deselect this option, a title will not display on your report(s).

Show graphic

Select this option to have an illustration display above the report title. For example, to display your company logo. You can use a bitmap (bmp) or an enhanced metafile (emf).

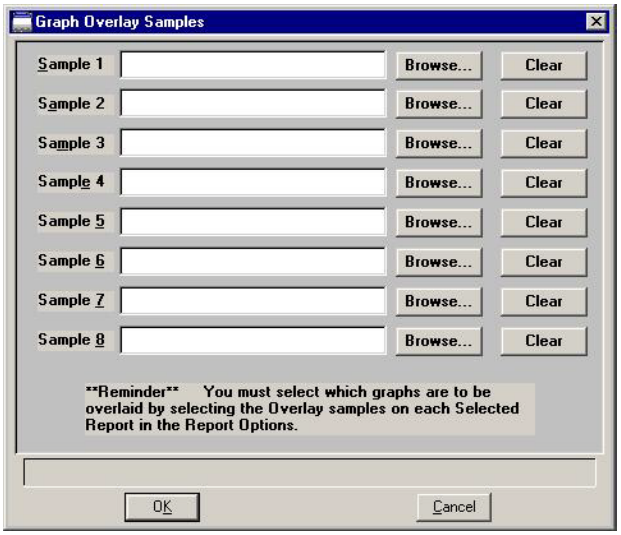
Click **Browse** to choose the graphic, then enter the height and width in the appropriate fields. This image can also be edited from the report window.

Replace

Allows you to replace the values in the current report options file with those from an existing file. A dialog is displayed so that you may select the file. Click **OK** and the values are copied into the current file automatically. You can edit the values in the new file; it will not change anything in the file from which they were copied.

Overlays

Displays the Graph Overlay Samples dialog so that you can choose the sample files to use in the overlay function.



Click **Browse** to the right of each field to choose the file; you may choose up to eight files.

Click **Clear** to clear the field of its entry.

Reports Selected

Lists the available reports. Choose a report by double-clicking on the report name or highlight the report name and press the **Spacebar**. A report is selected when it is preceded with a check (✓).

The following reports are available:

- Isotherm
- Analysis
- Langmuir surface area
- Freundlich
- Temkin
- Options
- Sample Log

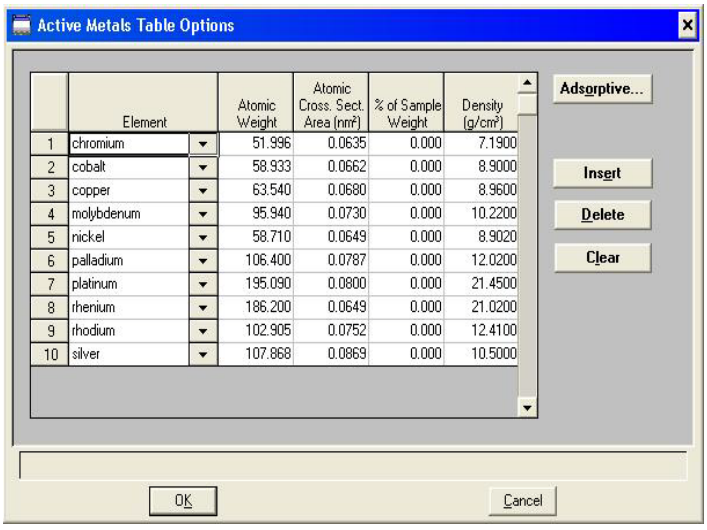
Edit

Displays an associated dialog for the selected report. Editing options for available reports are shown in the following sections.

This button is disabled for the Options and Sample Log reports.

Active Metals

Displays the Active Metals Table Options dialog allowing you to specify the characteristics of up to fifteen elements used in your analysis.



Element

The active metals appearing on the pull-down list are those specified in the Active Metals Table Defaults dialog. You can customize the Active Metals Table Options dialog by adding active metals listed in **Appendix F**, page **F-1**

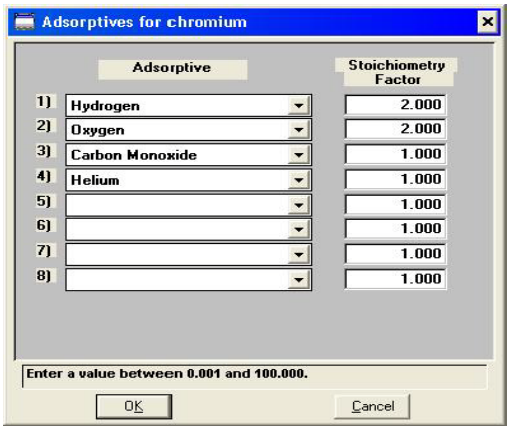
Atomic Weight The atomic weight of the element.

Atomic Cross. Sect. Area (nm2) The atomic cross-sectional area of the element.

% of Sample Weight The percentage of the sample weight occupied by the element.

Density Lists the element's density.

Adsorptive Use this button to specify the stoichiometry factors for each adsorptive associated with a selected element. The adsorptives and stoichiometry factors for the selected element appear:



Adsorptive
Gases available are those specified in the Gas Table Defaults dialog. Refer to **Gas Defaults**, page **8-14** for additional information.

Stoichiometry Factor
A factor which expresses the ratio between the number of active metal molecules and the number of adsorbate molecules.

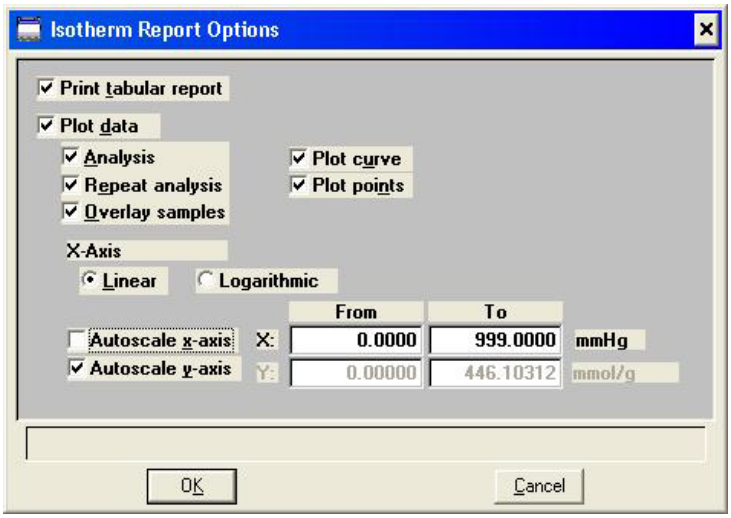
Insert Inserts a row above the current line.

Delete Deletes the current row.

Clear Removes all but one entry from the table; one entry is required. A warning message appears requesting confirmation before the table is cleared.

Isotherm Report Options

The isotherm report indicates adsorption of a gas by a solid held at constant temperature.



Print tabular report — Select this option to have collected data printed in a tabular format.

Plot data — Select this option to have data plotted as a graph.

- **Analysis** — Includes a line fit plot on the report for the primary analysis.
- **Repeat analysis** — Includes a line fit plot on the report for a secondary analysis.
- **Overlay samples** — Select this option to overlay the current plot with those of other samples. Click **Overlays** on the Report Options dialog to choose the sample files.
- **Plot curve** / **Plot points** — Use these options to specify in which manner to have the data plotted. You can plot data as a curve, points, or both.

X-Axis Scale — Enables you to have the x-axis displayed on a logarithmic or linear scale.

Autoscale Options

Select Autoscale to have the X- and/or Y-axes automatically scaled. Linear X-axes begin at zero, and logarithmic X-axes begin at an appropriate value. Y-axes begin at zero. The system uses the highest values collected during analysis as the ending points for axes ranges.

From/To fields

Enabled when you choose not (deselect) to **AutoScale**, allowing you to specify beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot.

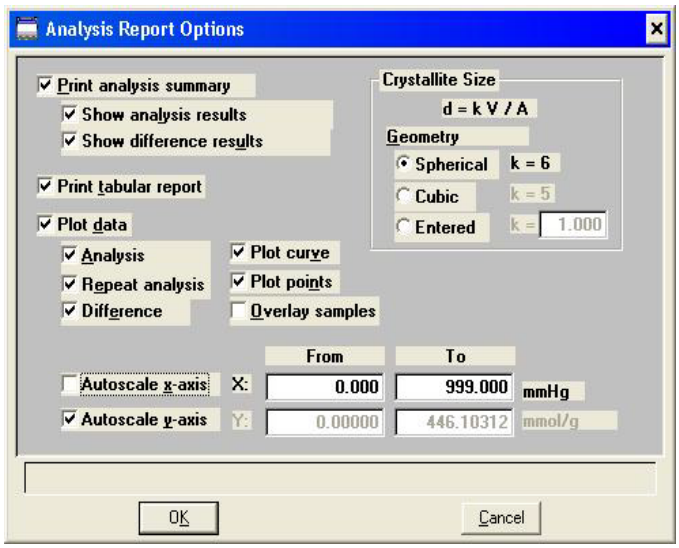
The value entered in the **To** field must be greater than the value entered in the **From** field.

The X-Axis Range fields show the pressure.

The Y-Axis Range fields show the volume of gas adsorbed.

Analysis Report Options

The analysis report consists of a summary page, a tabular report, and a line fit plot.



Print analysis summary

Select this option to print a summary of analysis and/or difference results.

Show Analysis results generates a summary of the following for the first analysis:

- Percent metal dispersion
- Metallic surface area
- Volume adsorbed
- Slope
- Correlation coefficient

Show difference results generates a summary of the differences between the following information for the first and repeat analyses:

- Percent metal dispersion
- Metallic surface area
- Average difference volume

Print tabular report

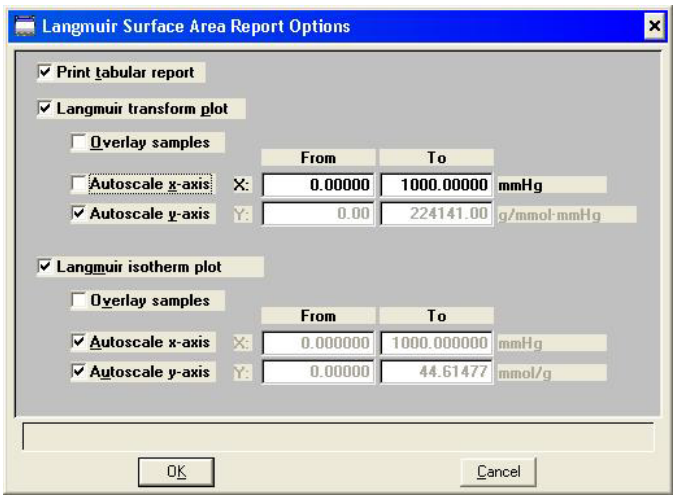
Select to have analysis data printed in a tabular format.

Plot Data

Select this option to produce a graph showing the line fit difference between the analysis and repeat analysis.

Analysis	Includes a line fit plot for the primary analysis.
Repeat analysis	Includes a line fit plot for the secondary analysis.
Difference	Plots the difference between the analysis and repeat analysis lines.
Overlay samples	Allows you to overlay data from the current sample with that of other samples. Click **Overlays** on the Report Options dialog to choose the other sample files.
Crystallite size	Allows you to choose the crystallite shape of your sample, or to enter one.
Autoscale Options	Select these options to have the X- and/or Y-axes automatically scaled. The system uses the highest values collected during analysis as the ending points for an axis range.
Axis Range	These fields are enabled if you deselect **AutoScale**, allowing you to specify beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot. The X-Axis Range fields show the pressure. The Y-Axis Range fields show the volume of gas adsorbed. Valid ranges for these fields are shown in the information bar across the bottom of the dialog.

Langmuir Surface Area Report



Print tabular report — Select this option to have a tabular report of the plotted data.

Langmuir transform plot — Generates a traditional Langmuir surface area plot that is used to determine monolayer volume and BET C constant.

Langmuir isotherm plot — Uses the Langmuir monolayer volume and constant to produce an isotherm.

Overlay samples — Select this option to overlay the current type plot with those of other samples. Click **Overlays** on the Report Options dialog to choose the other sample files.

Autoscale Options — Select these options to have the X- and/or Y-axes automatically scaled. The system uses the highest values collected during analysis as the ending points for an axis range.

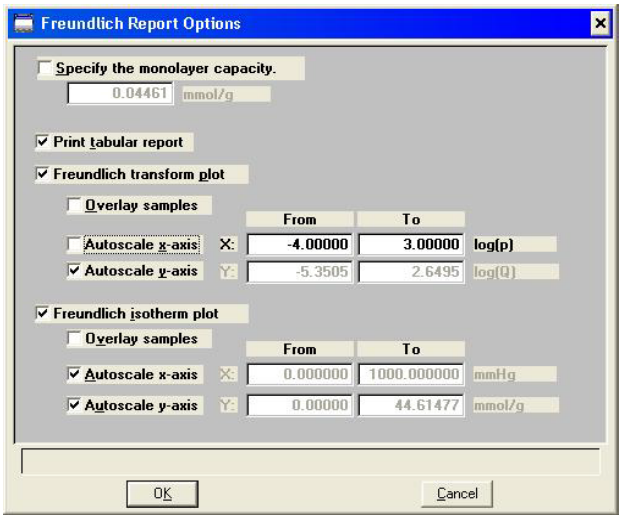
From/To — These fields are enabled if you choose not (deselect) to AutoScale, allowing you to specify beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the pressure.

The Y-Axis Range fields show the volume of gas adsorbed.

Valid ranges for these fields are shown in the information bar across the bottom of the dialog.

Freundlich Report



The Freundlich isotherm is an empirical isotherm that is used to model low-pressure adsorption data. It can also be applied to model some micropore isotherms.

Specify the monolayer capacity — In this field, enter the monolayer capacity of the sample.

Print tabular report — Select this option to have a tabular report of the pressure points generated.

Freundlich transform plot — Plots the linear form of the Freundlich equation.

Freundlich isotherm plot — Plots the absolute pressure vs quantity adsorbed. Shows best fit line.

Overlay samples — Choose this option to overlay data from the current file with the same type of data from other samples (files). Click **Overlays** on the Report Options dialog to choose the other files.

Autoscale Options — Select these options to have the X- and/or Y-axes automatically scaled. The system uses the highest values collected during analysis as the ending points for an axis range.

From/To

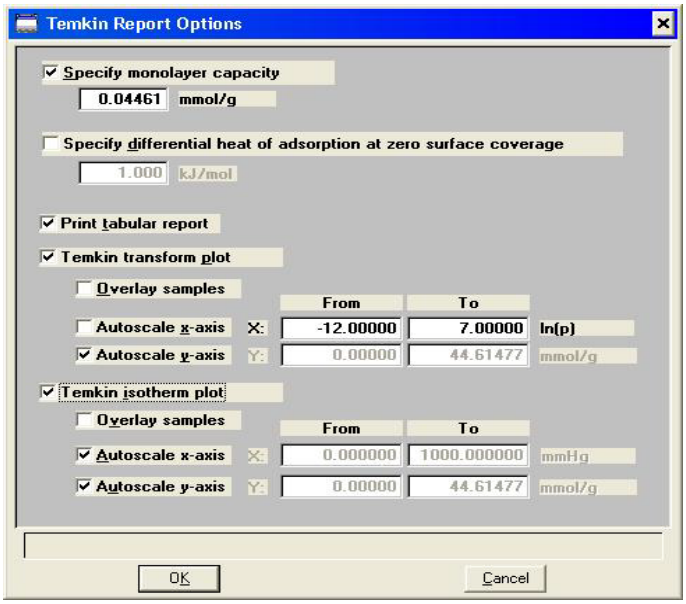
These fields are enabled if you choose not (deselect) to AutoScale, allowing you to specify beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the pressure.

The Y-Axis Range fields show the volume of gas adsorbed.

Valid ranges for these fields are shown in the information bar across the bottom of the dialog.

Temkin Report



The Temkin isotherm is used to model adsorption data where the heat of adsorption drops linearly with increasing coverage.

Specify monolayer capacity — In this field, enter the monolayer capacity of the sample.

Specify differential heat of adsorption at zero surface coverage — Enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.

Print tabular report — Select this option to have a tabular report of the pressure points generated.

Temkin transform plot — Plots a linear form of the Temkin isotherm.

Temkin isotherm plot — Overlays the Temkin isotherm with the analysis data.

Overlay samples — Choose this option to overlay data from the current file with the same type of data from other samples (files). Click **Overlays** on the Report Options dialog to choose the other files.

Autoscale Options

Select these options to have the X- and/or Y-axes automatically scaled. The system uses the highest values collected during analysis as the ending points for an axis range.

From/To

These fields are enabled if you choose not (deselect) to AutoScale, allowing you to specify beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the pressure.

The Y-Axis Range fields show the volume of gas adsorbed.

Valid ranges for these fields are shown in the information bar across the bottom of the dialog.

Options Report

The Options Report is a predefined collection of sample information file parameters printed by selecting **Options** from the Report Options dialog. You cannot edit this report.

If Type of Data is **Automatically collected**, the following information appears on the report:

Task Summary	Lists conditions you specified for each task selected.
Analysis Task Options	Details conditions you specified for the analysis task.
Experiment Log	Identifies actual conditions under which each task transpired.
Leak Test Results	Identifies outgas rates and the outcome for each leak test performed.

Redundant data appear in the **Task Summary** and the **Experiment Log** if the instrument is performing correctly.

If Type of Data is **Manually entered**, only the adsorptive gas, sample temperature, and free-space volume are reported.

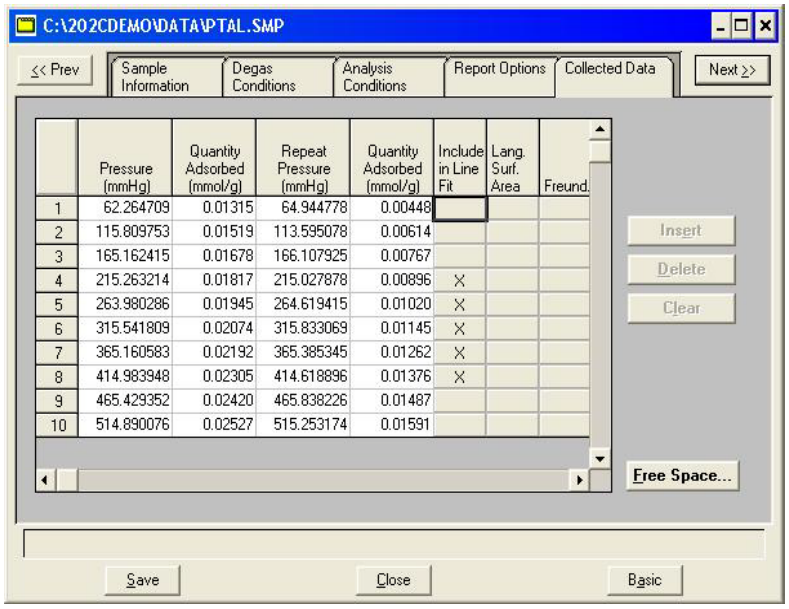
Sample Log

The Sample Log report provides the following statistics:

- manual control operations performed during analysis
- information entered using on the sample file editor
- warnings and/or errors that occurred during analysis

Collected/Entered Data

The Collected/Entered Data dialog displays after analysis is complete and when your option presentation is selected as Advanced.



- If **Automatically collected** was selected as the type of data in the Sample Information dialog, the Collected Data dialog contains the data points collected during analysis and the specified calculation assignments. You can edit calculation assignments during or after an analysis.

- If **Manually entered** was selected as the type of data in the Sample Information dialog, the Entered Data dialog contains the manually entered data and calculation assignments. You can edit calculation assignments as well as the points in the table during or after an analysis. **Insert**, **Delete**, and **Clear** become enabled allowing you to edit points.

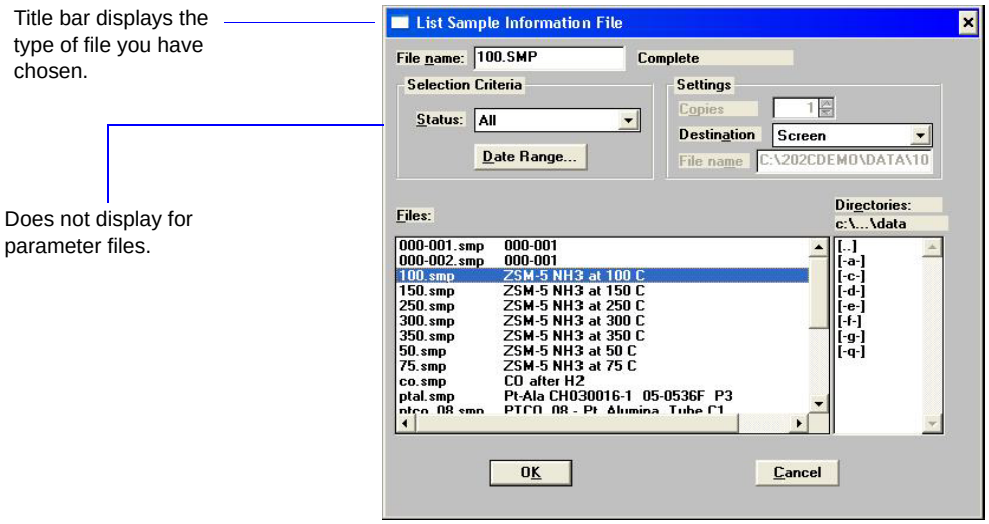
You cannot enter data if the analysis program is being used for offline data manipulation on a computer other than the one controlling the analyzer.

List

List enables you to display the following information on one or more sample or parameter files:

- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status (sample information files only)
- File name

The List dialog is common to all file types.



File name — If you select only one file from the **Files** list box, the name is copied to this field. If multiple files are chosen, the last one selected displays.

Copies — Enabled when **Printer** is selected as the print destination. You may print up to four copies.

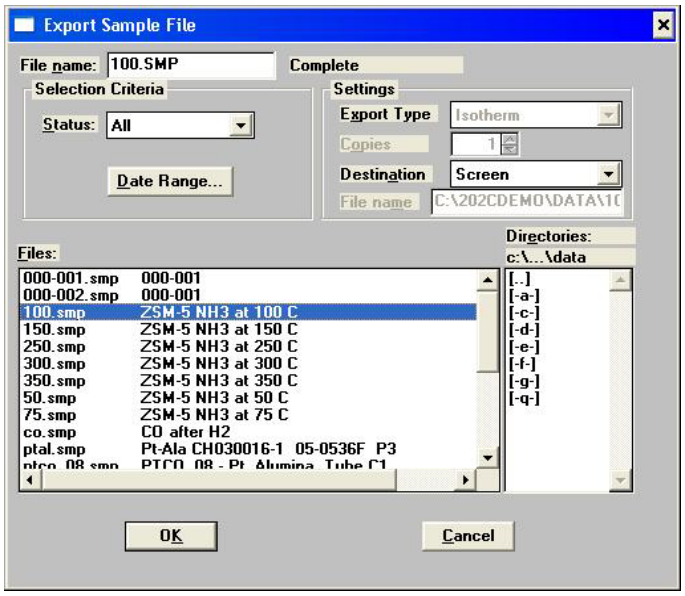
Destination — You may send the list to the printer, the screen, or to a file.

File name (print) — Enabled when you select **File** as the destination. A default file name appears. Use the default name or enter another file name. If you select multiple files, the information from all the files is copied into the file specified in this field.

Export

Export copies the isotherm data in sample information files and reformats it in ASCII format. Refer to **Appendix D**, **Format of Exported Data**, for record descriptions. The data can then be imported into other applications accepting ASCII format, such as spreadsheets.

Select **File > Export** to display the Export Sample File dialog.



File name — The name of the file you choose to export is copied to this field. If you choose multiple files, the name of the last one selected is displayed.

Settings — The options in this group box enable you to choose output options.

Copies — Enabled when **Printer** is selected as the print destination, allowing you to print up to four copies.

Destination — You may export files to a printer, to the screen, or to a file.

File name — Enabled when you choose **File** as the print destination. For the exported name, you can:

- Accept the default value where all selected files you choose to export will have the same name as those in the sample information file with a .**ISO** extension.

File name

(continued)

Enabled when you choose **File** as the print destination. For the exported name, you can:

- Accept the default value where all selected files you choose to export will have the same name as those in the sample information file with a .ISO extension.

For example, the following chart shows the selected files and export file names automatically assigned by the system when the default value is C:\DATA1*.EXP:

Selected Files	Export Names
C:\DATA1\SAMP1.SMP	C:\DATA1\SAMP1.ISO
C:\DATA1\SAMP2.SMP	C:\DATA1\SAMP2.ISO
C:\DATA1\SAMP3.SMP	C:\DATA1\SAMP3.ISO

- Enter the name of the file (full path name) receiving the ASCII data. The extension does not have to be .EXP. To export more than one file, wildcard characters may be entered. For example, the following chart shows the selected files and export file names resulting when you enter: C:\DATA2*.TXT:

Selected Files	Export Names
C:\DATA1\SAMP1.SMP	C:\DATA2\SAMP1.TXT
C:\DATA1\SAMP2.SMP	C:\DATA2\SAMP2.TXT
C:\DATA1\SAMP3.SMP	C:\DATA2\SAMP3.TXT

6. UNIT MENU

The Unit menu contains the options for the operations that can be performed with the ASAP 2020 Chemi analyzer. The main menu will contain a Unit menu for each attached analyzer. For example, if you have two attached analyzers, the main menu contains two Unit menus. The appropriate unit number and serial number display in the title bar of the operational windows. The status windows also display in different colors. This feature is especially useful when you have more than one analyzer attached to the same computer.

The Unit menu does not display on the menu bar if the analysis program is being used for offline data manipulation on a computer other than the one controlling the analyzer.

Description

Listed below are brief descriptions of the Unit menu options. Detailed descriptions are found later in this chapter.

Sample Analysis — Starts an analysis. This option is disabled if analyses are in progress. Page 6-3.

Analysis Sequence — Allows you to schedule a series of analyses. Page 6-7.

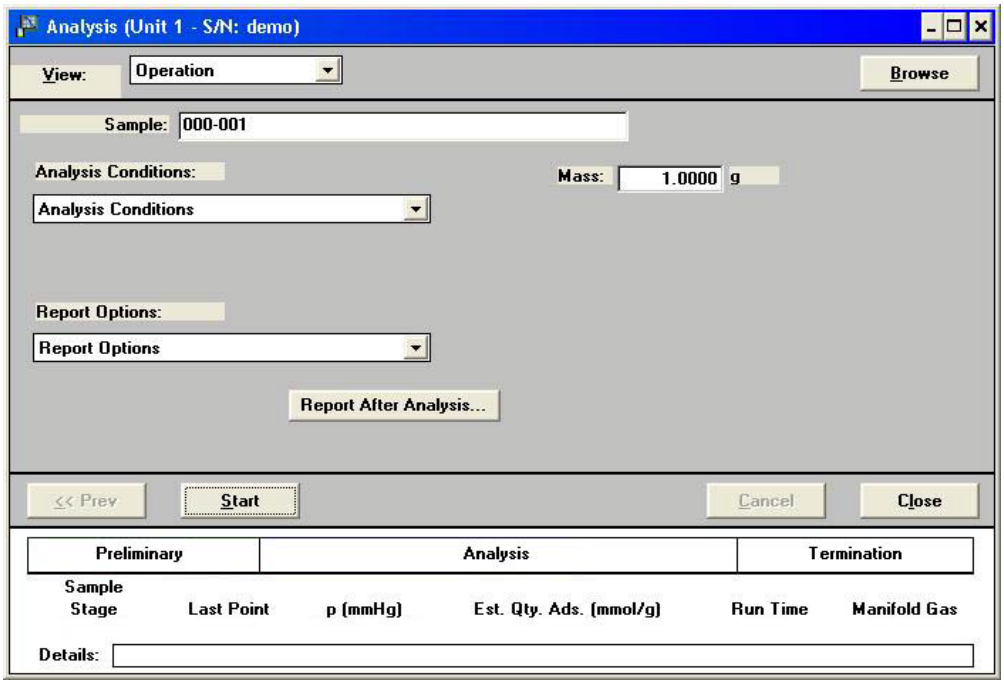
Start Degas — Allows you to begin the degassing operation on one or two samples. Page 6-9.

