ASAP 2420 Confirm

Accelerated Surface Area and Porosimetry System

Operator's Manual

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TABLE OF CONTENTS

1. GENERAL INFORMATION

Organization of the Manual 1-3
Conventions 1-4
Online Manual 1-5
- Using Bookmarks 1-5
- Using the Table of Contents, Index, and other Links 1-7
  - Table of Contents 1-7
  - Index 1-8
  - Cross References 1-8
- Using the Find Command 1-9
- Printing 1-10

Equipment Description 1-11
Gas Requirements 1-12
Analysis Program 1-13
Internet Access 1-14
Specifications 1-15

2. USER INTERFACE

User Levels 2-3
Controls, Indicators, and Connectors 2-4
- Front Panels 2-4
  - Lower Front Panel 2-5
  - Upper Front Panel 2-6
- Right Side Panel 2-8
  - Upper Right Side Panel 2-8
  - Lower Right Side Panel 2-9
- Back Panel 2-10

Turning On the System 2-11
Turning Off the System 2-11
Using the Software 2-12
- Logging In 2-12
- Shortcut Menus 2-12
- Shortcut Keys 2-13
- Dialog Boxes 2-14
- File Name Conventions 2-15

Common Software Commands 2-16
- Save 2-16
- Save as Template 2-17
- Close 2-17
- Cancel 2-17
- Replace All 2-17

Common Software Functions 2-18

File Selection . . . 2-19
Selecting a Directory . . . 2-19
Selecting a File Status . . . 2-20
Selecting a Range of Dates . . . 2-21
Report Settings . . . 2-22
Menu Structure . . . 2-23
Windows Menu . . . 2-24
Help Menu . . . 2-24

3. OPERATING PROCEDURES

Sample Information Files and Templates . . . 3-3
Sample Information Files . . . 3-3
Templates . . . 3-4
Creating Sample Information Templates . . . 3-5
Creating Parameter Templates . . . 3-7
Creating Parameter Files . . . 3-8
Sample Tube . . . 3-8
Degas Conditions . . . 3-10
Analysis Conditions . . . 3-12
Adsorptive Properties . . . 3-14
Report Options . . . 3-16
Creating Sample Information Files . . . 3-19
Analyst . . . 3-19
Developer . . . 3-22
Preparing Samples . . . 3-24
Choosing Sample Tubes . . . 3-24
Cleaning and Labeling Sample Tubes . . . 3-24
Determining Amount of Sample to Use . . . 3-28
Determining the Mass of the Sample . . . 3-28
Selecting Files for Degassing or Analyzing Samples . . . 3-31
Selecting a Predefined Sample Information File . . . 3-31
Creating a Sample Information File . . . 3-32
Degassing Samples . . . 3-33
Attaching Sample Tubes to Degas Ports . . . 3-33
Degassing a Single Sample . . . 3-35
Degassing Multiple Samples . . . 3-36
Filling Dewars . . . 3-38
Precautions . . . 3-38
Dewar Covers . . . 3-38
Filling an Analysis Dewar . . . 3-39
Transferring a Degassed Sample to an Analysis Port . . . 3-41
Degassing Samples on Analysis Ports . . . 3-45
Performing an Analysis . . . 3-51
Standard Analysis . . . 3-52
Krypton Analysis . . . 3-54
High Throughput Analysis . . . 3-56
Micropore Analysis . . . 3-58
Printing File Contents . . . 3-60
Print Sample Files or Templates . . . 3-60

Print Parameter Files or Templates . . . 3-61
Listing File Statistics . . . 3-62
List Sample File or Template Statistics . . . 3-63
List Parameter File or Template Statistics . . . 3-64
Importing Sample and Parameter Files . . . 3-65
Exporting Sample Data . . . 3-66
Generating Graph Overlays . . . 3-68
Multiple Samples Overlay . . . 3-68
Multiple Graphs Overlay . . . 3-71

4. FILE MENU

Description . . . 4-3
New . . . 4-5
Sample Information . . . 4-6
Analyst . . . 4-6
Developer . . . 4-10
Sample Tube . . . 4-14
Degas Conditions . . . 4-16
Analysis Conditions . . . 4-19
Adsorptive Properties . . . 4-34
Report Options . . . 4-37
Summary Report . . . 4-41
Isotherm Report Options . . . 4-42
BET/Langmuir Surface Area Report Options . . . 4-44
Freundlich Isotherm . . . 4-47
Temkin Isotherm . . . 4-49
t-Plot Report Options . . . 4-51
Alpha-S Plot . . . 4-57
f-Ratio Plot . . . 4-59
BJH Adsorption/Desorption Report Options . . . 4-61
Horvath-Kawazoe Report Options . . . 4-69
DFT Pore Size . . . 4-75
DFT Surface Energy . . . 4-78
Dubinin Report Options . . . 4-79
MP-Method Report Options . . . 4-84
Options Report . . . 4-88
Sample Log Report . . . 4-88
Collected or Entered Data . . . 4-89
Open . . . 4-91
Sample Information File . . . 4-91
Analyst . . . 4-93
Developer . . . 4-93
Templates . . . 4-94
Parameter Files . . . 4-94
Save . . . 4-95
Save As . . . 4-96
Save As Template . . . 4-98
Save All . . . 4-98
Close . . . 4-98

Close All 4-99
Print 4-99
List 4-101
Import 4-102
Export 4-104
Convert 4-105
Log In 4-106
Log Out 4-106
Exit 4-107

5. UNIT MENU

Description 5-3
Start Analysis 5-5
Start Krypton Analysis 5-9
Start High Throughput Analysis 5-13
Start Micropore Analysis 5-17
Start Single Degas 5-21
Start Multiple Degas 5-22
Evacuate Analysis Port 5-24
Evacuate Degas Port 5-25
Enable Manual Control 5-26
    Analysis System 5-26
    Degas System 5-29
Show Analysis Schematic 5-31
Show Degas Schematic 5-32
Show Analysis Status 5-33
Show Degas Status 5-34
Show Instrument Log 5-36
Unit Configuration 5-38
Diagnostics 5-40
Calibration Procedures and Service Tests 5-44

6. REPORTS MENU

Description 6-3
Start Report 6-4
Close Reports 6-5
Open Report 6-5
SPC Report Options 6-6
Regression Report 6-7
Control Chart 6-11
Printed Reports 6-14
    Header 6-14
    Onscreen Reports 6-15
        Tool Bar 6-16
        Shortcut Menus 6-19
    Zoom Feature 6-22
    Axis Cross Hair 6-23
Report Examples 6-23

Summary Report 6-24
Isotherm Tabular Report 6-25
Isotherm Linear Plot 6-27
Bet Surface Area 6-28
Bet Surface Area Plot 6-29
t-Plot Report 6-30
t-Plot 6-31
BJH Adsorption Pore Distribution 6-32
BJH Adsorption Cumulative Pore Volume 6-33
BJH Adsorption dV/dlog(D) Pore Volume 6-34
BJH Desorption Pore Distribution 6-35
BJH Desorption Cumulative Pore Volume 6-36
BJH Desorption dV/dlog(D) Pore Volume 6-37

7. OPTIONS MENU

Description 7-3
Add Archive Location 7-4
Edit Labels 7-5
Update File List 7-5
Units 7-6
Graph Grid Lines 7-6
Live Graph 7-7
Service Test Mode 7-7

8. TROUBLESHOOTING AND MAINTENANCE

Troubleshooting 8-3
Preventive Maintenance 8-7
Checking and Cleaning the Analysis Port Dewars 8-8
Replacing the Port Filters 8-9
Analysis Ports 8-10
Degas Ports 8-11
Replacing a Sample Tube O-ring 8-12
Accessing the Vacuum Pumps 8-14
Inspecting and Changing Vacuum Pump Fluid 8-16
Inspecting Fluid 8-16
Changing Fluid 8-17
Replacing the Alumina in the Oil Vapor Traps 8-20
Lubricating the Elevator Screws 8-25
Cleaning the Analyzer 8-26
Checking the Analysis Manifold Outgassing Rate 8-26
Checking the Degas Manifold Outgassing Rate 8-29
Testing the Analyzer for Leaks 8-30
Replacing the Vacuum Pump Exhaust Filter 8-31
Routine Maintenance 8-33
Changing Gases 8-33
Guidelines for Connecting Gases to the Analyzer 8-33
Connecting a Gas Regulator to the Gas Supply 8-33
Connecting the Gas Delivery Tubing to the Analyzer 8-35

Specifying Gas Ports 8-36
Performing a Reference Material Analysis 8-36

9. ORDERING INFORMATION

Analysis System Components and Accessories 9-3
Sample Tubes and Components 9-6

APPENDICES

A. ASAP SERIES SAMPLE DATA WORKSHEET

B. ERROR MESSAGES

2200, 2300, and 2400 Series B-3
2500 Series B-19
4000 Series B-28
6100 Series B-44
6200 Series B-45
6500 Series B-54

C. CALCULATIONS

Saturation Pressure (Po) C-3
Relative Pressure C-3
Free Space C-4
Volume Adsorbed C-5
Equilibration C-6
Thermal Transpiration Correction C-7
BET Surface Area C-8
Langmuir Surface Area C-9
t-Plot C-10
BJH Pore Volume and Area Distribution C-12
Explanation of Terms C-12
Calculations C-13
Compendium of Variables C-20
Horvath-Kawazoe C-21
Slit Pore Geometry (original HK) C-21
Cylinder Pore Geometry (Saito/Foley) C-22
Sphere Pore Geometry (Cheng/Yang) C-23
Cheng/Yang Correction C-24
Interaction Parameter C-24
Additional Calculations C-25
Dubinin-Radushkevich C-28
Dubinin-Astakhov C-29
MP-Method C-33
Freundlich Isotherm C-35
Temkin Isotherm C-35
DFT (Density Functional Theory) C-36
The Integral Equation of Adsorption C-36

Application to Surface Energy Distribution . . . C-37
Application to Pore Size Distribution . . . C-37
Performing the Deconvolution . . . C-38
Regularization . . . C-38
Summary Report . . . C-38
SPC Report Variables . . . C-40
Regression Chart Variables . . . C-40
Control Chart Variables . . . C-41
References . . . C-42

D. TESTING THE ANALYZER FOR LEAKS

Valve Locations . . . D-3
Degas Manifold . . . D-3
Analysis Manifold . . . D-5
Degas Manifold Valves . . . D-7
Analysis Manifold Valves . . . D-9
Gas Inlet Valves . . . D-11
Po Valves . . . D-13
What To Do If You Detect a Leaking Valve . . . D-15
Removing Differential Pressure from a Leaking Valve . . . D-15
Repairing or Replacing a Leaking Valve . . . D-15
Repairing Valves on the Analysis Manifold . . . D-15
Replacing Valves on the Degas and Gas Inlet Manifold . . . D-16
Replacing a Servo Valve . . . D-17
Repairing the Degas Solenoid Valve . . . D-17
Valve Test Data Sheet . . . D-18

E. DFT MODELS

Models Based on Statistical Thermodynamics . . . E-3
Theoretical Background . . . E-3
Molecular Simulation Methods . . . E-4
Molecular Dynamics Method . . . E-4
Monte Carlo Method . . . E-4
Density Functional Formulation . . . E-5
Models Included . . . E-10
Non-Local Density Functional Theory with Density Independent Weights . . . E-10
Modified Non-Local Density Functional Theory with Density-Dependent Weights . . . E-10
Models Based on Classical Theories . . . E-14
Surface Energy . . . E-14
Pore Size . . . E-14
Models Included . . . E-15
Kelvin Equation with Halsey Thickness Curve . . . E-15
Kelvin Equation with Harkins and Jura Thickness Curve . . . E-16
Kelvin Equation with Broekhoff-de Boer Thickness Curve . . . E-16
References . . . E-17

INDEX

1 GENERAL INFORMATION

Organization of the Manual	1-3
Conventions	1-4
Equipment Description	1-11
Gas Requirements	1-12
Analysis Program	1-13
Internet Access	1-14
Specifications	1-15

GENERAL INFORMATION

This manual provides a description of the ASAP 2420 analyzer and the menu options and functions available for the ASAP 2420 Confirm program. This manual covers operations for a Developer and an Analyst. A Developer is authorized to perform all functions listed on the menu bar. An Analyst is restricted to certain functions which are enabled; the functions not authorized for an Analyst are disabled.

Organization of the Manual

The manual is divided into the following chapters:

1. GENERAL INFORMATION
Provides a general description of the ASAP 2420 system as well as its specifications.

2. USER INTERFACE
Provides basic instrument and software interface.

3. OPERATING PROCEDURES
Provides step-by-step instructions for operating the ASAP 2420.

4. FILE MENU
Provides a description of the commands on the File menu.

5. UNIT MENU
Provides a description of the commands on the Unit menu.

6. REPORTS MENU
Provides a description of the commands on the Reports menu and samples of reports.

7. OPTIONS MENU
Provides a description of the commands on the Options menu.

8. TROUBLESHOOTING AND MAINTENANCE
Provides instructions for troubleshooting hardware problems and for performing routine maintenance procedures.

9. ORDERING INFORMATION
Provides part numbers and descriptions of the ASAP 2420 System components and accessories.

A. ASAP SERIES SAMPLE DATA WORKSHEET
Contains a form to assist you in determining the sample mass.

B. ERROR MESSAGES
Lists the error messages that may be displayed in the analysis program; includes a cause and action for each.

C. CALCULATIONS
Contains the calculations used by the system to produce reports.

D. TESTING THE ANALYZER FOR LEAKS
Describers the procedure for checking each valve for leaks.

E. DFT MODELS
Provides information on DFT models.

Conventions

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

Notes contain a tip or important information pertinent to the subject matter.

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

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

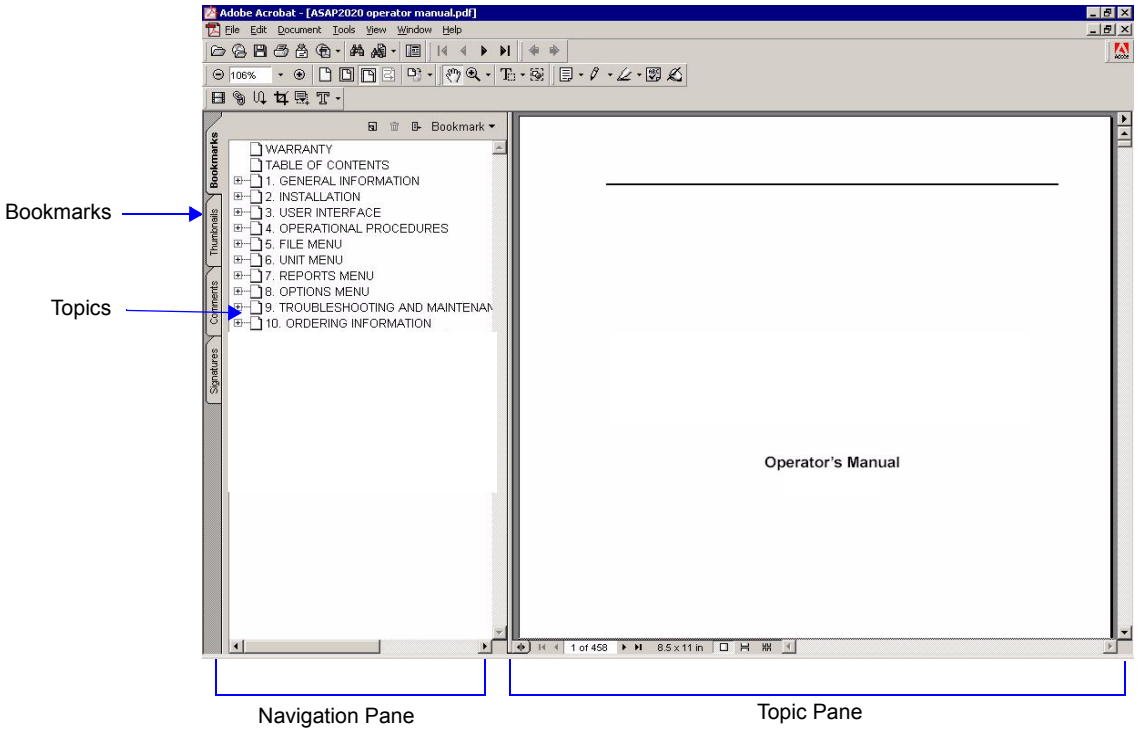
Online Manual

For your convenience, the Operator's Manual is available online. You can access the manual by selecting **Help**, then **Operator's Manual** from the analysis program main menu. The manual appears in an Adobe® Acrobat® Reader®.

Following are some tips to help you quickly locate the information you need in the manual. Refer to the Adobe Acrobat Help system (click the **Help** button on the Acrobat menu) for more information on the Acrobat features you can use while viewing the manual.

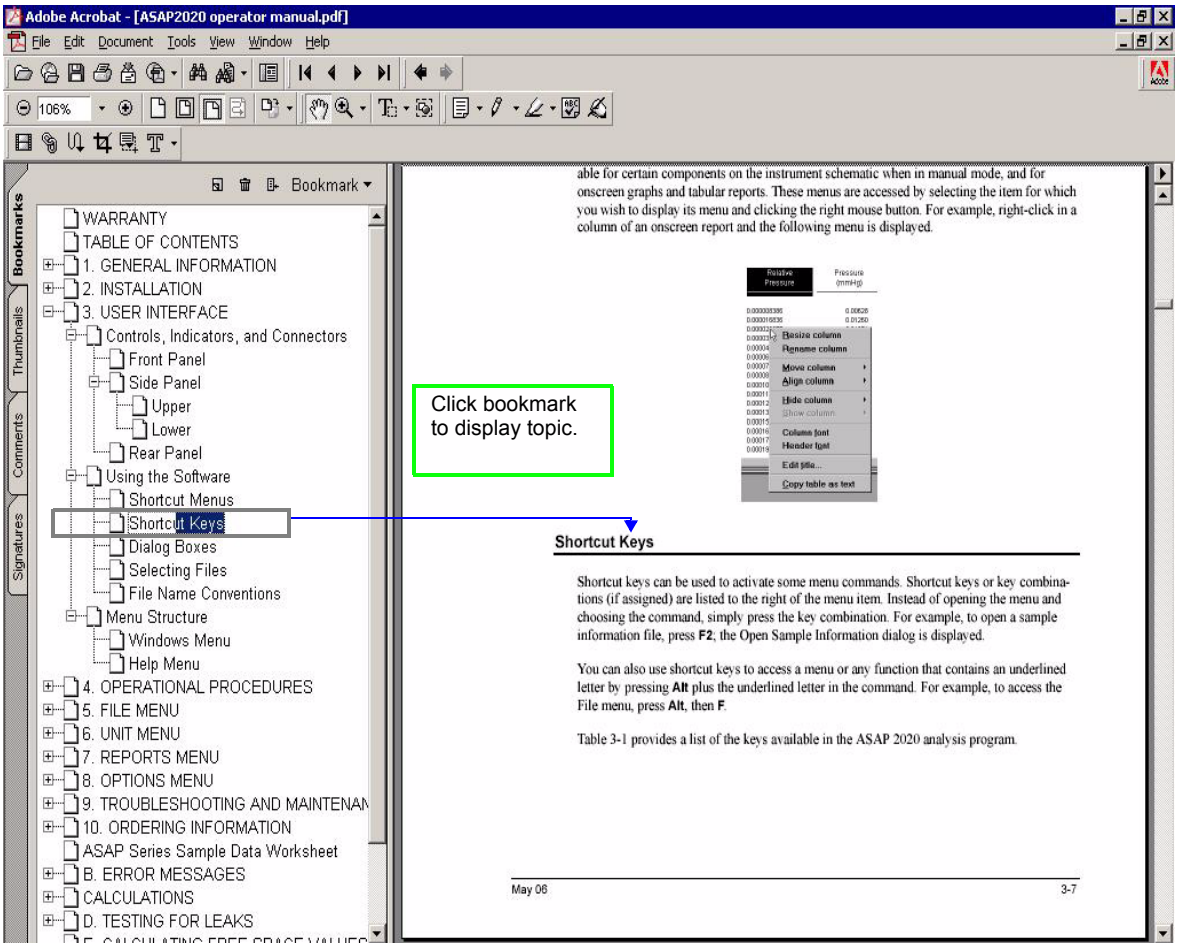
Using Bookmarks

Click the **Bookmarks** tab to list and access the topics included in the manual.