Enable Manual Control	Allows you to control certain components of the system manually. Page **6-10**.
Show Instrument Schematic	Displays a schematic of the analyzer components. Page **6-14**.
Show Status	Shows the status window of the operation in progress. Page **6-15**.
Show Instrument Log	Displays a log of recent analyses, calibrations, and error messages. Page **6-16**.
Unit Configuration	Displays the configuration of the analyzer. Page **6-18**.
Diagnostics	Enables you to perform certain diagnostic tests. Page **6-21**.
Calibration	Enables you to calibrate certain components of the analyzer. Page **6-23**.
Degas	Enables you to perform operations associated with degassing the sample. Enabled only if the SmartVac is installed. Page **6-25**.
Service Test	Enables you to perform certain troubleshooting procedures. This option is enabled only with the direction of a Micromeritics service representative. Page **6-30**.

Sample Analysis

When you select this option from the Unit menu, the Analysis dialog is displayed with all fields disabled (greyed) and the Start Analysis dialog positioned on top. This allows you to select an existing sample file for your analysis or to create a new one.

After a sample file has been designated, the Analysis dialog is fully displayed. The fields now contain the values for the selected file or, if creating a new file, the specified defaults.



View

Allows you to view one of the following in the current window:

- the current operation
- the instrument schematic
- the instrument log

Refer to **Show Instrument Log**, page **6-16** and **Show Instrument Schematic**, page **6-14** for additional information.

Browse

Displays the Open Sample Information dialog allowing you to choose a different sample file for your analysis.

Sample

Displays an identification of the sample file you choose.

Mass

Allows you to enter an accurate value for the sample mass.

Analysis Conditions

Displays the name (identification) of the Analysis Conditions file for the current sample file.

If this is a new file, this field contains the name you specified as the default.

The drop-down list contains a list of predefined parameter files that were saved to the Parameter Files Directory (Chapter 8). Some files were also shipped with the analysis program. You may choose a different file from this list.

Report Options

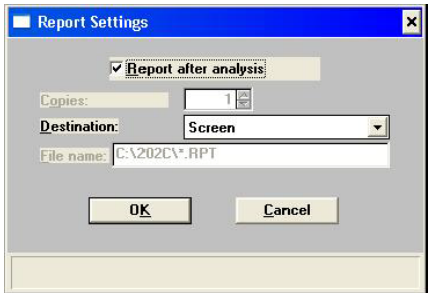
Displays the name (identification) of the Report Options file for the current sample file.

If this is a new file, this field contains the name you specified as the default.

The drop-down list contains a list of predefined parameter files that were saved to the Parameter Files Directory (Chapter 8). Some files were also shipped with the analysis program. You may choose a different file from this list.

Report After Analysis

Displays the Report Settings dialog so that you may specify report output options. If you choose **Screen**, reports have many options for being customized and manipulated.



Report after analysis

Select this option to have reports generated automatically after analysis. If you do not select this option, you can generate the report using the **Start Report** option on the **Reports** menu.

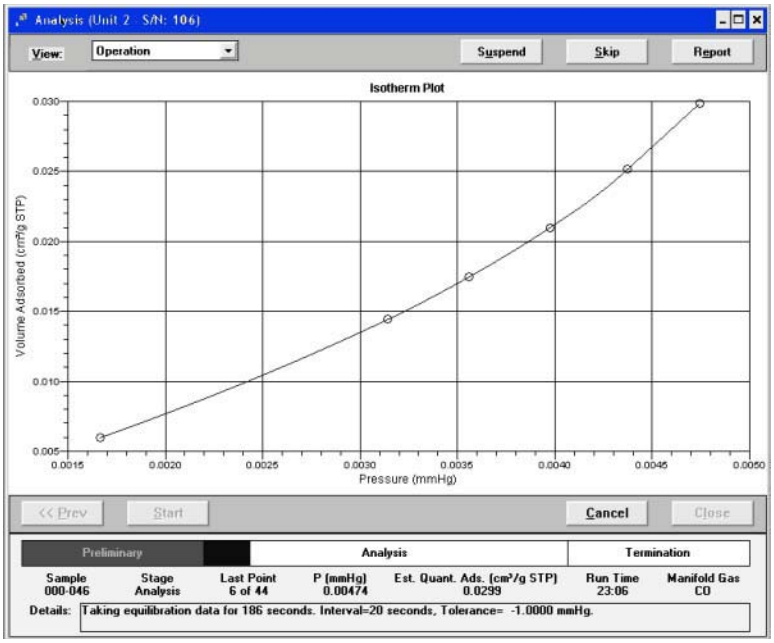
Copies

Enabled when **Printer** is selected as the destination, allowing you to print up to four copies.

Destination Allows you to have reports printed to the Screen, Printer, or to a File. If you choose Screen, many options are available for customizing your report.

File name Enabled when you choose File as the destination for report output. You can enter a name or accept the default, which is the same name as the sample file with an **RPT** extension.

Start Begins the analysis; displays an analyzing view of the Analysis dialog. A graph will be drawn as data are collected.



Skip Skips the current step of the analysis.

Use the Skip function with caution; the ASAP performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.

Suspend Suspends the analysis.

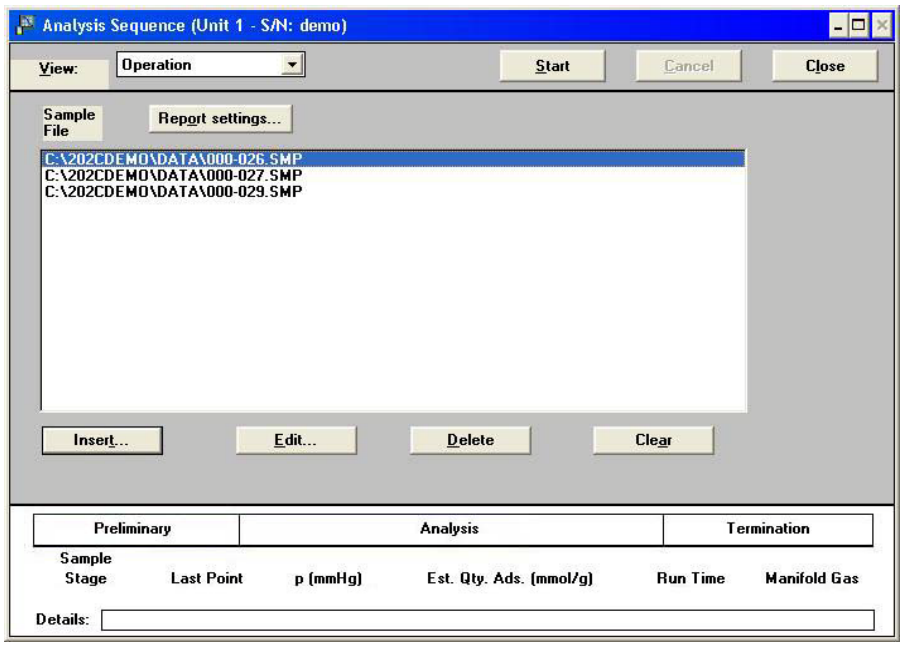
Resume Resumes a suspended analysis.

Cancel Cancels the analysis.

Start	On this view of the Analysis dialog, this button changes to **Next** when the analysis is finished.
Next	Returns you to the first view of the Analysis dialog so that you may schedule another analysis.
Close	Closes the dialog.

Analysis Sequence

This option allows you to perform a series of analyses using sample files ready for analysis. Sample files cannot be created at the time of analysis when performing sequenced analyses. When you select this option, the Analysis Sequence dialog is displayed.



View

Allows you to view one of the following in the current window:

- the current operation
- the instrument schematic
- the instrument log
- results

Report Settings

Displays the Report Settings dialog so that you can specify report output. The options on this dialog are explained in the previous section.

Sample File window

Contains the files you choose to use with your analyses.

Insert

Displays the Select Sequence File dialog so that you can choose the sample files to use with your analyses. The file you choose is inserted above the line you have selected in the Sample File window. This button is not available while analyses are in progress. If you need to add a file name to the sequence, click **Suspend**, insert the new file name, then click **Start** to restart the sequence.

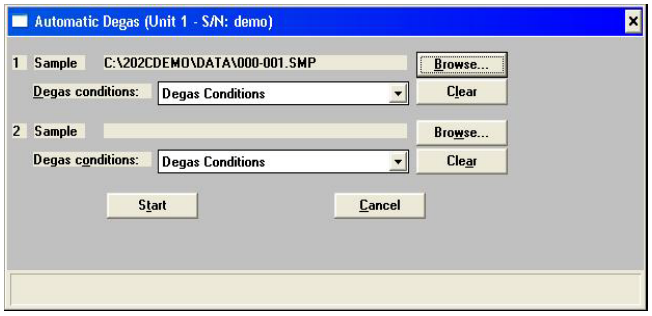
Edit	Allows you to choose a different file to replace the selected file name.
Delete	Deletes the selected file name from the sequence. You cannot delete the file name for an analysis in progress because the system removes the file name from the sequence as soon as the analysis begins. This button is not available while the sequence is in progress. Click **Cancel** to stop the sequence, select the file name you want to delete, click **Delete**, then click **Start** to restart the sequence.
Clear	Removes all file names from the sequence. You cannot clear the sequence while analyses are in progress. Click **Cancel** to stop the sequence and then click **Clear**.

If you weigh the sample after analysis, make sure you enter the new sample's mass in the Sample Information file before starting reports.

Status window	Refer to **Show Status**, page **6-15** for an explanation of this window's contents.
Start	Starts the sequence of analyses.
Cancel	Stops a sequence in progress. If an analysis is in progress, it continues.

Start Degas

Displays the Automatic Degas Operation dialog, allowing you to choose files and start degas operations on one or two samples.



Sample — Click **Browse** to the right of this field to select a sample file that will be used with the current sample.

Degas Conditions — Displays the description of the degas file that is part of the chosen sample file. You can choose a different Degas Conditions file from the drop-down list. If you choose a different file, the values in the current file will be overwritten with the values of the new one.

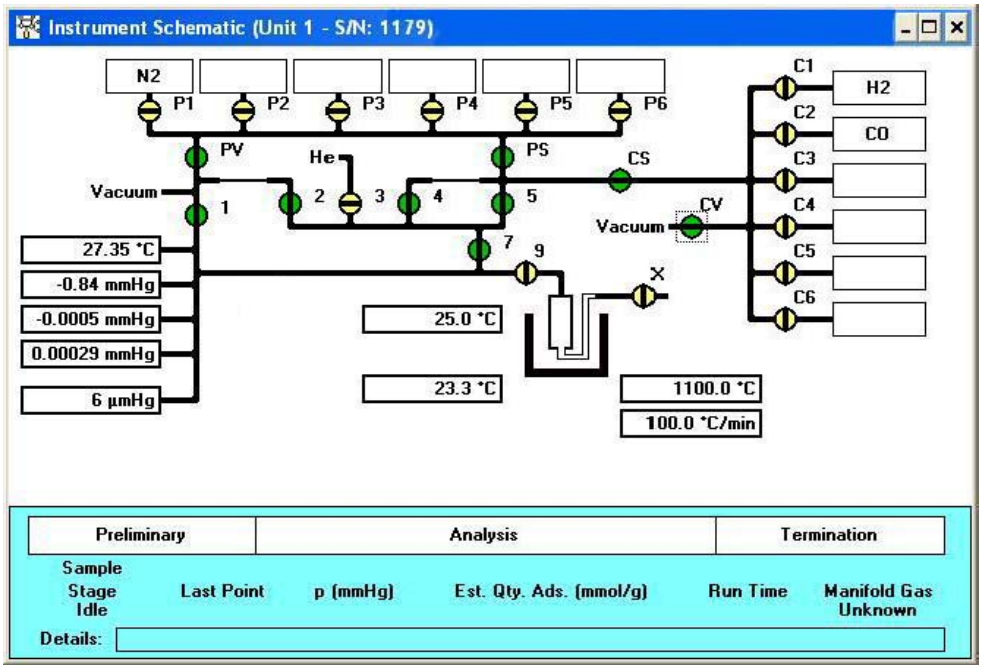
Clear — Clears the sample file from the port assignment. If you plan to use another sample file, click **Browse** to choose the file.

Start — Starts the degassing operation.

Cancel — Closes the dialog, cancelling any information entered.

Enable Manual Control

Select this option to control certain components of your system manually. If the instrument schematic is not displayed, select **Show Instrument Schematic**.



When you enable manual control, the color for the valve symbols changes to yellow if closed, and green if open.

Use the mouse pointer to select a component. A component is selected when it is surrounded by a thin line. Each component has a shortcut menu displaying the operations available for that particular component. These menus may be accessed by right-clicking on the component, or by using the shortcut keys **Shift + F9**.

You can open and close valves, and raise or lower the elevator by using one of the following methods:

- access the shortcut menu and select the appropriate action
- double-click on the valve or elevator symbol
- select the valve or elevator symbol and press the spacebar

Valves

System valves and their functions are listed in Table 6-1.

Actions: Open, Close, Pulse

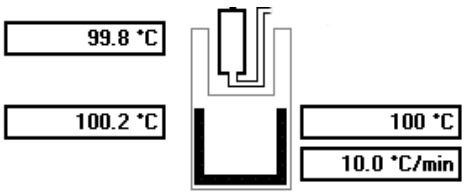
Pulse briefly opens and closes (or vice versa) the valve.

Table 6-1. System Valves

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Free-space measurement gas (helium)
4	Restricted analysis gas
5	Unrestricted analysis gas
7	Lower manifold isolation
9	Sample port
CV	Vacuum valve, allows evacuation of chemisorption gas inlet manifold
CS	Supply valve, supplies gas to manifold for chemisorption operation
C1 through C6	Gas inlet port valves for chemisorption operation
X	Exhaust from sample tube to gas flow meter, located at sample port above furnace
PV	Vacuum valve, allows evacuation of physisorption gas inlet manifold
PS	Supply valve, supplies gas to manifold for physisorption operation
P1 through P6	Gas inlet port valves for physisorption operation

Furnace

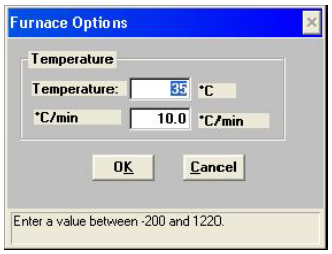
The furnace resides on the elevator. The following illustration shows the furnace and its displays



Actions: Set, Raise, Lower, Stop

Set

Displays the Furnace Options dialog allowing you to specify a furnace temperature and heat rate.



The temperature and heat rate you enter are displayed to the right of the furnace symbol (shown on the previous page).

Raise, Lower, and Stop

Allow movement of the furnace on the elefvator.

 The furnace is being raised; note position of arrow in the center of the furnace symbol.

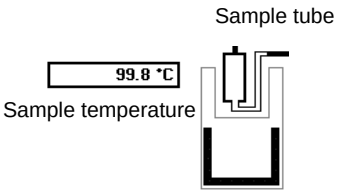
 The furnace is being lowered; note the position of arrow in the center of the elevator symbol.

 The furnace is stopped while being raised; it stops in the uppermost position.

 The furnace is stopped while being lowered; it stops in the lowermost position.

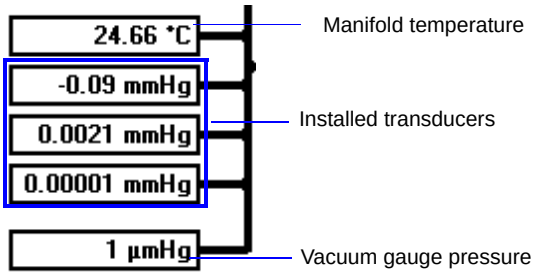
Sample Tube

The current sample tube temperature is displayed to the left of the sample tube.



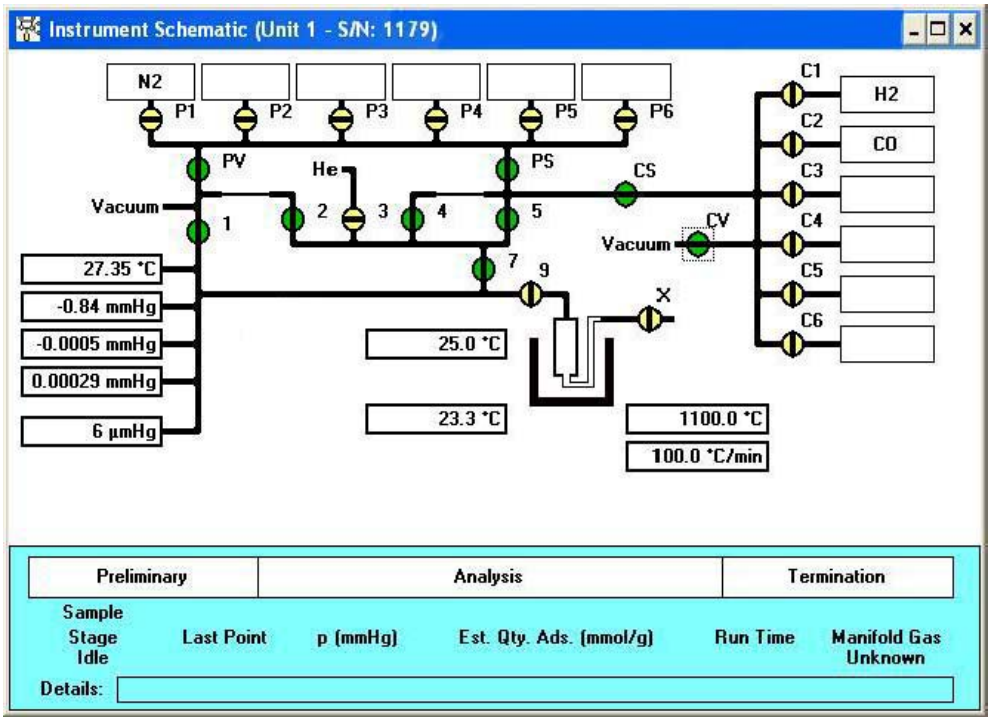
Manifold Temperature Transducers Vacuum Gauge Pressure

The group of rectangles on the left side of the schematic displays the temperature, the vacuum gauge pressure, and the transducers currently installed.



Show Instrument Schematic

Select this option to display a schematic of the ASAP 2020 Chemi analyzer. The schematic is a graphical representation of the plumbing system including system valves, the analysis station, and the furnace position. The valves and furnace can be controlled from the schematic when Manual control is enabled. Refer to the previous section for an explanation of the components displayed on the instrument schematic.



The state of the valves can be determined quickly by color representation even when manual control is not enabled:

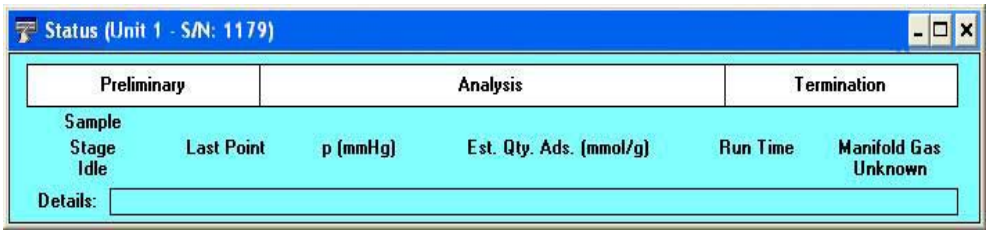
- Green = open
- White = closed

To change the state of a valve, you must enable manual control.

Show Status

A status window is shown across the bottom of all operational dialogs as well as the instrument schematic. The Show Status option enables you to show only the status window. This frees up your computer screen allowing you to perform other tasks (such as creating or editing sample files), while still monitoring the progress of your analysis.

If you have multiple instruments attached to your computer, the status bar for each instrument is displayed in a different color.



Analysis status bar

Shows the progression of the analysis. This bar contains three stages:

- **Preliminary**: sample preparation; displays green during progression
- **Analysis**: data are collected; displays blue during progression
- **Termination**: the furnace lowers, and the sample is cooled and backfilled; displays grey during progression

All stages are displayed in red if the analysis has been suspended.

Analysis details

The following analysis details are displayed:

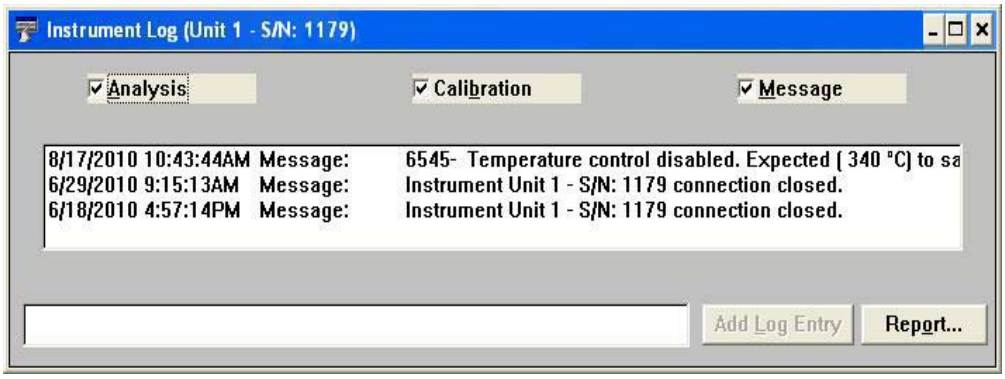
Sample: Sample file number
Stage: Stage of the analysis (as in status bar)
Last Point: Last point and number of points requested
P: Pressure of last point
Est. Qty. Ads.: Quantity adsorbed
Run Time: Elapsed time since the start of analysis
Manifold Gas: Current manifold gas

Step details

Provides details of the current step of the analysis.

Show Instrument Log

Displays a log of recent analyses, calibrations, and errors or messages. By default, this information is logged for a 7-day period for analyses and a 30-day period for messages and calibrations. You may change the time for which this information is retained in the Unit section of the ASAP202C.INI file.



Analysis
Calibration
Messages

These options allow you to choose the type of entries displayed in the window. For example, select only the Calibration check box to display only calibration information.

Add Log Entry

Enables you to make an entry in the instrument log that cannot be recorded automatically through the application software. For example, you may change the port filter. The field adjacent to the button allows you to enter the operation; the button is enabled when you make an entry in the field, allowing you to add the entry.

Report

Generates a report of the Instrument log.

Report

Allows you to generate the log contents to a specified destination; the Log Report Settings dialog is displayed.

Use the **Start Date** field to specify a date at which to start the printout. You can specify a date using one of the following methods:

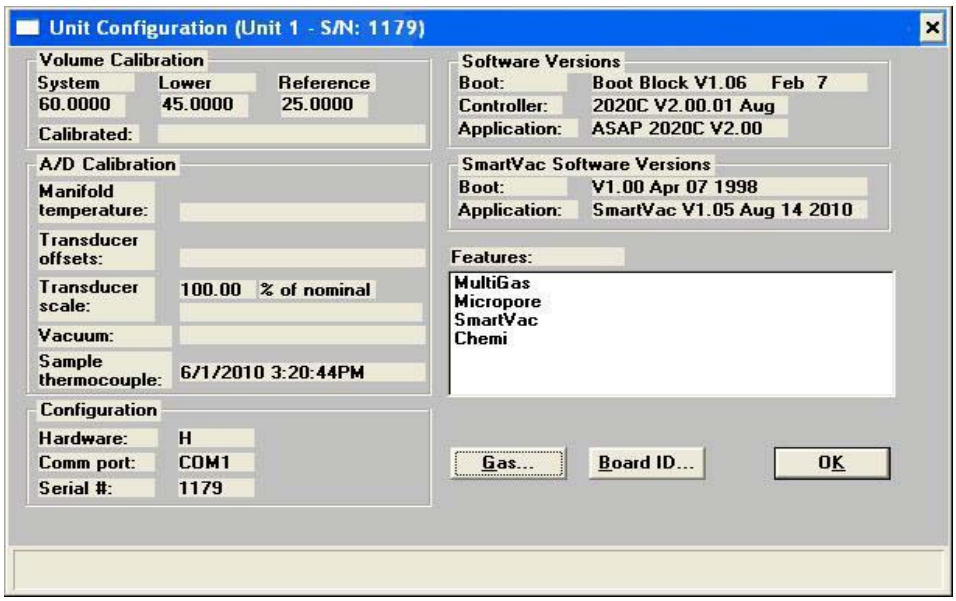
- Press **F2** to clear the field so that you can enter a date
- Double-click in the field (or press **F4**) to display a calendar to choose a date
- Press **F3** to insert the current date

The **Copies** field is enabled when you select **Printer** as the destination, enabling you to print up to four copies.

Choose the report destination in the **Destination** field. If you choose **File** as the destination, the **File name** field is enabled, allowing you to enter a name for the printed file (or you may accept the default).

Unit Configuration

Select this option to view the current calibration settings, the date on which calibration was performed, and the software and hardware configuration of your system.



Volume Calibration

Displays the system, lower, and reference volumes from the most recent calibration. It also displays the date and time the calibration was completed.

A/D Calibration

Displays the following:

- The date and time the manifold temperature was last calibrated.
- The date and time the transducer offsets were last calibrated.
- The percent of nominal transducer scale in use, and the date and time the transducer scale was last calibrated.
- The date and time the vacuum gauge was calibrated.
- The date and time the sample thermocouple was last calibrated.

Configuration

Displays the following:

The hardware code specifying the hardware components (for example transducers) present in your analyzer. This code is provided so the Service Department may quickly identify your hardware configuration and provide prompt assistance. Hardware codes translate as follows:

E = 1000-mmHg transducer
G = 10- and 1000-mmHg transducers
H = 1-, 10-, and 1000-mmHg transducers

- The identification number of the port being used to control the analyzer.
- The serial number of the attached analyzer (active unit).

Software Versions

The software versions of the MIC BIOS, Controller, and ASAP 2020 Chemi System software.

SmartVac Software Versions

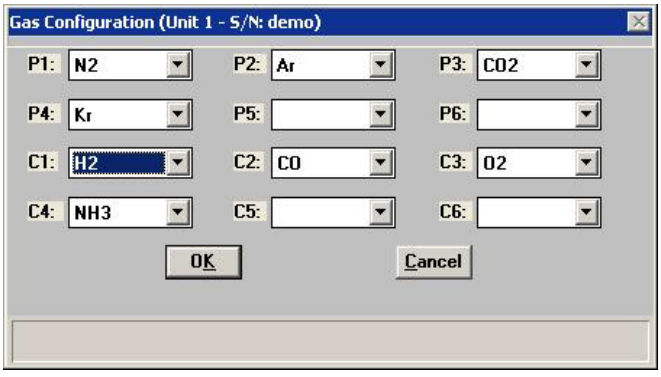
The software versions of the MIC BIOS and SmartVac software. This group box is not displayed if the SmartVac is not installed.

Features

The options currently enabled on your system (for example, Micropore or MultiGas).

Gas

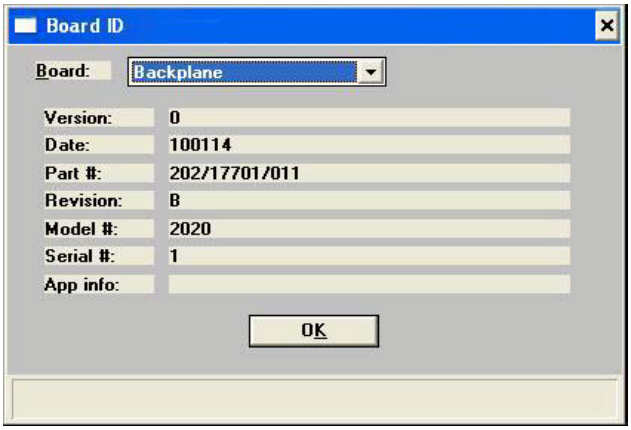
Displays the Gas Configuration dialog.



This dialog allows you to choose which gases are attached to each port of the analyzer. The numbers preceded with P are the physisorption gases and the ones preceded with C are the chemisorption gases.

Board ID

Displays the Board ID dialog so that you can view the statistics of the board contained in the requested slot of the computer. For example, this dialog shows statistics of the backplane board.

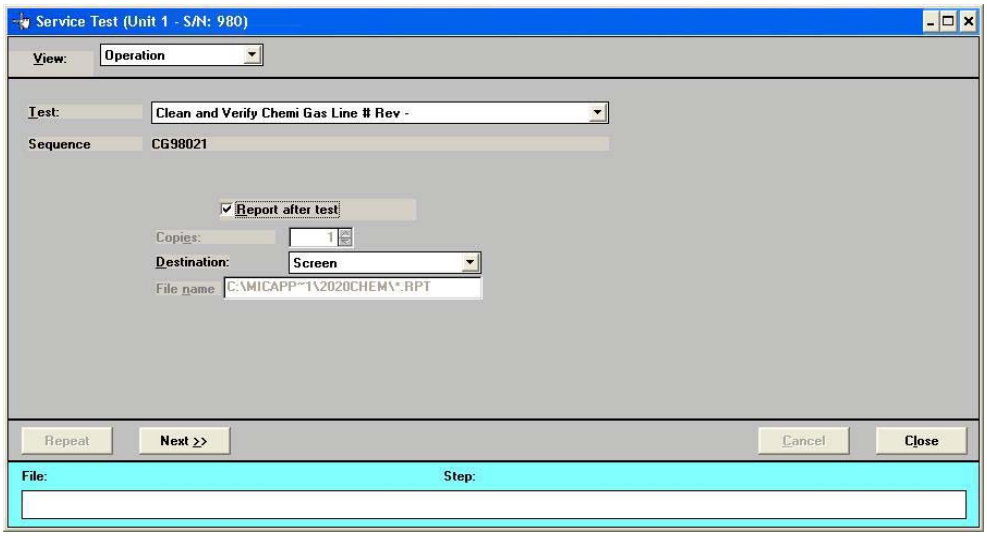


Diagnostics

This option enables you to clean and verify gas lines when connecting or changing a gas. This option also allows you to perform diagnostic tests which your service representative may request. The data generated from these tests may be insignificant to you as a user, but can be very helpful to your service representative. Your service representative can view the results and may be able to resolve the problem, eliminating downtime and repair costs.

These tests generate files to the **2020Chem\Service\userdiag** directory. Your service representative will request that you E-mail or fax the files to him so that he may examine them.

When you select **Unit [n] > Diagnostics**, the Service Test dialog is displayed:



View — Allows you to view the current operation, the instrument schematic, or the instrument log in the Service Test dialog. **Operation** is the appropriate choice for tests.

Test — Contains a list of diagnostic tests. These tests will always include the latest revision letter at the end of the test name.

Sequence — Displays the test file number. This number is assigned automatically and incrementally sequenced by the software each time a test is performed. This also serves as the name of the file that is generated to the **userdiag** directory, and will be appended with SVT. For example, the name of the file for the test shown in the above dialog is **0006.SVT**.

Report after test

Select this option to have a report generated automatically after the test; page **6-4** provides an explanation of output options.

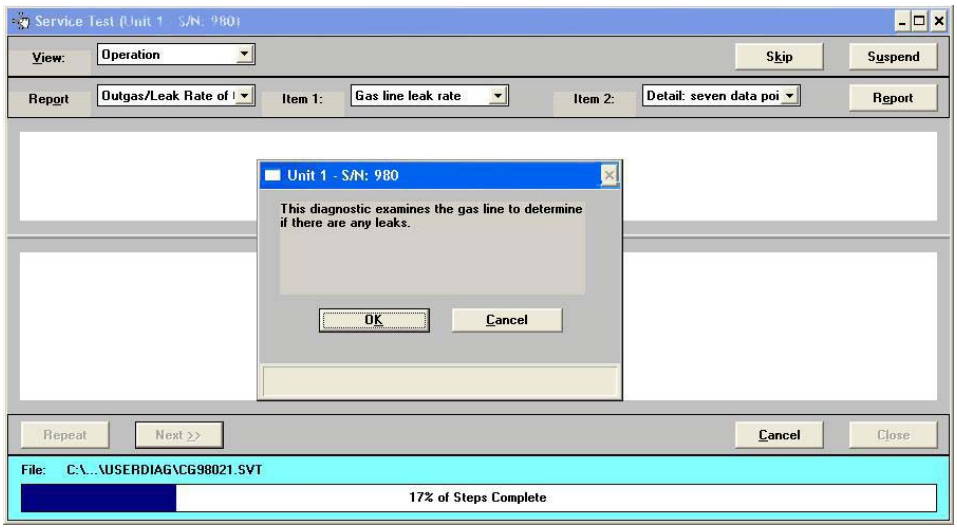
If you do not select this option, you can still have a report generated from the report window.

Cancel

Cancels the test.

Next

Begins the test; displays the next view of the Service test dialog. The second view of the Service test dialog may contain a single pane or two panes, depending on the test selected.



Report (field)

Contains a list of the reports that will be generated during this test.

Item [n]

Lists the data in the two panes. **Item 1** is the upper pane and **Item 2**, the lower pane.

Report

Enables you to generate a report.

Cancel

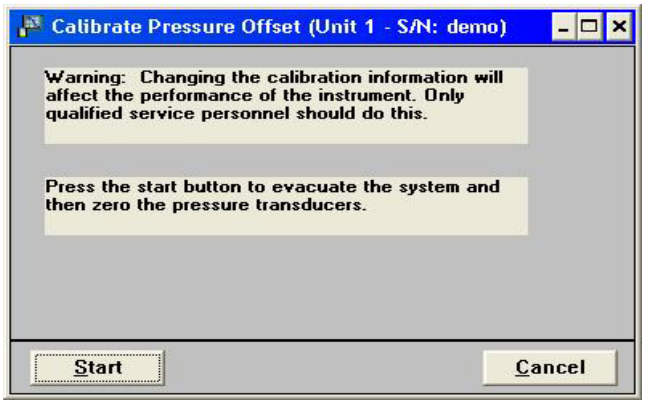
Cancels the test.

Calibration

The options on this menu allow you to perform system calibrations. Only one calibration is enabled and can be performed without direction of a Micromeritics service representative. You must contact your service representative for performing other calibrations.

Pressure Zero

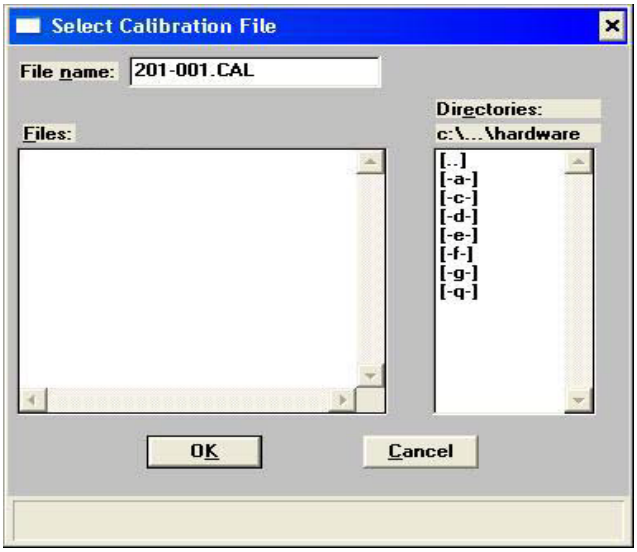
Displays the Calibrate Pressure Offset dialog.



This dialog allows you to evacuate the manifold and zero all transducers in the system.

Save to File

Use this option to save the current calibration settings to a file; the Select Calibration File dialog is displayed.



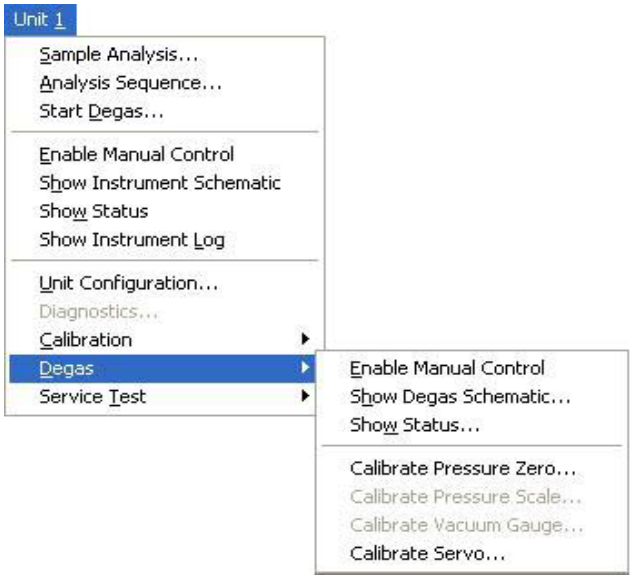
File name Defaults to the next sequence number for calibration files. The sequence number consists of: xxx-yyy.CAL, where xxx = serial number of unit and yyy = sequence number. For example, the number listed in the above dialog represents the first calibration file for unit serial number 201.

Load from File

Displays the Select Calibration File dialog (shown above) so that you can load a previously saved calibration file

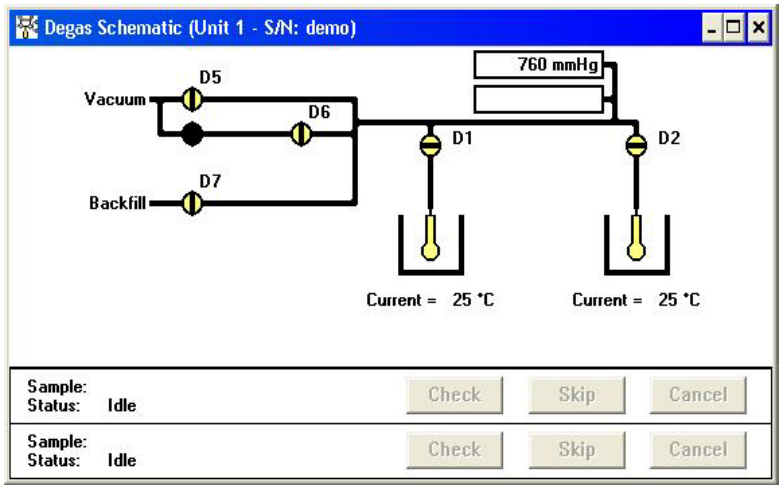
Degas

This option on the Unit menu is enabled if your ASAP 2020 Chemi is equipped with the SmartVac degassing option. The choices on this menu allow you to select operations for the SmartVac degasser.



Enable Manual Control

Select this option to control manually certain components of the degas operation. If the instrument schematic for the SmartVac is not displayed, select **Unit > Degas > Show Instrument Schematic**.



Valves Degasser valves and their function are listed in Table 6-2.

Actions: *Open, Close*

Table 6-2 Degasser Valves

Valve	Description
D1 and D2	Sample port valves
D5	Vacuum valve
Unnamed	Servo valve
D6	Servo isolation valve
D7	Gas inlet port valve

Heaters

Actions: Set, Disable

Set displays the Port [n] Heater Settings dialog so that you can specify a ramp rate and target temperature. After you click **OK** and close the dialog, the settings are displayed directly below the heater symbol.

Disable cancels the settings.

Status window

Displays the status of the current degassing operation(s). Refer to Show Status on the following page for information on the Status window.

Show Degas Schematic

Displays the schematic (shown in the previous section) for the SmartVac.

Show Status

This option allows you to monitor the degassing operation.

Sample Displays the sample file being used with the degassing operation for each port.

Status Displays the current stage of the degassing operation for each port.

Check Allows you to check the outgassing rate of the sample on the related port; the following actions occur:

- current degassing step is suspended (on both ports). Degassing can be checked after the vacuum setpoint has been attained, or during a temperature ramp and hold. If you choose this option during any other step, a message indicating the SmartVac is not in a valid state is displayed.
- vacuum valves are closed and the vacuum level monitored
- the Status window is displayed (if not already displayed). The Status window will indicate that the degas operation is being checked and will display the outgassing rate as it becomes available.

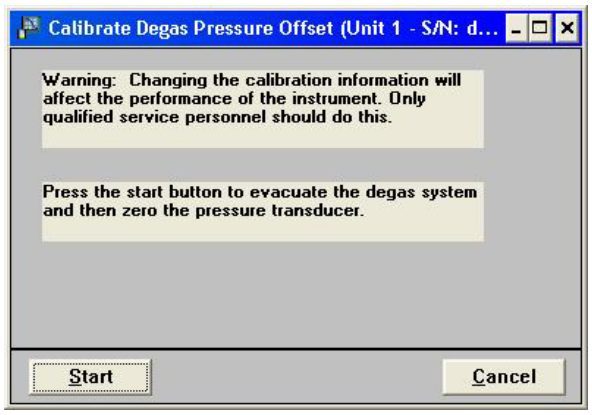
During the degas check, this button changes to **Continue**. When **Continue** is clicked, the valves open, the temperature ramp or hold continues, and the degassing operation resumes. If your outgassing rate indicated that the sample is rid of contaminants (minimal pressure increases), you can click **Skip** to advance to the next stage of the degassing operation. For example, if you check degassing after the setpoint is attained, **Skip** advances you to the ramping stage.

Stop Skips the current stage of the degassing operation for the port in which this button was selected.

Cancel Cancels the degassing operation for the port in which this button was selected.

Calibrate Pressure Zero

Use this option to evacuate the manifold and zero the transducer.

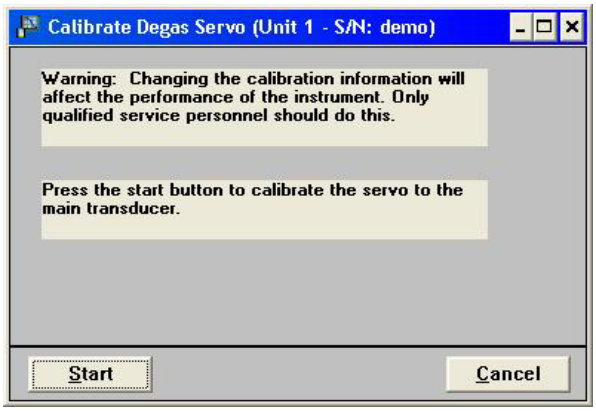


Start Begins the calibration.

Status messages are displayed during this procedure, then the dialog closes automatically.

Calibrate Servo

Use this option to calibrate the servo valve to the manifold pressure transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The Calibrate Servo Valve dialog is displayed.



Start Begins the calibration. The servo valve is used to fill and equilibrate the manifold. The pressure transducer readings are used to calibrate the servo set point. Status messages are displayed during this procedure, then the dialog closes automatically.

The other calibrations on the Degas menu require direction from a Micromeritics service representative.

Service Test

Various service tests are included in the ASAP 2020 Chemi operating program. These tests can be enabled and performed only with the assistance of a trained Micromeritics service representative. These tests are designed to provide your service representative with instrument readouts, as well as to assist him in troubleshooting potential problems and, perhaps, eliminating unnecessary repair services. This service strategy allows you to conduct expert tests in less time than it would take to be trained in servicing the instrument properly.

7. REPORTS MENU

This chapter describes:

- how to start reports, page 7-2
- how to close reports, page 7-5
- how to open reports saved from the report window, page 7-5
- how to generate Heat of Adsorption reports, page 7-6
- the appearance of reports, page 7-9
- manipulation tools for onscreen reports, page 7-9

This chapter also contains examples of some of the reports available for the ASAP 2020 Chemi analysis program beginning on page 7-19.

Description

Listed below are brief descriptions of the options contained on the Reports menu. Detailed descriptions follow this section.

Start Report — Allows you to generate a report on data for a completed sample analysis, a sample analysis that is in progress, or on manually entered data. Page 7-2.

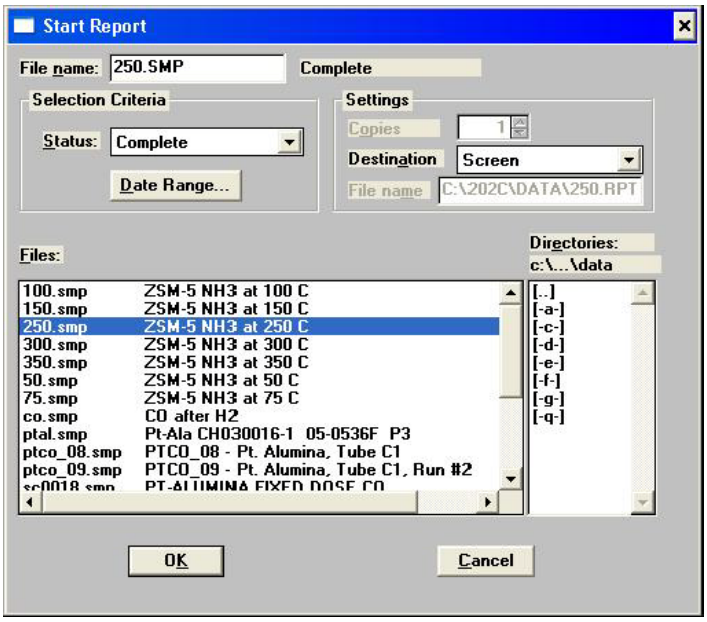
Close Reports — Closes all open report windows. Page 7-5.

Open Report — Enables you to open a report that was saved from the report window. Page 7-5.

Heat of Adsorption — Allows you to generate a heat of adsorption report. Page 7-6.

Start Report

Select this option to generate a report on a sample analysis; the Start Report dialog is displayed.



File name — The name you select from the Files list box is copied to this field. If you select multiple files, only the last one selected is displayed.

Status — This drop-down list determines the type of sample files displayed in the **Files** list window.

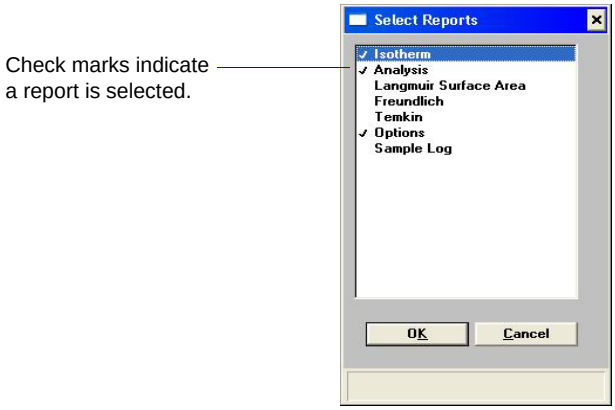
Date Range — Use this button to specify a range of dates in which to display sample files.

Settings — This group box contains print options.

Copies — Enabled when you select **Print** as the destination; you may print up to four copies.

Destination	Displays a drop-down list of output destinations. **Printer** generates reports to the default printer. **Screen** generates reports to the screen. You can print a paper copy from the report window. Using this option allows flexibility for manipulating and customizing reports. Refer to **Onscreen Reports**, page **7-9**. **File** prints tabular reports of the requested file to a text file which can be viewed with a text editor or other text file manipulation tool.
File name	Enabled when you select **File** as the destination. Allows you to enter a name, or you may accept the default.
Files list window	Displays a list of the available sample files for the choice shown in the **Status** field.
Directories	Displays a list of available drives and directories. The drive and directory last accessed is displayed immediately above the **Directories** list window.

If you choose a single sample file, the Reports to Generate dialog is displayed. The reports selected are the ones you specified in the sample file. This dialog allows you to deselect reports or select additional ones.



Double-click on the report(s) to select (or deselect). Alternatively, you can highlight the report and press the **Spacebar**. A report is selected when it is preceded with a check mark. After you make your report selection and click **OK**, the requested reports are sent to the specified destination.

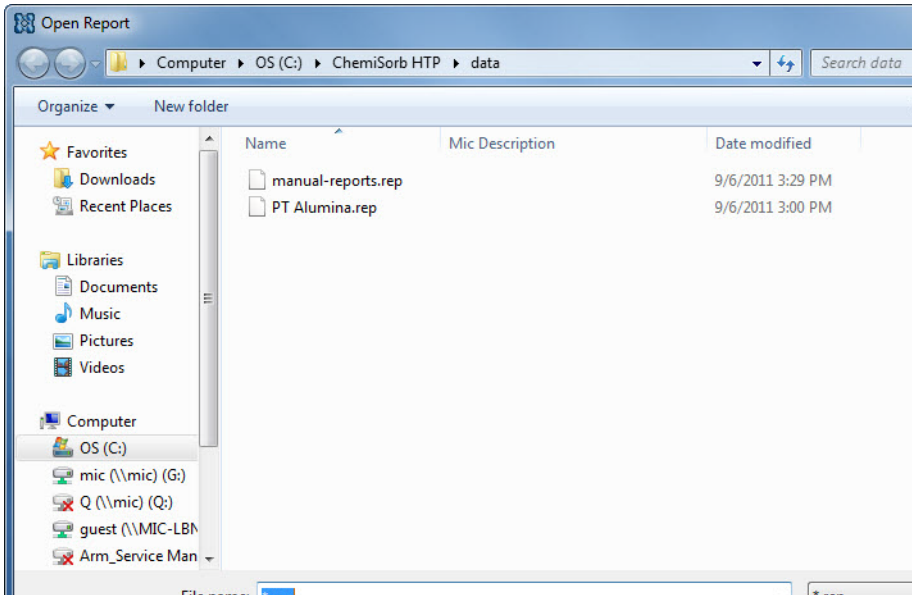
If you choose more than one file, this dialog is not displayed. The reports you specified in each sample file will be generated.

Close Reports

This option enables you to close all open report windows at one time. This avoids having to select close on each report window. This option is unavailable if reports are being generated.

Open Report

This option enables you to open a report that was saved from the Report window; the Open dialog is displayed.



After you navigate to the directory, select your file, and click **Open**, your report is displayed in the Report window.

Heat of Adsorption

The isosteric heat of adsorption is an important parameter for characterizing the degree of catalyst surface heterogeneity, providing information about catalyst activity under specific chemical reaction conditions. Multiple adsorption isotherms are obtained on the same sample using the same adsorptive but at different temperatures to obtain the heat of adsorption.

This option allows you to choose the sample files, define the quantities, and generate a Heat of Adsorption report; the Heat of Adsorption dialog is displayed.

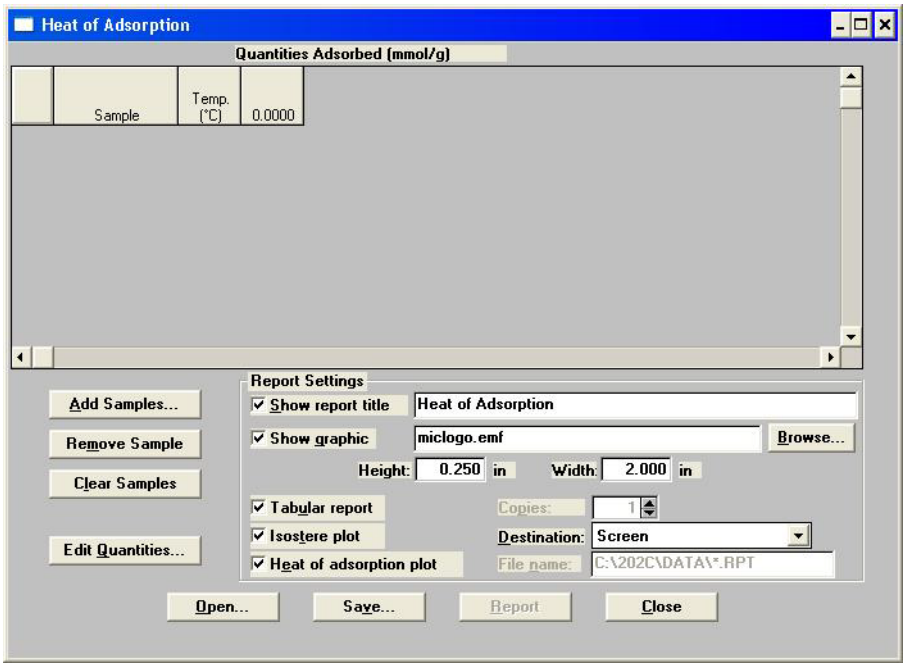


Table — Contains the files you choose; also lists the quantity adsorbed.

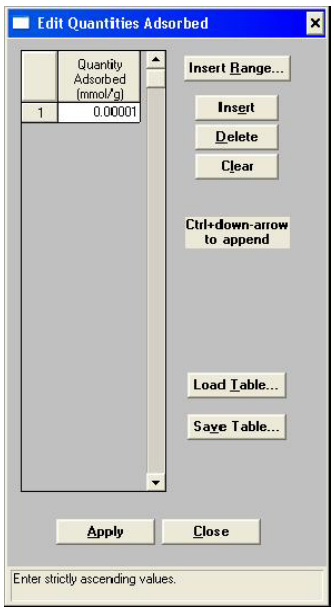
Add Samples — Displays the Select Samples dialog so that you can choose files.

Remove Sample — Removes the selected sample from the list. If no samples are selected, the last one is removed.

Clear Samples — Removes all samples from the list.

Edit Quantities

Displays the Edit Quantities Adsorbed dialog so that you can specify the range of surface coverage to include in the heat of adsorption report.

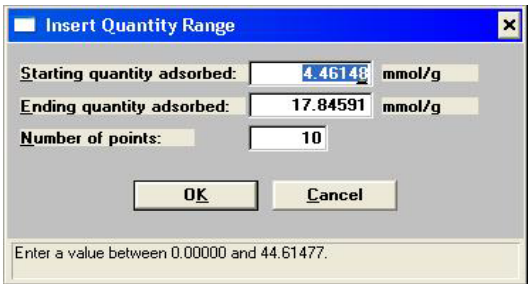


Quantity Adsorbed table

Allows you to enter the points.

Insert Range

Displays the Insert Quantity Range dialog.



This dialog allows you to specify the starting and ending quantities adsorbed, as well as the number of points to insert within the specified range.

Insert

Inserts a row above the selected row.

Delete

Deletes the selected row.

Clear	Clears the entire table of all entries except one; one is required.
Load Table	Allows you to import a previously saved table.
Save Table	Allows you to save the current table as a file (QNT).
Report Settings	The options in this group box allow you to choose report criteria.
Show report title	Choose this option to have a title display in the header of your report; use the adjacent field to enter the title.
Show graphic	Allows you to have a graphic appear in your title; for example, to show your company logo. You can use a bitmap (bmp) or an enhanced metafile (emf). Use the **Height** and **Width** fields to specify a size.
Copies	Enabled when you choose **Printer** as the destination, allowing you to print up to four copies of your report(s).
Destination	You can generate reports to the **Screen**, a **Printer**, or to a **File**.
File name	Enabled when you choose **File** as the destination, allowing you to enter a name (or you can accept the default).
Tabular report	Choose this option to have a tabular report generated.
Isostere plot	Choose this option to generate a graph showing quantities of gas adsorbed versus the temperature.
Heat of adsorption plot	Choose this option to have the heat of adsorption data plotted.
Open	Enables you to open a previously saved report.
Save	Saves the current report.
Report	Generates the report.

Printed Reports

Header

All printed reports (either to the screen or to a printer) contain a header displaying file statistics.

- Tabular and graphical reports contain sample and instrument statistics such as date and time of analysis, analysis conditions, background data, and so forth.

  The headers for these reports also contain notes of any changes to the sample file which occur after analysis.

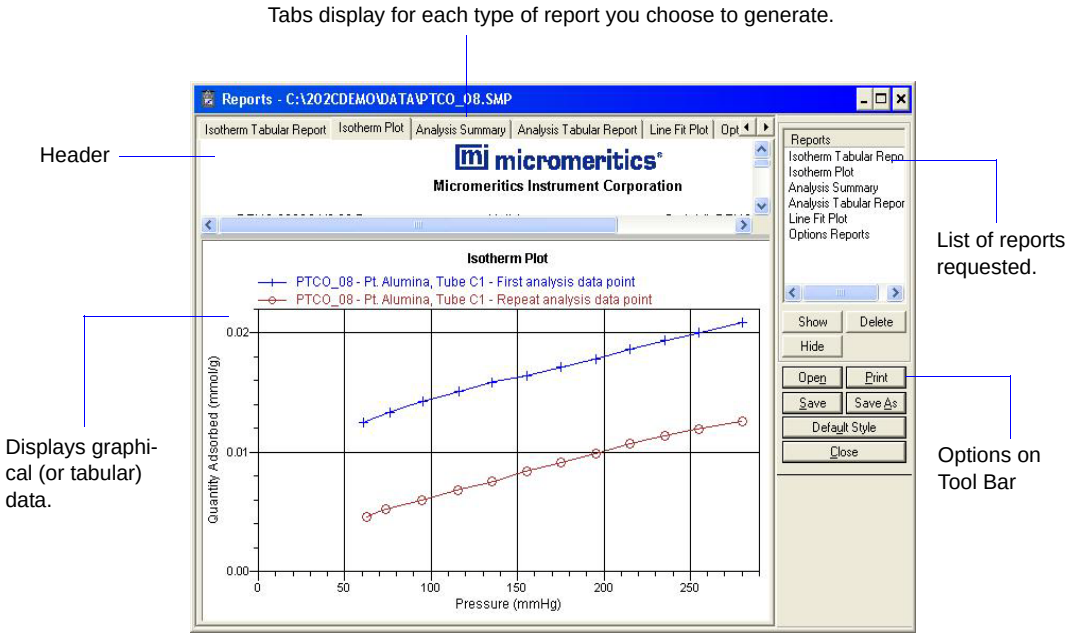
- Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.