You can use the + and − buttons next to topics as they are used in Windows Explorer to expand or collapse the topic list.

To display a topic, click the topic name in the Bookmarks section. The related information appears in the topic pane of the window as shown in the following example.



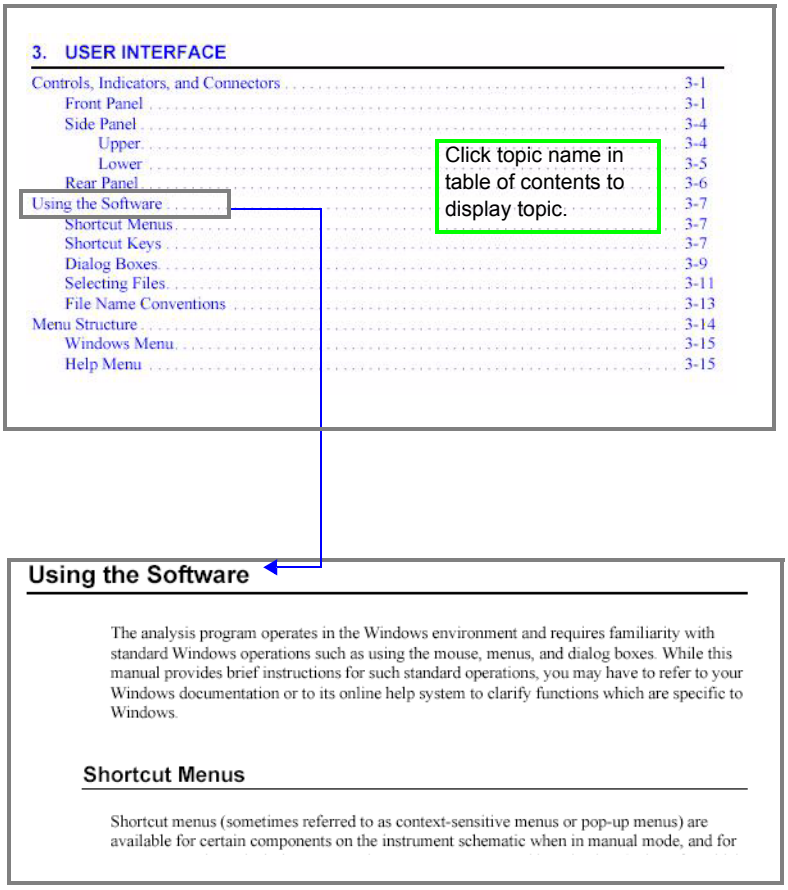
Using the Table of Contents, Index, and other Links

Links provide direct access to selected information. All links appear in blue type. Links are contained in:

- the table of contents
- index entries
- cross-references within the manual

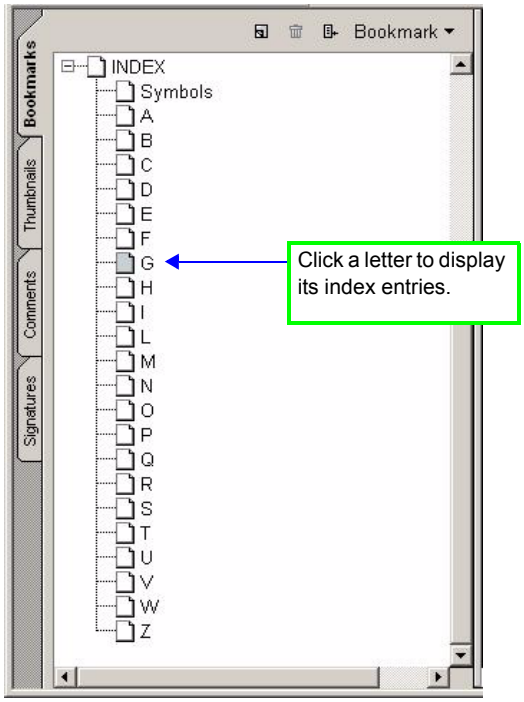
Table of Contents

To display the table of contents, click **Table of Contents** in the **Bookmarks** section. When the table of contents is displayed, you can click an entry to display its associated page. For example, clicking **Using the Software** in the table of contents, displays the page containing information about the software.



Index

To use the index in the online manual, click the **Bookmarks** tab, scroll down to **INDEX** (the last topic in Bookmarks), then click the + button to expand the index. The letters A through Z are displayed. Click a letter to display its corresponding index entries as shown in the following example.



After you display the entries, locate the item of interest and click on the page reference to access the information.

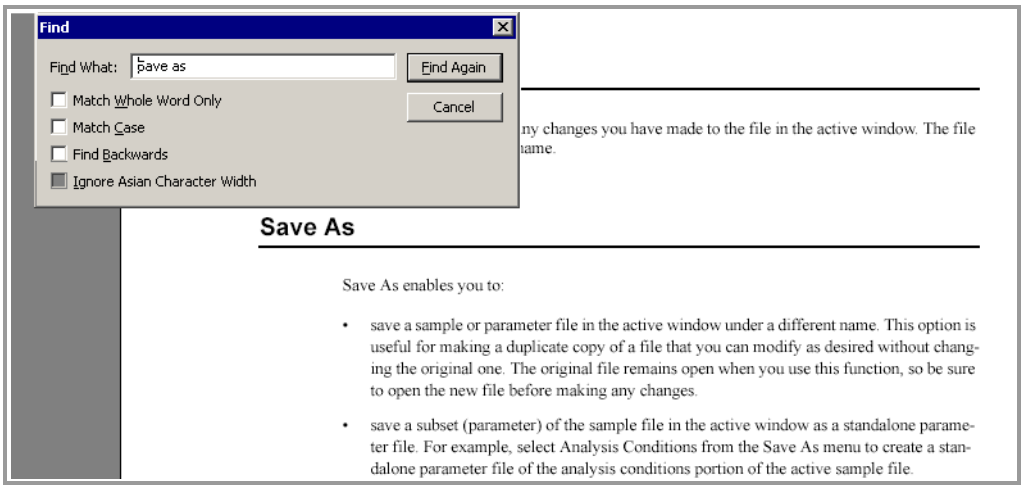
Cross References

Cross-references work in the same manner. In the example below, clicking on the cross-reference, **FILE MENU** (shown on the screen in blue type) will display the first page of the chapter describing the commands found on the File menu.

> **FILE MENU**
>
> Provides a description of the commands available on the File menu.

Using the Find Command

The Adobe Acrobat **Find** command provides another method of easily accessing specific information. For example, suppose you want to know how the **Save as** command works. You could select **Edit > Find** from the Adobe Acrobat menu, then enter **Save as** in the Find dialog. The following example shows the results.

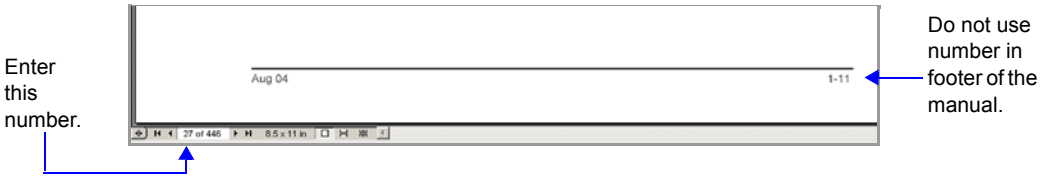


Printing

You can print the entire manual, a selected page, or range of pages. There are several options for printing. You can:

- Select the printer icon ( ) on the Adobe Acrobat toolbar.

  A standard Print dialog is displayed. Select the page(s) to print, then click **OK**. When using this option (or the next one), be sure to enter the page number(s) displayed in Adobe Acrobat; do not use the page number(s) listed in the footer(s) of the manual.



- Select **File > Print**.

  A standard Print dialog is displayed. Select the page(s) to print, then click **OK**.

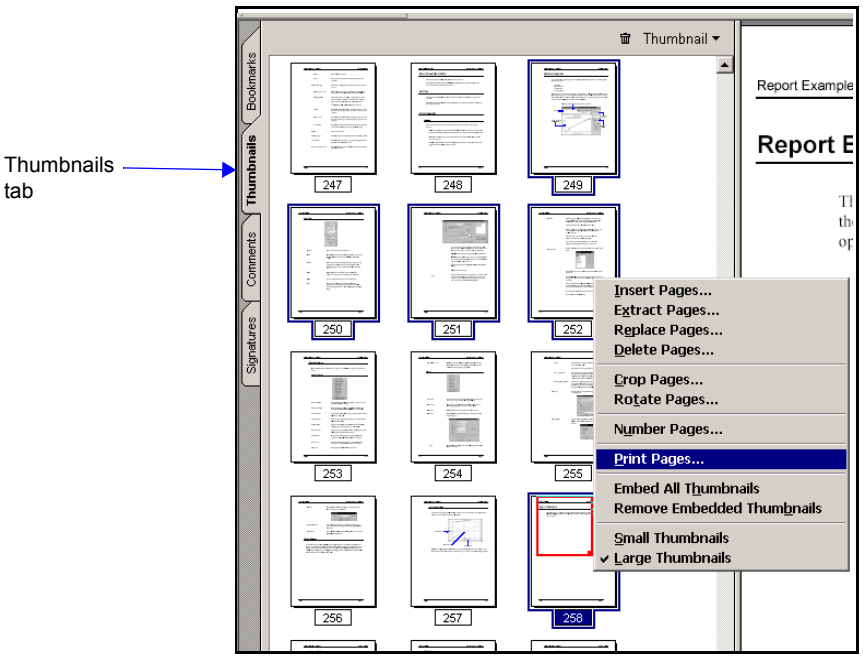
- Click the **Thumbnails** tab.

  Thumbnails of manual pages are displayed.

  a. Click the pages you want to print.

  b. Right-click to display a shortcut menu, then select **Print Pages**.

  c. A standard Print dialog is displayed; click **OK**.



Equipment Description

The ASAP 2420 surface area analyzer is actually two independent instruments in a single cabinet — one to degas samples and the other to analyze samples. A computer monitors and controls analyses, and reduces, reports and archives data for each sample.

The degas system has 12 automatically-controlled sample heat/vacuum ports. Each port operates independently. Samples may be added to or removed from degas ports with minimal disturbance to the treatment of other samples.

Temperature and rate of temperature change at each degas port can be set and monitored individually. Indicator lights above each degas port show the current state of the port. In addition, the degas operation can be monitored on the computer screen with the Instrument Schematic and Status windows.

The analysis system has six ports. The ports can be operated independently or concurrently and can perform analyses of at least 50 hours without refilling the Dewars. Each port can accept a range of sample tube neck diameters (1/4-, 3/8-, or 1/2-in. OD). Analysis operations can be monitored on the computer screen with the Instrument Schematic and Status windows.

For very low surface area analyses, a Krypton option is available. The option consists of a high-vacuum pump, low pressure transducer, and software. If this option is purchased with the instrument, it is factory-installed. It is also available as a field upgrade.

For analyses that require measurement and equilibration at pressures as low as 1.0 μm, the Micropore upgrade is available. When purchased with the instrument, this upgrade is factory-installed. The upgrade can also be added to 2420 krypton units in the field.

The ASAP analysis program supports four analysis modes:

- Standard
- Krypton
- High Throughput
- Micropore

In **Standard** mode, the system performs nitrogen, or similar gas, analyses at 77 K. All analyses must use the same gas. Samples can be removed and added to any of the six ports without disturbing the analyses being performed on other ports.

In **Krypton** mode, up to five Krypton analyses are started together. Port 6 is used to store purified Krypton for dosing. The data collection is done sequentially — one analysis starts and completes before the next is started.

In **High Throughput** mode, up to six nitrogen, or similar gas, analyses are started together. All analyses must use the same analysis gas and the same Psat gas, which may be different from the analysis gas. Once a set is started, no other samples can be started until the set is complete. The data collection is done in parallel. This mode also supports measured freespace.

In **Micropore** mode, up to six analyses are started together. Once a set is started, no other samples can be started until the set is complete.

Each analysis port of the ASAP 2420 is equipped with an elevator that raises and lowers the analysis bath fluid Dewar automatically. A removable shield to enclose the Dewar is also included for safety purposes.

The sample saturation pressure (Psat) tube is located next to the sample analysis port. Gas inlet ports and cable connections are located on the side panel of the analyzer for easy access.

The ASAP 2420 system includes Micromeritics' patented Isothermal Jackets for the sample tube. The Isothermal Jacket maintains a stable thermal profile along the full length of the sample and Psat tubes.

Gas Requirements

Compressed gases are required for analyses performed by the ASAP 2420 analyzer. Gas bottles or an outlet from a central source should be located near the analyzer. Up to five

different non-reactive adsorptives, for example, N_2, Ar, CO_2, and Kr, plus helium for free space can be attached to the analyzer simultaneously. A separate gas inlet is provided for the degas system. Helium or nitrogen are typically used for degas.

Appropriate two-stage regulators which have been leak-checked and specially cleaned are required. Pressure relief valves should be set to no more than 30 psig (200 kPag). Gas regulators are available from Micromeritics; refer to Chapter 9, **ORDERING INFORMATION**.

Analysis Program

The ASAP 2420 Confirm analysis program is designed to operate in a Windows 2000 or Windows XP Professional environment. The Windows environment provides a user-friendly interface for performing analyses and generating reports.

The analysis program monitors and controls the analyzer. It enables you to perform automatic analyses with just a few key strokes, 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 analysis program includes a report system which allows you to manipulate and customize reports. You can 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. Reports can be customized with your choice of fonts and a company logo added to the report header for an impressive presentation.

Internet Access

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

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Online Catalog	Catalog of instruments and accessories, allowing you to place your order online
News and Press	Press releases, Events calendar, microReports, and latest Micromeritics news updates
Lab Service	Provides laboratory tips and access to the Micromeritics Analytical Services web site
Customer Support	Customer support contacts, product registration, instrument training information, Material Safety Data Sheets, and account registration
Grant Program	Details of the Grant Program established for non-profit organizations and universities
Contact Us	Contact information, office locations, maps and driving directions to the Micromeritics facility, and registration for the microReport newsletter

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

Specifications

The ASAP 2420 system has been designed and tested to meet the specifications provided below.

Characteristic	Specification
ELECTRICAL	
Voltage:	100/115/230 VAC (± 10%)
Frequency:	50 or 60 Hz
Power:	800 VA, exclusive of vacuum pumps, which are powered separately
ENVIRONMENT	
Temperature:	10 to 30° C operating -10 to 55° C storage or shipping
Humidity:	Up to 90% (non-condensing) for instrument
CAPACITY	
Analysis Manifold:	6 sample ports (5 for krypton) each with a constantly-monitored saturation pressure port
Degas Manifold:	12 degas ports, each with an independently-controlled heating mantle
ANALYSIS MANIFOLD TEMPERATURE TRANSDUCER	
Type:	Platinum resistance device (RTD)
Accuracy:	± 0.10° C by keyboard entry
Stability:	± 0.10° C per month

Characteristic	Specification
PRESSURE MEASUREMENT	
Analysis Manifold Transducer:	
Range:	0 to 950 mmHg
Resolution:	1000-mmHg Transducer: 0.001 mmHg 10-mmHg Transducer*: 0.00001 mmHg
Accuracy (analysis system only), includes nonlinearity, hysteresis and nonrepeatability:	1000-mmHg Transducer: within 0.15% of reading 10-mmHg Transducer*: within 0.15% of reading Note: These are transducer manufacturer's specifications.
* Readings from the 10-mmHg transducer are actively taken only in systems with the krypton or micropore option. The 10 mmHg transducer, even when present, is not utilized when running samples in standard (N2, etc.) or high-throughput modes.	
Vacuum Transducers:	
Type:	Thermocouple
Range:	0.001 to 1 mmHg
VACUUM SYSTEM	
Pumps:	Nitrogen: 2 oil-based pumps: 1 analysis, 1 degas* Krypton: 4 pumps: 2 oil-free, 2 high-vacuum Oil-based mechanical pump: 5 x 10^{-3} mmHg ultimate vacuum. Oil-free and high vacuum pump: ultimate vacuum 3.8 x 10^{-9} mmHg. Ultimate vacuum measured by pump manufacturer according to Pneurop Standard 5608. *Can also be ordered with 4 pumps: 2 oil-free and 2 high-vacuum.

Characteristic	Specification
DEGAS SYSTEM	
Capacity:	12 degas ports
Vacuum Control:	Selectable target pressure controls switchover from restricted to unrestricted evacuation
Evacuation:	Selectable evacuation rate from 1.0 to 50.0 mmHg/s
Temperature Range:	Ambient to 450° C (Programmable)
Temperature Control:	1 ramp during evacuation phase, 5 additional selectable ramps during heating phase
Selection:	Digitally set, 1° C increments from computer
Accuracy:	Deviation less than ±10° C of set point at the sensing thermocouple embedded in the heating mantle
Backfill Gas:	User-selectable at dedicated port, typically nitrogen or helium
PHYSICAL	
Height:	159 cm (62.5 in.)
Width:	103 cm (40.5 in.)
Depth:	51 cm (20.2 in.)
Weight:	160 kg (350 lb)
COMPUTER	
Minimum requirements:	Pentium CPU (or equivalent) CD-ROM drive 128 megabytes of main memory 20-gigabyte hard drive SVGA monitor (1024 x 768 minimum resolution) Windows 2000 or Windows XP Professional Ethernet port, capable of communicating with a 10 base-T ethernet card

2 USER INTERFACE

Controls, Indicators, and Connectors	2-4
Using the Software	2-12
Common Software Commands	2-16
Common Software Functions	2-18
Menu Structure	2-23

USER INTERFACE

User Levels

The ASAP 2420 Confirm software is accessed by three different levels of users:

- **Administrator**: installs and maintains software and updates, establishes user accounts and rights (the Administrator responsibilities are covered in a separate manual).
- **Developer**: has access to all functions of the software. A Developer's primary function is to create templates for an Analyst.
- **Analyst**: creates sample files from predefined templates; some fields, such as the Mass field, are enabled and can be edited. An Analyst is also allowed to perform other tasks as designated throughout this manual. Tasks not allowed are disabled.