Onscreen Reports

The report window containing onscreen reports provides many options for customizing and manipulating reports:

- a tool bar, page **7-10**
- shortcut menu, page **7-13**
- zoom feature, page **7-17**
- axis cross hairs, page **7-18**

When reports are printed to the screen, they are printed in a window like the one shown below. Each requested report is listed in the Reports window on the tool bar; they are also indicated by selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports window and click **Show**.



Tool Bar

Reports Contains a list of all requested reports.

Show Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible.

Delete Deletes the selected report. A deletion confirmation dialog is displayed since this function cannot be undone. The deleted report(s) will have to be regenerated if deleted in error.

Hide

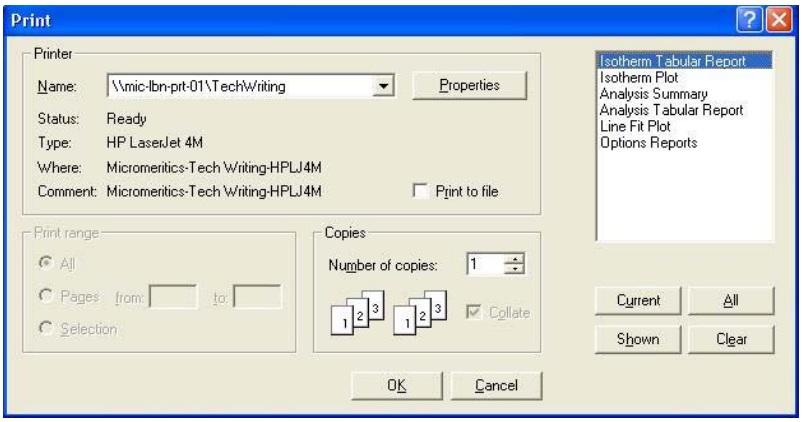
Hides (removes) the selected report and its tab from the report window (greys in the Reports list).

Open

Allows you to open a previously saved report file.

Print

Displays a print dialog so that you can choose an appropriate printer for report output. A list of available reports is displayed in the window on the right side of the dialog.



For convenience in selecting reports to print, buttons are provided beneath the report window. Or, you can make your selection by clicking on the reports.

Current selects the report displayed in the report window.

Shown selects only the shown reports; any unhighlighted reports indicate they are hidden. You can still select hidden reports from this window to print.

All selects all reports, including those that may have been hidden.

Clear clears all selections.

Save

Saves all reports of the currently open file in a report format using the same name as the sample file, only with an rep extension. To specify a name and/or specific reports to save, use the **Save As** button.

Save As

Allows you to save all or specified reports from the currently open file. The buttons displayed on this dialog function in the same manner as the print dialog (explained above).

Save As

(continued)

Allows you to save all or specified reports from the currently open file. The buttons displayed on this dialog function in the same manner as the print dialog (explained above).

Reports can be saved in three different formats:

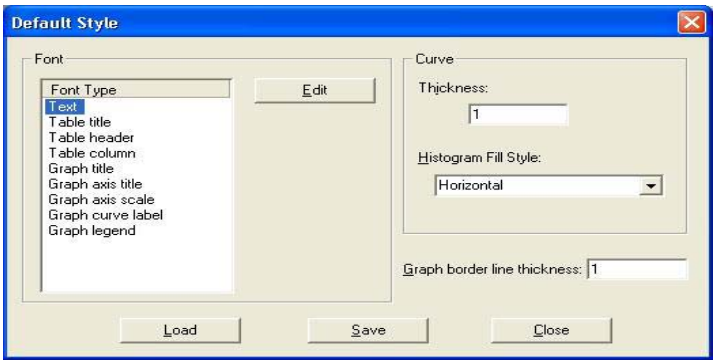
Report system (*.rep): Saved in a format which allows you to reopen the file using the button on the Report window tool bar.

Spreadsheet (*.xls): Saved in a format which can be imported into most spreadsheet programs.

Ascii Text (*.txt): Saved in ASCII text which can be imported into programs accepting this type of file.

Default Style

Displays the Default Style dialog so that you can specify default parameters for report fonts and curve properties.



Font

Contains a list of report elements for which the font can be edited. Simply highlight the element and click **Edit**; a font dialog enabling you to specify the font and attributes is displayed.

Curve

The items in this group box enable you to specify a thickness for report curves and, when using histograms, the type of fill to apply.

Graph border line thickness

Enables you to specify a thickness for the border of the graph.

Load

Loads the last saved defaults.

Save Saves the changes as the defaults. If you do not click **Save**, the changes will apply to the current report set only. The next reports will revert to the defaults.

Close Closes the Default Style dialog and applies the changes. If you clicked **Save**, the changes become the defaults. If you did not click **Save**, the changes apply only to the current report set.

Shortcut Menus

Shortcut menus are accessed when you right-click on the tabular or graphical portion of a report.

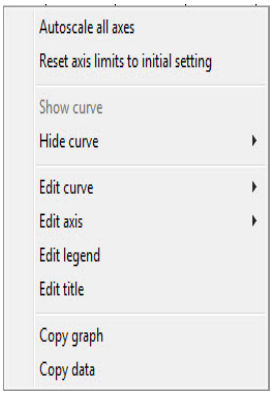
Tabular Reports

Resize column Displays a dialog so that you can specify the width of the selected column (in inches).

Rename column Displays a dialog so that you can edit the name of the selected column. Use **Ctrl + Enter** to insert line feeds.

Move column	Allows you to move the location of the selected column to the left or to the right.
Align column	Enables you to right-align, left-align, or center the data in the selected column.
Hide column	Displays a list of all columns, enabling you to select the one to hide.
Show column	Displays a list of all hidden columns, enabling you to select the one to have shown again.
Column font	Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.
Header font	Displays a Font dialog, allowing you to change font attributes for column headers in the current report.
Edit title	Allows you to edit the table title and font.
Copy table as text	Enables you to copy the entire table (column headers and data) and then insert it into another program. Columns are tab-delimited, allowing easy alignment.

Graphs



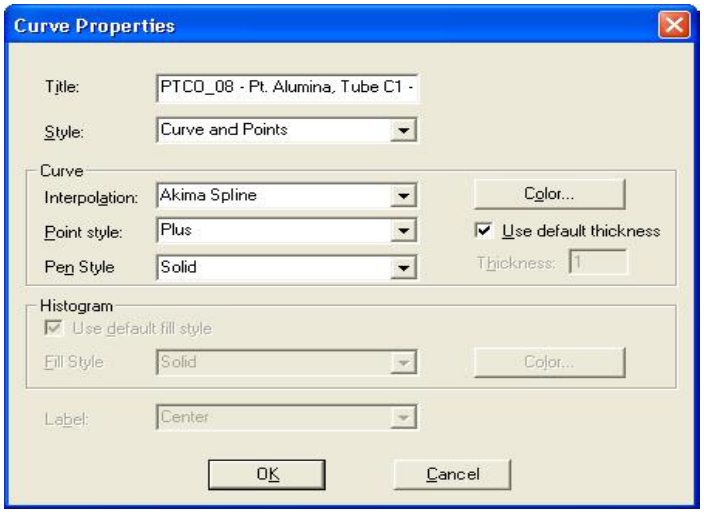
Autoscale Autoscales all axes of the graph. This option is useful for returning to a full view after a zooming operation.

Redraw Sets axis boundaries to its original view. You can also use this option to remove cross-hairs.

Show curve Allows you to show curves that have been hidden. This option is disabled (greyed) if no curves have been hidden.

Hide curve Allows you to hide (remove) unwanted curves.

Edit curve Displays the Curve Properties dialog, allowing you to edit curve properties.



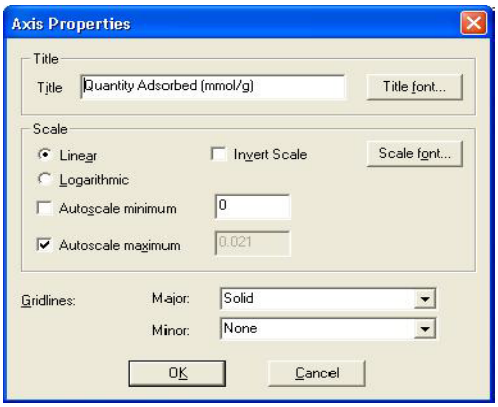
Title Displays the title of the curve you are editing.

Style Drop-down list containing styles in which collected data can be displayed.

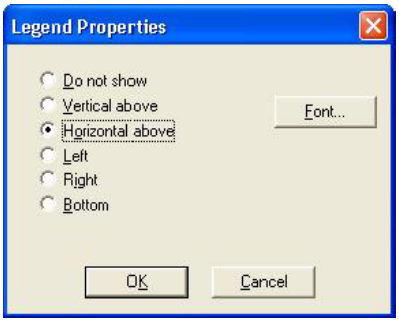
Curve group box Contains options for curves and points. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color. The options in this group box are disabled if Histogram is chosen in the **Style** drop-down list.

Histogram group box Allows you to specify the type of fill as well as the color if Histogram is chosen as the style for collected data.

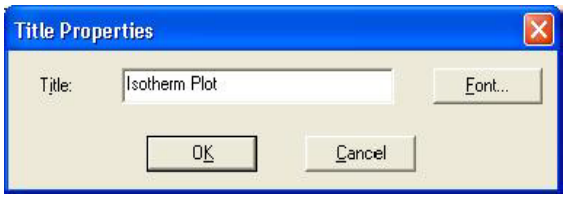
Edit axis Displays the Axis Properties dialog, allowing you to edit axis properties.



Edit legend Displays the Legend Properties dialog, allowing you to edit the placement of the legend.



Edit title Displays the Title Properties dialog, allowing you to edit the current graph's title and font.



Copy Graph Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

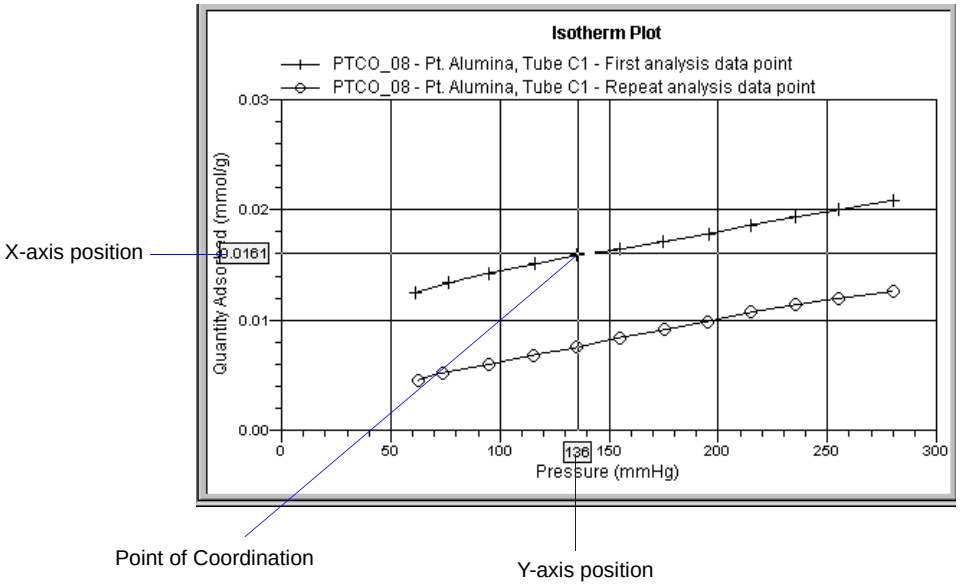
Copy Data Copies the data used to generate the graph as a series of tab-delimited columns of text.

Zoom Feature

A zoom feature is included with the report system so that you can zoom in to examine fine detail of the distribution. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a rectangle) across the area to view; then release the button. The enlarged area immediately fills the graph area. You can return to the normal view by right-clicking on the graph and selecting Autoscale.

Axis Cross Hair

A cross-hair function is available so that you can view axis coordinates. To use this feature, simply left-click in the area of the graph.



Right-click in the graph area and select **Autoscale** or **Redraw** from the shortcut menu to return to the normal view; alternatively, you can click outside of the graph area.

Report Examples

This section contains examples of the types of reports available in the ASAP 2020 Chemi analysis program.

Isotherm Report

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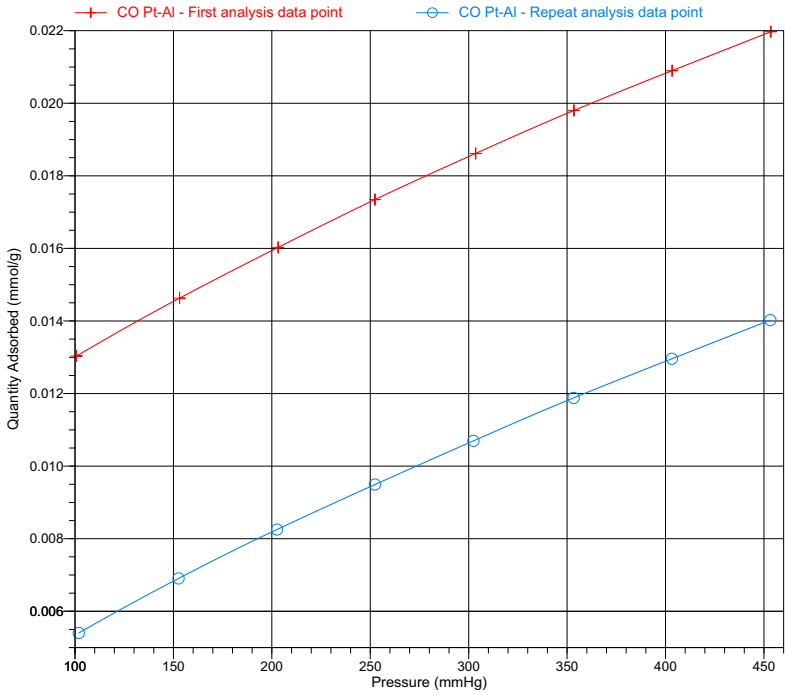
2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Isotherm Plot



Analysis Report

The Analysis report provides a summary of analysis statistics, a tabular report, and a line fit plot; the line fit plot is shown here.

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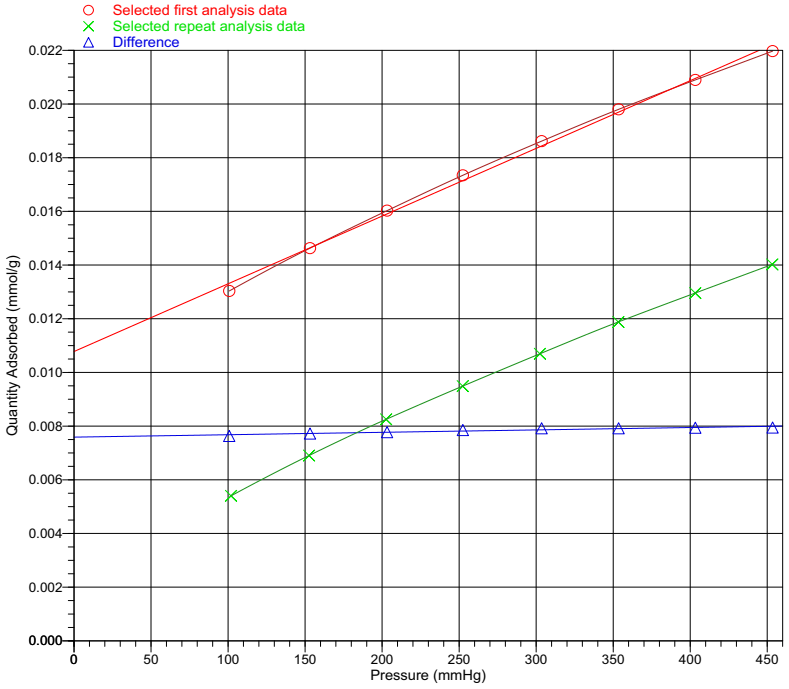
2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Line Fit Plot



Langmuir Surface Area Report

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Micromeritics Instrument Corporation

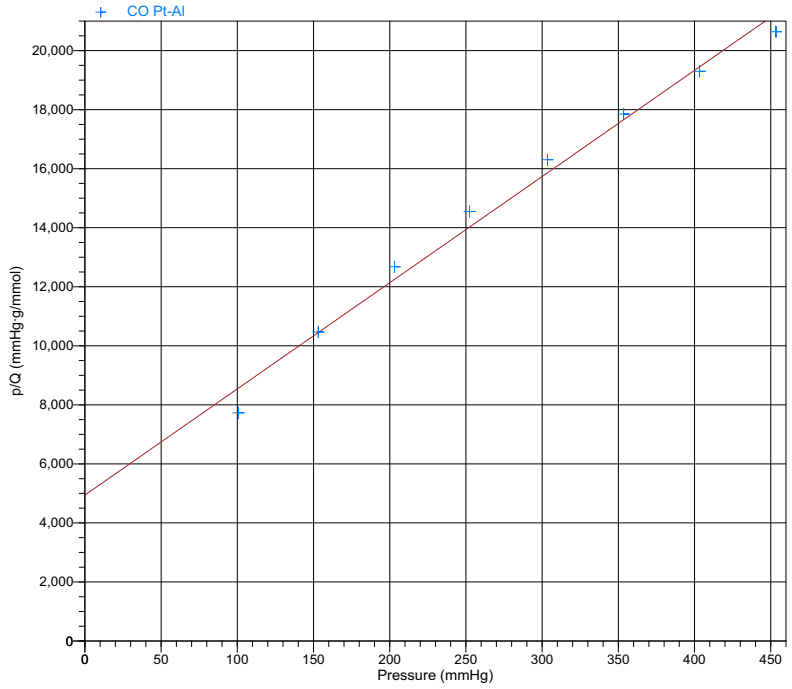
2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Langmuir Surface Area Plot



Freundlich Report

micromeritics®

Micromeritics Instrument Corporation

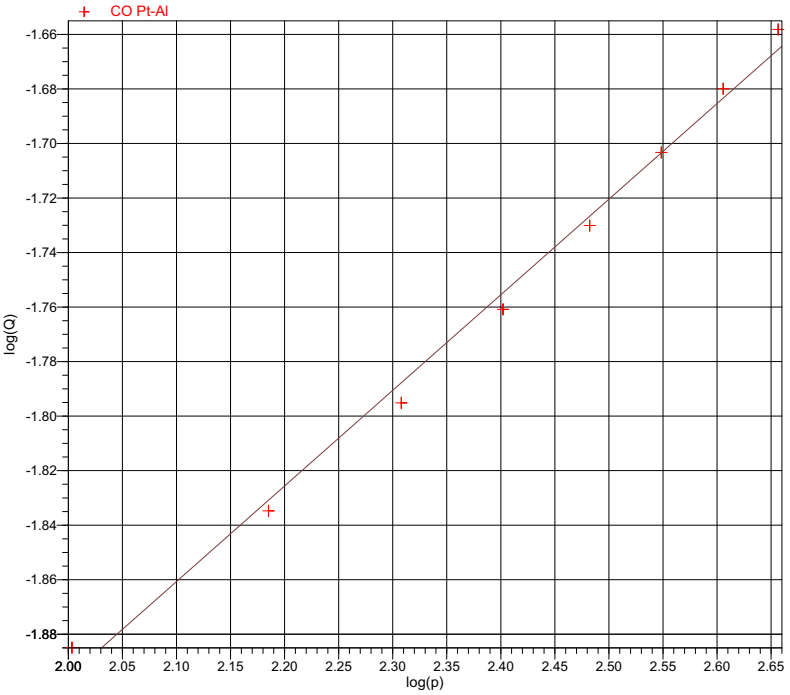
2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Freundlich Plot



Temkin Report

micromeritics®

Micromeritics Instrument Corporation

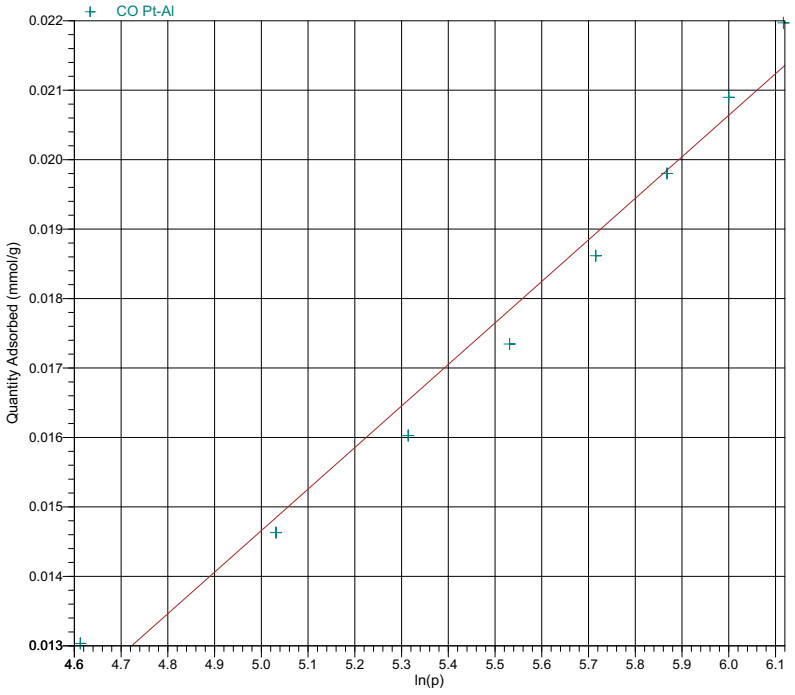
2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Temkin Plot



Options Report

This example shows the first page of a 2-page Options Report.

micromeritics®

Micromeritics Instrument Corporation

2020C V2.00 H Unit 1 Serial #: 1179 Page 1

Sample: CO Pt-Al
Operator: jch
Submitter: PT
File: F:\...\DATA\000-005.SMP

Started: 10/10/2010 1:42:06PM
Completed: 10/10/2010 11:30:11PM
Report Time: 10/29/2010 11:21:53AM
Sample Mass: 0.8453 g
Measured free space: 6.9283 cm³

Analysis Gas: CO
Analysis Temp: 307.1 K
Equilibration Interval: 20 s
Low Pressure Dose: None
Automatic Degas: No

Options Report

Task Summary

Task Number	Task Name	Gas	Temp (K)	Rate (K/min)	Time (min)	Pressure (mmHg)
1	Evacuation	He	383	10.0	30	
2	Flow	H2	373	10.0	10	
3	Flow	H2	623	10.0	120	
4	Evacuation		623	10.0	120	
5	Evacuation		308	100.0	10	
6	Leak Test		308	10.0		
7	Evacuation		308	10.0	20	
8	Analysis	CO	308	10.0		

Task Number: 8
Adsorptive: Carbon Monoxide
Temperature: 308.1 K
Heat rate: 10.0 K/min
Equilibration interval: 20 s
Relative target tolerance: 2.5 %
Absolute target tolerance: 2.500 mmHg

Repeat analysis: Yes
Fast evacuation: Yes
Unrestricted evac. pressure: 30.0 mmHg
Evacuation time: 30 min

Free space group: Measured
Estimated free space: 7.7000 cm³

Incremental dosing: No

Experiment Log

Task Number	Task Name	Start Time (h:min)	Gas	Furnace Temp. (K)	Sample Temp. (K)	Time (min)	Pressure (mmHg)
1	EVAC	0:02	Hel	403.5	382.8	30	
2	FLOW	0:53	H2	391.8	372.7	10	777.8066
3	FLOW	1:07	H2	662.1	622.7	120	778.5252
4	EVAC	3:36		662.8	622.6	120	
5	EVAC	5:38		308.4	308.7	10	
6	LEAK	6:18		307.9	308.1	1	0.0898
7	EVAC	6:22		309.6	308.2	20	
8	ANL	6:42	CO	308.5	307.3	184	516.0168

8. OPTIONS MENU

The ASAP 2020 Chemisorption program provides various options that enable you to configure the system to your laboratory's requirements. You can select report formats, enter a default sample information file, and enter information to be printed in the free form area of reports. These options are selected from the Options menu.

Description

Option Presentation — Allows you to display the sample file dialog in Advanced, Basic, or Restricted mode. Page **8-3**.

Sample Defaults — Enables you to enter default sample information file values. When the system automatically generates a new sample information file name, the file contains the default values. Page **8-6**.

Active Metals Defaults — The Active Metals Table Defaults dialog enables you to specify options for the chemisorption elements. Page **8-12**.

Gas Defaults — The Gas Table Defaults dialog enables you to specify options for the adsorptives. Page **8-14**.

Units — Allows you to choose the types of units to use for measurement, pressure, and temperature. Page **8-15**.

Graph Grid Lines	Enables you to choose the types of grid lines to display for the X- and Y-axes. Page **8-15**.
Parameter Files Directory	Allows you to specify a location for the parameter files used in the Basic sample file editor. Page **8-16**.
Service Test Mode	Enables you to perform certain troubleshooting procedures. This option is available only under the direction of a Micromeritics service representative. Page **8-16**.

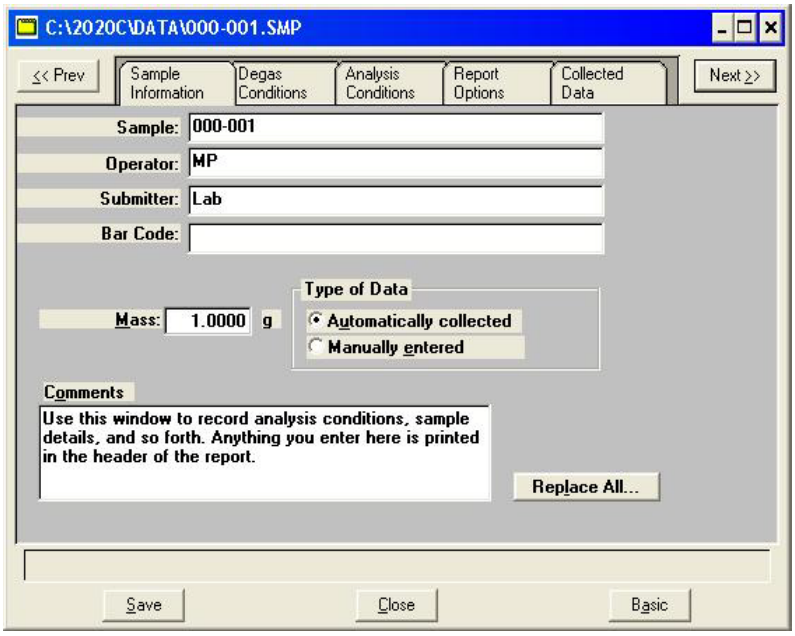
Option Presentation

The sample editing dialogs for the ASAP 2020 Chemi analysis program may be presented in three modes: Restricted, Basic, and Advanced. Each format displays sample information and menu options differently.

- **Advanced**: displays all parts of the sample information file in a tabbed dialog similar to that of an index card file. You just click on the tab of the file to edit. This format also allows you to switch to the Basic mode.
- **Basic**: displays all parts of the sample file as a single dialog. You select from predefined parameter files. This format also allows you to switch to the Advanced mode if editing of parameters is needed.
- **Restricted**: displays in the same manner as the Basic format. Certain menu options, however, become disabled and you cannot switch to the Advanced mode. You also must enter a password to access and exit this format.

Advanced

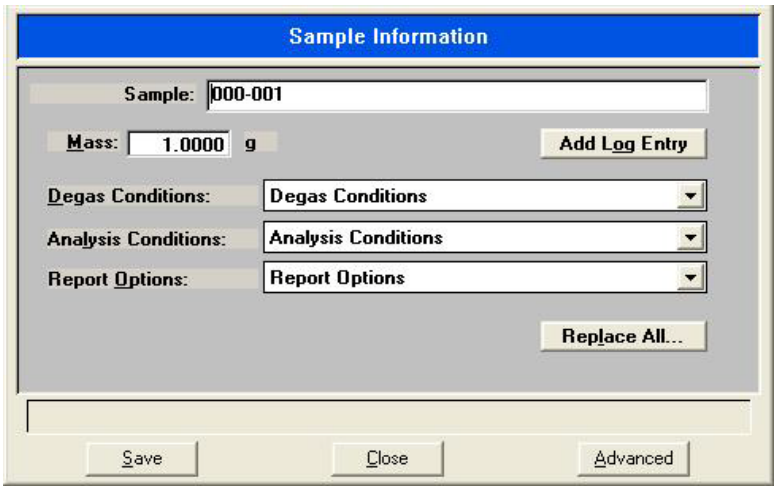
The Advanced format presents all parts of the sample information file in a tabbed dialog. Each tab opens an associated dialog. For example, if you are using the Advanced format and you open or create a sample information file, the following dialog is displayed.



The Advanced format can be used to edit sample parameters and customize files.

Basic

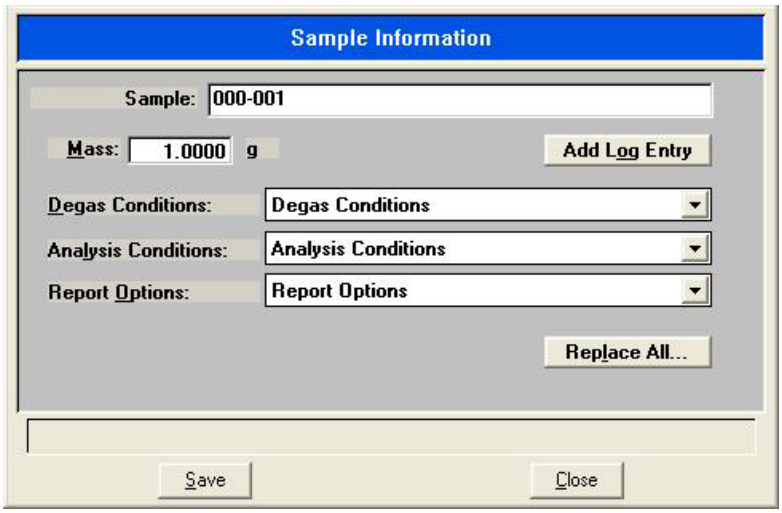
The Basic format presents the sample information file and its parameter files as a single dialog. You also have access to all menu options. For example, if you are using the Basic format and open or create a sample information file, the dialog is displayed in this manner.



The Basic format is used to quickly create sample information files using previously defined parameter files. You also can easily switch to the Advanced format to view or edit details of parameter files.

Restricted

The Restricted format is identical to the Basic format, except that certain options are disabled. You also cannot switch to the Advanced format. This format is password-protected and is typically used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.



When you select Restricted, a dialog prompting you to enter a password is displayed.

You can enter any password to enable the Restricted format. You must enter the same password to exit the Restricted format. For example, if you enter “password” to enable the Restricted format, then you must enter “password” to exit. If you forget the password, open the system INI file and navigate to the Private section. The current password is shown immediately following “OptionPresentationPassword.” Make a note of the password, exit the INI file, and enter the password where requested. Deleting the password from the INI file will not disable the Restricted mode; you must enter the password using the Password dialog to exit the Restricted format.

Sample Defaults

Sample defaults are the values you see in the sample information editors when you create a new sample file. This option allows you to specify the default values. This feature makes it easy for you to apply the same conditions to many samples.

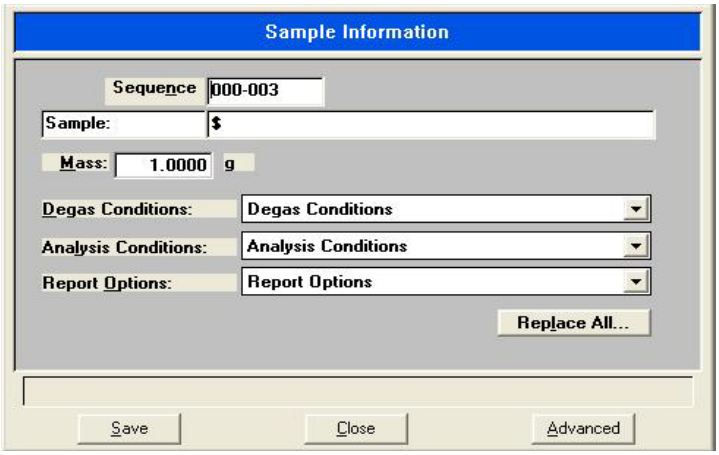
For efficiency, it is best to specify defaults for materials you most commonly analyze. You can always edit the values in the sample file when it is created.

The analysis program contains one complete default sample information file. When you select Open, Sample Information from the Main Menu, a new file with these default values is generated. Default files for analysis conditions, adsorptive properties, and reports options also are included. Details of system default files are found in Appendix E.

Sample defaults can be specified using the Advanced or Basic format. The Advanced or Basic format is chosen by selecting Options, Option Presentation from the main menu.

Basic

When you select Sample Defaults while using the Basic format, the Basic Sample Defaults dialog is displayed.



Sequence Allows you to specify a default sequence for the sample file name. The number you specify is incrementally sequenced each time you create a sample file. It is the number that appears in the **File name** field when you select **File > Open > Sample information**.

Sequence

(continued)

- Use numbers, letters, or other printable characters, such as dashes. At least three numbers must be included.
- Do not use characters such as * or ?.

Sample

Allows you to enter an additional identification that provides more information than the sample file name itself.

In the field on the left, you can edit the prompt for **Sample**. For example, you may prefer to use **Test** or **Material**.

In the field on the right, specify a format for the sample identification.

- Use numbers, letters, or other printable characters, such as dashes.
- Maximum number of characters is 42, plus the $ symbol.
- Include the automatically generated file name as part of the identification by using the $ symbol where you want the sequence number to appear.

  For example, if the sequence number is 000-001, enter the identification as follows:

  Lab #25 - $

  The resulting sample identification for the first sample information file would be:

  Lab #25 - 000-001

  for the second file:

  Lab #25 - 000-002, and so on.

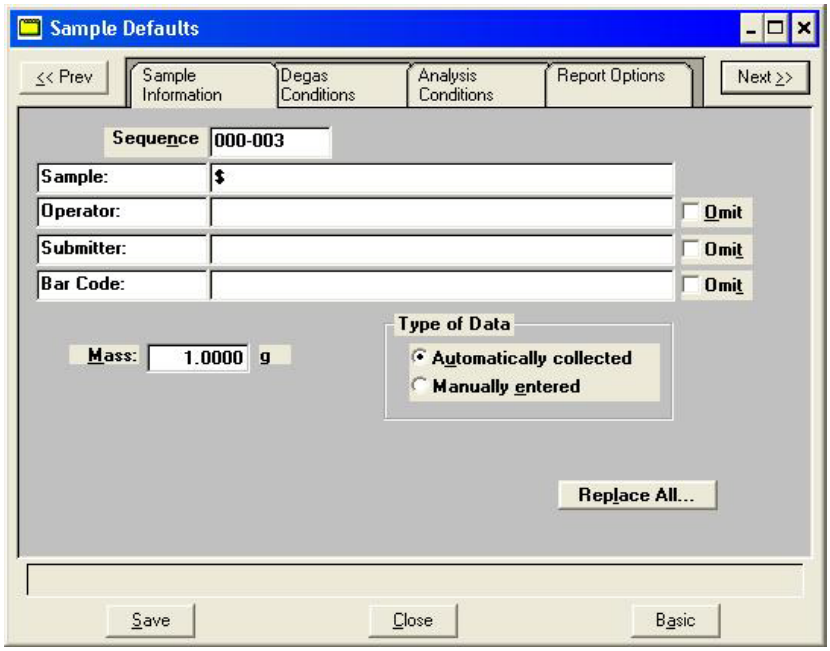
Mass

Enter a value that represents the mass of your most commonly analyzed material. This value can be edited in the sample file at the time of analysis.

Degas Conditions **Analysis Conditions** **Report Options**	Each parameter has a drop-down list containing predefined parameter files. Several default files are included with the analysis program. For files to appear in this drop-down list, they must be saved to the Parameter Files directory; the default directory is params. Refer to **Parameter Files Directory**, page **8-16** for additional information.
Replace all	Use this button to replace the current default values with those from an existing sample file.
Save	Saves the current definition as the defaults.
Close	Closes the dialog.
Advanced	Switches the sample editor to the Advanced format.

Advanced

When you select Sample Defaults while using the Advanced format, the Advanced Sample Defaults dialog is displayed.



Sequence

Allows you to specify a default sequence for the sample file name. The number you specify is incrementally sequenced each time you create a sample file. It is the number that appears in the **File name** field when you select **File > Open > Sample information**.

- Use numbers, letters, or other printable characters, such as dashes. At least three numbers must be included.
- Do not use characters such as * or ?.

Sample

Allows you to enter an additional identification that provides more information than the sample file name itself.

In the field on the left, you can edit the prompt for **Sample**. For example, you may prefer to use **Test** or **Material**.

Sample

(continued)

In the field on the right, specify a format for the sample identification.

- Use numbers, letters, or other printable characters, such as dashes.
- Use up to 42 characters, plus the $ symbol.
- Include the automatically generated file name as part of the identification by using the $ symbol where you want the sequence number to appear.

  For example, if the sequence number is 000-001, enter the identification as follows:

  Lab #25 - $

  The resulting sample identification for the first sample information file would be:

  Lab #25 - 000-001

  for the second file:

  Lab #25 - 000-002, and so on.

Operator
Submitter
Bar code

These fields enable you to enter defaults for the operator, submitter, and bar code information.

The fields on the left can be edited to display a different label for the prompts.

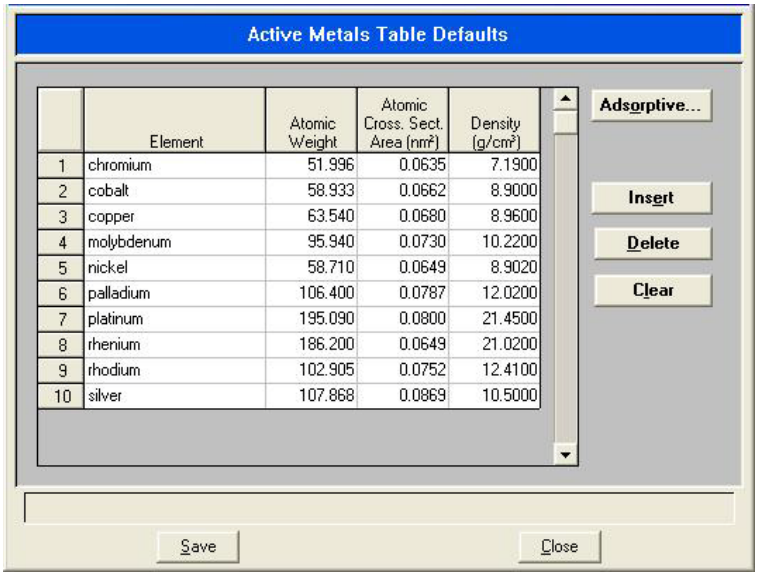
The fields on the right allow you to specify default names or titles, and bar code information.

- Include the automatically generated file number/name as part of the identification by using the $ symbol where you want the name to appear.
- Use up to 43 characters (including the $ symbol) for the identification.
- Select **Omit** adjacent to any field to omit from displaying on the sample information dialog.

Mass	Enter a value that represents the mass of your most commonly analyzed material. This value can be edited in the sample file at the time of analysis.
Type of Data	This group box allows you to choose the manner in which data are collected. Select **Automatically collected** if you want the ASAP 2020 Chemi System to collect data during analysis; this is the standard setting. Select **Manually entered** if you plan to enter pressure data collected from another source.
Comments	Use this window to enter sample characteristics, analysis conditions, etc. Anything you type in this window is printed in the report header.
Replace all	Use this button to replace the current default values with those from an existing sample file.
Save	Saves the current values as the defaults.
Close	Closes the dialog.
Basic	Switches the sample editor to the Basic format.

Active Metals Defaults

This option enables you to specify characteristics of up to twenty elements. You must enter at least one element in the table.



Element — Displays the element. You may insert or delete as needed. Refer to **Appendix F**, page **F-1** for a list of elements. This appendix provides a list of elements, their atomic weights, cross-sectional area, and density.

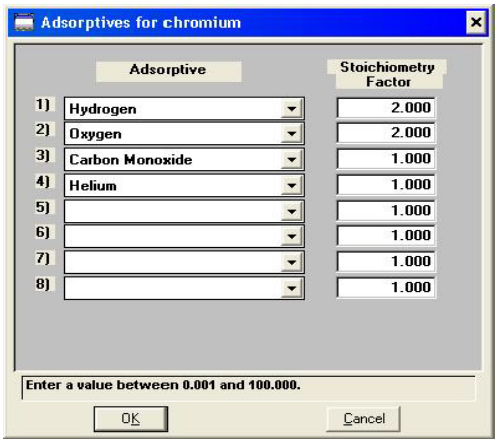
Atomic Weight — The atomic weight of the element.

Atomic Cross. Sect. Area (nm2) — The atomic cross-sectional area of the element.

Density — The density of the element.

Adsorptive

Enables you to specify adsorptive options for a selected element; displays the Adsorptives dialog for the selected element (in this example, cobalt is shown).



Adsorptive

Gases shown in the list are the ones specified in the Gas Table Defaults dialog. Refer to **Gas Defaults**, page **8-14** for information on adding gases.

Stoichiometry Factor

A factor which expresses the ratio between the number of active metal molecules and the number of adsorbate molecules. This value is used in report calculations but not in the analysis itself.

Insert

Inserts a row above the selected row, allowing entry of a new element to the table.

Delete

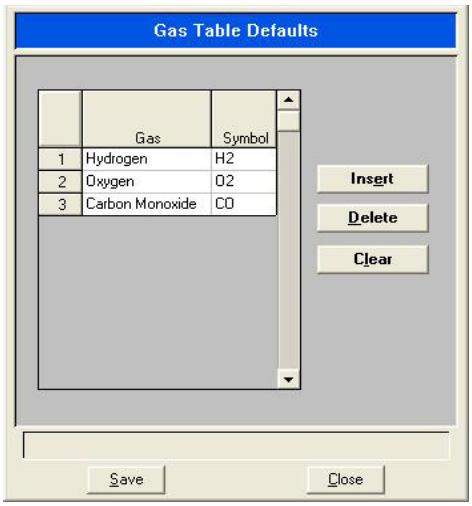
Deletes the selected row.

Clear

Clears the table of all entries except one; one is required.

Gas Defaults

This option enables you to specify the gases that appear in the drop-down list of the Gas Configuration dialog; the Gas Table Defaults dialog is displayed.



Gas — Provides a list of gases; at least one adsorptive gas must be present in the table.

Even though helium may not appear on the list, it is always available as a default gas.

Symbol — The mnemonic for the adsorptive gas.

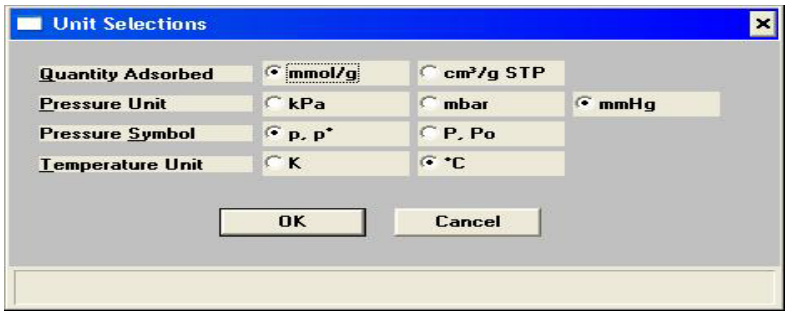
Insert — Inserts a row above the selected row, allowing entry of a new gas to the table.

Delete — Deletes the selected row.

Clear — Clears the table of all entries except one; one is required.

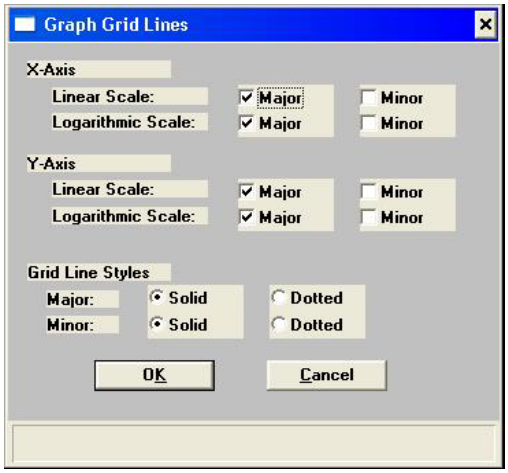
Units

This menu command displays the Units Selections dialog which allows you to choose the manner in which to display data on dialogs and in reports.



Graph Grid Lines

Graph Grid Lines enables you to choose the type(s) of grid lines to show on your reports; the Graph Grid Lines dialog is displayed.



X-Axis
Y-Axis

Enables you to choose **Major** and/or **Minor** lines to display in printed reports for the Logarithmic and Linear scales.

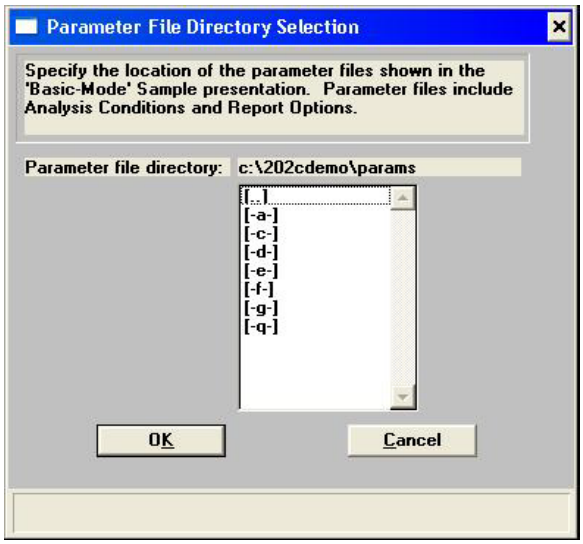
If you deselect these items (remove the check marks), your report will not display grid lines.

Grid Line Style

Allows you to choose the type of grid line to display if grid lines are being shown.

Parameter Files Directory

This option allows you to specify a location for the predefined Degas conditions, Analysis conditions, and Report options files displayed in the drop-down lists on the Basic and Restricted Sample Information dialogs.



The default directory is **params** and includes several parameter files supplied with the analysis program. If you specify a different directory, these files will not be included in the drop-down lists unless you copy (or move) them to the new directory.

To continue using the params directory for parameter files, be sure to save any files you create to this directory; otherwise they will not be included in the drop-down lists of the Basic and Restricted sample file editors.

Service Test Mode

Various service tests are included in the ASAP 2020 Chemi operating program. These tests can be performed only with the assistance of a trained Micromeritics service representative. When you select Service Test Mode from the Options menu, a dialog prompting you to enter a password is displayed. This password is coded to change on a regular basis. You must contact your local service representative to obtain the correct password. You will not be able to perform these tests without his guidance.

9. TROUBLESHOOTING AND MAINTENANCE

The ASAP 2020 Chemi System has been designed to provide efficient and continuous service. However, to get the best results over the longest period of time, certain maintenance procedures must be followed. This chapter describes recommended maintenance procedures and includes troubleshooting information.

Troubleshooting

Most operational problems are caused by leaks (commonly around the sample tube O-ring at the analysis port), sample weighing errors, or entry of incorrect system volume for analysis. Always check these first when expected analysis results are not obtained. Some common operational problems and their respective causes and solutions are provided in Table 9-1.

Table 9-1. Troubleshooting Chart

What Happened	Why	What To Do
Furnace used for analysis cannot be raised (or lowered).	Elevator that moves furnace stuck in up position, down position, or somewhere in between.	Check for possible obstruction to elevator movement.
Vacuum pump gurgles continuously.	Sample tube or cold trap tube O-ring or fitting loose.	Tighten fitting. Replace O-ring.
	Sample tube cracked.	Replace with new sample tube.
	No sample tube loaded on a selected port.	Install plug or empty sample tube.
	Gas inlet valve open while vacuum valve open.	With manual control enabled, use the instrument schematic to close gas inlet valve.
Valves cannot be operated.	Circuit opened by circuit breaker.	Reset breaker (depress breaker button) located on the side of the instrument (refer to **Side Panel**, page **3-5**). If it does not stay in or if it continues to trip, contact appropriate service personnel.
	Cable from computer to the instrument is loose.	Make sure the cable is seated properly.

What Happened	Why	What To Do
Vacuum gauge shows reading above 20 µmHg, even after extended pumping through unrestricted valve with analysis or degas ports closed.	Vacuum pump turned off or unplugged.	Check pump power plug and power switch.
	Vacuum pump oil is low, causing ineffective evacuation.	Add or change vacuum pump oil. Be sure to add oil to proper level according to indicator window on the pump.
	The alumina in the oil vapor trap is holding moisture.	Replace or dry the alumina. Refer to **Replacing the Alumina in the Oil Vapor Trap**, page **9-11**.
	Cold trap obstructed by condensation.	Clean the cold trap tube. Refer to **Cleaning the Cold Trap Tubes**, page **9-17**.
	Filter in port being used is dirty.	Replace filter in port. Refer to **Replacing the Port Filters**, page **9-4**.
	Leak in vacuum plumbing.	Check and tighten all connections in vacuum plumbing, including cold traps.

Preventive Maintenance

Table 9-2 lists the preventive maintenance procedures you should perform to keep your analyzer operating at peak performance. Instructions for each procedure follow the table.

Table 9-2. Preventive Maintenance Schedule

Maintenance Required	Frequency
Clean the analyzer, page **9-6**	As required or every 6 months
Lubricate elevator screw, page **9-4**	As required or every 6 months
Replace port filters, page **9-4**	Every 30 days
Replace vacuum pump exhaust filter, page **9-6**	Annually (heavy use may require more frequent maintenance.)
Inspect and change vacuum pump fluid, page **9-8**	As required or every 3 months
Replace alumina in oil vapor traps, page **9-11** (if installed)	As required or every 3 months
Clean cold trap tubes, page **9-17**	As required or every 3 months
Calibrate manifold temperature sensor, page **9-16**	Every 12 months
Test analyzer for leaks, page **I-1** (Appendix I)	As required or every 12 months
Perform reference material run	As required or every 3 months

Lubricating the Elevator Screw

Apply a light coat of lithium grease to the elevator screw as needed.

Replacing the Port Filters

A 20-µm filter is located in the analysis and exhaust ports, and in each degas port of the analyzer.

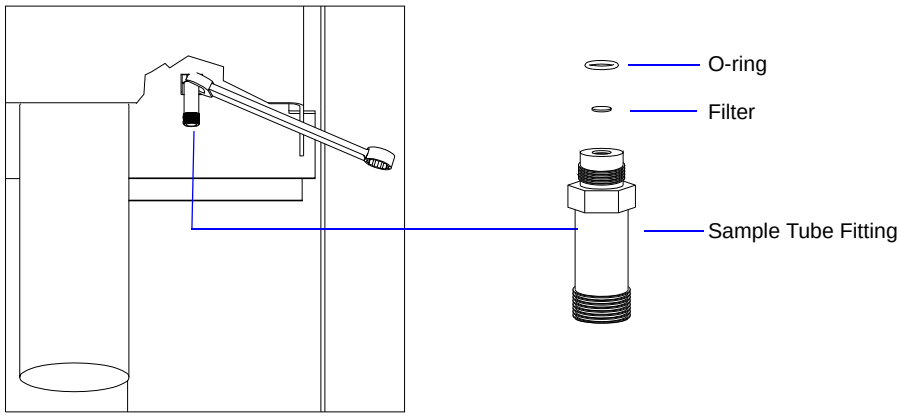
Analysis Port

Using a contaminated filter on the analysis port may extend the time required to achieve a vacuum at that port. More importantly, the contaminant may adsorb or desorb during analysis, affecting the analysis results. A contaminated filter on the analysis port may be detected by a leak test (if the contaminant outgasses) or by a free space reading much lower than normal.

Perform the following steps to replace the filters:

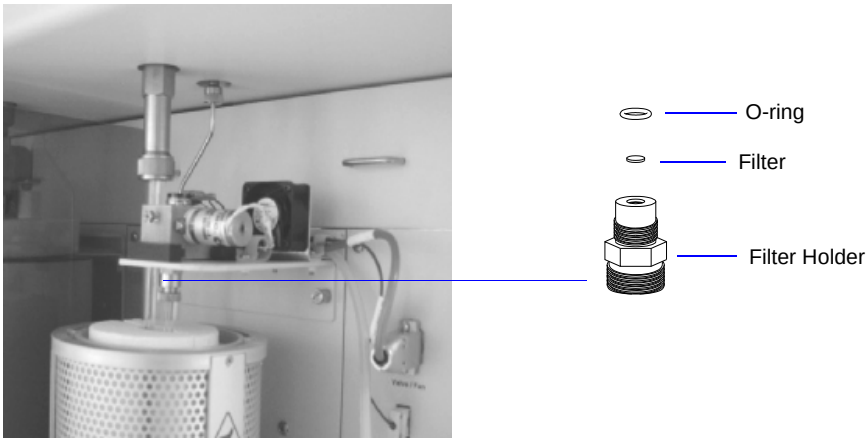
1. Make sure the furnace and sample tube or plug are removed. Make sure the analysis port (sample valve) is closed.

2. Using a wrench, remove the sample tube fitting from the manifold connector and remove the filter and O-ring.



3. Replace the filter and the O-ring. Carefully reassemble the sample tube fitting, filter, O-ring and manifold connector, and tighten by hand. Then tighten with the wrench to prevent leaks when evacuated.

Exhaust Port



1. Using a 9/16-in. wrench, unscrew the filter holder from the exhaust port block.
2. Remove the filter and O-ring by pushing them out from the opposite end of the filter holder.
3. Clean the filter holder in acetone or alcohol. The filter may be similarly cleaned or replaced with a new filter.
4. Inspect the O-ring; replace if damaged.
5. Reassemble the filter, O-ring and filter holder, and reinstall them in the exhaust port block.

Degas Port

If a filter on a degas port is contaminated, the contaminant may adsorb atmospheric gases when the port is not plugged (with either a sample tube or a plug), resulting in extended degassing time for samples on that port.

Perform the following steps to replace the filter:

1. Using a wrench, remove the degas port fitting and remove the filter and O-ring.
2. Replace the filter and the O-ring. Carefully reassemble the sample tube fitting, filter, O-ring and manifold connector, and tighten by hand. Then tighten with the wrenches to prevent leaks when evacuated.

To avoid degassing problems, the new filter and O-ring must be clean. Do not touch them with bare hands.

Cleaning the Analyzer

Use a clean cloth dampened with isopropyl alcohol, a mild detergent in water, or a 3% hydrogen peroxide solution to clean the outside casing of the analyzer.

Use only a mild detergent in water to clean the shields; do not use isopropyl alcohol.

Do not use isopropyl alcohol to clean the shields. Isopropyl alcohol could damage the surface of the shield.

Testing the Analyzer for Leaks

Refer to Appendix I, page **I-1** for instructions on testing the ASAP 2020 Chemi for leaks.

Replacing the Vacuum Pump Exhaust Filter

This procedure does not apply, and is not required, for dry vacuum pumps.