The following chart shows the ASAP 2420 program functions that can be performed by Developers and Analyses.

Function	Developer	Analyst
Create sample records from templates	✓	✓
Analyze samples	✓	✓
Generate reports	✓	✓
List and print sample records and templates	✓	✓
Perform routine maintenance	✓	✓
Enable manual control when the instrument is idle	✓	✓
Change limited analysis conditions before performing an analysis	✓	✓
Change report options after an analysis	✓	✓
Create analysis methods (templates) for analyst use	✓	
Perform all other Micromeritics application functions	✓	

This manual is written for the Developer and Analyst user levels. Functions for the Administrator level are covered in the Administrator Utility User's Guide.

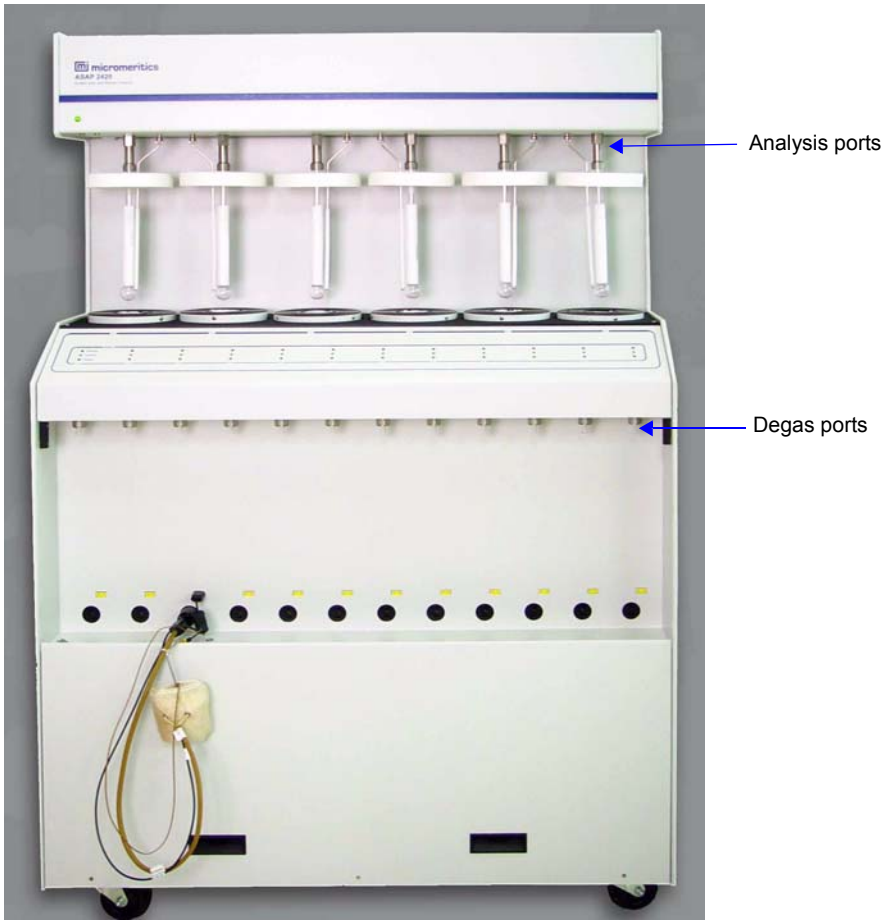
This chapter contains information to familiarize you with the hardware and software components of the ASAP 2420 system. It is recommended that you read this chapter before attempting to operate the ASAP 2420 system.

Controls, Indicators, and Connectors

This section contains a description of the controls, indicators, and connectors located on the front and side panels of the ASAP 2420 system.

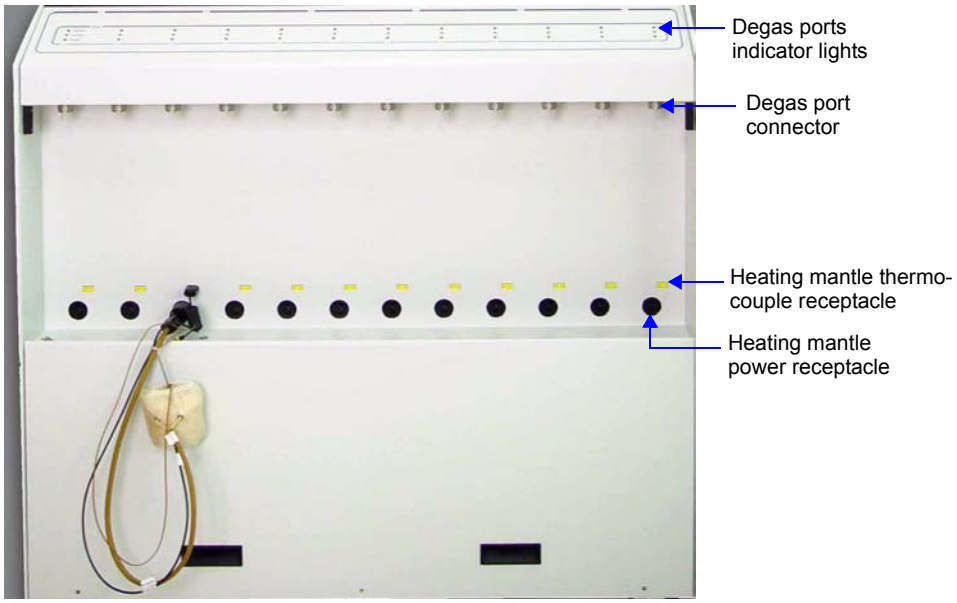
Front Panels

The upper front panel contains the analysis ports.The lower front panel contains the degas ports and indicator lights that show the status of each port.



Lower Front Panel

The lower front portion of the unit contains 12 degas stations, and an indicator light and heating mantle connections for each station.



Degas Port Indicator Lights Each degas port has three indicator lights located above the port. The lights show the current state of the port.

When an indicator light is red, do not touch the sample tube, heating mantle, or mantle clip on the associated port. These components may be hot enough to cause burns.

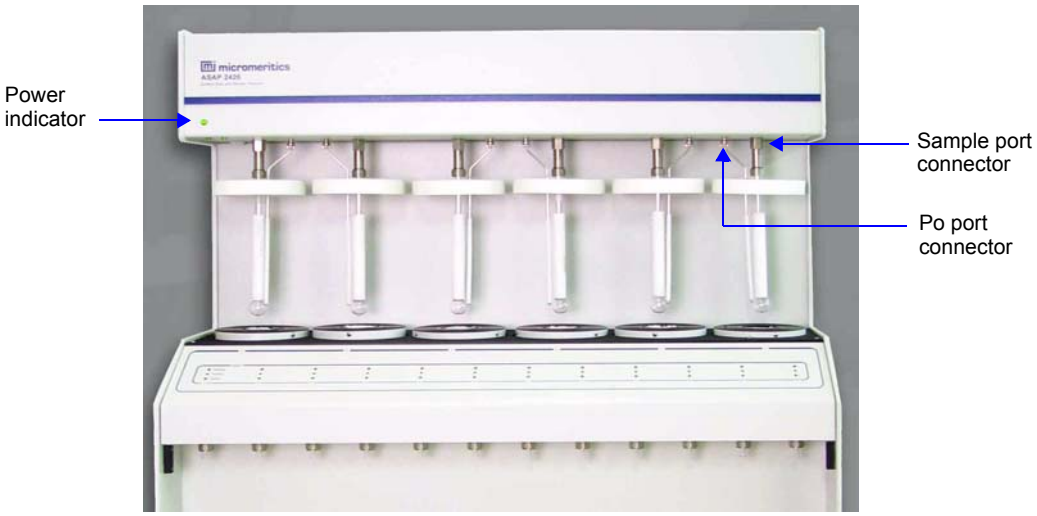
A blinking indicator light means the port valve is Open. Do not remove a sample tube or port plug while a valve is open. Doing so could contaminate the degas manifold.

Indicator Light Color	Description
Red	Heat is being applied to the port. Do not touch the sample tube, heating mantle, or heating mantle clip.
Yellow	Cool down is in progress. Heat is no longer being applied to the port, but the sample tube, heating mantle, and mantle clip may still be hot. Avoid touching these components.
Green	The port has cooled down and is idle. A degas operation can be started.

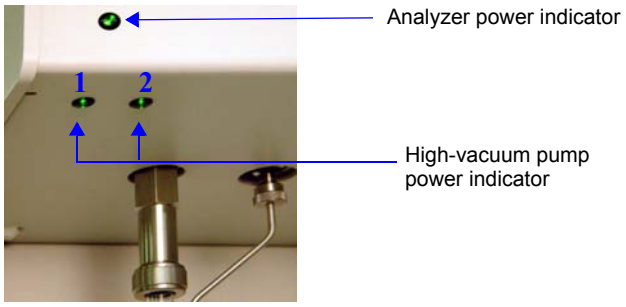
Degas port connector	Allows connection of a sample tube to the degas port.
Heating mantle thermocouple receptacle	Allows connection of a heating mantle thermocouple (one for each degas port).
Heating mantle power receptacle	Allows connection of the heating mantle power cord (one for each heating mantle).

Upper Front Panel

The upper portion of the unit contains six analysis stations and six saturation pressure ports. A power indicator is located on the left of the panel (as you face the unit).



Sample port connector	For installing the sample tube containing the material you wish to analyze.
Po port connector	For installing a Po (saturation pressure) tube.

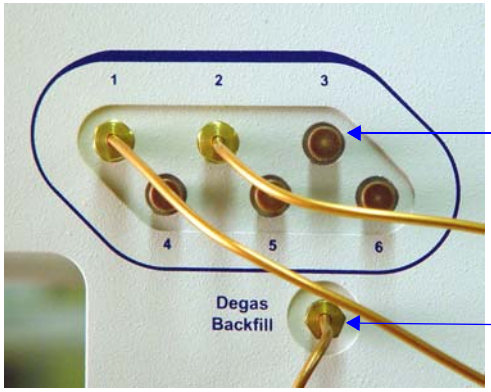


Analyzer power indicator Turns on and flashes green while the analyzer is attempting to communicate with the computer. Once communications are established, remains solid green.

High-vacuum pump power indicator These indicator lights turn on (and remain solid green) when power is being applied to the high-vacuum pump(s) and they are at full operating speed. Applies only to units with high-vacuum pumps installed.

Right Side Panel

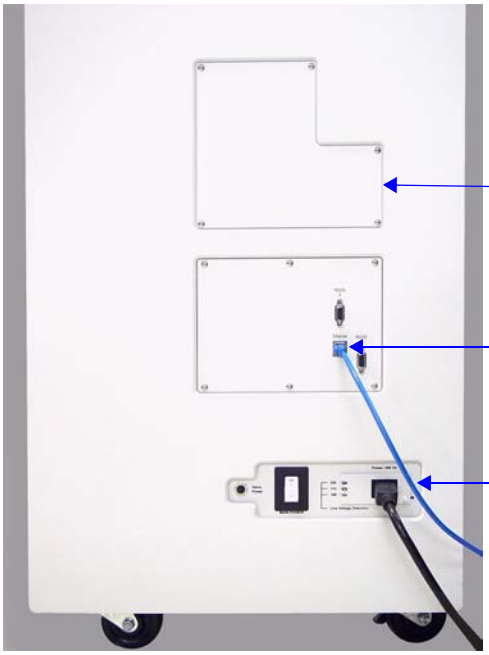
Upper Right Side Panel

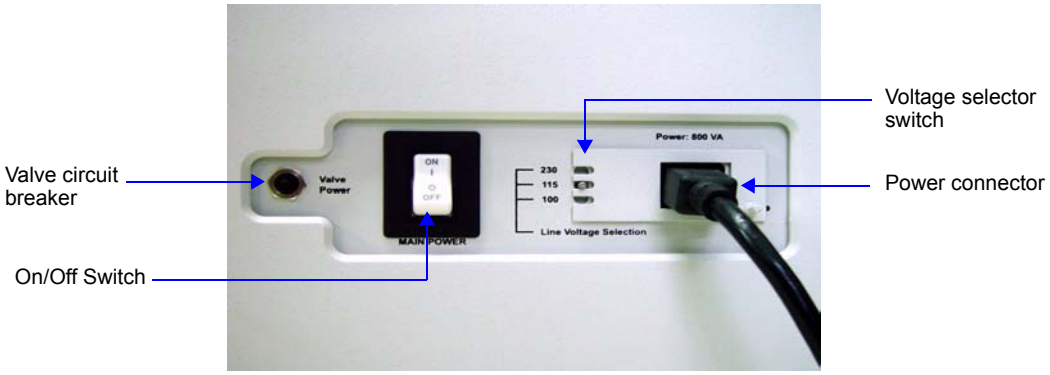


Gas inlet ports (1 through 6) — Used to connect up to six analysis gas supplies to the analyzer. When measuring free space, any one of the ports may be used for helium.

Degas Backfill port — Allows connection of a gas to use after degassing the sample to backfill the sample tube with a clean dry gas.

Lower Right Side Panel





Power Panel

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 power supply.

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.

Voltage selector switch For setting the analyzer to the correct incoming AC line voltage.

Back Panel

Vacuum pump housing The vacuum pumps are located on a shelf at the bottom on the unit. The pumps can be accessed by removing the lower back panel or the lower front panel of the unit.

Vacuum pump power connectors For connecting the vacuum pumps to the power supply.

Turning On the System

1. Place the ON/OFF switches for the computer and all peripheral devices in the ON position.
2. Place the analyzer ON/OFF switch in the ON position.

Turning Off the System

Always exit the ASAP 2420 program and/or Windows before turning off the computer. Failure to do so could result in loss of data.

1. Select **Close** from the System Menu or **Exit** from the File menu.
2. If you exit the ASAP 2420 program with analyses in progress, you will be warned of the operation. If desired, you can continue exiting the application and the analyses will proceed and continue to collect data. Reports that are queued under the Print Manager will print. If, however, a power failure occurs and an uninterruptible power supply (UPS) is not attached to the computer, the data collected after exiting the ASAP 2420 System program are lost.
3. Place the computer, monitor, and printer ON/OFF switches in the OFF position.
4. Place the analyzer Power switch in the OFF position.

Using the Software

The analysis program operates in the Windows environment and requires familiarity with standard Windows operations such as using the mouse, menus, and dialog boxes. While this manual provides brief instructions for such standard operations, you may have to refer to your Windows documentation or to its online help system to clarify functions which are specific to Windows.

Logging In

Each user — Administrator, Developer, or Analyst — is required to enter a user name and password when opening the application. Initially, the Administrator establishes passwords for the Developer and Analyst(s) when the software is installed. These passwords are temporary and are used for initial log-in; at that time, each user is prompted to specify a password that only he/she knows.

Your password must consist of at least six characters and expires after a specified time (designated by the Administrator). You will be locked out of the application if you fail to enter the correct password after three (default) tries. This number can be changed by your Administrator. The Administrator is also required to unlock the application in the event it becomes locked after failure to enter the correct password.

Shortcut Menus

Shortcut menus (sometimes referred to as context-sensitive menus or pop-up menus) are available for certain components on the instrument schematic when in manual mode, and for onscreen graphs and tabular reports. These menus are accessed by selecting the item for which you wish to display its menu and clicking the right mouse button. For example, right-click in a column of an onscreen report and the following menu is displayed.

Shortcut Keys

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (if assigned) are listed to the right of the menu item. Instead of opening the menu and choosing the command, simply press the key combination. For example, to open a sample information file, press **F2**; the Open Sample Information dialog is displayed.

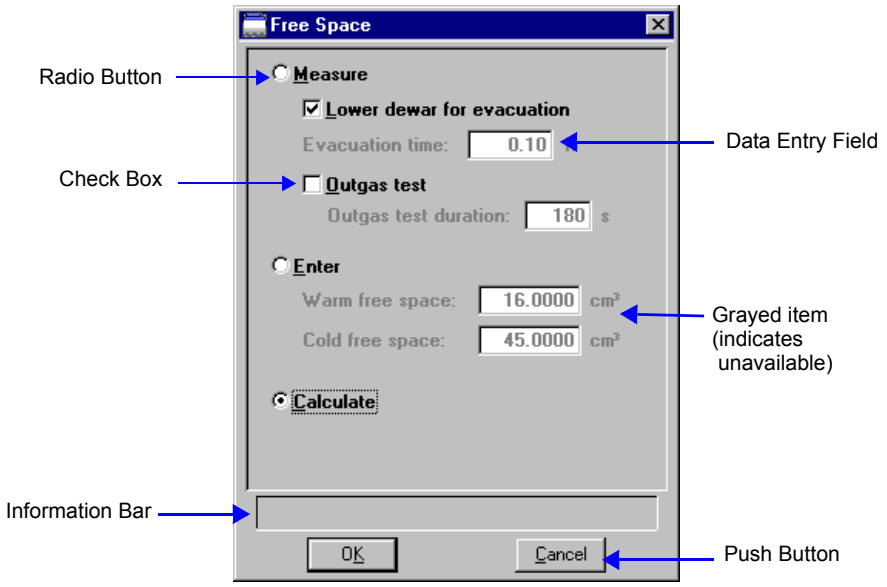
You can also use shortcut keys to access a menu or any function that contains an underlined letter by pressing **Alt** plus the underlined letter in the command. For example, to access the File menu, press **Alt**, then **F**.

Table 2-1: Shortcut Keys

Key(s)	Function
F1	Access online 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	Open an adsorptive properties file
	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
Alt + F4	Exit the ASAP 2420 program
Shift + F2	List sample information files
Shift + F3	List analysis conditions files
Shift + F4	List adsorptive properties files
Shift + F5	List report options files
Shift + F9	Access shortcut menu of (1) selected component on instrument schematic, or (2) onscreen reports

Dialog Boxes

Dialog boxes are displayed when a menu item or command push button is selected.



Elements that are often included in dialog boxes are described below. If an element is shown in gray instead of black, the element is currently unavailable.

Data Entry Fields

A Data Entry field is used to enter text; either numeric (numbers only) or alphanumeric (numbers, letters, or printable characters).

If you enter an invalid entry, for example, if you attempt to enter a number outside of the specified range, the field turns red.

Information Bar

Some dialog boxes contain information pertinent to the selected field in an information bar across the bottom of the dialog. For example, a range is shown for fields in which numeric entries are required.

Radio buttons

Radio buttons are provided in groups of two or more and are used to make a choice; only one radio button can be selected.

Check boxes

Check boxes allow you to choose multiple options from a group of options.

Push Buttons

A push button is used to display a subdialog box in which to enter additional information about the subject matter, or to cause an action.

For example:

- If you click the **Date Range** button on the Open Sample Information dialog, the Select Dates subdialog box is displayed.
- If you click **Cancel** on the Open Sample Information dialog box, your entries are canceled and the dialog is closed.

Drop-down list

A drop-down list contains a list of options and is indicated by a down arrow to the right of the field. If there are more items than can fit in the box, a scroll bar is provided for navigating through the list.

File Name Conventions

For sample information and report options files, a wildcard character (*) and a default extension are displayed.

The following table shows the file name extensions for the ASAP 2420 program.