The gases used by the ASAP 2020 Chemi System are exhausted by vacuum pumps. If no exhaust line is connected, an exhaust filter is installed on the exhaust port of each oil-based vacuum pump. The filter minimizes the release of oil vapor. The gases used are diluted substantially upon being released from the vacuum pumps. However, it may be desirable in some locations to provide a fume hood for added protection from hazardous gases and vapors released into the work area. Chemisorption instruments are provided with a hose connector which may be used to connect the analysis vacuum pump directly to a ventilation system.

Exhaust filters are used on the vacuum pump to minimize the release of oil vapors. The gases used are diluted substantially upon being released from the vacuum pumps. However, it may be desirable in some locations to provide a fume hood for added protection from hazardous gases and vapors released into the work area.

Perform the following steps to replace the filter:

1. Using a flat-blade screwdriver, remove the screws from the vacuum pump panel on the front of the analyzer, then remove the panel to access the pump.

2. Loosen the wing nut of the clamp at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.

3. Remove and discard the exhaust filter; do not remove the centering ring.

4. Make sure the centering ring is in place on the exhaust port.

5. Place the new filter on the centering ring.

6. Open the clamp and place it around the flange of the exhaust port and the flange of the exhaust filter. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

7. Reinstall the vacuum pump panel.

Inspecting and Changing Vacuum Pump Oil

This procedure does not apply, and is not required, for dry vacuum pumps.

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

Inspecting the Oil

1. Remove the vacuum pump panel (located on the left front side of the analyzer).

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

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

Changing or Adding Oil

Use oil supplied by Micromeritics, or refer to the manual for the vacuum pump for other acceptable oils.

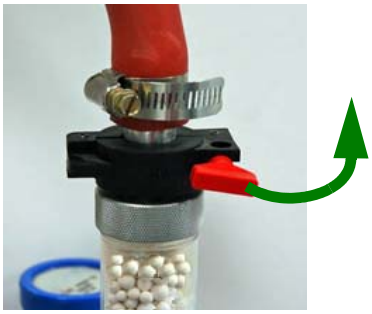
Always drain the vacuum pump while the pump is warm and switched off (unplugged).

1. Place the analyzer ON/OFF switch in the OFF position.

2. Remove the vacuum pump panel (if not already removed).

1. Unplug the vacuum pump from the power source.

2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.



3. Grasp the handle on top of the vacuum pump and place it on a work table.

4. Drain the used oil:

If you are adding oil, skip this step and continue with Step 5.

a. Place a waste container under the drain spout.

b. Remove the plug from the drain spout; allow the oil to drain into the waste container.

d. Replace the drain plug.

5. Remove the plug from the oil-fill port.

6. Slowly add oil to the port until the level is midway between the indicator lines in the oil-level window.

Do not allow oil to rise above the midway position. Doing so may cause oil to splash into the oil filter and contaminate it.

7. Insert the oil-fill plug and turn counterclockwise to tighten.

8. Check the alumina in the oil vapor trap. If most of the pellets are no longer white, replace the alumina in the oil vapor trap before reattaching the vacuum pump. Refer to **Replacing the Alumina in the Oil Vapor Trap**, page **9-11** for instructions.

9. Reconnect the vacuum pump hose.

10. Reconnect the power cord to the power source.

11. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

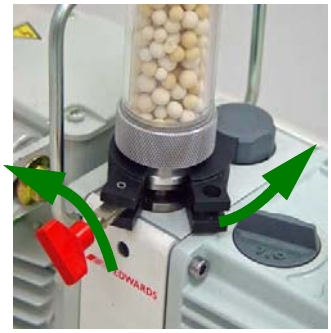
Replacing the Alumina in the Oil Vapor Trap

The activated alumina in the oil vapor trap becomes saturated during use. The alumina should be inspected periodically and replaced when most of the alumina pellets are no longer white.

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire or an explosion.

1. Disconnect the vacuum pump from the analyzer and place it on a work table (refer to **Changing or Adding Oil**, page **9-8**, steps 1, 2, and 3 for instructions).

2. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Swing the clamp open and remove the trap.



3. Remove one end fitting from the trap body; dispose of the used alumina in an appropriate manner.

4. Wash the trap body with a detergent-based soap. Rinse with water, then with isopropyl or ethyl alcohol. Set the trap aside and allow to dry thoroughly.

Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.

5. Prepare fresh alumina as follows:

   a. Preheat the oven to 300 °C.

   b. Pour approximately 180 grams of fresh alumina into a glass or metal container (approximately 250 mL if a graduated beaker is used). Place the container in the oven.

   c. Bake the alumina for two hours.

   d. Remove the baked alumina from the oven and allow it to cool until lukewarm. A desiccator may be used to speed the cooling process.

6. Using a small spatula, gently pry the O-ring from the end fittings of each end of the trap body.

7. Inspect the O-rings.

   - If dusty, clean with a lint-free tissue.
   - If damaged, replace with a new O-ring.

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

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

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

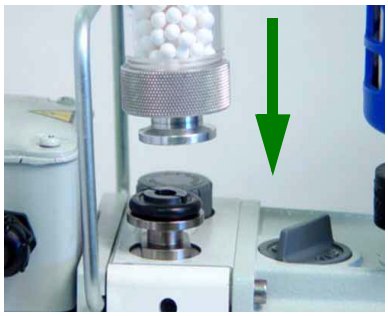
11. Lightly tap both ends of the trap body on the work surface. This will remove any remaining dust from the pellets.

12. Inspect the centering ring before placing it back on the intake port. If it appears to have flattened, replace it. A flattened centering ring can cause vacuum leaks.

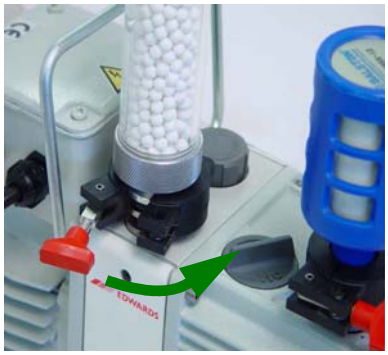
    There are two types of centering rings, use the one with the smaller opening at the intake port

Centering Ring

13. Place the trap on the centering ring.



14. Open the clamp and place it around the flange of the intake port and the flange of the trap. Swing the clamp fastening screw toward the intake port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.



15. Reconnect the hose from the analyzer to the oil vapor trap.

    a. Inspect the centering ring before placing it on the trap body. If it appears flattened, replace it with a new one. A flattened centering ring can cause vacuum leaks.

       Use this type of centering ring for the trap body.

    b. Place the clamp around the flange of the vacuum pump hose and vapor trap.

c. Swing the clamp fastening screw around until it fits into the slot on the other half of the clamp. Tighten the wing nut securely by hand.

16. Plug the pump power cord into the power source.

17. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh oil and to produce efficient vacuum operations

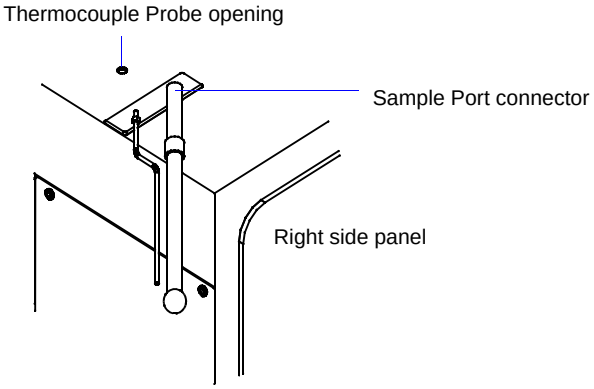
18. Replace the vacuum pump access panel.

Calibrating the Manifold Temperature Sensor

You should calibrate the manifold temperature on an annual basis. This allows you to correct for any changes in the manifold temperature sensor that occur with time.

You will need a small diameter probe, such as a 1/16-in. (1.5-mm) thermocouple, to calibrate the manifold temperature sensor.

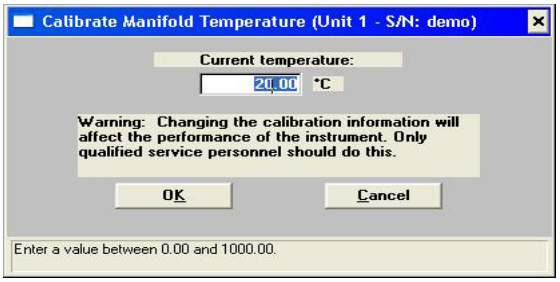
1. Insert the thermocouple probe into the opening located to the left of the sample port connector.



2. Move the thermocouple back and forth until you locate the small opening on the underside of the manifold. Push the thermocouple into the opening until the thermocouple stops.

3. Allow the temperature gauge reading to stabilize.

4. Select **Unit** > **Calibration > Temperature**; the Calibrate Manifold Temperature dialog is displayed.



5. Enter the manifold temperature indicated on the temperature gauge. Click **OK** to store the new value.

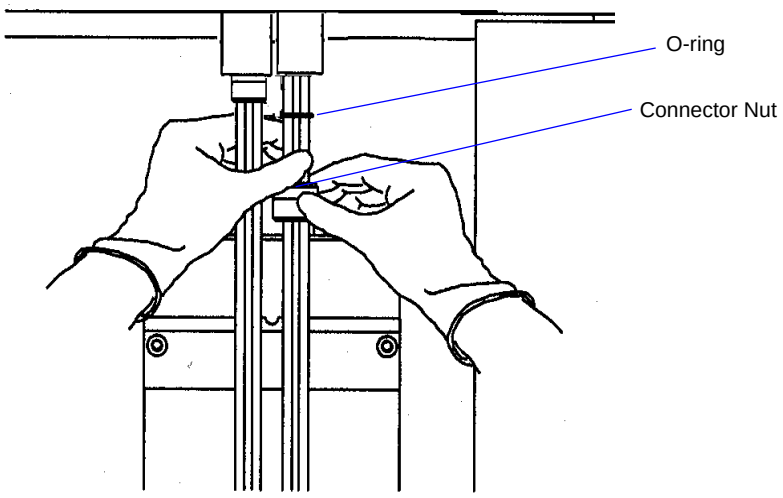
6. Remove the thermocouple probe.

Cleaning the Cold Trap Tubes

Oil vapor from the vacuum pumps accumulates in the cold trap. Clean the tubes as follows:

If the high vacuum pump is installed, wait for it to completely stop (approximately 10 minutes).

1. Remove the vacuum pump panel.
2. Disconnect the vacuum pump power cord from its power source.
3. Display the **[unit] Instrument Schematic** and select **Enable Manual Control**.
4. Open analysis valves in the following order: **P1**, **PS**, **5**, **7**, and **1**. Allow the pressure to stabilize until around atmospheric. Then, close the valves in the same order as they were opened. This step fills the analysis vacuum section before disconnecting.
5. Display the **Degas Instrument Schematic** and select **Enable Manual Control**.
6. Open degas valves in the following order: **D5** and **D7**. Allow the pressure to stabilize until around atmospheric. Then, close valve **D7** and **D5**. This step fills the degas vacuum section before disconnecting
7. Remove the connector nut from the glass tube and inspect the O-ring. Replace the O-ring if it is cracked or worn.



8. Carefully slide the glass tube down over the metal tube and remove the glass tube.
9. Rinse the tube with acetone; then dry it.
10. Reinstall the cold trap tube. Make sure the O-rings are in place.

11. Repeat Steps 2 through 6 for the other cold trap.
12. Reconnect the vacuum pump power cord
13. Replace the vacuum pump access panel.

10. ORDERING INFORMATION

The ASAP2020 Chemi system components and accessories can be ordered using one of the following methods:

- Call our Customer Service Department at (770) 662-3636
- Access our web site at www.micromeritics.com
- Contact your local sales representative

When ordering, please use the information provided in this chapter to place your order.

System Components and Accessories

Part Number	Item and Description
	Peripheral Accessories
202-33008-00	Chemisorption software and operator's manual
202-33054-00	Vapor Option, factory installed
060-00030-00	FlowPrep 060, degasses up to six samples at up to 400 ºC with flowing gas.
061-00030-00	VacPrep 061, degasses up to six samples at up to 400 ºC with either flowing gas or by evacuation (evacuation requires a vacuum pump).
021-00000-00	Model 021 LN_2 transfer system, for easy filling of sample Dewars; includes 47 L Dewar, mobile platform, and pump with 100 cm discharge line
	System Components
004-25103-00	Ferrule, front, Teflon 1/4 in.
004-25104-00	Ferrule, rear, Nylon 1/4 in.
290-25846-00	Copper tube, for gas inlet, 1/8 in. diameter, 6 ft. long
290-25846-01	Copper tube, for gas inlet, 1/8 in. diameter, 16 ft. long
201-25818-00	Gas inlet line assembly, stainless steel, 1/8 in. diameter, 6 ft. long
201-25818-01	Gas inlet line assembly, stainless steel, 1/8 in. diameter, 16 ft. long
004-25549-00	Reducer, brass; 1/8 in. tube x 1/4 in. tube, accepts 1/8 in. tube, connects to 1/4 in. swage fittings
004-25318-00	Reducer, stainless steel; 1/8 in. tube x 1/4 in. tube, accepts 1/8 in. tube, connects to 1/4 in. swage fittings

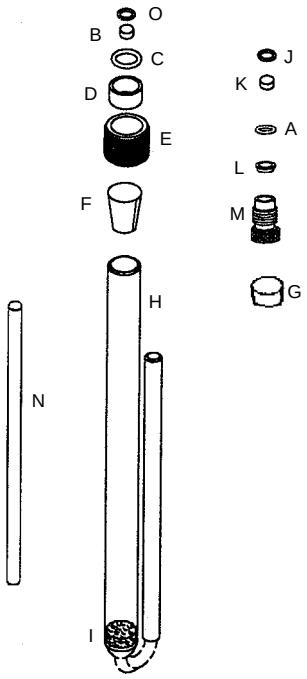
004-62230-58	Gas pressure regulator, CGA 580 fitting (Ar, He, Kr, N_2), 30 psig

Part Number	Item and Description
004-62230-35	Gas pressure regulator, CGA 350 fitting (CO, H_2), 30 psig
004-62230-54	Gas pressure regulator, CGA 540 fitting (O_2), 30 psig
004-62230-32	Gas pressure regulator, CGA 320 fitting (CO_2), 30 psig
004-62230-326	Gas pressure regulator, CGA 326 fitting (N_2O), 30 psig
004-62230-705	Gas pressure regulator, CGA 705 fitting (NH_3), 30 psig
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator, includes fittings and instructions
004-33602-00	Pressure Relief Kit; prevents excessive gas pressure in the event of regulator failure (not to be used with noxious gases)
202-33053-00	Dewar option kit; includes 4-liter, stainless-steel Dewar, stainless-steel cold trap, and elevator adapter
003-26043-00	Heating mantle with type K thermocouple, 450 ºC maximum, 24 V
230-25808-00	Heating mantle clip
004-54609-00	Brush, for cleaning sample tube
004-54609-01	Brush, for cleaning exhaust tube
240-25853-00	Sample tube funnel
240-14855-00	Sample tube rack
280-32800-00	Sample tube support, assists sample weighing
248-31702-00	Insulator disk, for oven
004-16825-00	Reference material, chemisorption
202-25903-00	Mat, rubber; for front shelf
250-25608-00	Valve gasket, Kel-F, for analysis manifold
250-25627-00	Valve plunger, Buna-N seal, for analysis manifold
250-25628-00	Valve spring, for Buna-N plunger
202-42808-00	Operator’s manual
	Vacuum System Components
062-00000-11	Vacuum pump, oil-sealed, with built-in anti-suckback valve, 100/120 VAC; includes hose kit
062-00000-23	Vacuum pump, oil-sealed, with built-in anti-suckback valve, 220/240 VAC; includes hose kit

062-33002-00	Activated alumina oil vapor trap, for one vacuum pump

Part Number	Item and Description
004-16003-01	Vacuum pump oil, 1 liter
200-25879-00	Vacuum pump oil funnel
004-27040-00	Vacuum pump exhaust filter
004-16830-00	Activated alumina, 500 grams, for oil vapor trap
004-25652-00	O-ring, size 217, for oil vapor trap

Sample Tube and Components



Part Number		Item and Description
004-25465-00	A	O-ring, size 010, Kalrez; for sample tube exhaust port fitting
004-27041-00	B	Port filter, 20 µm, 1/4 in. diameter
004-25474-00	C	O-ring, size 013, Kalrez for sample tube fitting
260-25843-00	D	Ferrule, 1/2 in. stainless steel
260-28804-00	E	Sample nut
240-32000-00	F	Stopper, for 1/2 in. sample tube
240-32000-00	G	Cap (stopper), for 1/4 in. sample tube
202-61001-00	H	Sample tube, quartz, use with temperatures up to 1100 ºC
004-32164-01	I	Wool, quartz, use with quartz sample tubes to constrain sample
004-25673-00	J	O-ring, size 008, Kalrez for exhaust port filter
004-27056-00	K	Exhaust filter, 20 µm, 3/16 in. diameter
275-25803-00	L	Ferrule, 1/4 in. stainless steel
201-25822-00	M	Exhaust nut
004-61701-02	N	Filler rod, quartz
004-25465-00	O	O-ring, size 010, Kalrez, for sample port filter

Cold Trap Tube and Components

Part Number		Item and Description
004-25469-00	A	O-ring, Buna-N, size 014
004-25979-00	B	Nut, cold trap
004-61063-00	C	Tube, 1/2 in. OD stem, cold trap

A. FORMS

This appendix contains the following form:

- Sample Data Worksheet

Copy and use this form as needed.

micromeritics®

ASAP Chemi Series Sample Data Worksheet

This form is provided to assist you in obtaining an accurate sample mass for report calculations. You may use the *After Degas* value (Step 5) or the *After Analysis* value (Step 7), provided they are close to the same.

Sample tube:_____ **Sample:** _____

Before Degas:

1. Mass of empty sample tube _____ (g)
2. Mass of sample tube plus sample _____ (g)
3. Mass of sample (Step 2) – (Step 1) _____ (g)

After Degas:

4. Mass of sample tube plus sample _____ (g)
5. Mass of sample (Step 4) – (Step 1) _____ (g)

After Analysis:

6. Mass of sample tube plus sample (g) _____ (g)
7. Mass of sample (Step 6) – (Step 1) _____ (g)

Compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5). These two values should be close in range. If a significant difference is noted, analysis problems may exist or the sample may have been improperly degassed.

B. ERROR MESSAGES

Error messages are listed numerically. If the **Action** response instructs you to contact your service representative, record the error message and make backup copies of any files involved in the operation.

The 1000-series error messages (used primarily for software testing) are not included in this appendix. These errors should not occur during normal operation. If you receive a 1000 series error message or an error message not listed in this appendix: record the error message, make backup copies of any files involved, then contact your service representative.

2200 and 2400 Series

2201- Cannot execute report subsystem.

Cause: **Start Report** failed to execute the report subsystem (which is a separate process).

Action: Restart the computer. If the problem persists, reinstall the application (this will not affect any of your sample files). If the problem continues, contact a Micromeritics service representative.

2401- FATAL ERROR: (error message)

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2430- Error accessing file (file name), error code = (number).

Cause A: A computer or network problem occurred.

Action A: Check the performance of your computer devices or network.

Cause B: A software error occurred when the file was accessed.

Action B: Contact your service representative.

2431- Error writing file (file name), error code = (number).

Cause: The hard disk does not have enough space left to perform the operation.

Action: Copy files not used regularly from the hard disk to a diskette, CD, or network directory, delete them from the hard disk, and then try the operation again.

2432- Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2433- New entries have been found in this directory. Refresh the directory information?

Cause: Program files (sample information, analysis conditions, or report options) have been added to this directory by some function other than this application.

Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** if you do not want to spend the time updating the directory information. This option may be feasible if a large number of files have been copied into the directory and you know the name of the file.

2434- File (file name) — Subset # (number) wrote wrong amount of data.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2436- Path specification (path name) is invalid.

Cause: You entered an invalid path name and/or extension.

Action: Type a valid path name (including the proper extension) and press **Enter**.

2437- File (file name) does not exist.

Cause: You entered a file specification that does not exist.

Action: Enter an existing file specification or select a file name from the list box.

2438- Disk drive (letter): is inaccessible.

Cause: You selected a disk drive that is not presently accessible.

Action: Ensure that the disk is not write-protected.

2439- Could not register file.

2440- Subset not found.

2441- Seek within file failed.

2442- Bad header in subset file.

2443- Subset owner denied access.

2444- Not a valid file format.

2445- Subset wrote the wrong amount of data.

2446- Error reading data.

2447- Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.

Action: Contact your service representative.

2448- File directory (path name) is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

Action: The installation directory will be substituted. The next time you open a file, use the Directories: list to move to the correct directory.

2449- This field does not contain a valid file specification.

Cause: You entered an invalid file name.

Action: See the description of file naming conventions in your DOS or Windows manual and re-enter the name.

2450- Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: You are in the process of initiating an automatic analysis (an analysis in which sample files are created using the defaults) while editing the defaults.

Action: Finish your edit session of the defaults and close the dialog. Then restart the automatic analysis.

2452- Attempt to write MICATTR.DIR in read only mode. (file name)

2453- Attempt to append MICATTR.DIR in read only mode. (file name)

Cause: The Read-Only attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file and disable the Read-Only option.

2454- Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

2455- Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: You selected too many files for this operation.

Action: Select only the number of files specified in the message.

2456- Insufficient file handles available. Application cannot continue.

Cause: You have more than 50 files open at the same time.

Action: Refer to the manual for your operating system and set the limit for open files to 50 or greater.

2457- Results cannot be displayed. More than (number) windows are currently displaying or printing results.

Cause: You have too many windows open in the application.

Action: Close some of the open windows.

2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: You attempted to exit the application while the analyzer is performing a critical operation. This operation must be completed before the application can be stopped.

Action: Wait a few minutes before attempting to exit the application again.

2459- An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit?

Cause: You attempted to exit the application while the analyzer was performing an operation.

Action: Rather than exiting the application you may choose to minimize it.

2460- Fatal Communications error on (unit n).

Cause: Repeated attempts to maintain communication with the analyzer have failed.

Action: Check the communications cable connecting the analyzer to the computer. Confirm that the analyzer is turned on and that the status light is not blinking.

If these checks are okay, exit the analysis program and then restart the application. If this error persists, contact your service representative.

2461- No instruments are in operation. This application will unconditionally terminate.

Cause: At least one analyzer must be active for the application to operate. The initialization of the analyzers configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the analyzer's failure to initialize. See the instructions for that message.

Action B: Check the cable connection between the analyzer and the computer. Verify that the analyzer has the power switch in the **ON** position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the analyzer, contact your service representative.

2461- No instruments are in operation. This application will unconditionally terminate.

Cause: At least one analyzer must be active for the application to operate. The initialization of all of the analyzers configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the analyzer's failure to initialize. See the instructions for that message.

Action B: Check the cable connection between the analyzer and the computer. Verify that the analyzer power switch is in the **ON** position. If the application continues to fail in its attempts to initialize the analyzer, contact your service representative.

2471- Unit n - S/N; nn has an invalid communications port specified. It cannot be initialized.

Cause: The communications port specified for this analyzer during installation (or configuration) is invalid.

Action: Use the setup program to change the analyzer configuration.

2474- (Unit n) Communications port COM (port number) specified in the program control files is already in use. Unit cannot initialize.

Cause: The communications port assigned to the indicated unit is in use by another program.

Action: End the program using the busy port or use the Setup program to change the communications port assigned to this program.

2475- (Unit n) Communications port COM (port number) specified in the program control files cannot be accessed. Unit cannot initialize.

Cause: The operating system has prevented the application from accessing the communications port.

Action: Review the hardware configuration of the computer, ensure that no other application is using the port. Contact your service representative if you continue to receive this error message.

2476- Unit startup failed. Cannot initialize the communications port.

Cause: The communications port specified during installation is invalid.

Action: Use the setup program to change the analyzer configuration. Contact your service representative if you continue to receive this error message.

2477- (Unit n; Serial nn) did not properly initialize.

Cause: The software was unable to initialize the analyzer.

Action A: Run the Setup program and ensure that a valid port is specified; if not, specify a valid one when prompted.

Action B: Reinstall the software, then restart application.

Action C: Contact your Micromeritics service representative if you continue to get this message.

2478- Error copying sequential data segment.

Cause: An internal processing and/or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors; for example, use a utility such as ScanDisk. Contact your service representative if you continue to receive this error message.

2479- (Unit n; Serial nn) The instrument is busy performing an operation of which this application is unaware. Do you want to cancel? (Yes, No)

Cause: During initialization of the application, the status of the analyzer was found to be in a different state than expected.

Action: Click **Yes** to cancel the operation in process, allowing the analyzer to reset and continue with initialization.

Click **No** to cancel the initialization process.

If you continue to get this message, verify that files in the application directory structure are not being changed or removed.

2480- File (file name) cannot be analyzed. It is currently being edited.

Cause: You attempted an analysis using a sample file that is being edited.

Action: Save the changes and close the dialog box.

2481- Error accessing the sample information file (file name).

Cause A: You attempted to open a file that is already open, possibly minimized.

Action A: View the minimized icons, locate and maximize the file.

Cause B: A computer or network problem occurred.

Action B: Check the performance of your computer devices or network.

Cause C: A software error occurred when the file was accessed.

Action C: Contact your service representative.

2482- File cannot be opened for writing. It is already in use.

Cause: You are attempting to open a file that is currently being used (either by this application or another).

Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all windows, one of which may contain this file).

2483- An analysis cannot be performed on (file name). It is open for editing and contains errors.

Cause: You attempted to use a sample file containing errors that is currently open.

Action: Go to the window containing the file, correct the errors, and save it.

2484- The edit session for (file name) must be saved before the analysis. Save changes and continue with the analysis.

Cause: You attempted an analysis using a sample file that contains unsaved changes.

Action: Select **Yes** to save the changes and continue with the analysis.

Select **No** to abort the analysis and return to the sample file.

2485- The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected service test file has a status other than **No Analysis**.

Action: Select a different service test file or create a new one and use **Replace All** to copy parameters from the file you originally selected.

2486- Cannot construct (name) report type. Program will terminate.

2487- Cannot start report generator. Error code (number). Program will terminate.

Cause A: You may not have full rights to the application's folders and files.

Action A: Contact your system administrator and have him grant you full rights.

Cause B: An internal processing and/or hardware error has occurred.

Action B: Contact your service representative if you continue to receive this error message.

2488- File (file name) cannot be opened for editing. It is already in use.

Cause: The file you specified is being used in another edit operation.

Action: Check the Windows list to locate the other edit session.

2489- File (file name) cannot be opened for writing. It is already in use.

Cause: The file you specified in a **Save As** operation is already open for edit.

Action: Select a different file for the **Save As** operation.

2490- No '.INI' file present. Application will terminate.

Cause: The ASCII (.INI) file containing initialization and system options information cannot be found. The .INI file may have become corrupted. The application cannot operate without this file.

Action: Use the Setup CD to uninstall the ASAP 2020 Chemi application. When you uninstall the application, only the application files are deleted; data files remain intact.

After the uninstall operation is complete, reinstall the application.

2491- Highlighted fields contain errors. Please correct the errors before closing dialog box.

Cause: The highlighted fields contain invalid entries. You will not be able to close the dialog until you correct the errors.

Action: Check the entries, correct the errors, and close the dialog.

2492- This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493- An entry is required for this field.

Cause: This field requires a valid entry for you to proceed.

Action: Enter or select an appropriate value.

2494- Value is out of the valid range.

2495- Value is out of the valid range. Enter a value between (value) and (value).

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter or select an appropriate value.

2496- Invalid number.

Cause: The number you entered in the highlighted field is invalid.

Action: Check the entry and enter or select a valid number.

2497- This field contains an invalid character.

Cause: You entered an invalid character in the highlighted field.

Action: Check the entry and enter valid characters.

2498- The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status (for example, from automatically collected to manually entered) could not be done.

Action: Contact your service representative if you continue to receive this error message. Record the name of the sample file in which the problem occurred.

2499- Sequence number must contain at least 3 digits.

Cause: You tried to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500 Series

2500- All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.

Action: Select **Options** > **Sample Defaults** from the Main Menu and enter a new sequence number.

2501- System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows on the screen. Contact your service representative if you continue to receive this error message.

2502- Error writing to file (name) during print. Error code: (number).

Cause: An error occurred in the file being written to during a print operation.

Action: Ensure that there is sufficient space on the drive containing the file.

2503- Error converting file (file name). Could not create DIO intermediate file.

Cause A: Insufficient space is available on the hard disk. The DIO file is placed in the directory specified by the TEMP environment variable.

Action A: Determine if there is sufficient space on the drive where the TEMP directory is located.

Cause B: An internal processing and/or hardware error has occurred.

Action B: Contact your service representative if you continue to receive this error message.

2504- Cannot create output file for sample (sample name).

Cause: Insufficient space may be available on the hard disk.

Action: Ensure that sufficient space is available. Contact your service representative if you continue to receive this error message.

2505- Error Logger cannot be initialized! Error code (number). Program will exit.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative.

2506- (sample file) Output device (name) is not installed. Printing cannot be accomplished.

Cause: The selected output device is not installed in Windows.

Action: Select a different output device in the System Configuration dialog box. Install the device using the Control Panel, Printers operation.

2508- (sample file) Overlay file (name) was not found. It will not be included in the reports.

Cause: The specified overlay file could not be found.

Action: Ensure that the file specified as an overlay does exist.

2509- (sample file) Error opening file (name): (error). Reports cannot be produced.

Cause: An error occurred while the program was opening a file necessary to the report operation.

Action: Use the name given in the error message to investigate. Contact your service representative if you continue to receive this error message.

2510- (sample file) Error parsing reports from file (name). Reports cannot be produced.

Cause A: One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).

Action A: Review the data entry fields (for example, the Sample field) and remove the invalid character.

Cause B: The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.

Action B: Check the space available on the hard disk.

Cause C: An internal processing error occurred.

Action C: Contact your service representative.

2511- Print job (name) has been cancelled due to insufficient disk space. Delete unnecessary files and restart the report.

Cause: The disk drive does not have enough space for the temporary file required by the Windows Print Manager. Therefore, printing of the requested report has been canceled.

Action: Delete unnecessary files from the disk. You will require at least five megabytes of free space for normal operation.

2512- Print job (name) has been cancelled.

Cause: The requested print job was canceled at your request.

Action: None required.

2513- Unable to read the calibration file (file name).

Cause: You selected an invalid calibration file or one that cannot be read.

Action: Be sure the media containing the calibration file has no problems.

2514- Unable to write the calibration file (file name).

Cause: An attempt to Save calibration data has failed due to possible media problems.

Action A: Be sure the media you want to Save the file to has no problems.

Action B: Choose an alternate media to Save the calibration data.

2515- Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause: You have started the process of performing a calibration operation.

Action: Calibration operations should only be done by or under the direction of qualified service personnel.

2516- Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause: You have performed a calibration operation; a backup copy is recommended.

Action: Perform a calibration **Save** operation.

2517- Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause: You have not accepted the calibration you performed.

Action: If the calibration operation was successful, click **Accept**.

2518- Analysis data are not suitable for smoothing. Smoothing has been disabled.

Cause: The Analysis data contains volume adsorbed values that are less than or equal to zero.

Action: No action required; this is a status message only.

2519- Data sets with fewer than 11 data points cannot be smoothed. Smoothing has been disabled.

Cause: Primary or repeat data with less than eleven data points cannot be smoothed. This message may refer to the number of points in the repeat analysis if it is selected for reporting.

Action: No action required; this is a status message only.

2520- No data points available for reporting.

Cause: You requested a report for a data file which contained no data points relevant to the report.

Action: The affected report will not be produced. If the data file contains no points, select another file for the report. If the data file contains points, verify that you selected a sufficient number of points for the report on the Collected/Entered Data dialog.

2521- Unable to program controller.

Cause: A hardware malfunction has occurred.

Action: Contact your local Micromeritics service representative.

2522- Invalid controller application file.

Cause: The application’s control file has been corrupted or deleted.

Action: Reinstall the ASAP 2020 analysis program.

2523- Programming the controller failed.

2524- CRC check failed on programming controller.

2525- Unknown error programming controller.

2526- Controller download was not successful.

2527- Controller CRC error on boot block.

2528- Controller DRAM error.

2529- Controller Com1: error.

2530- Controller Com2: error.

2531- Controller debug port error.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2532- The instrument contains a different software version. Do you want to reset it?

Cause: The application has discovered a different version of software operating in the analyzer.

Action: If there are no analyzers other than the ASAP 2020 connected to the computer, select **Yes** and allow the updated software to load.

2533- Analyzer initialization failed.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this error message.

2534- Error opening file (name) for printing. Error code: (number).

Cause: An error occurred in the selected file for print output.

Action: Ensure that sufficient space is available on the drive containing the file.

4000 Series

4000- Memory Allocation Error.

Cause: An internal processing and/or hardware error occurred during report generation.

Action: Contact your service representative if you continue to receive this error message.

4003- Error Converting Pressures.

4004- Error Computing Volume Adsorbed.

Cause: An internal processing and/or hardware error occurred during report generation.

Action: Contact your service representative if you continue to receive this error message.

4006- Report Type Not Found.

4007- Error Processing Report.

Cause: An internal processing and/or hardware error occurred during report generation.

Action: Contact your service representative if you continue to receive this error message.

4008- Report requested on sample file with no data points.

Cause: You selected a file for reporting which contains no collected data.

Action: Select another file which contains collected data and restart the report.

4009- Error, No Reports Selected.

Cause: You attempted to generate reports for a sample that has no reports selected in its report options.

Action: Select the reports in the sample's report options and save the sample file.

4011- Analysis gas in sample file does not match analysis gas in unit.

Cause: You attempted to start an analysis using a sample information file in which the analysis gas specified does not match the analysis gas entered in the unit configuration.

Action: If necessary, attach the appropriate gas bottle, then enter the gas in the Unit Configuration dialog.

4015- Error creating export file for sample <sample file name>.

Cause: A file error occurred during creation of an export output file.

Action: The output file name may be invalid. Ensure that the target directory exists. Ensure that the target diskette is not full or write protected. The target disk drive may be damaged or inoperative. Verify that other files may be created on the same drive. Contact your service representative if you continue to receive this error message.

4016- Sample (file name) has no data for export.

Cause: The file selected for export has a status of **No Analysis**. No export file will be created.

Action: Select a file which contains analysis data.

4017- Damage to the instrument will result if the sample has not been manually evacuated. Have you evacuated the sample?

Cause: You did not select **Backfill sample at start of analysis** on the Sample Backfill Options dialog box. The sample tube is normally at atmospheric pressure when an analysis is started, and it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: If you have manually evacuated the sample tube, select **Yes**. If you have not, select **No** and then either perform a manual evacuation or go to the Sample Backfill Options dialog box and select **Backfill sample at start of analysis**.

4020- Disabling this option may damage the instrument. Are you sure that the sample should not be backfilled?

Cause: You did not select **Backfill sample at start of analysis** on the Sample Backfill Options dialog. The sample tube is normally at atmospheric pressure when an analysis is started; it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: If you want to manually evacuate the sample prior to the start of the analysis, select **Yes**. Otherwise, select **No**, go to the Sample Backfill Options dialog, and select **Backfill sample** at start of analysis.

4023- The file (sample file) cannot be prepared for analysis. It is open for editing and contains errors.

Cause: You attempted an analysis using a sample file that is being edited and has invalid values in one or more of its fields.

Action: Enter valid values into the fields that have errors, save the changes, and close the dialog box.

4024- Backfill gas in sample file does not match any gas in unit.

Cause: You selected a backfill gas that is not included in the gas configuration.

Action: Make sure that gas selections match the gases that are attached to the instrument. Choose one of the gases as the backfill gas, or edit the sample file and choose one of the existing gases as the backfill gas.

4025- There is no Helium attached to the unit.

Cause: A measured free space has been requested but helium does not appear to be available.

Action: Make sure the helium cylinder is attached to the unit at its designated port.

6200 Series

6201- Communications port (port name) specified in the program control files is not a valid device. Unit cannot initialize.

Cause A: The communications port assigned to this program cannot be made available for proper communications.

Action A: Run the Setup program, select **Change analyzer setup**, and enter the correct port information. If the message continues, contact your service representative.

Cause B: The program's control file was manually modified, and an invalid communications port was specified.

Action B: Use the Setup program to change the communications port assigned to this program.

6202- No serial number available. Unit cannot initialize.

Cause: The program control file does not contain a serial number for the unit.

Action: Run the Setup program, select **Change analyzer setup**, and define the serial number for the instrument.

6203- File cannot be opened for editing. It is already in use.

Cause: The file you specified is being used in another edit operation.

Action: Check the Windows list to locate the other edit session.

6204- File cannot be opened for writing. It is already in use.

Cause: The file you specified in a **Save As** operation is already open for edit.

Action: Select a different file for the **Save As** operation.

6211- The gas selected for the (task name) task is different than the gas selected for the preceding (task name) task. Do you wish to insert an evacuation task in between?

Cause: A task in the **Selected Tasks** list uses a different gas than the previous or next task. Mixing gases may be dangerous.

Action: Select **Yes** to evacuate the sample before introducing the new gas. Select **No** to exclude evacuation between the two tasks.

6212- A leak test is specified without a preceding evacuation. Do you wish to insert an evacuation task?

Cause: By inserting or deleting a task in the **Selected Tasks** list on the Analysis Conditions dialog box, you left a leak test task without a preceding evacuation task. The leak test will fail if the sample pressure exceeds 0.5 mmHg.

Action: Select **Yes** to evacuate the sample between the two tasks. Select **No** to exclude evacuation

6213- The maximum allowed number of tasks is selected. Delete some tasks before proceeding.

Cause: You attempted to insert more than twenty tasks.

Action: You may specify no more than twenty tasks in one sample file. If more preparation steps are required, use two sample files. The first sample file should contain only preparation tasks; the second file should contain any remaining preparation tasks and an analysis task.

6214- An analysis task is specified without a preceding evacuation. Do you wish to insert an evacuation task?

Cause: You inserted or deleted a task in the **Selected Tasks** list of the Analysis Conditions dialog box, leaving the analysis task without a preceding evacuation task.

Action: Select **Yes** if you want the system to automatically insert an evacuation task. Selecting **No** causes the analysis to produce incorrect results.

6215- The Selected Tasks list contains adjacent tasks using different gases. Do you

still wish to save this file?

Cause: You attempted to save a sample file in which one task in the **Selected Tasks** list uses a different gas than the previous/next task. Mixing gases may be dangerous.

Action: Select **No** if you do not want to save the file. Return to the Analysis conditions dialog and insert an evacuation task between all flow, soak and analysis tasks which use different gases.

6216- The selected task has already been executed. It may not be changed or deleted.

Cause: You attempted to edit or delete a task after it was already performed.

Action: Edit or delete a task not yet performed.

6217- New tasks may only be inserted following task (number).

Cause: You attempted to insert a task before the task currently being executed.

Action: Insert new tasks following the task currently being executed.

6218- An analysis task may not be inserted while an experiment is in progress.

Cause: You attempted to insert an analysis task in the **Selected Tasks** list for a sample file which is currently being processed by the analyzer.

Action: If you want to insert an analysis task, first cancel the experiment in progress. Then delete the tasks which had executed up to the time of the cancellation, insert an analysis task, and restart the experiment

6219- No new tasks may be inserted. Analysis is complete.

Cause: You attempted to insert a new task for a sample file which is currently being processed by the analyzer. All tasks have been processed and the analyzer is performing the termination sequence.

Action: Construct a new sample file containing the tasks and submit it for processing.

6220- Tasks may not be deleted after an experiment has been started.

Cause: You attempted to delete a task after the sample file was submitted for processing.

Action: Reduce the duration of the task to minimum value.

6221- Error opening file (file name) for conversion.

Cause: A file error occurred while the system was attempting to open a MICMOS chemisorption data file and convert it to the ASAP 2010C file format. The disk drive is malfunctioning.

Action: Contact your service representative.

6222- (Unit {number}) (gas name) required by task (number) is not configured on the selected unit.

Cause: You attempted to start an analysis which requires a gas not entered in the unit configuration information. The analysis is canceled.

Action: Select **Unit [n] > Unit Configuration** to display the Unit Configuration dialog. Click **Gas** to display the Gas Configuration dialog; then specify the valve to which the required gas is connected.

6223- Sample has no Analysis Task entry. Do you wish to proceed with the analysis?

Cause: You are attempting to analyze a sample file with no analysis task entry.

Action: Select **Yes** to proceed with the analysis; all preparation tasks are performed but no data points are collected. Select **No** to cancel the analysis.

6224- Sample has no pressure table entries. Do you wish to proceed with the analysis?

Cause: You attempted to start an analysis using a sample file for which no pressure table was specified.

Action: Select **Yes** to proceed with the analysis; all preparation tasks and the free space measurement are performed, but no data points are collected. Select **No** to cancel the analysis; enter a pressure table for the sample.

6225- The Selected Tasks list contains adjacent tasks using different gases. Do you still wish to close this file? (yes, no)

Cause: You attempted to close a file that has adjacent experiment tasks which specify different gases. Incompatible gases may be mixed during sample preparation.

Action: Select **No** if you do not want gases to be mixed on the sample. Select **Yes** to return to the Analysis conditions dialog box and add evacuation tasks if necessary.

6226- (gas symbol) required for backfilling the sample at the end of the analysis is not configured on the selected unit.

Cause: The gas specified for use in backfilling the sample at the end of the experiment is not configured on the instrument.

Action: Select **Unit > Unit Configuration** to display the Unit Configuration dialog. Click **Gas** to display the Configuration Gas Options dialog; select the gas inlet port to which the required gas is connected.

6230- The Active Metals Table Options dialog does not specify a stoichiometry factor for the adsorptive (gas name) of the element (metal name). One (1.0) was substituted for this value in the calculations.

Cause: You specified a non-zero percent of sample weight for an element in the Active Metals Table of the sample report options but you did not specify a stoichiometry factor for the adsorptive used in the analysis.

Action: The calculation proceeds with the default value and the program produces a report. Edit the sample information file to include the correct value and run the report again.

6231- No element in the Active Metals Table Options has a "% of Sample Weight" value greater than zero. Metallic Dispersion and Metallic Surface Area will no be calculated.

Cause: All elements in the Active Metals Table of the sample report options have the value zero entered for the percentage of sample weight.

Action: The calculations proceed and the program produces an incomplete report. Edit the sample information file to include the correct value and run the report again

6232- Line Fit Plot cannot be produced: Fewer than two data points were selected for inclusion in the calculations.

Cause A: You selected fewer than two data points for inclusion in the line fit plot calculation. The plot requires at least two data points to be produced.

Action A: Edit the sample information file to select the data points used in the calculation.

Cause B: The second temperature entered while calibrating the sample thermocouple differs from the first by less than 75 ºC and will produce an unsatisfactory calibration.

Action B: Select **Yes** to use the entered value. Select **No** to repeat the second temperature measurement.

6234- Leak test failure - the observed outgas rate of [VAC0] [VAC-U] exceeds the limit of [VAC0] [VAC-U].

Cause: The outgas rate specified in the leak test criteria was exceeded.

Action: Check sample tube fitting to ensure that it is securely attached to the port. Then start the analysis again.

6235- Leak test failure in task (number): The sample was not fully evacuated prior to the test.

Cause: The sample was at a pressure greater than 0.5 mmHg at the start of the leak test. A leak test can not be performed unless the sample has been fully evacuated.

Action: Insert an evacuation task immediately before the leak test. If an evacuation task is already present, ensure that the evacuation is being performed at the same temperature as the test and that the duration of the evacuation is adequate.

6236- Pressure exceeded [PR0] [PR-U] while flowing (gas).

Cause: The pressure in the manifold exceeded the indicated maximum, while flowing gas over the sample.

Action: Verify that the exhaust line is not plugged or restricted. Reduce the pressure of the flowing gas at the tank regulator. Resume the experiment while monitoring the manifold pressure and flow rate. Adjust the regulator pressure to achieve the flow rate with a manifold pressure of less than the indicated maximum.

6237- (Unit {number}) Analysis canceled in task (number): Time limit exceeded while attempting to reach temperature (number).

Cause A: The furnace has malfunctioned.

Action A: Refer to **Chapter 9**, **Troubleshooting and Maintenance**, to diagnose the malfunction and correct it, or contact your service representative.

Cause B: The pressure within the cooling line is too low.

Action B: Increase the pressure of the cooling gas to between 10 and 20 psi.

6238- Furnace is not in the raised position. This is not the recommended operating configuration. Do you wish to proceed with the analysis?

Cause: The elevator holding the furnace is not in the fully raised position. Unless a custom furnace or Dewar is being used, it is not the correct operating position.

Action: Select **No** to stop the analysis initialization; then correctly position the furnace. Select **Yes** if you are using custom equipment and are certain it is installed correctly.

6248- The selected task is currently executing. It may not be changed or deleted at this time.

Cause: You attempted to edit or delete a task which is currently in operation.

Action: None required for this task. For future reference, you must suspend the analysis to edit or delete a task.

6249- The sample file (number) is already present in the sequence.

Cause: You attempted to insert a sample file that is already in the sequence.

Action: Create or choose another file.

6500 Series

6500- Failed to evacuate manifold to VAC SET in (number of) seconds. Calibration canceled.

Cause A: The vacuum set point is set too low.

Action A: Reset VAC SET point to 5.0 mmHg. If the VAC SET point is already at 5.0 mmHg or above, the vacuum gauge may need servicing.

Cause B: Leak in manifold.

Action B: Locate the leak and repair it. Refer to **Chapter 9**, **Troubleshooting and Maintenance**. Restart calibration.

Cause C: Valve failure.

Action C: Identify the leaking valve. Contact your service representative.

6501- The 1000 mmHg transducer offset exceeds recommended limits: %8.4f

Cause: The Pressure Gauge Calibration operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the Pressure Gauge Calibration operation, if this message occurs again contact a Micromeritics service representative.

6502- The 10 mmHg transducer offset exceeds recommended limits: %8.5f

Cause: The Pressure Gauge Calibration operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the Pressure Gauge Calibration operation, if this message occurs again contact a Micromeritics service representative.

6503- The 1 mmHg transducer offset exceeds recommended limits: %8.6f

Cause: The Pressure Gauge Calibration operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the Pressure Gauge Calibration operation, if this message occurs again contact a Micromeritics service representative.

6504- Unable to write the calibration file (name).

Cause: A **Save to File** operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6505- Unable to read the calibration file (name).

Cause: A **Load from File** operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6506- Calibration file for (file name) is invalid.

Cause: A **Load From File** operation failed due to invalid information in the file.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists contact a Micromeritics service representative.

6509- The sample has an invalid status and cannot be used for degassing.

Cause: A file selected for degassing has a status other then **No Analysis** or **Prepared**.

Action: Select a different file.

6510- Error evacuating.

Cause: An evacuation error occurred during a degas operation.

Action: If the problem persists, contact a Micromeritics service representative.

6511- Error dosing.

Cause: A dosing error occurred during a degas operation.

Action: If the problem persists, contact a Micromeritics service representative.

6512- Error calibrating the servo.

Cause: An error occurred while calibrating the servo valve.

Action: If the problem persists, contact a Micromeritics service representative.

6513- Error waiting for pressure to drop.

Cause: A pressure error occurred during a degas operation.

Action: If the problem persists, contact a Micromeritics service representative.

6514- Problem encountered dosing to target [PR2] [PR-U], last pressure = [PR4] [PR-U], elapsed time = [0]:[0].

Cause: Dosing during an analysis did not come within the allowed range of the target.

Action: Check that the outlet stage of the gas regulator is within specification. If the problem occurs frequently contact a Micromeritics service representative.

6515- The furnace reports an error. Sample temperature control during an analysis will not be possible.

Cause: An error occurred while the system was setting the target temperature of the furnace. The software will not be able to control the sample temperature.

Action: The furnace controller has malfunctioned. An error code of one (1) indicates that the furnace controller is not responding to any commands. An error code of two (2) implies that the furnace controller is responding but the responses are garbled. Contact your service representative.

6516- (Unit [number]) Analysis canceled: Sample pressure greater than (pressure) mmHg is not allowed.

Cause: An absolute pressure greater than (pressure) mmHg was attained during Low pressure dosing (either fixed dose mode or incremental dose mode).

Action: The analysis was canceled. All previously collected data were stored.

6517- (Unit [number]) Analysis canceled: Total volume dosed greater than (volume) cm^3 is not allowed.

Cause A: More than (volume) cm3 has been dosed onto the sample, possibly due to leaks in the system.

Action A: Perform leak checks on the system. Refer to **Appendix F**, **Testing for Leaks**.

Cause B: The amount of sample you used may be too large. Multiply the maximum value from the Volume Adsorbed column of the Isotherm Report by the sample weight. If the result exceeds (volume) cm^3, the amount of sample is too large.

Action B: Reduce the amount of sample.

6518- (Unit [number]) Analysis canceled: Pressure of (pressure point) mmHg exceeds the maximum manifold pressure of (pressure point) mmHg.

Cause: An absolute pressure greater than (pressure) mmHg was attained that exceeded the specified maximum manifold pressure.

Action: The analysis was canceled. All previously collected data were stored. Change the maximum manifold pressure value in the Adsorptive Properties file.

6520- Unit (number): Power failure detected.

Cause: A power failure occurred in the specified unit, and any analyses in progress were terminated.

Action: If the program was running and an Uninterruptible Power Supply (UPS) was attached, data points were collected and stored in the sample file. If enough data points were not collected, create another sample information file and start a new analysis.

6521- Transducer overrange detected.

Cause: The transducer in the specified unit has detected a pressure equal to or greater than 1000 mmHg. The exhaust port on the sample tube may be blocked.

Action A: Inspect for and clear any blockage.

Action B: Observe caution when operating the analyzer manually. If the problem persists contact a Micromeritics service representative.

6522- Analysis canceled: Time limit exceeded while evacuating manifold.

Cause A: Maximum manifold evacuation time was exceeded before the vacuum set point was achieved. Vacuum pump may be turned off.

Action A: Turn on vacuum pump switch. Then restart the analysis.

Cause B: The vacuum pump oil level is low.

Action B: Check the vacuum pump oil level and add more oil if necessary. Then restart the analysis.

Cause C: The manifold is contaminated or leaking.

Action C: Correct the problem. Refer to **Appendix F**, **Testing for Leaks**. Then restart the analysis.

6523- Analysis canceled: Time limit exceeded while evacuating sample (unrestricted).

Cause: The maximum time for evacuating the sample through the unrestricted valve was exceeded. Possible causes are a leak in the sample tube fitting or a crack in the sample tube.

Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Then restart the analysis.

6524- Analysis canceled in task (number): Time limit exceeded while evacuating sample (restricted).

Cause: The maximum time allowed for evacuating the sample through the restricted valve was exceeded.

Action: Check the sample tube and the sample tube fitting to ensure that the tube is securely attached to the port. Verify that the sample is properly degassed. Then start the analysis again.

6526- Time limit exceeded while backfilling manifold to [PR4] [PR-U] with (gas).

Cause A: The maximum time was exceeded before the target pressure point was reached. The gas regulator may be set too low or turned off.

Action A: Set the gas regulator to 10 psig (0.7 bar). Then resume the analysis.

Cause B: The gas bottle is empty.

Action B: Connect a new gas bottle. Then resume the analysis.

6527- Analysis suspended: Time limit exceeded while dosing manifold to (target pressure point) mmHg with (gas name).

Cause A: The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.

Action A: Set the analysis gas regulator to 10 psig (0.7 bar). Then resume the analysis.

Cause B: The analysis gas bottle is empty.

Action B: Connect a new analysis gas bottle. Then resume the analysis.

6528- Low pressure gauge offset too high - [PR4] [PR-U].

Cause: A check of the 1000 mmHg gauge's offset during an automatic operation indicated it was too high.

Action: If this message occurs repeatedly, contact a Micromeritics service representative.

6529- Master pressure gauge offset is too high - [PR1] [PR-U].

Cause: A check of the 10 mmHg or 1 mmHg gauge's offset during an automatic operation indicated it was too high.

Action: If this message occurs repeatedly contact a Micromeritics service representative.

6530- Volume calibration canceled due to failure (code [0]).

Cause: A problem occurred during volume calibration.

Action: Contact a Micromeritics service representative.

6531- The gas configuration file for %s is invalid.

Cause: The contents of the gas configuration file are not valid.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted (run ScanDisk). If the problem persists, contact a Micromeritics service representative.

6532- The analyzer is currently running the 2020 Physisorption software. Do you want to reset it?

Cause: The analyzer is under the control of the 2020 Physisorption software.

Action: Selecting **Yes** will reset the analyzer and download the 2020 Chemisorption control software.

6533- The analyzer is currently running the 2020 Chemisorption software. Do you want to reset it?

Cause: The analyzer is under the control of the 2020 Chemisorption software.

Action: Click **Yes** to reset the analyzer and download the 2020 Physisorption software.

Click **No** to continue running the 2020 Chemisorption software.

6534- Instrument (unit number) is not calibrated.

Cause: Calibration information for various analyzer components are missing.

Action: Run the application setup program and reinstall calibration information for the specified unit.

6535- Problem encountered evacuating.

Cause: Evacuation during the manifold dosing operation did not come within the allowed range of the target.

Action: Check that the outlet stage of the gas regulator is within specification. If the problem occurs frequently, contact a Micromeritics service representative.

6537- Error reading SmartVac ADC.

Cause: There was a problem reading one of the signals on the SmartVac board.

Action: Exit the application, turn off the power switch on the analyzer, then turn the power back on. Restart the application and the degas operation. If the problem persists, contact your service representative.

6538- Power failure detected. The sample is in an unknown condition. A run termination will be performed for safety.

Cause: A total power failure occurred (a UPS was not connected) while an analysis was in progress.

Action: Allow the analysis to terminate.

6539- Error overheating Current = (no.), Target = (no.), Limit = (no.)

Cause A: The heating mantle is reporting a higher temperature than expected.

Action A: Ensure that the heating mantle and thermocouple cables are plugged in fully on the port(s).

Cause B: An internal failure of the degas system may have occurred.

Action B: Unplug the heating mantle power connector; do not unplug the thermocouple. Allow the heating mantle to cool down (approximately 15-30 minutes). Then exit the analysis program and turn off the analyzer. Wait a couple of minutes, then turn the instrument on and restart the program. If the problem recurs, contact your Micromeritics service representation.

6540- Error thermocouple unplugged Current = (no.), Target = (no.)

Cause: The heater was enabled, but the thermocouple was unplugged.

Action: Plug in the thermocouple and try again.

6541- Error: SmartVac is not in a valid state to check degas.

Cause: You clicked Check but the sample was not in an appropriate state. You can only check the degassing operation after the vacuum setpoint has been attained, or during a temperature ramp or hold.

Action: Wait until an appropriate time during the degassing operation, and check the degassing operation again.

C. CALCULATIONS

This appendix contains the calculations used in the ASAP 2020 Chemi program.