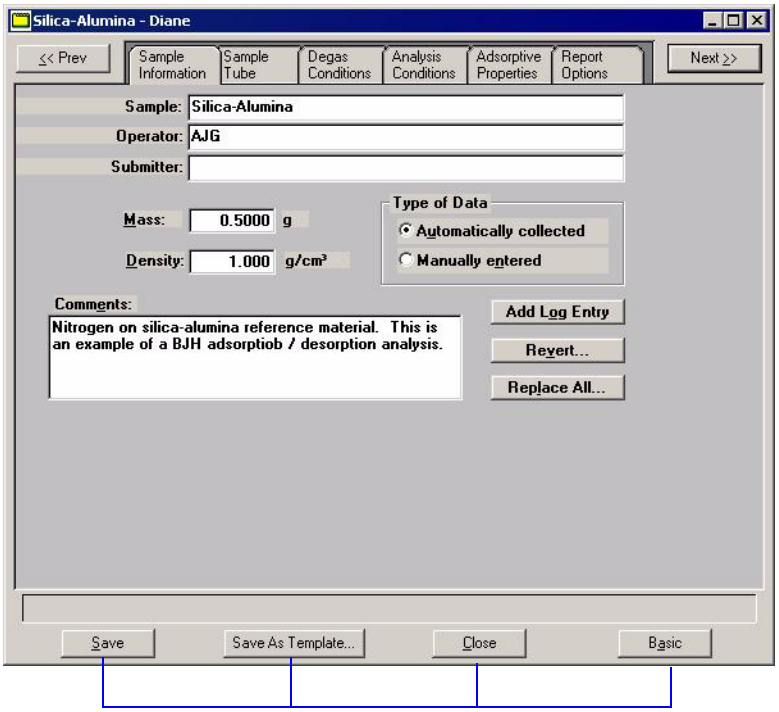
Table 2-2: Default File Name Extensions

File Type	Extension
Sample Information	SMP
Degas conditions	DEG
Analysis Conditions	ANC
Report Options	RPO
Adsorptive Properties	ADP
Export to disk (ASCII)	ISO
Sample information files from the ASAP 2000 analyzers; may be converted to an ASAP 2420-compatible format	DAT

File Type	Extension
Report to disk	RPT
List to disk	LST
Thickness curve	THK
Alpha-s curve	ALS
The following types are available for reports saved from the Report window	
Report	REP
Spreadsheet	XLS
ASCII	TXT

Common Software Commands

Many of the dialog boxes contain command push buttons, such as **Save**, that provide the same function in each dialog. These commands are described below.



Command Push Buttons

Save

Saves the information entered in the current session; the dialog box remains open.

Save as Template

Saves a sample information or parameter file as a template.This command is available only when a Developer is logged in.

Close

Closes the active dialog box. If the dialog box contains unsaved changes, you will be prompted to save them before the dialog box closes.

Any information entered in *subdialog* boxes is discarded also.

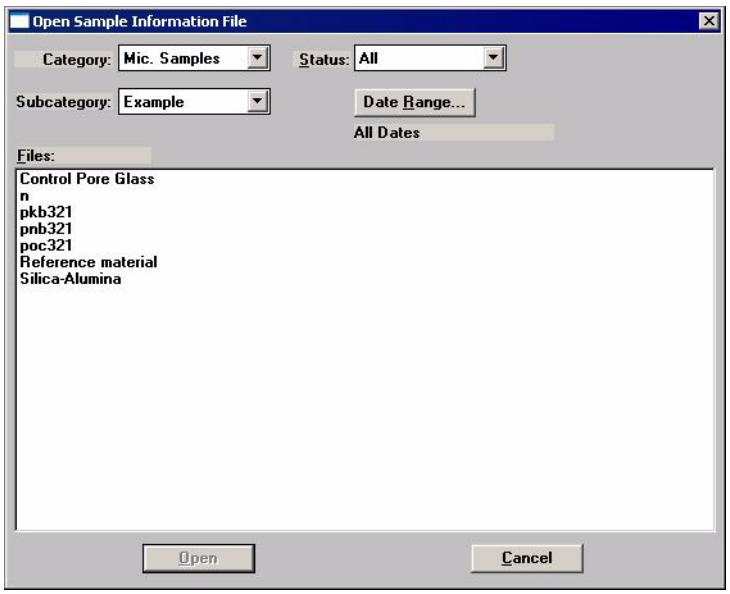
Cancel

Discards everything you entered in the dialog box and any subdialog boxes, and closes the dialog box. A warning message is displayed before closing.

Replace All

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

When you select **Replace All**, an Open dialog, similar to the one shown below, is displayed.

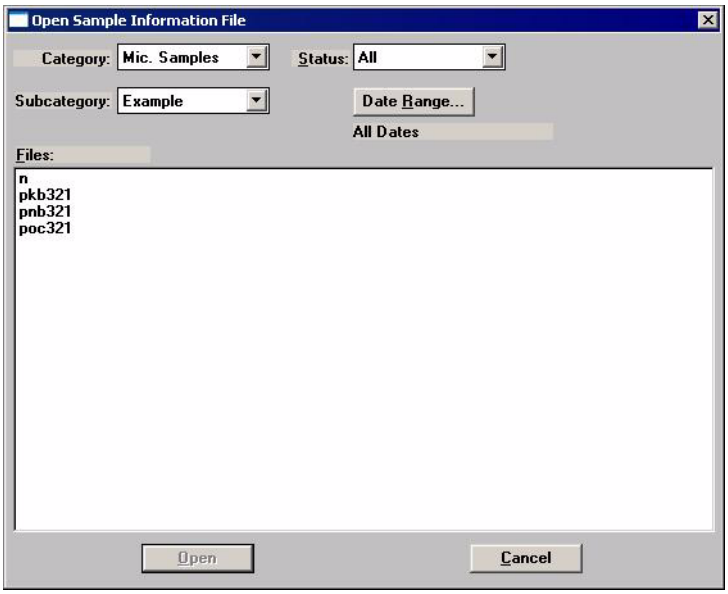


Select the file you wish to use, then click **OK**.

Common Software Functions

There are several software functions that are accessed on numerous dialogs.

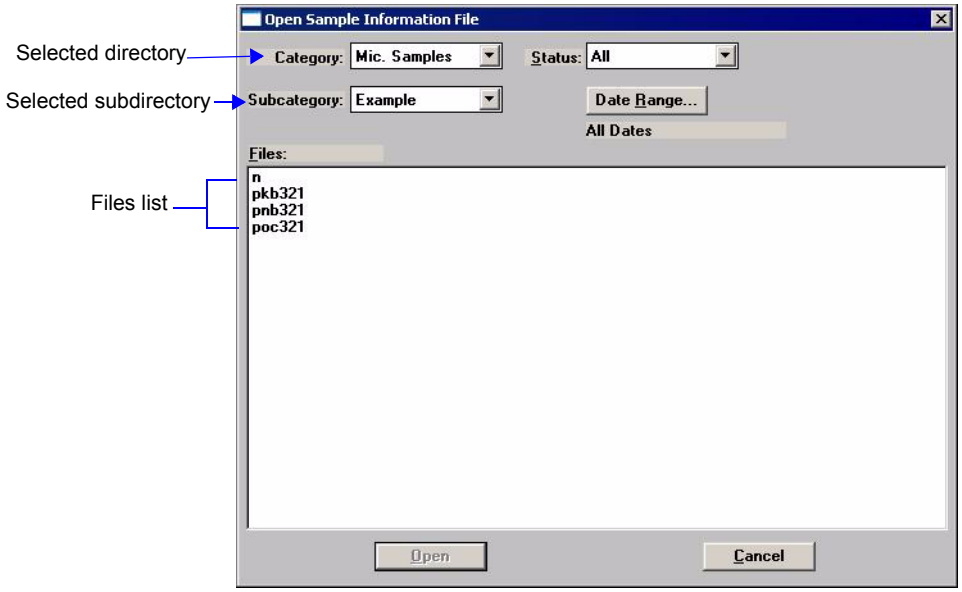
For example, there are dialogs from which you select a file to view, edit, or report on. The file is selected from a list of files that reside in the current directory. This list may be lengthy; so you may limit the files displayed by entering **Selection Criteria**.



This section of the manual describes the functions that are common to numerous dialogs in the 2420 analysis program.

File Selection

Certain dialogs contain a list of files available for that particular operation. For example, the Open Sample Information File dialog contains a list of sample files.

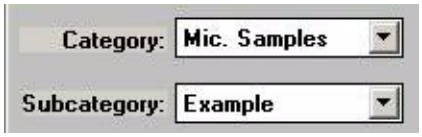


The files displayed in the **Files list** are those contained in the selected directory and having the status shown in the **Status** field. You can change or limit the list of files displayed by:

- Selecting another directory or subdirectory
- Selecting another file status
- Selecting a date or range of dates

Selecting a Directory

The selected directory and subdirectory are displayed in the **Category** and **Subcategory** fields. You can select another directory or subdirectory from the drop-down lists.



Selecting a File Status

You can select a file status from the **Status** drop-down list. The following table describes each file status.

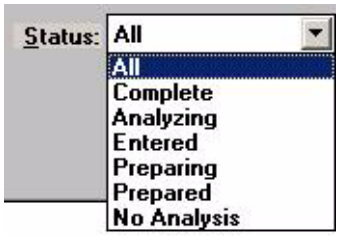
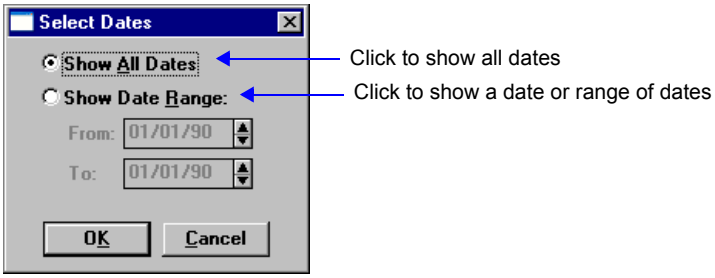


Table 2-3: File Status and Description

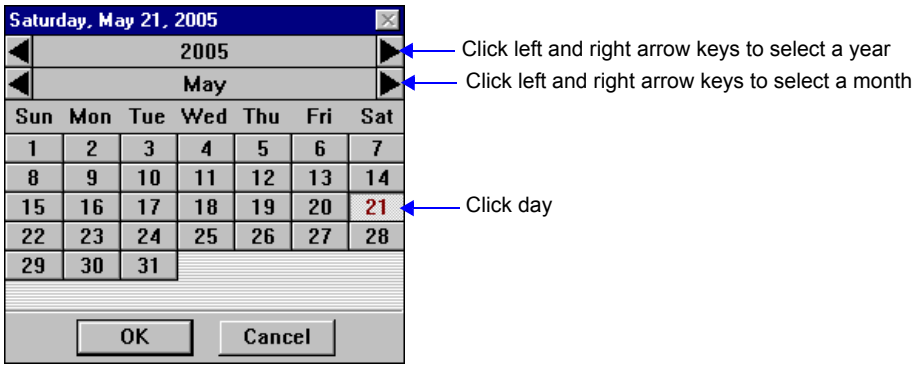
Status	Description
All	All sample information files in the specified directory and within the specified range of dates
Complete	Sample information files that have been used with an analysis
Analyzing	Sample information files that are currently being used with an analysis
Entered	Sample information files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

Selecting a Range of Dates

You can select to show all dates or a specific date or range of dates in the Select Dates group box.



- Click **Show All Dates** to show all files.
- Click **Show Date Range** to enter a date or range of dates. The From and To fields become enabled. You can either enter a beginning and ending date in these fields or double-click in each field to display a calendar to select a date.



a. Use the left and right arrow keys to select the year and month.

b. Click the day on the calendar.

c. Click **OK.**

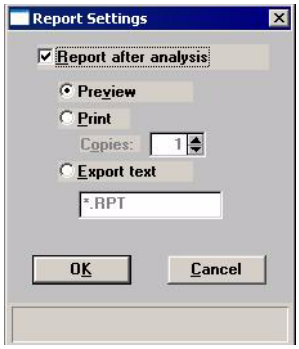
The range of dates remains the default until you change the dates or select **Show All Dates**.

For convenience, the following shortcut keys are available for selecting dates:

F2 Clears the date
F3 Inserts the current date
F4 Displays a calendar from which you may select a date

Report Settings

You can select whether or not to send reports to the screen, printer, or a file at the completion of an analysis. The **Report After Analysis** button on the analysis screens displays the Report Settings dialog.



Report after analysis

Select **Report after analysis** to generate reports when the analysis is complete and to select the reports' destination; deselect if you do not want to generate reports upon analysis completion.

If you do not select **Report after analysis**, you can generate reports later using the **Start Report** command on the Reports menu.

Preview

Click **Preview** to display reports on the screen.

Print

Click **Print** to send reports to your default printer.

Copies

If you selected **Print**, use the up and down arrows to select a number or enter a number in the field. You may print from 1 to 4 copies.

Export text
(An Analyst is not authorized to export text)

Click **Export text** to save report data in a text file. Graphical data cannot be sent to a File.

Enter a file name in the field below the radio button. The default report name is the name of the sample file with an .RPT extension. When you export a file, the file is saved in the **Export** directory.

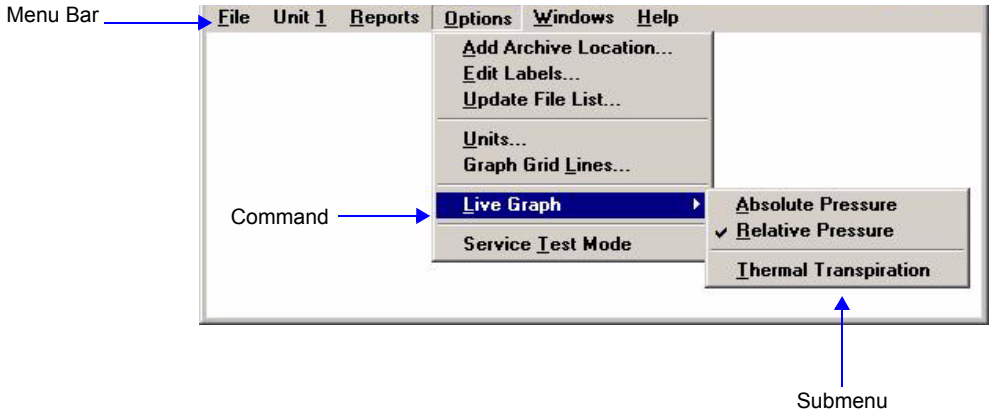
This function is displayed only when a Developer is logged into the application.

OK

Click **OK** after you have made a selection to close the Report Settings dialog.

Menu Structure

All functions for the ASAP 2420 are located on menus which are accessed from the Menu bar. Each menu contains commands, and in some cases a submenu. A submenu is indicated when the command is followed by an arrow.



Brief descriptions of each menu are provided below; refer to the chapter given in parentheses for a detailed description of the commands contained on that menu.

File — Enables you to maintain system files.
(Chapter 4, **FILE MENU**)

Unit [n] — Enables you to perform analyses and other instrument operations.
(Chapter 5, **UNIT MENU**)

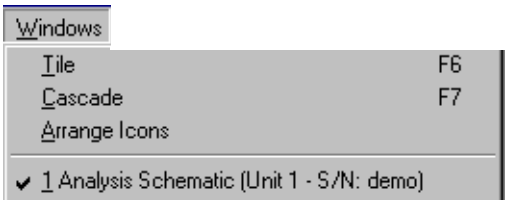
Reports — Enables you to generate, customize, and close reports. Also provides examples of reports.
(Chapter 6, **REPORTS MENU**)

Options — Enables you to add archive locations, edit labels for sample files, and select data presentation formats
(Chapter 7, **OPTIONS MENU**)

Windows — Enables you to arrange the windows and icons on your screen. It also displays the names of all open files.
(this chapter, page **2-24**)

Help — Displays Help information.
(this chapter, page **2-24**)

Windows Menu



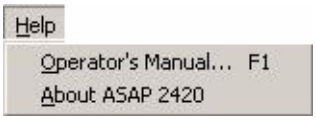
Tile — Resizes all open windows and arranges them side by side so that the contents of all open windows are visible.

Cascade — Resizes all open windows and arranges them in a stacked fashion. The active window is positioned on top of the stack. Each window's title remains visible, making it easy to select other windows.

Arrange Icons — Arranges the symbols for all minimized windows in an orderly manner.

Open Files — Displays all open files; the active window is preceded with a check mark.

Help Menu



Operator's Manual — Displays the operator's manual in PDF format.

About ASAP 2420 — Displays information about the ASAP 2420 analysis program

3 OPERATING PROCEDURES

Sample Information Files and Templates	3-3
Creating Sample Information Templates	3-5
Creating Parameter Templates	3-8
Creating Parameter Files	3-8
Creating Sample Information Files	3-19
Preparing Samples	3-24
Selecting Files for Degassing or Analyzing Samples	3-31
Filling Dewars	3-38
Performing an Analysis	3-51
Printing File Contents	3-60
Listing File Statistics	3-62
Importing Sample and Parameter Files	3-65
Exporting Sample Data	3-66
Generating Graph Overlays	3-68

OPERATIONAL PROCEDURES

This chapter contains step-by-step procedures for operating the ASAP 2420 Confirm program. It does not provide detailed descriptions of the fields in the dialogs used to perform these procedures. Refer to Chapters 4 through 7 for field descriptions.

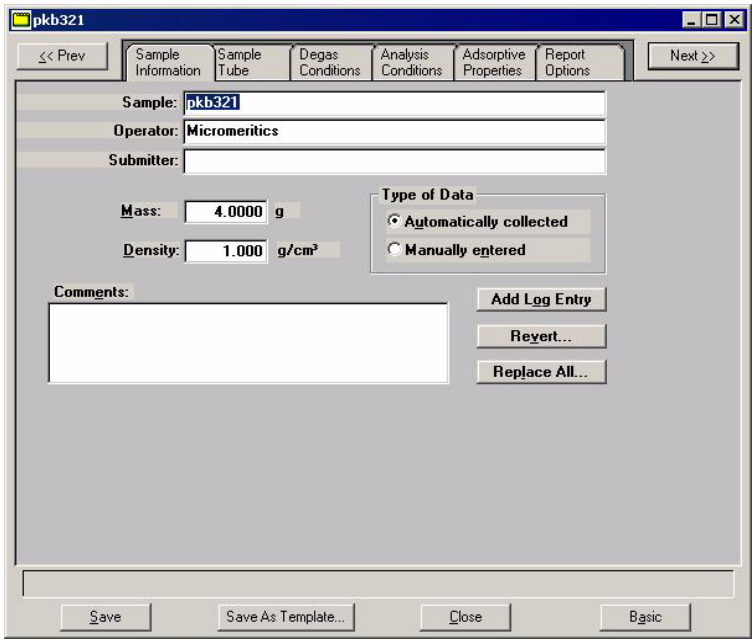
Some procedures cannot be performed by an Analyst; these procedures are marked accordingly.