Free Space

Free-space volumes are calculated using the following equations:

$$V_{FS} = \frac{V_{LOW}}{T_1} \times \left[\frac{P_1}{P_2} - 1\right] \times T_{STD}$$

where:

P_1	=	system manifold pressure before dosing helium onto sample (mmHg)
P_2	=	system manifold pressure after dosing helium onto sample (mmHg)
T_{STD}	=	standard temperature (273.13 K)
T_1	=	system manifold temperature before dosing helium onto sample (K)
T_2	=	system manifold temperature after raising Dewar and equilibrating with helium (K)
V_{LOW}	=	lower manifold volume (cm^3)
V_{FS}	=	volume of free space (cm^3 at standard temperature)

Volume Adsorbed

For the I^{th} dose:

$$Ngas_I = Ngas_{I-1} + V_{LOW} \times \left[\frac{Psys1_I}{Tsys1_I} - \frac{Psys2_I}{Tsys2_I}\right] \times \frac{T_{STD}}{P_{STD}}$$

$$Nads_I = Ngas_I - P_{sam} \div P_{STD} \times V_{FS}$$

$$V_I = \frac{Nads_I}{Wsam}$$

where:

$Ngas_I$	=	total amount of gas dosed into sample tube after I^{th} dose (cm^3 STP)
$Nads_I$	=	amount of gas adsorbed after equilibrating I^{th} dose (cm^3 STP)
P_{STD}	=	standard pressure (760 mmHg)
$Psys1_I$	=	system manifold pressure before I^{th} dose of gas onto sample (mmHg)
$Psys2_I$	=	system manifold pressure after I^{th} dose of gas onto sample (mmHg)
$Psam_I$	=	sample pressure after equilibrating I^{th} dose of gas onto sample (mmHg)
$Tsys1_I$	=	system manifold temperature before I^{th} dose of gas onto sample (K)
$Tsys2_I$	=	system manifold temperature after I^{th} dose of gas onto sample (K)
Wsam	=	weight of sample (g)
V_I	=	amount of gas adsorbed per gram of sample (cm^3/g STP)

Equilibration

Equilibration is reached when the pressure change per equilibration time interval (first derivative) is less than 0.01% of the average pressure during the interval. Both the first derivative and average pressure are calculated using the Savitzky-Golay[1] convolution method for polynomial functions. The equations below are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

$$P_{AVG} = \frac{-36(P_{11} + P_1) + 9(P_{10} + P_2) + 44(P_9 + P_3) + 69(P_8 + P_4) + 84(P_7 + P_5) + 89(P_6)}{429}$$

$$P_{CHG} = \frac{5(P_{11} - P_1) + 4(P_{10} - P_2) + 3(P_9 - P_3) + 2(P_8 - P_4) + (P_7 - P_5)}{110}$$

where the numerical constants are from the Savitzky-Golay[1] convolution arrays, and

P_{AVG} = average pressure (mmHg)
P_{CHG} = change in pressure (mmHg)
P_I = I^{th} pressure reading taken at equilibrium intervals (mmHg)

After equilibration has been reached, if the user-entered minimum equilibration time from the Low Pressure Options dialog box has not elapsed, the equilibration continues until the entered time has elapsed.

If the user entered a non-zero maximum equilibration time from the Low Pressure Options dialog box, and this time period has elapsed before equilibration has been reached, the equilibration ends as if equilibration had been reached, and the point is collected.

If a non-zero value that is too small is entered for the maximum equilibration time, the points are collected before equilibration is reached.

If P_{AVG} is greater than 0.995 times the current Po, equilibration will not take place until the *Minimum equilibration delay for P/Po 0.995* has expired, in addition to the standard equilibration criteria.

1. Savitzky, A. and Golay, M.J.E., *Anal. Chem. 36*, 1627 (1964).

Data Smoothing

Introduction

The data smoothing feature of the chemisorption program was developed to cope with noise and drift of pressure readings at very low pressures - usually at pressures well under 1 mmHg and very low gas volumes adsorbed. Data smoothing in the chemisorption program requires eleven points to satisfy the technique employed. However, most chemisorption analyses are performed with fewer than eleven points and at pressures higher than 1 mmHg, and so the data smoothing option is turned off by default. It may be enabled if required.

Technique

The analysis information consists of absolute pressure and volume adsorbed data pairs (P,V). In order to account for negative transducer shift, all coordinate pairs are scanned for points with pressures less than or equal to zero. If such points exist, then all pressure data are shifted by an amount large enough to convert the smallest pressure to a nominally small positive value.

After examination of the low pressure data is complete, the entire data set is subdivided into equally spaced intervals along the volume axis and corresponding pressures for these volumes are interpolated. The pressure data are smoothed by passing them through an eleven-point Blackman[2] convolution array. Once the smoothing is complete, the volume adsorbed values from the original data set are used to interpolate back to the corresponding smoothed analysis pressures. This amended set of data, consisting of the original volume adsorbed values and the fully smoothed pressure values, forms the isotherm data from which all subsequent report data are based. Coefficients for the Blackman convolution array are given below.

$$\begin{aligned} P_{SMOOTHED} = {} & (0.0029918)P_{I-5} + (0.020005)P_{I-4} + (0.0618580)P_{I-3} + \\ & (0.1250300)P_{I-2} + (0.1850900)P_{I-1} + (0.2100500)P_{I} + \\ & (0.1850900)P_{I+1} + (0.1250300)P_{I+2} + (0.061850)P_{I+3} + \\ & (0.0200050)P_{I+4} + (0.0029918)P_{I+5} \end{aligned}$$

where

$P_{SMOOTHED}$ = I^{th} smoothed pressure

P_I = pressure coordinate from the I^{th} equally spaced data point

Five data points are constructed on both the left and right ends to allow the filter to operate.

2. Blackman, R.B. and Tukey, J.S., “The Measurement of Power Spectra,” Dover (1958).

Stoichiometry Factor

The calculated stoichiometry factor is a weighted average. It is dependent on both the individual stoichiometry factor and number of moles of each active metal.

$$SF_{CALC} = \frac{\frac{\%weight1 \times SF1}{W_{ATOMIC1}} + \frac{\%weight2 \times SF2}{W_{ATOMIC2}} + \frac{\%weight3 \times SF3}{W_{ATOMIC3}} + \ldots + \frac{\%weightn \times SFn}{W_{ATOMICn}}}{\frac{\%weight1}{W_{ATOMIC1}} + \frac{\%weight2}{W_{ATOMIC2}} + \frac{\%weight3}{W_{ATOMIC3}} + \ldots + \frac{\%weightn}{W_{ATOMICn}}}$$

where

SF_{CALC}	=	calculated stoichiometry factor
SF1	=	stoichiometry factor for first metal
SF2	=	stoichiometry factor for second metal
SF3	=	stoichiometry factor for third metal
SFn	=	stoichiometry factor for nth metal
%weight1	=	% of sample weight for first metal
%weight2	=	% of sample weight for second metal
%weight3	=	% of sample weight for third metal
%weightn	=	% of sample weight for nth metal
$W_{ATOMIC1}$	=	atomic weight of first metal (g/mole)
$W_{ATOMIC2}$	=	atomic weight of second metal (g/mole)
$W_{ATOMIC3}$	=	atomic weight of third metal (g/mole)
$W_{ATOMICn}$	=	atomic weight of nth metal (g/mole)

Metal Dispersion

The metal dispersion is the percentage of the active metal available for interaction with the adsorbate.

$$\%M_{DISP} = \frac{100\% \times 100\%}{22414^*} \times \frac{V \times SF_{CALC}}{\frac{\%weight1}{W_{ATOMIC1}} + \frac{\%weight2}{W_{ATOMIC2}} + \frac{\%weight3}{W_{ATOMIC3}}}$$

where

$\%M_{DISP}$	=	metal dispersion (%)
V	=	(cm^3/g STP) If $\%M_{DISP}$ is being calculated from analysis data, then V is the volume intercept derived from the best line fit of the points selected for line fit. If $\%M_{DISP}$ is being calculated from difference data, then V is V_{DIFF})
SFCALC	=	calculated stoichiometry factor
%weight1	=	% of sample weight for first metal
%weight2	=	% of sample weight for second metal
%weight3	=	% of sample weight for third metal
$W_{ATOMIC1}$	=	atomic weight of first metal (g/mole)
$W_{ATOMIC2}$	=	atomic weight for second metal (g/mole)
$W_{ATOMIC3}$	=	atomic weight of third metal (g/mole)
V	=	volume intercept derived from the best line fit to the volume differences between the selected points of the first analysis and the repeat analysis

*The volume one mole of gas occupies (cm^3 STP/mole of gas)

Metallic Surface Area

The metallic surface area per gram of sample is the total active metal surface area available for interaction with the adsorbate.

$$Msa = \frac{6.023 \times 10^{23}}{22414*} \times V \times SF_{CALC} \times A_{AREA}$$

where

Msa	=	metallic surface area (m^2/g sample)
V	=	(cm^3/g STP)) If Msa is being calculated from analysis data, then V is the volume intercept derived form the best line fit of the points selected for line fit by the user. If Msa is being calculated from difference data, then V is the average of the volume differences (V_{DIFF}) for the points selected for line fit.
A_{AREA}	=	effective area of 1 active metal atom (m^2/atom)
SF_{CALC}	=	calculated stoichiometry factor
V_{DIFF}	=	difference in volume between the first analysis and the repeat analysis

*The volume one mole of gas occupies (cm^3 STP/mole of gas)

Assuming that all species of metals disperse evenly, the metallic surface area per gram of metal is

$$Nsa = Msa \cdot \frac{100\%}{\%weight1 + \%weight2 + \%weight3}$$

where

Nsa	=	metallic surface area (m^2/g metal)
Msa	=	metallic surface area (m^2/g sample)
%weight1	=	% of sample weight for first metal
%weight2	=	% of sample weight for second metal
%weight3	=	% of sample weight for third metal

Freundlich Isotherm

The Freundlich isotherm has the form:

$$\frac{Q}{Q_S} = CP^{1/m}$$

where

Q	=	quantity of gas adsorbed
Q_S	=	quantity of gas in a monolayer
C	=	temperature-dependent constant
m	=	temperature-dependent constant

The pressure is absolute; typically, m > 1. In terms of quantity adsorbed,

$$Q = Q_S CP^{1/m}$$

Taking the log of both sides yields:

$$\log Q = \log Q_S C + \frac{1}{m} \log P$$

Temkin Isotherm

The Temkin isotherm has the form,

$$\frac{Q}{Q_S} = \frac{RT}{q_0\alpha} \ln(A_0 P)$$

where

Q	=	quantity of gas adsorbed
Q_S	=	quantity of gas in a monolayer
q_0	=	the differential heat of adsorption at zero surface coverage
A_0	=	$a_0 \exp\{-q_0/RT\}$, where α_0 and a_0 are adjustable constants

In terms of quantity adsorbed,

$$Q = \frac{RTQ_S}{q_0\alpha}\left[\ln A_0 + \ln\left(\frac{P}{Po}\right)\right]$$

Thus, the plot of the natural log of absolute pressure vs. quantity adsorbed yields a straight line with slope RTQ_S/q_0 and intercept (ln A_0) $RTQ_S/q_0\alpha$.

D. FORMAT OF EXPORTED DATA

This appendix details the format and meaning of data in unreduced reports. Each record is terminated by a carriage return and line feed. Character strings are delimited by double quotation marks.

Record position indicates the relative position of a record within a group. The different types of counter records indicate those sections of the file having a variable number of records; counter records specify the number of entries (types of information) contained in a table.

Record Position	Information Conveyed	Form
1	Sample ID	quoted string (50 characters)
2	Submitter ID	quoted string (40 characters)
3	Operator ID	quoted string (40 characters)
4	Bar code	quoted string (40 characters)
5	Sample mass	floating point
6	Type of data 0 = Automatically collected 1 = Manually entered	integer
7*	Count of data points	integer
	(Each entry for this counter appears on one line, separated with a comma.)	
	Pressure value	floating point
	Elapsed time	integer
	Quantity dosed	floating point
	Repeat Pressure value	floating point
	Repeat Elapsed time	integer
	Repeat Volume dosed	floating point
	Line fit flag 0 = no 1 = yes	integer
	(All the repeat measurements are not valid if a pressure was skipped on the second analysis. Invalid entries have a zero value for the pressure, elapsed time, and volume dosed.)	

E. DEFAULT FILES AND SYSTEM FILES

The following tables describe the default files provided with the ASAP 2020 Chemi software.

Table E-1. Default Sample Information Files

File	Description
100.SMP	ZSM-5 NH3 at 100 C
150.SMP	ZSM-5 NH3 at 150 C
250.SMP	ZSM-5 NH3 at 250 C
300.SMP	ZSM-5 NH3 at 300 C
350.SMP	ZSM-5 NH3 at 350 C
50.SMP	ZSM-5 NH3 at 50 C
75.SMP	ZSM-5 NH3 at 75 C
CO.SMP	CO after H2
PTCO_08.SMP	PTCO_08 - Pt. Alumina, Tube C1
SC0018.SMP	Pt. Alumina, Fixed Dose CO
SC0019.SMP	Pt. Alumina, Fixed Dose CO, 1st Run, No repeat
SC0020.SMP	Pt. Alumina, Fixed Dose CO, 2nd Run, No repeat
SC0022.SMP	Ag Method #3
SC0023.SMP	Ag Method #3
SC0028.SMP	Nickel Method 4
SC0036.SMP	0.5% pt p2 m2 un2 3rd run
SC0038.SMP	0.5% pt p2 m2 un2 1st run

Table E-2. Default Report Options Files

File	Description
FULL.RPO	Full: includes Isotherm, Analysis, and Options.
PT_CO.RPO	Report options for platinum-alumina sample.

Table E-3. Default Analysis Conditions Files

File	Description
AG_O2.ANC	Silver using oxygen.
NI_H2.ANC	Nickel surface area using hydrogen.
PD_H2.ANC	Palladium surface area using hydrogen.
PD_TITR.ANC	Palladium surface area using hydrogen-oxygen titration.
PT_CO.ANC	Platinum or palladium using carbon monoxide.
PT_H2.ANC	Platinum surface area using hydrogen.
PT_TITR.ANC	Platinum surface area using hydrogen-oxygen titration.

Table E-4. Other Files Created by the ASAP 2020 Chemi Program

File	Description
*ASAP202C.INI	ASCII file containing initialization information used during program startup as well as system options information.
*ASAP202C.SMP	Binary file containing 2020 Chemi sample defaults.
GASDATA.MDB	Material database containing the default Active Metals Table and the default Gas Table.
(serial number).SST	Binary file containing 2020 Chemi instrument status information and calibration data.
	**Do not attempt to edit this file. Personal injury or damage to the ASAP 2020 could result because the operator may be given erroneous status information.**
ASAP202C.LOG	ASCII file containing a recorded log of all error messages displayed on the screen.
*(serial number).LOG	Binary log file for analyzer with serial number contents accessible using the Show Instrument Log option on the Unit menu.
*You should back up these files periodically to ensure data integrity. You may want to store each backup set on a separate media device so you have access to different versions of the files. For example, ASAP202C.SMP contains the default sample information. Any time the sample defaults are changed, an updated ASAP202C.SMP is created. Keeping version backups of this file on a separate media device allows restoration of any version needed.	

F. ATOMIC WEIGHTS AND CROSS-SECTIONAL AREAS FOR SELECTED METALS

Metal	Symbol	Atomic Weight (g/mole)	Cross-Sectional Area (sq nm)	Density (g/mL)
chromium	Cr	51.996	0.0635	7.19
cobalt	Co	58.933	0.0662	8.9
copper	Cu	63.546	0.0680	8.96
gold	Au	196.967	0.08696	18.9
hafnium	Hf	178.490	0.0862	13.3
iridium	Ir	192.220	0.0769	22.4
iron	Fe	55.847	0.0613	7.89
manganese	Mn	54.938	0.0714	7.43
molybdenum	Mo	95.940	0.0730	10.2
nickel	Ni	58.710	0.0649	8.8
niobium	Nb	92.906	0.0806	8.57
osmium	Os	190.220	0.0629	22.6
palladium	Pd	106.400	0.0787	12.0
platinum	Pt	195.090	0.0800	21.4
rhenium	Re	186.207	0.0649	21.0
rhodium	Rh	102.906	0.0752	12.4
ruthenium	Ru	101.070	0.0613	12.4
silver	Ag	107.868	0.0869	10.5
tantalum	Ta	180.947	0.0800	16.6
thorium	Th	232.038	0.1350	11.7
tin	Sn	118.710	0.1082	4.54
tungsten	W	183.850	0.0741	19.3
vanadium	V	50.942	0.0680	6.11
zirconium	Zr	91.220	0.0877	6.51

G. CHEMISORPTION METHODS

This appendix describes the preparation steps for standard methods of chemisorption. These procedures, which have been developed over a number of years, are recommended starting points for creating other methods. Methods 1 and 2 require a repeat analysis. Methods 3 and 4 do not.

Method 1 Prepares the sample for carbon monoxide as the analysis gas. Procedure is given for platinum.

Method 2 Prepares the sample for hydrogen as the analysis gas. Procedures are given for platinum, palladium, and nickel.

Method 3 Prepares the sample for oxygen as the analysis gas: usually used with silver.

Method 4 Prepares the sample for hydrogen-oxygen titration. Procedures are given for platinum and palladium.

For each of the methods, the recommended vacuum set point is 5 µmHg, and the recommended temperature increase rate is 10 ºC per minute.

Samples are evacuated to the vacuum set point, and evacuation continues for the specified TIME.

FLOW (soak) = ramp up to the specified TEMPERATURE, and then continue flowing (soaking) for the specified TIME.

TIME = total time below the vacuum set point that the sample spends at the specified TEMPERATURE or PRESSURE.

Method 1 - Carbon Monoxide Analysis of Platinum

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-1. Carbon Monoxide Analysis of Platinum

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Flow oxygen		100	5
3	Flow oxygen	10	350	30
4	Evacuate		350	15
5	Evacuate	10	100	15
6	Flow hydrogen		100	5
7	Flow hydrogen	10	350	120
8	Evacuate		350	30
9	Evacuate	10	35	30
10	Analysis		35	

Analysis

Gas: Carbon monoxide
Temperature: 35 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: Yes
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min

Free space: Measure
Incremental dosing: No
Line Fit: Enabled for all pressure points
Pressure table (mmHg): 200, 250, 300, 350, and 400

Method 2 - Hydrogen Analysis

Method 2 contains three subsets of instructions for hydrogen analysis. The metals analyzed are platinum, palladium, and nickel.

Platinum

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-2. Hydrogen Analysis of Platinum

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Flow oxygen		100	5
3	Flow oxygen	10	350	30
4	Evacuate		350	15
5	Evacuate	10	100	15
6	Flow hydrogen		100	5
7	Flow hydrogen	10	350	120
8	Evacuate		350	30
9	Evacuate	10	35	30
10	Analysis		35	

Analysis

Adsorptive: Hydrogen
Temperature: 35 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: Yes
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min*

Free space:	Measure
Incremental dosing:	No
Line fit:	Enabled for all pressure points
Pressure table (mmHg):	75, 110, 140, 170, and 200

*Experience may indicate the need for a longer evacuation time.

Palladium

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-3. Hydrogen Analysis of Palladium

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Flow oxygen		100	5
3	Flow oxygen	10	350	30
4	Evacuate		350	15
5	Evacuate	10	100	15
6	Flow hydrogen		100	5
7	Flow hydrogen	10	350	120
8	Evacuate		350	30
9	Evacuate	10	100	30
10	Analysis		100	

Analysis

Adsorptive: Hydrogen
Temperature: 100 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: Yes
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min

Free space: Measure
Incremental dosing: No
Line fit: Enabled for all pressure points
Pressure table (mmHg): 120, 155, 190, 225, and 260

Nickel

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-4. Hydrogen Analysis of Nickel

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Evacuate	10	450	5
3	Flow hydrogen		450	120
4	Evacuate		450	30
5	Evacuate	10	35	30
6	Analysis		35	

Analysis

Adsorptive: Hydrogen
Temperature: 35 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: Yes
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min

Free space: Measure
Incremental dosing: No
Line fit: Enabled for all pressure points
Pressure table (mmHg): 100, 150, 200, 250, and 300

Method 3 - Oxygen Analysis of Silver

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-5. Oxygen Analysis of Silver

Task Number	Action	Temperature (ºC)	Time* (minutes)
1	Evacuate	100	30
2	Flow O_2	100	10
3	Ramp temperature while flowing O_2	170	10 º/min
4	Hold temperature while flowing O_2	170	60
5	Evacuate	170	30
6	Flow H_2	170	60
7	Evacuate temperature <10 mmHg	170	30
8	Analysis	170	

*Time for evacuation is the number of minutes below set point at the specified temperature. Time for gas flow is the number of minutes at the specified temperature.

Analysis

Gas:	Oxygen
Temperature:	170 ºC
Line fit:	Enabled for all pressure points
Repeat analysis:	No
Pressure table (mmHg):	40, 67, 93, and 120

Method 4 - Hydrogen-Oxygen Titration

Method 4 contains two subsets of instructions for hydrogen-oxygen titration. The metals analyzed are platinum and palladium.

Platinum

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-6. Hydrogen-Oxygen Titration of Platinum

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Flow oxygen		100	5
3	Flow oxygen	10	350	30
4	Evacuate		350	15
5	Evacuate	10	100	15
6	Flow hydrogen		100	5
7	Flow hydrogen	10	350	120
8	Evacuate		350	30
9	Evacuate	10	35	30
10	Flow oxygen		35	60
11	Evacuate		35	30
12	Analysis		35	

Analysis

Adsorptive: Hydrogen
Temperature: 35 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: No
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min

Free space:	Measure
Incremental dosing:	No
Line fit:	Enabled for all pressure points
Pressure table (mmHg):	75, 110, 140, 170, and 200

Palladium

Preparation

Rate of temperature increase = 10 ºC per minute
Vacuum set point = 5 µmHg

Table G-7. Hydrogen-Oxygen Titration of Palladium

Task Number	Action	Ramp Rate (ºC/min)	Temperature (ºC)	Time (minutes)
1	Evacuate	10	100	30
2	Flow oxygen		100	5
3	Flow oxygen	10	350	30
4	Evacuate		350	15
5	Evacuate	10	100	15
6	Flow hydrogen		100	5
7	Flow hydrogen	10	350	120
8	Evacuate		350	30
9	Evacuate	10	100	30
10	Flow oxygen		100	60
11	Evacuate		100	30
12	Analysis		100	

Analysis

Adsorptive: Hydrogen
Temperature: 35 ºC
Heat rate: n/a

Equilibration interval: 10 sec
Relative target tolerance: 5.0%
Absolute target tolerance: 5.0 mmHg

Repeat analysis: No
Fast evacuation: No
Unrestricted evac pressure: 30 mmHg
Evacuation time: 30 min

Free space: Measure
Incremental dosing: No
Line fit: Enabled for all pressure points
Pressure table (mmHg) 120, 155, 190, 225, and 260

H. FLOWMETER CONVERSION FACTORS

The gas flowmeter on the front panel displays the gas flow rate for air in standard cubic centimeters per minute (sccm). You must divide the displayed gas flow rate by a conversion factor from the table below to obtain the actual flow rate for gases other than air.

Example: Oxygen is flowing through the meter at a displayed rate of 30 sccm (air). The actual flow rate is:

30 ÷ 1.05 = 28.6 sccm

Example: You require a helium flow rate of 40 sccm. In this case, multiply the required flow rate by the conversion factor as follows:

40 x 0.37 = 14.8 sccm (air)

Adjust the gas flow until the gas flowmeter displays 14.8 sccm.

Table H-1. Flowmeter Conversion Factors

Gas	Factor
Air	1.00
Argon	1.18
Butane	1.42
Carbon dioxide	1.23
Carbon monoxide	0.98
Cyclopropane	1.21
Ethane	1.02
Helium	0.37
Hydrogen	0.26
Isobutane	1.42
Methane (natural gas)	0.75
Neon	0.83
Nitrogen	0.98
Nitrous oxide	1.23
Oxygen	1.05
Propane	1.23
Sulfur hexafluoride	2.25

I. TESTING FOR LEAKS

Introduction

This appendix contains general instructions for testing the ASAP 2020 Chemi and the Smart-Vac degasser (if installed) for leaks. If the analyzer successfully performs a blank tube analysis using nitrogen, you do not need to test for leaks.

These procedures may be performed individually or sequentially. Record pressure readings on the Valve Test Data Sheet included at the end of this appendix. After you finish testing for leaks, replace any leaking valves.

You will need the following items to perform tests:

- Stopwatch
- Valve Test Data Sheet (included in this appendix)

Testing Individual Valves

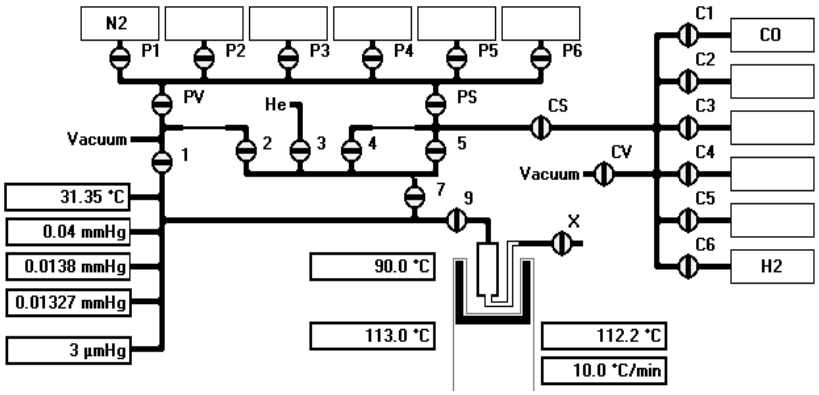
Analysis Valves

This procedure removes differential pressure from all valves in the analysis system and establishes that there are no leaks in the analysis system. It should be performed before testing for individual valves.

1. Ensure that the analyzer is idle.
2. Close the regulator outlet valve for each gas supply line to the analyzer.
3. Attach a clean, empty sample tube to the analysis port.

Ensure that a Po tube is installed on the Po port and a plug is inserted into the vapor inlet.

4. Select **Unit > Show Instrument Schematic**, then **Unit > Enable Manual Control**. (For clarity, this illustration shows only the valve portion of the schematic.) Table I-1 lists descriptions of the analysis valves.



5. Close all valves.
6. Open valve CV, then open valves C1, C2, C3, C4, C5, and C6.
7. Wait 30 minutes.
8. Close valves C1, C2, C3, C4, C5, C6, and CV.
9. Open valves CS, 5, 1, 2, and 7.
10. Open valve 9.
11. Evacuate the system to below 10 µmHg, then continue evacuation for 30 minutes.

Table I-1. Analysis Valve Descriptions

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Helium inlet port
4	Restricted analysis gas
5	Unrestricted analysis gas
7	Lower manifold isolation
9	Sample port
CV	Vacuum valve, allows evacuation of chemisorption gas inlet manifold
CS	Supply valve, supplies gas to manifold for chemisorption operation
C1 through C6	Gas inlet port valves for chemisorption operation
X	Exhaust from sample tube to gas flow meter, located at sample port above furnace
PV	Vacuum valve, allows evacuation of physisorption gas inlet manifold
PS	Supply valve, supplies gas to manifold for physisorption operation
P1 through P6	Gas inlet port valves for physisorption operation

Valve X

1. Close valves 1 and 7.
2. Take a pressure reading; record it as P1 for valve X.
3. Wait 10 minutes and take another pressure reading; record it as P2 for valve X.
4. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve X. This value should be less than 0.5 µmHg/min.

Valve 9

1. Close valve 9.
2. Open valve X.
3. Take a pressure reading; store it as P1 for valve 9.
4. Wait 10 minutes and take another pressure reading; record it as P2 for valve 9.
5. Subtract P1 from P2 and divide the difference by 10; record the value as the 1st outgassing rate for valve 9. This value should be less than 0.5 µmHg/min.

Chemi Gas Inlet Manifold

1. Open valves 1, 2, 5, 7, CS, and CV.
2. Evacuate to below 10 µmHg, then allow evacuation for 15 minutes.
3. Close valves 1, 2, and CV.
4. Take a pressure reading; record it as P1 for the chemi gas inlet manifold.
5. Wait 10 minutes and take another pressure reading; record it as P2 for the chemi gas inlet manifold.
6. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for the chemi gas inlet manifold. This value should be less than 10 µmHg/min.

Valves C1 through C6

1. Open the regulator outlet valve for the gas connected to port C1.
2. Take a pressure reading; record it as P1 for valve C1.
3. Wait 10 minutes and take another pressure reading; record it as P2 for valve C1.
4. Subtract P1 from P2 and divide the difference by 10; record this value as the outgassing rate for valve C1. This value should be less than 0.7 µmHg/min.
5. Repeat Steps 1 through 4 for each gas inlet port (C2 through C6) to which a gas is attached. Record the values on the Valve Test Data Worksheet in the spaces provided.
6. Close valves 5 and CS.
7. Open valves 1 and 2.

Leak testing for physisorption-related valves and the degas manifold are explained in Appendix D of the ASAP 2020 physisorption operator's manual.

Valve Test Data Sheet

Make a copy of this form to record pressure readings and outgassing rates when leak-testing system valves.

VALVE(s)	P1 (1st pressure reading)	P2 (2nd pressure reading)	OUTGASSING RATE
Analysis Valves			
X			
9			
Chemi Gas Inlet Manifold			
C1			
C2			
C3			
C4			
C5			
C6			

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