Sample Information Files and Templates

Sample Information Files

Sample information files contain parameters used to control degassing and analysis. After you perform an analysis, the sample information file stores the results. You cannot degas or analyze a sample until you assign it a sample information file. A sample information file consists of the following parameters:

- sample identification
- sample tube properties
- degas conditions
- analysis conditions
- adsorptive properties
- report options



Portions 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 you wish.

For example, if you typically use the same analysis conditions, you can create an analysis conditions file containing the desired conditions. Then when you create a sample file, click the **Replace** button and select the previously-created analysis conditions; the values will be copied into the current sample file and you can edit them if needed. The file from which the values were copied remains intact and ready for the next use.

Sample information files reside in a folder, or 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 500. You may create additional directories as needed.

Templates

A template is a predefined sample information file used to create new sample information files. Developers create templates to be used by Analysts (or Developers) when performing analyses.

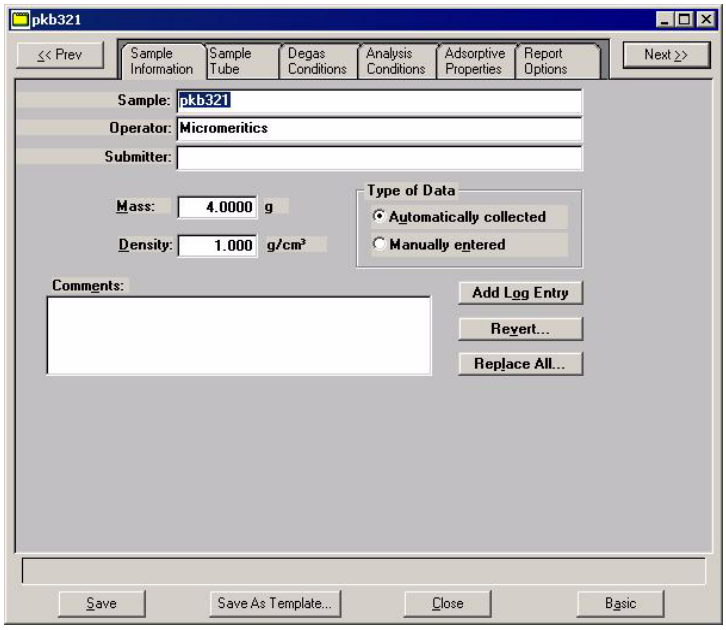
Several templates have been included with the Confirm program. You can create additional templates as described in the next section. Once a template has been created, it cannot be edited or deleted.

An Analyst is not authorized to create sample and parameter file templates.

Creating Sample Information Templates

A sample information template can be created by opening a sample information file, editing it if desired, and saving it as a template. Additionally, when creating a new sample information file (refer to **"Creating Sample Information Files" on page 3-19**), you may save it as a template.

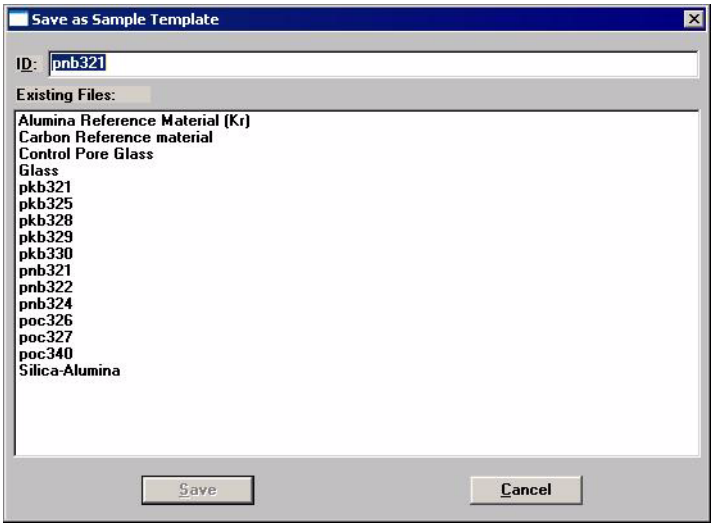
1. Edit the fields on the Sample Information dialog and parameter dialogs if desired (click the associated tab to access a parameter).



2. Choose one of the following:

   - Click the **Save As Template** button on the Sample Information dialog, or
   - Select **File > Save as Template > Sample Information** from the main menu.

   The Save as Sample Template dialog is displayed.



3. Enter an appropriate description in the **ID** field; you can use up to 42 characters. Be sure to use an intuitive description.

4. Click **Save**; the dialog closes.

   If **Save** does not become enabled, a template with the same description currently exists. The ASAP 2420 Confirm program does not allow duplicate template descriptions. Edit the description to enable **Save**.

5. On the Sample Information dialog, click **Close.**

   If you made changes to the sample information file, the following message is displayed:

   (file name) has been changed. Save changes before closing?

   The (file name) is the name of the file you opened to create the template. Click **No** (unless you wish to save the changes in the sample information file as well as the template) to close the dialog box.

Creating Parameter Templates

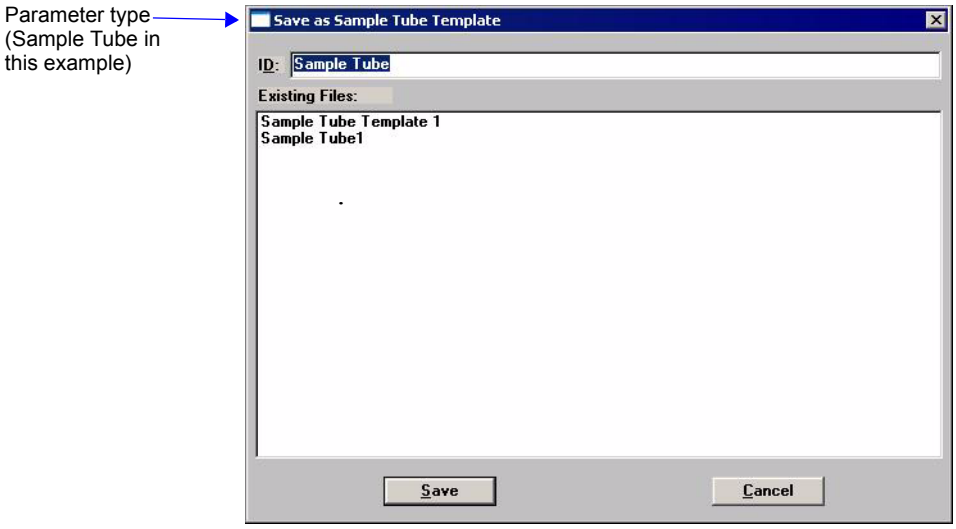
As mentioned earlier, parameter sets can also exist as standalone parameter files. In addition, you may save a parameter set as a template as described below.

For more information about the fields in the parameter dialogs, refer to **"Creating Parameter Files" on page 3-8**.

1. Choose one of the following:
   - In a parameter file dialog, click the **Save as Template** button.
   - In a sample information file, select the parameter tab, then select **File > Save as Template > (template type)** from the main menu.

In a sample information file, do not click the *Save as Template* button at the bottom of the screen to save a parameter file. The *Save as Template* button saves the entire Sample Information file as a template, not the parameter set.

2. The Save as (parameter type) Template dialog is displayed.



3. Enter an appropriate description in the **ID** field; you can use up to 42 characters. Be sure to use an intuitive description.

4. Click **Save**; the dialog closes.

   If **Save** does not become enabled, a template with the same description currently exists. The ASAP 2420 Confirm program does not allow duplicate template descriptions. Edit the description to enable **Save**.

5. On the Sample Information or Parameter dialog, click **Close.**

If you made changes to the sample information or parameter file, the following message is displayed:

(file name) has been changed. Save changes before closing?

The (file name) is the name of the file you opened to create the template. Click **No** (unless you wish to save the changes in the parameter file as well as the template) to close the dialog box.

Creating Parameter Files

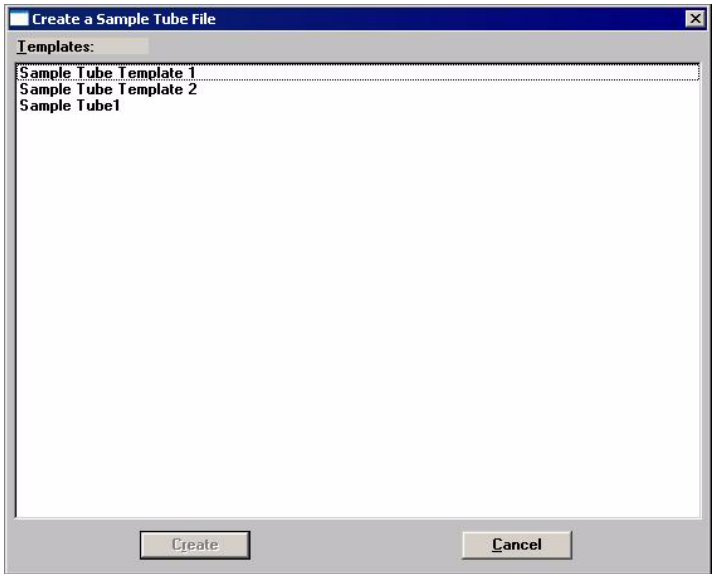
An Analyst is not authorized to create parameter files.

The following parameters can exist as part of the sample information file, as well as individual parameter files:

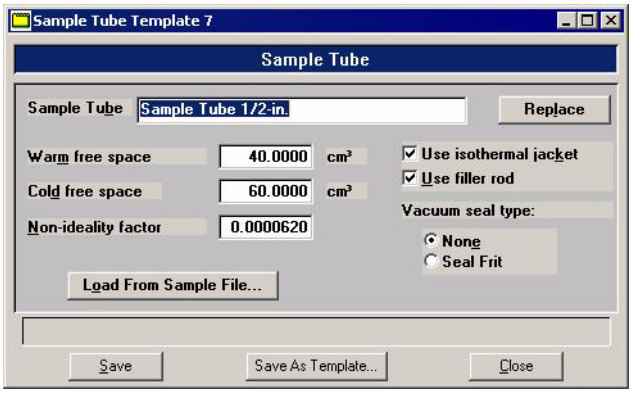
- Sample tube
- Degas conditions
- Analysis conditions
- Adsorptive properties
- Report options

Sample Tube

1. Select **File > New > Sample tube**; the Create a Sample Tube File dialog containing the available templates is displayed.



2. Choose a template, then click **Create**; the Sample Tube dialog is displayed.



3. Enter a description in the Sample Tube field. Be sure to use an intuitive description so that you can recognize it easily. You may enter up to 42 characters.

4. If you want to import free space from a blank tube run:

   a. Click **Load from Sample File**; the Open Sample Information File dialog is displayed.

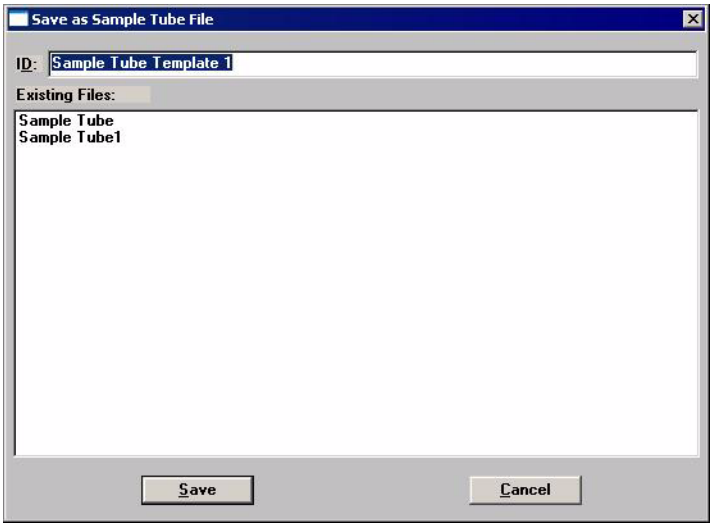
   b. Select the file you used in the blank run with this sample tube.

   c. Click **Open** to copy the warm and cold free space, and the non-ideality factor values into the Sample Tube file.

5. Select whether an isothermal jacket and/or filler rod are to be used.

6. If a vacuum seal is to be used, click **Seal Frit;** otherwise leave **None** selected.

7. Click **Save**; the Save as Sample Tube File dialog is displayed.



8. The description you entered in step 3 is displayed in the **ID** field. You may change it if desired.

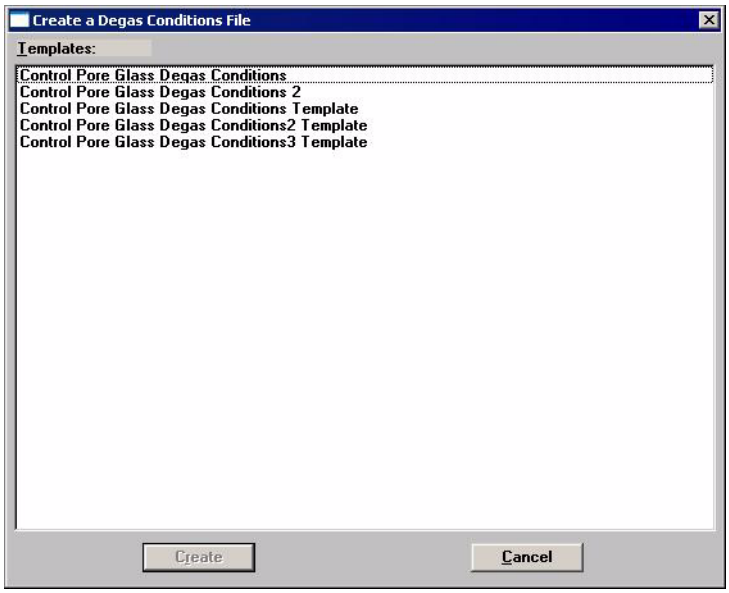
   Click **Save** to save the file and return to the Sample Tube dialog. If **Save** is disabled, the description is a duplicate one and must be edited. The ASAP 2420 Confirm program does not allow duplicate descriptions.

9. Click **Close** to close the Sample Tube dialog box.

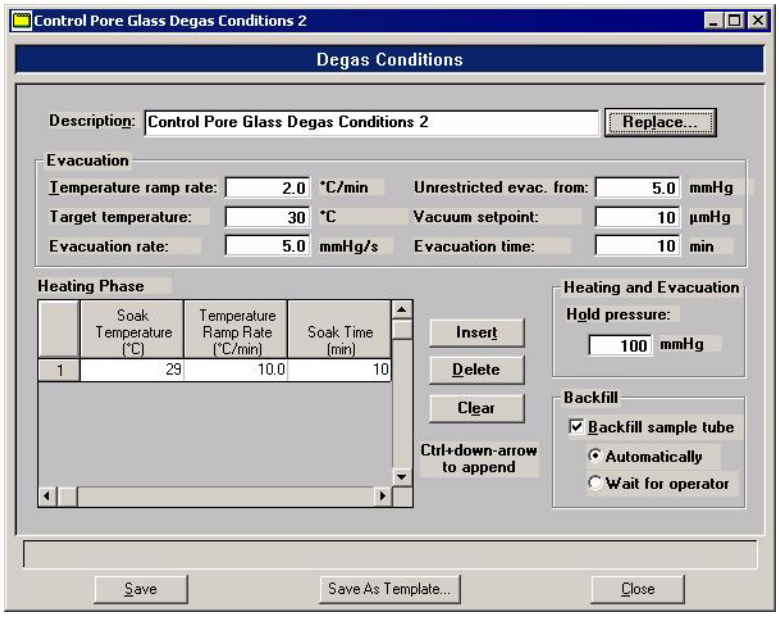
Degas Conditions

Degas Conditions files contain degassing information for sample preparation.

1. Select **File > New > Degas Conditions**; the Create a Degas Conditions File dialog containing the available templates is displayed.



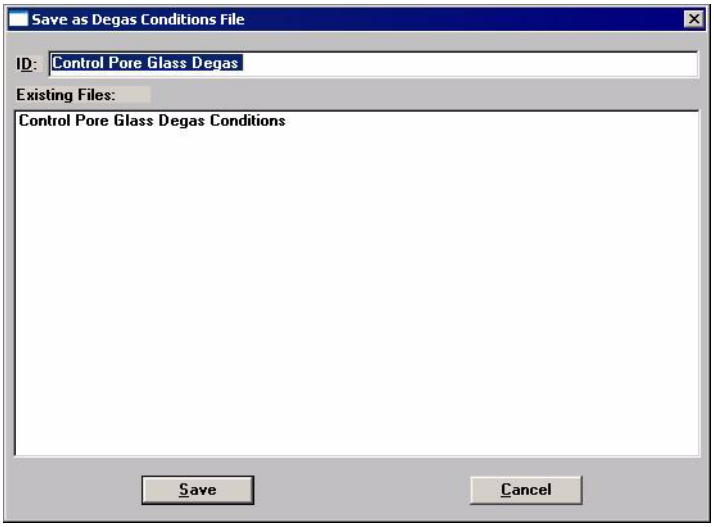
2. Choose a template, then click **Create**; the Degas Conditions dialog is displayed.



3. Enter a description in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily.

4. Specify values for the evacuation and heating phases of degassing.

5. Click **Save**; the Save As Degas Conditions File dialog is displayed.



6. The description you entered in step 3 is displayed in the **ID** field. You may change it if desired.

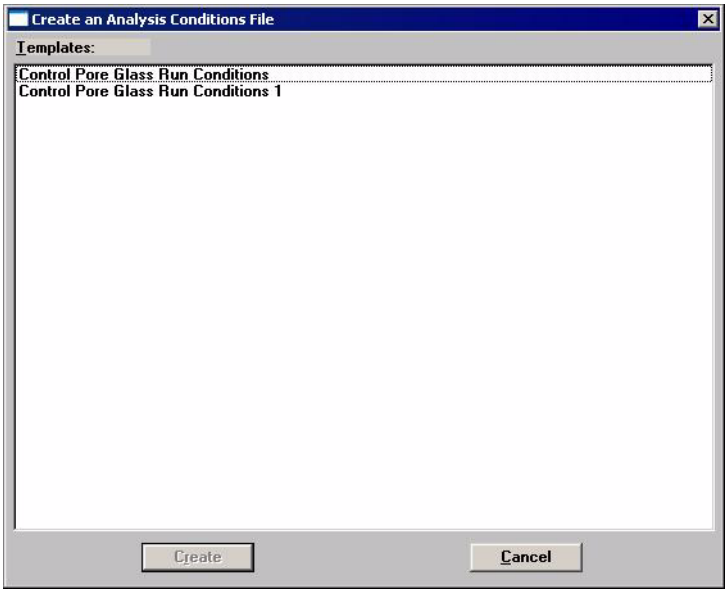
Click **Save** to save the file and return to the Degas Conditions dialog. If **Save** is disabled, the description is a duplicate one and must be edited. The ASAP 2420 Confirm program does not allow duplicate descriptions.

7. Click **Close** to close the Degas Conditions dialog box.

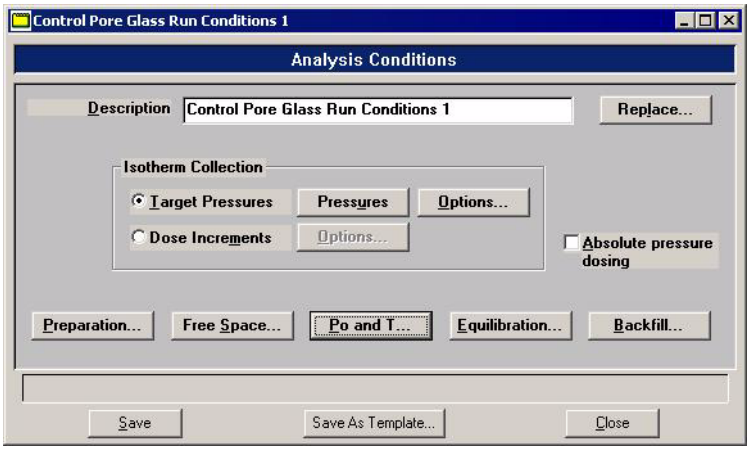
Analysis Conditions

Analysis Conditions files specify the data used to guide an analysis.

1. Select **File > New > Analysis Conditions**; the Create an Analysis Conditions File dialog containing the available templates is displayed.



2. Choose a template then click **Create**; the Analysis Conditions dialog is displayed.



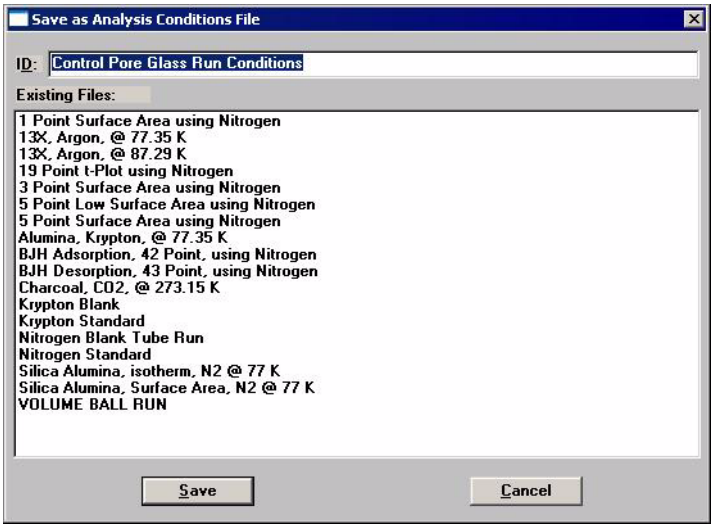
3. Enter a description in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily. You may enter up to 42 characters.

4. Select the method to be used for isotherm collection; either **Target Pressures** or **Dose Increments**.

5. Click the following push buttons to define associated analysis conditions.

   Preparation
   Free Space
   Po and T
   Equilibration
   Backfill

6. Click **Save**; the Save As Analysis Conditions File dialog is displayed.



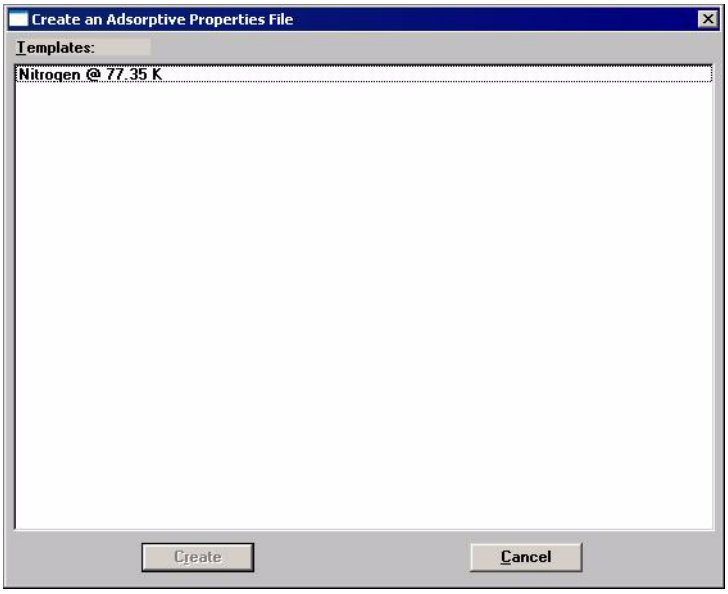
7. The description you entered in step 3 is displayed in the ID field. You may change it if desired.

   Click **Save** to save the file and return to the Analysis Conditions dialog. If **Save** is disabled, the description is a duplicate one and must be edited. The ASAP 2420 Confirm program does not allow duplicate descriptions.

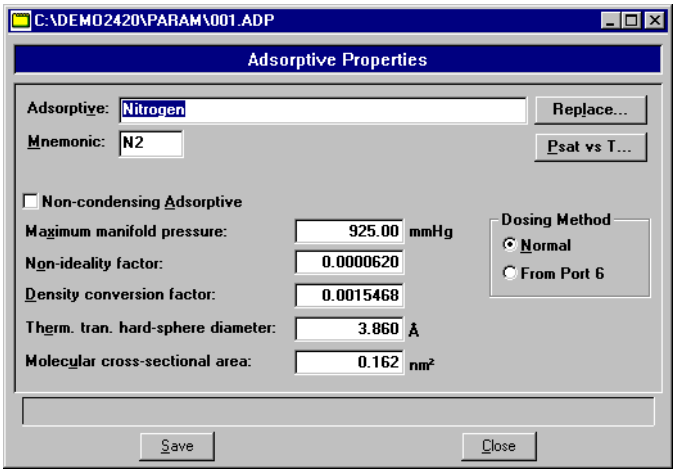
8. Click **Close** to close the Analysis Conditions dialog box.

Adsorptive Properties

1. Select **File > New**> **Adsorptive Properties**; the Create Adsorptive Properties dialog containing the available templates is displayed.



2. Choose a template, then click **Create**. The Adsorptive Properties dialog is displayed.

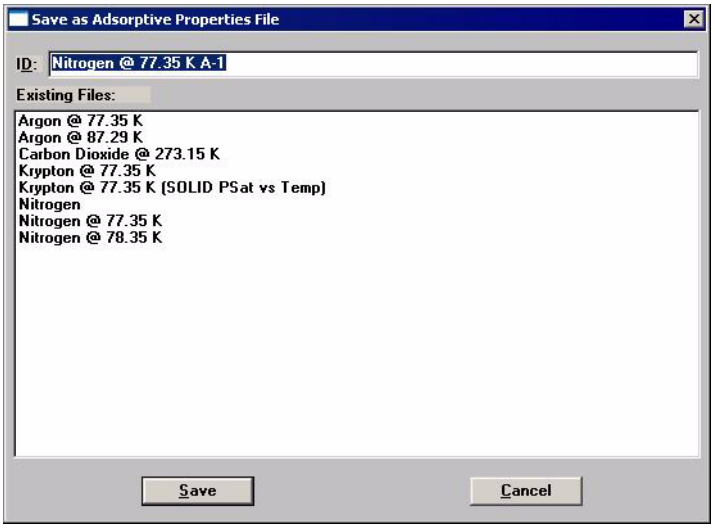


3. Enter a description of the adsorptive gas in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily. You may enter up to 42 characters.
4. Enter the **Mnemonic** name for the adsorptive in the **Mnemonic** field.
5. Click **Psat vs T** to edit the Psat vs Temperature table if required.
6. Select **Non-condensing Adsorptive** if the gas is non-condensing.
7. Enter the appropriate information (or accept the defaults) in the following fields.
   - Maximum manifold pressure

- Non-ideality factor
- Density conversion factor
- Thermal transpiration hard-sphere diameter
- Molecular cross-sectional area

8. Choose the dosing method: **Normal** for standard and high throughput analyses or **From Port 6** for krypton analysis.

9. Click **Save**. The Save as Adsorptive Properties File dialog is displayed.



10. The description you entered in step 3 is displayed in the ID field. You may change it if desired.

    Click **Save** to save the file and return to the Adsorptive Properties dialog. If **Save** is disabled, the description is a duplicate one and must be edited. The ASAP 2420 Confirm program does not allow duplicate descriptions.

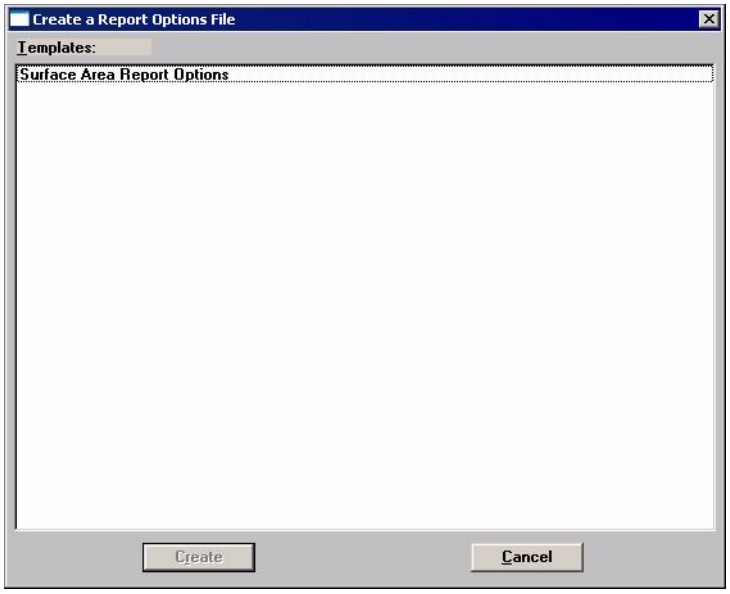
11. Click **Close** to close the Adsorptive Properties dialog box.

Report Options

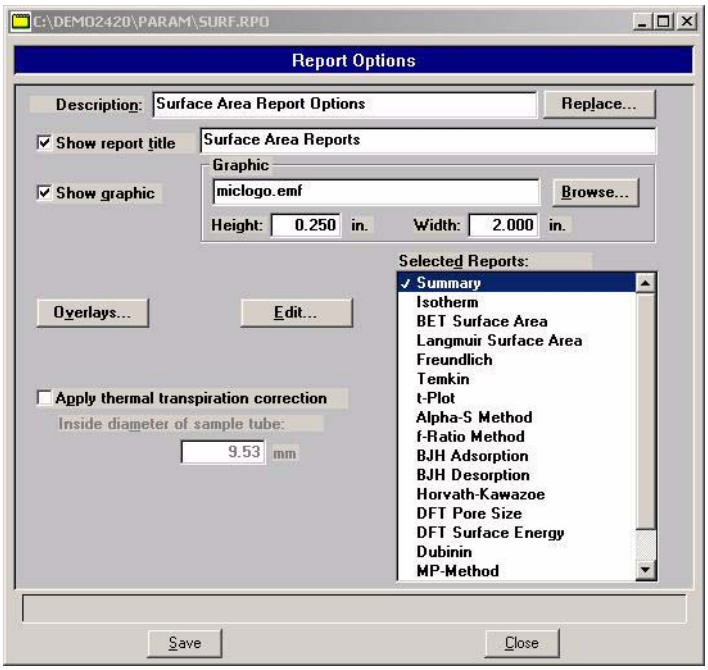
Report options specify the types of reports to be generated from an analysis or from manually entered data. They also help you customize details of reports such as axis scale, axis range, column headings, and components of thickness curve equations.

You can tailor report options files to accommodate the requirements of your analyses. For example, you can generate a simple report that lets you determine the basic characteristics of the sample. Then use that report to make choices about the variables you want to include in lengthier, more sophisticated reports. You can specify for reports to be generated automatically after each analysis, or you can 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.

1. Select **File > New** > **Report Options**; the Create a Report Options File dialog box is displayed.



2. Select a template, then click **Create;** the Report Options dialog is displayed.



3. Enter a description (up to 42 characters) in the **Description** field. Enter an identifier that gives a more intuitive description of the file's contents. For example, **BJH Adsorption Report Options**.

4. Select **Show report title** and enter the title you wish 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 (bmp) or enhanced metafile (emf), you can have it display in the report header by selecting *Show graphic*. Click *Browse* to select the file; use the *Height* and *Width* fields to specify the size.

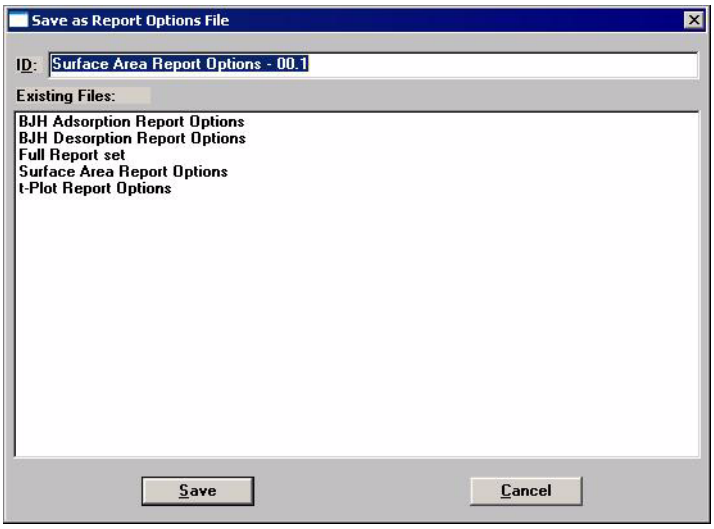
5. If you wish to compare the same type of graph from multiple files, click **Overlays** and choose the files. Then be sure you edit the graph from the Selected Reports window and choose **Samples** from the Overlay drop-down list. (Refer to **"Generating Graph Overlays" on page 3-68**).

6. Select **Apply thermal transpiration correction** if you want correction made for thermal transpiration in the sample tube, then enter the inside diameter of the sample tube.

7. The reports that may be generated are listed in the Selected Reports list. Select reports by double-clicking on the desired report. Reports are deselected in the same manner. A report is selected when it is preceded by a check mark.

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

8. Click **Save**; the Save as Report Options File is displayed.



9. The description you entered in step 3 is displayed in the ID field. You may change it if desired.

   Click **Save** to save the file and return to the Report Options dialog. If **Save** is disabled, the description is a duplicate one and must be edited. The ASAP 2420 Confirm program does not allow duplicate descriptions.

10. Click **Close** to close the Report Options dialog box.

Creating Sample Information Files

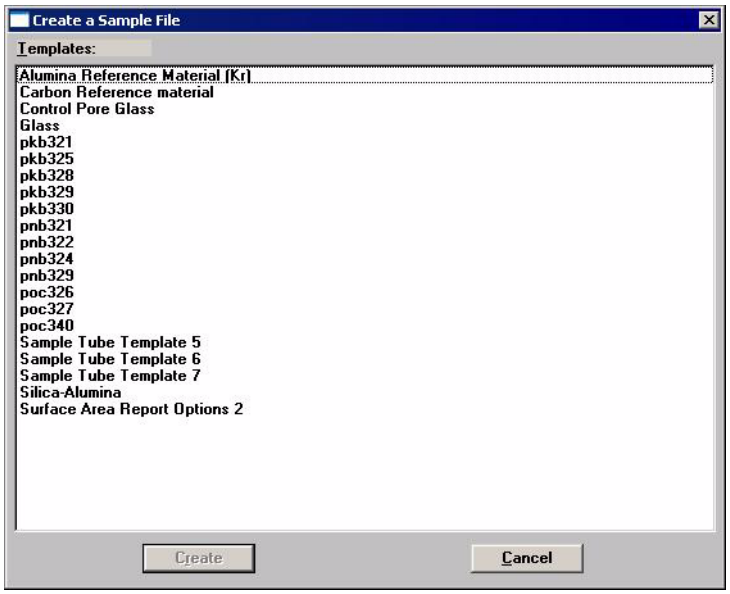
A sample information file must be created for each analysis. The sample information file consists of information groups which, collectively, identify the sample, guide the analysis, and specify report options.

A sample file can be created by a Developer or an Analyst.

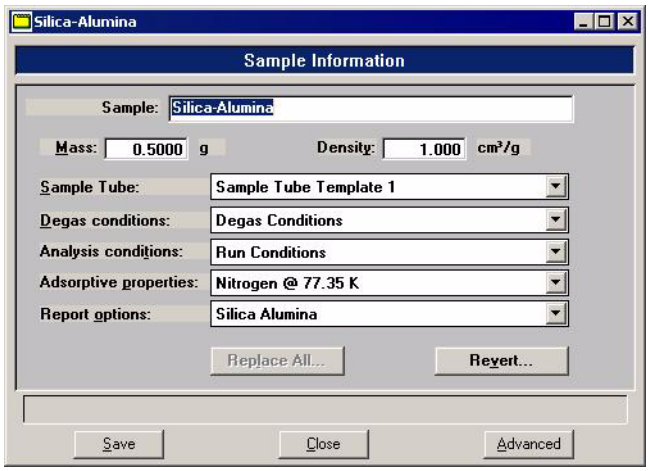
Analyst

An Analyst creates a sample information file by choosing predefined parameters.The Analyst can switch to Advanced format to view details of a parameter file, but cannot edit the file.

1. Select **File > New > Sample Information;** the Create a Sample File dialog containing the available templates is displayed.



2. Choose the desired template, then click **Create**; the Sample Information dialog is displayed.

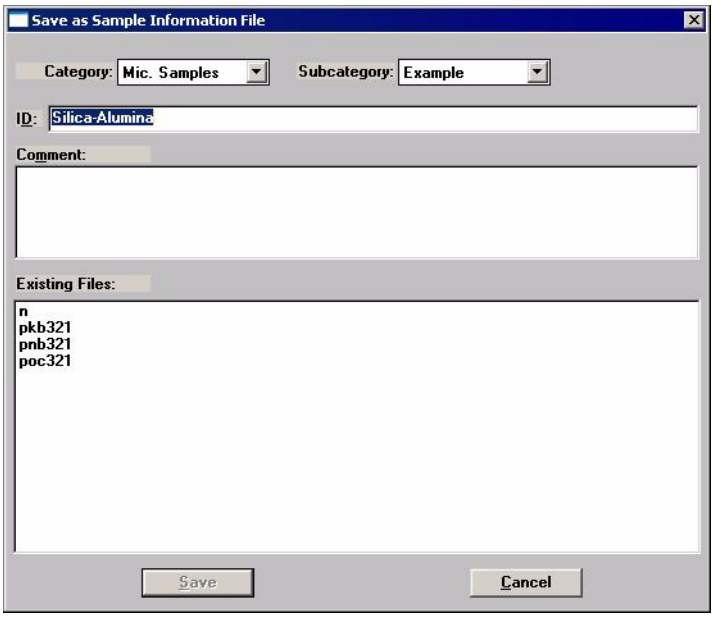


3. Enter a description in the **Sample** field. You may enter up to 42 characters.
4. Enter the sample's mass in the **Mass** field.
5. If you plan to use a calculated free space, enter the sample's density in the **Density** field.
6. Click on the down arrow to the right of each of the following fields to choose appropriate parameter files, or you can accept the defaults displayed in the template:

   - Sample Tube
   - Degas Conditions
   - Analysis Conditions
   - Adsorptive Properties
   - Report Options

You can click **Advanced** to view details of parameter files, but you cannot edit them.

7. After you have selected all parameters, click **Save**: the Save As Sample Information File dialog is displayed.



8. Choose the directory into which the file is to be saved using the Category and Subcategory drop-down lists.

9. The description you entered in step 3 is displayed in the **ID** field. You may change it if desired.

10. Enter a comment in the **Comment** window; for example, you may wish to enter the date and time the file is created. This is a required field.

11. Click **Save** to save the file and return to the Sample Information dialog.

    If **Save** is disabled, the description is a duplicate one or the Comment window is blank. Correct as necessary, then click **Save**.

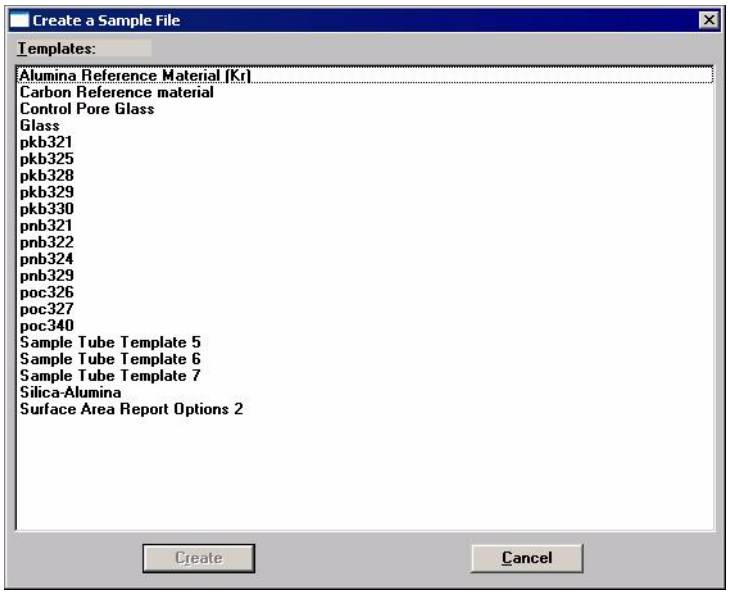
12. Click **Close** to close the Sample Information dialog box.

Developer

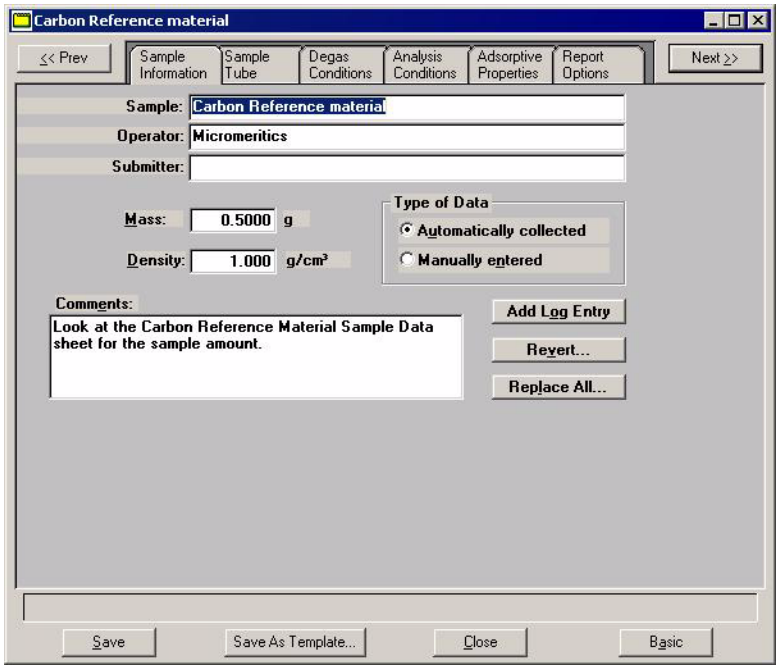
A Developer's view of the sample information dialog is more flexible than that of an Analyst. A Developer is allowed to view and edit (if desired) all parameters of the file.

Perform the following steps to create a sample information file:

1. Select **File** > **New > Sample Information**; the Create a Sample Information File dialog containing the available templates is displayed.



2. Choose a template, then click **Create**; the Sample Information dialog is displayed.



3. Enter the description in the **Sample** field; you can use up to 42 characters.

4. In the **Operator** field, enter the name of the Analyst who will be performing the analysis.

5. In the **Submitter** field, enter the name of the department, company, etc. submitting the sample for analysis.

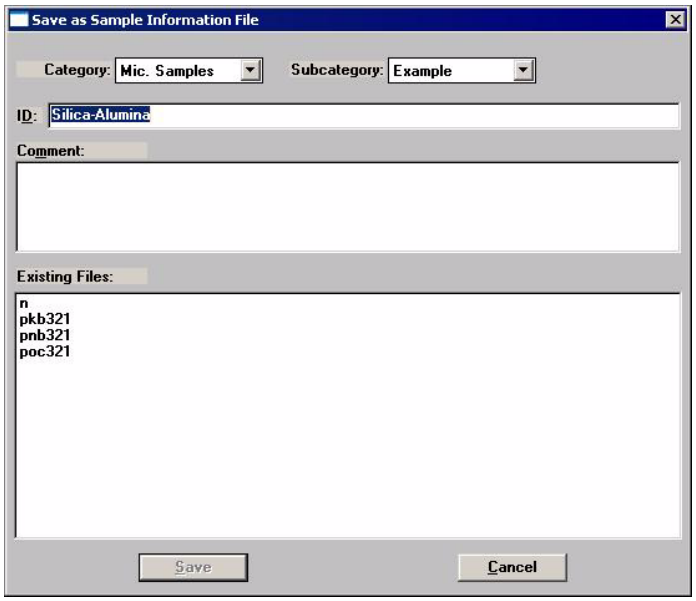
6. Enter the mass of the sample in the **Mass** field; this value can be edited later if desired.

7. If you plan to use a calculated free space, enter the sample's density in the **Density** field.

8. Choose whether you are going to enter data or have it automatically collected.

9. After you complete the Sample Information dialog, click on the parameter tabs to open associated dialogs and edit as desired. Refer to **"Creating Parameter Files" on page 3-8**.

10. After you have completed all parameters, click **Save**; the Save As Sample Information File dialog is displayed.



11. Choose the directory into which the file is to be saved using the Category and Subcategory drop-down lists.

12. The description you entered in step 3 is displayed in the **ID** field. You may change it if desired.

13. Enter a comment in the **Comment** window; for example, you may wish to enter the date and time the file is created. This is a required field.

14. Click **Save** to save the file and return to the Sample Information dialog.

    If **Save** is disabled, the description is a duplicate one or the Comment window is blank. Correct as necessary, then click **Save**.

15. Click **Close** to close the Sample Information dialog.

Preparing Samples

Care should be taken in choosing, conditioning, and filling sample tubes. The following guidelines will help you obtain accurate, reproducible analysis results.

Choosing Sample Tubes

A sample tube set consists of the following parts:

- Sample tube
- Stopper or seal frit
- Filler rod

Standard sample tubes for the ASAP 2420 degas and analysis stations have a 1.27-cm (1/2-in.) outside diameter (OD). Two other sizes are available: 0.64 cm and 0.95 cm (1/4 in. and 3/8 in.) OD. Stepped ferrules, smaller O-rings, and filler rods are available for adapting the smaller stems to the degas or analysis ports.

A rubber stopper may be used with all size sample tubes; however, seal frits are recommended for 1.27-cm (1/2-in.) OD sample tubes or when helium is used as the degas backfill gas.

Filler rods help ensure accuracy in samples with lower total surface areas by reducing the free-space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters.

Cleaning and Labeling Sample Tubes

Sample tubes and filler rods 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.

Supplied by Micromeritics	Supplied by User
Sample tube Sample tube brush Stoppers for sample tube Sample tube rack Sample weighing support Funnel Sample data worksheet (copied from Appendix A of this manual) Pipe cleaners	Drying oven Ultrasonic cleaning unit Alconox Rubber gloves Lint-free cloth Acetone or isopropyl alcohol Safety glasses Forceps Balance

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

2. Rinse the sample tubes and filler rods.

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

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

5. Fill the sample tubes with warm water and place the tubes and filler rods in the reservoir of the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit. Leave the glassware in the cleaning unit until the walls of the tubes are free of sample material residue.

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

7. Clean the interior of the sample tubes with the brush supplied with the ASAP 2420 System. Rinse the sample tubes and filler rods thoroughly with hot water.

8. After rinsing with hot water, rinse the sample tubes and filler rods with isopropyl alcohol or acetone.

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

9. Stand the sample tubes on the sample tube rack and place the filler rods in a basket or in the 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), place a filler rod (if used) in each sample tube by holding the sample tube horizontally and sliding the filler rod into the sample tube slowly.

12. Wipe a rubber stopper (or seal frit, if used with 1.27-cm (1/2-in.) tubes) with a lint-free cloth.
13. Label the sample tube and stopper or seal frit for identification.
14. Insert the stopper or seal frit.

Determining Amount of Sample to Use

The amount of sample to use can be determined best by experiment. In general, a sample providing 40 to 120 square meters of total surface area is recommended for nitrogen analysis. Less than this may cause variability of results; considerably more than this extends unnecessarily the time required for analysis.

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

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

Determining the Mass of the Sample

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

1. Weighing the sample tube set (sample tube, filler rod, and stopper or seal frit) before degas.
2. Weighing the sample tube set with sample before degas and subtracting the weight from the step 1 weight.
3. Weighing the sample tube set with sample after degas and subtracting the weight from the step 1 weight.
4. Weighing the sample tube set with sample after analysis and subtracting the weight from the step 1 weight.

A Sample Data Worksheet for recording the weights and calculating the mass is included in Appendix A. You may make copies as needed.

To weigh the sample before degas:

1. Write the *Sample Tube* number on the Sample Data Worksheet.
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).

3. Place the sample tube set (sample tube, stopper or seal frit, and filler rod, if used) on the sample support.

4. Record the stabilized weight on the Sample Data Worksheet as *Mass of empty sample tube*. Remove the sample support and sample tube set from the balance.

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

If handling toxic sample material, perform steps 5 though 8 under a vent hood.

5. Slowly add the sample to a sample container.
6. Remove the rubber stopper or seal frit and filler rod from the sample tube.
7. Using a funnel, pour sample from the container into the sample tube; or, if analyzing large granules or chunks, use forceps to load the sample tube.

If some sample clings to the inside of the sample tube above the top 3 in. (7.5 cm) of the tube, use a pipe cleaner or lint-free wipe to remove it.

8. Replace the filler rod and insert the rubber stopper or seal frit.
9. Weigh the sample tube set containing the sample and record the weight on the Sample Data Worksheet as *Mass of sample tube plus sample* (Before Degas).

10. Subtract the *Mass of empty sample tube* from the *Mass of sample tube plus sample*; record this value as the **Mass of sample**.

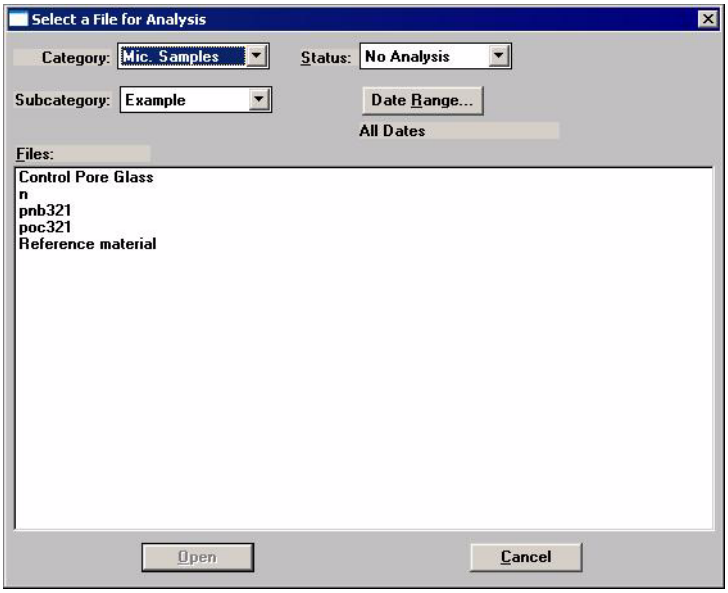
Selecting Files for Degassing or Analyzing Samples

All degas and analysis operations require a sample information file. You may use a predefined sample information file (refer to **"Creating Sample Information Files" on page 3-19**) or create a new file when you begin the operation.

All degas and analysis dialogs contain **Browse**, **New**, and **Clear** buttons. A **Browse** button enables you to select a predefined sample file. A **New** button enables you to create a new sample file. A **Clear** button clears the sample field and enables you to make another selection.

Selecting a Predefined Sample Information File

1. Click a **Browse** button on the analysis or degas dialog. The Select a File for Analysis dialog is displayed.

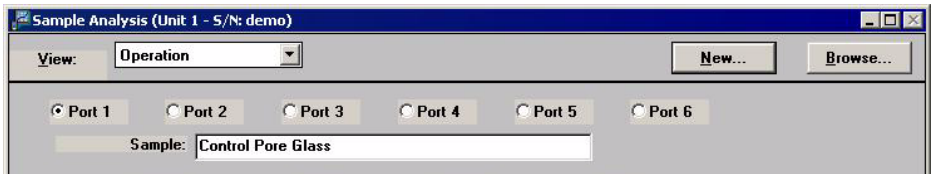


2. Select a file from the **Files** list.

   Refer to **"File Selection" on page 2-19** for an explanation of how to limit the files displayed by selecting a file category, status, or date range.

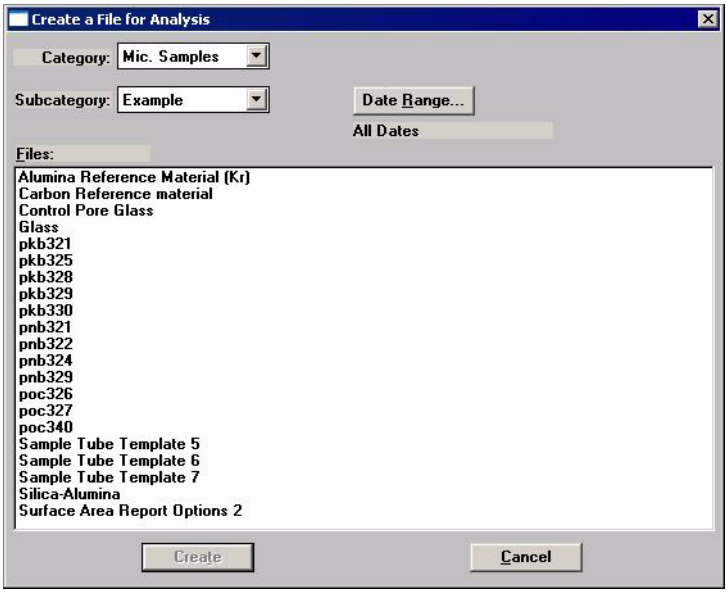
3. Click **Open**.

4. The Select a File for Analysis dialog closes and the description of the file you selected appears in the **Sample** field in the degas or analysis dialog.

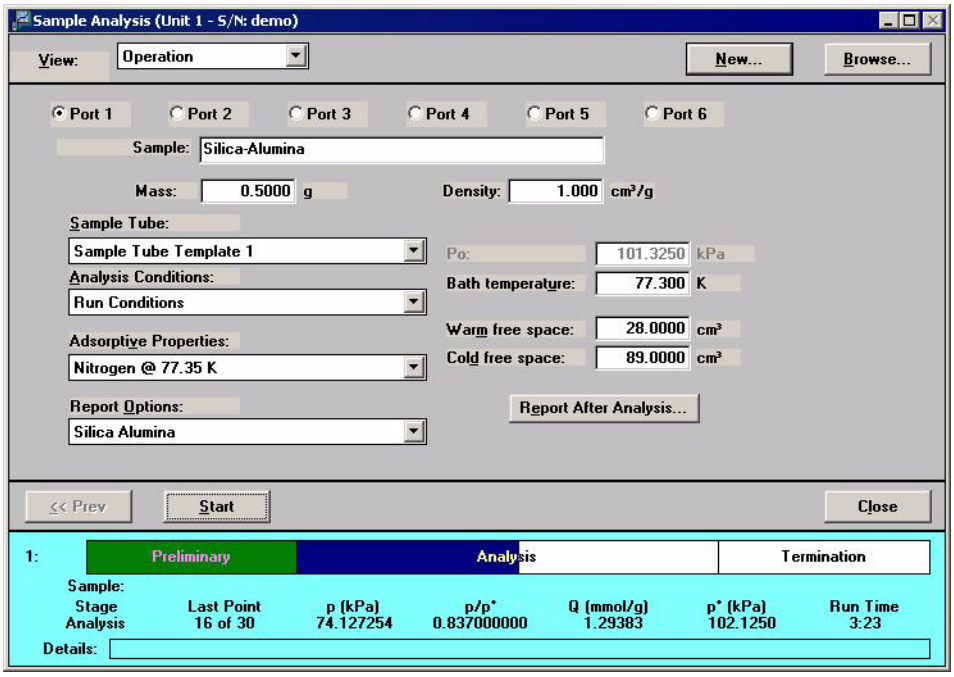


Creating a Sample Information File

1. Click a **New** button on the degas or analysis dialog. The Create a File for Analysis dialog is displayed, with the available templates listed.



2. Select a template on which to base the sample information file from the **Templates** list.
3. Click **Create**.
4. The Create a File for Analysis dialog closes and the name of the template you selected appears in the **Sample** field in the degas or analysis dialog.

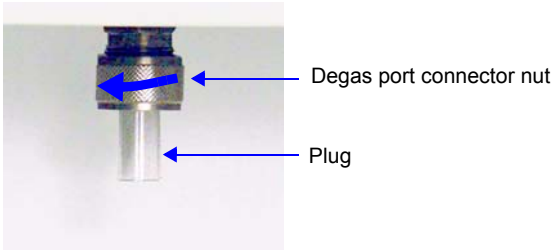


Degassing Samples

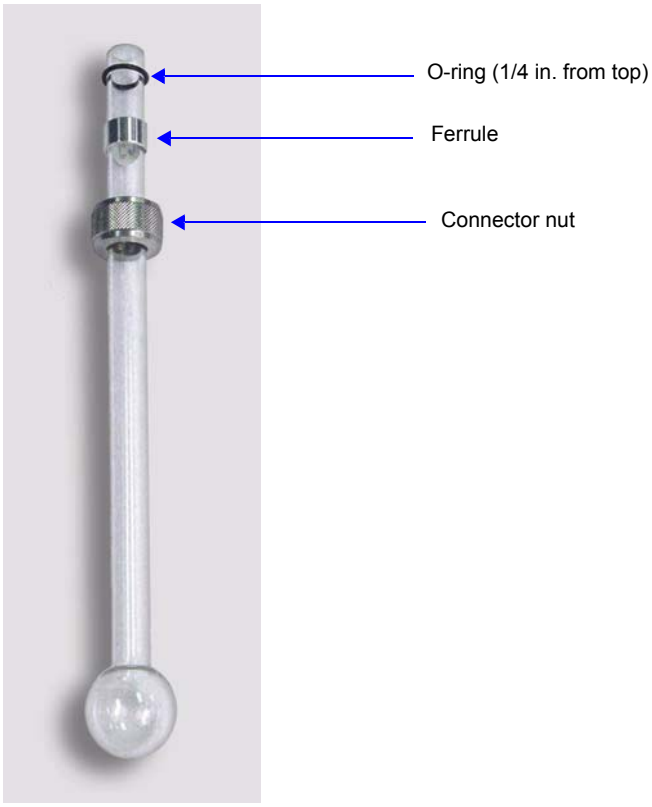
Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The degas process heats the sample and places it under vacuum to remove moisture and other contaminants, thus providing a clean sample for analysis.

Attaching Sample Tubes to 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.



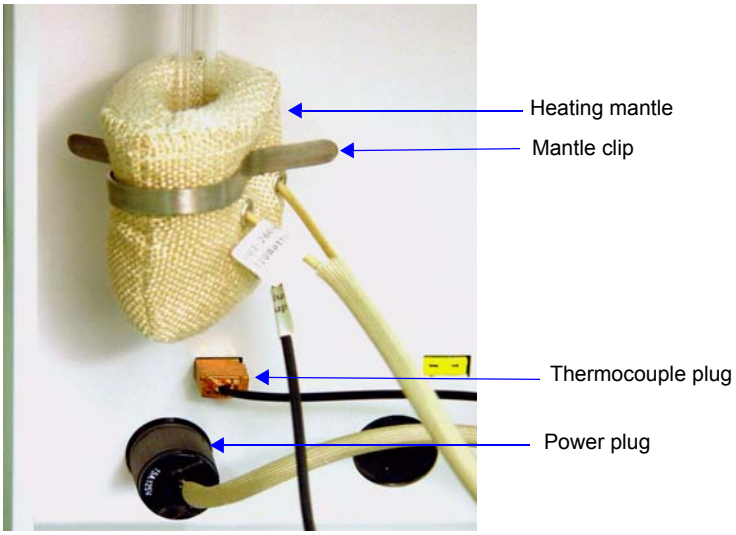
2. Remove the rubber stopper (if used instead of a seal frit) from the sample tube.
3. Place the degas port connector nut, ferrule, and O-ring onto the sample tube set as shown below.



4. Attach the sample tube set to the degas port. Be sure to push the sample tube in to a full stop. 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.

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

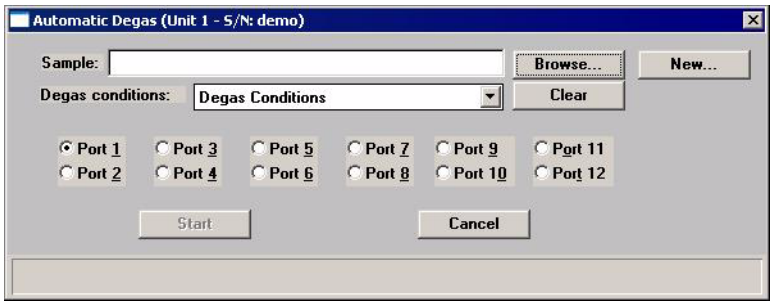
6. Insert the heating mantle thermocouple plug into the thermocouple connector on the analyzer. Insert the heating mantle power plug into the power connector on the analyzer and twist to lock securely.



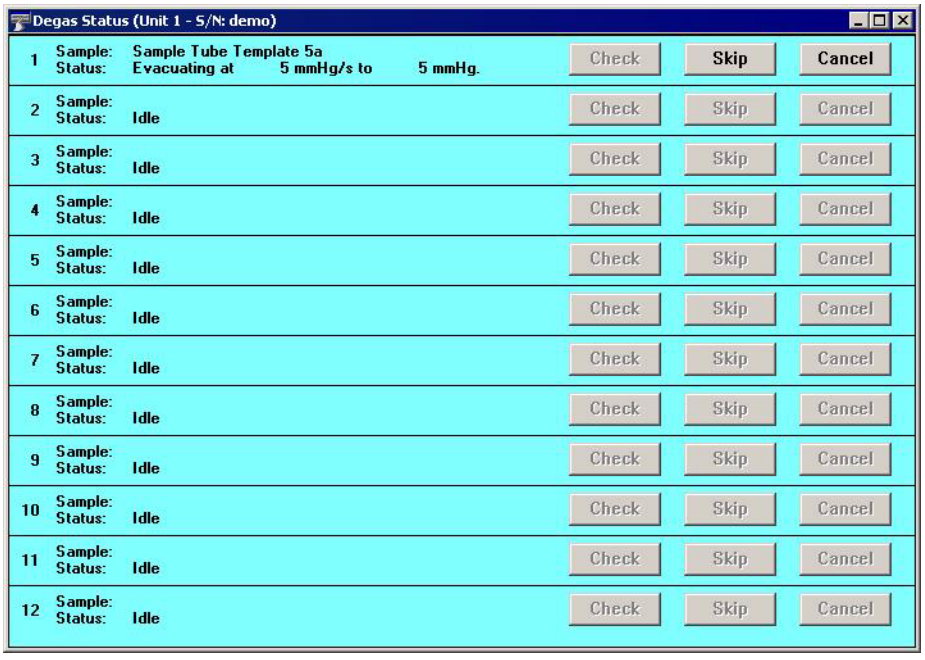
Degassing a Single Sample

You can degas a single sample as described below.

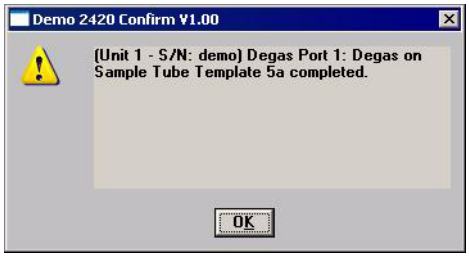
1. Attach the sample tube to the port you wish to use as described in the previous section.
2. Select **Unit > Start Single Degas**; the Automatic Degas dialog is displayed.



3. Select or create a sample file (refer to **"Filling an Analysis Dewar" on page 3-39**).
4. The degas conditions file contained in the sample file is displayed. Click the down arrow to display a list of degas conditions files if you wish to choose another file.
5. Click the port containing the sample tube.
6. Click **Start** to begin the degassing operation The Degas Status screen is displayed.



7. When the degas operation is complete, a message dialog is displayed.



8. Click **OK** to close the message box.

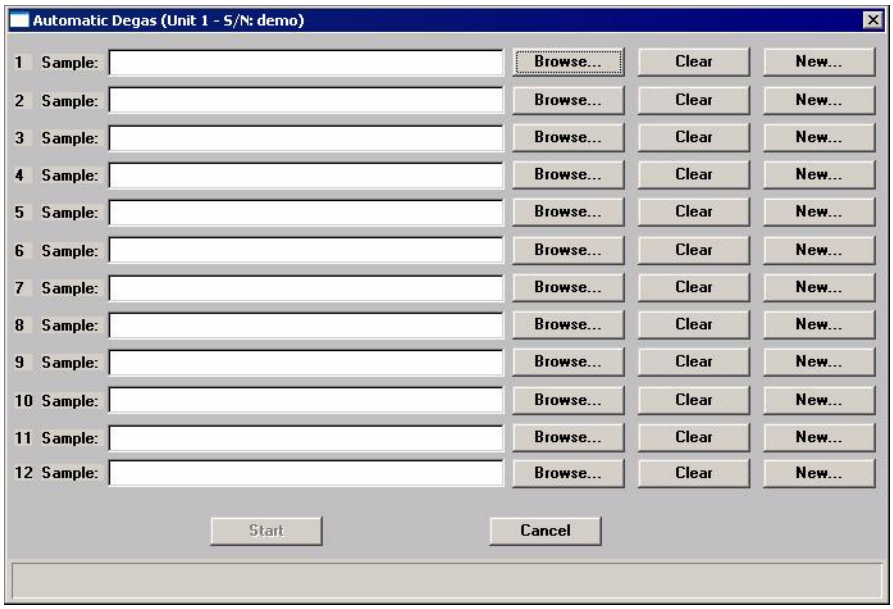
9. Allow the sample tube to cool before removing it.

Degassing Multiple Samples

You can degas up to 12 samples as described below. At any time you can add one or more new samples to degas ports. Whenever a sample (or set of samples) is added, the initial evacuation will interrupt the degassing of the other samples. If any other samples are still in the initial evacuation phase, the new ones will wait until the fast evacuation is started. Starting several samples as a batch will allow them to perform the initial evacuation together.

1. Attach sample tubes to the ports you wish to use as described in **"Attaching Sample Tubes to Degas Ports" on page 3-33**.

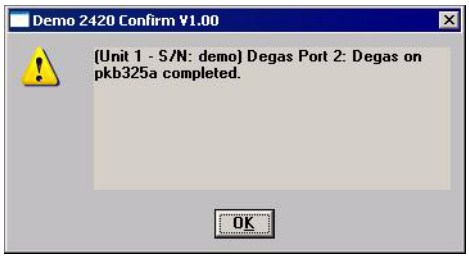
2. Select **Start Multiple Degas** from the Unit menu; the Automatic Degas dialog is displayed.



3. Select or create a sample file (refer to **Selecting Files for Degassing or Analyzing Samples**) for each port to be used.

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

5. When degas is complete, a message dialog is displayed for each port:



6. Click **OK** to close each message box.

7. Allow the sample tubes to cool before removing them.

Filling Dewars

Precautions

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

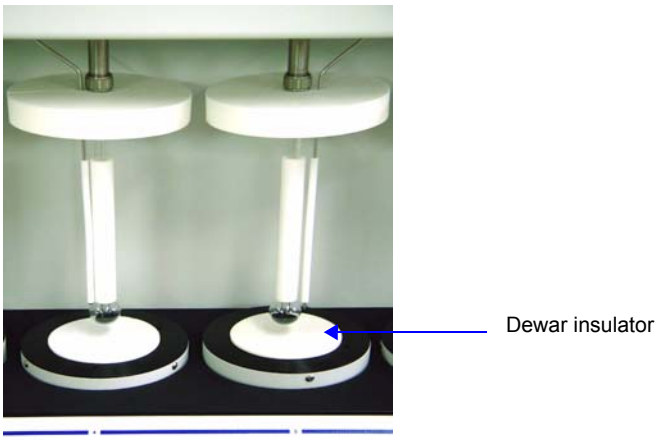
We recommend the following be observed when handling Dewars containing liquefied gases:

- 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 handle heavy objects above the Dewar. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

Dewar Covers

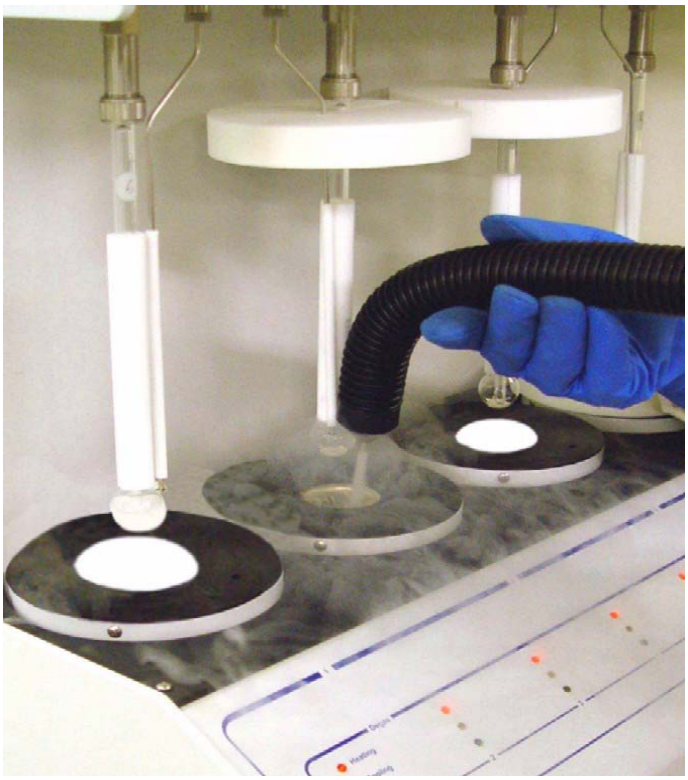
Dewar insulators, which cover the opening in the top of a Dewar, are shipped with the instrument.These covers help maintain the purity of the liquid cryogen, as well as preventing objects from falling into the Dewar.

Whenever a Dewar is not in use, make sure the Dewar elevator is fully lowered and the top of the Dewar is covered with an insulator.



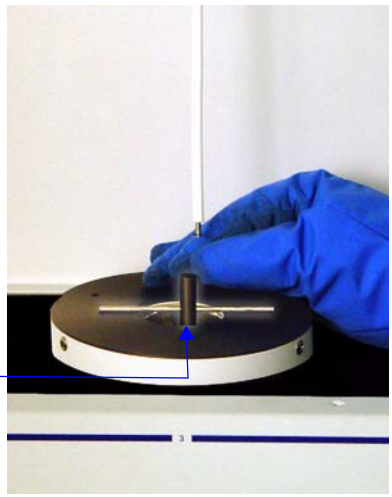
Filling an Analysis Dewar

1. With the Dewar elevator fully lowered, fill the analysis Dewar with the analysis bath fluid to about 5 cm (2 inches) from the top.

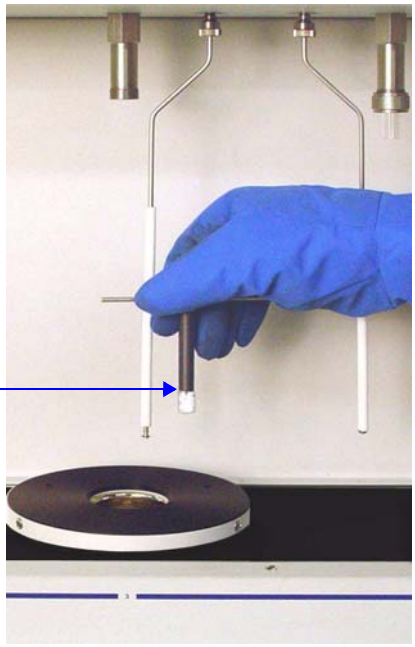


Incorrect fluid levels can lead to measurement errors. Do not overfill the Dewar.

2. To check the fluid level, insert the dipstick in the Dewar as shown below.



3. Check the level of the frozen condensation or wetness. It should not be above the maximum level indicator mark on the dipstick.



4. Allow the analysis bath liquid to equilibrate for at least 30 minutes (one hour is preferable).

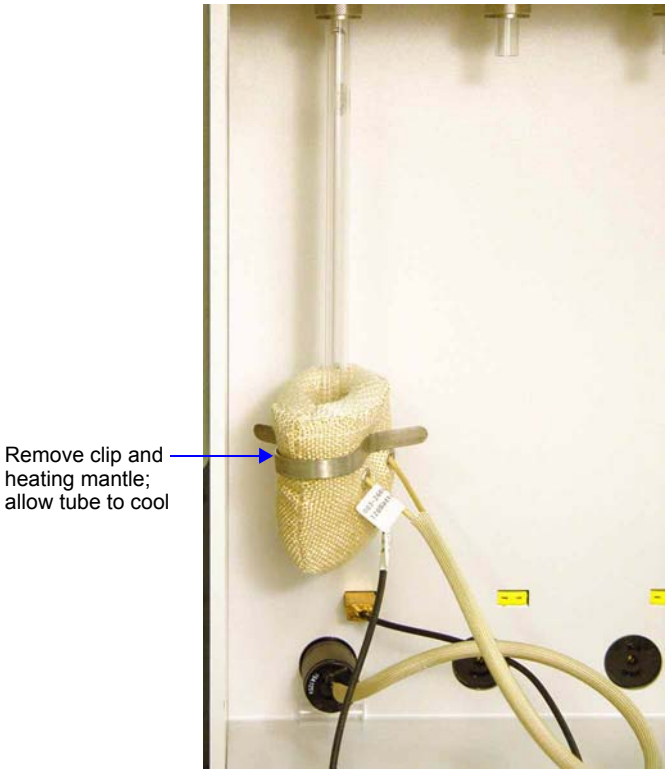
Transferring a Degassed Sample to an Analysis Port

After degas, a sample tube must be removed from the degas port and weighed before attaching it to the analysis port.

If the sample tube is not mounted to the analysis port immediately after cooling, either leave it on the degas port or remove it and insert the rubber stopper (unless you are using a seal frit) into the sample tube opening.

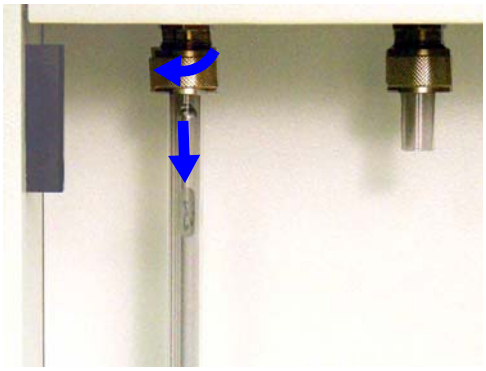
Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube, heating mantle, or heating mantle clip before they have cooled could result in burns.

1. Carefully remove the heating mantle clip and the heating mantle from the sample tube when they are cool enough to handle.



2. Allow the sample tube to cool to room temperature (approximately fifteen minutes).

3. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If you are not using a seal frit, insert a stopper immediately.



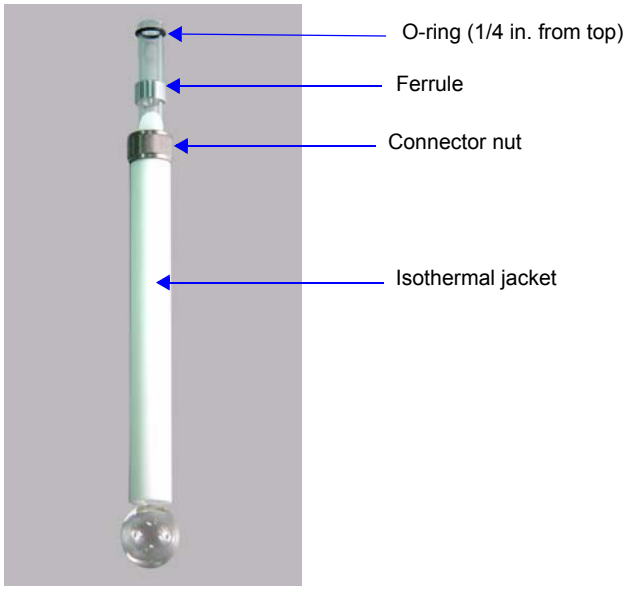
4. Remove the connector nut, ferrule, and O-ring from sample tube stem.

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

6. 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)*.

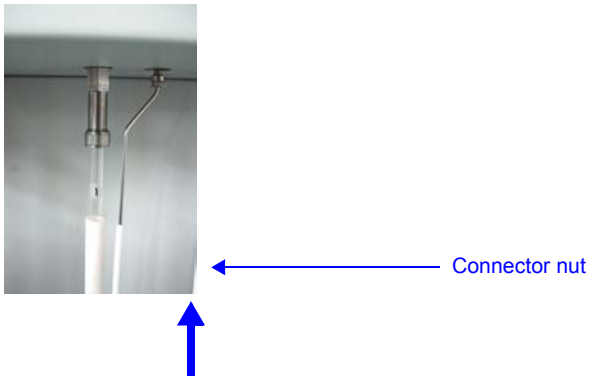
7. Slide an isothermal jacket down over the sample tube stem until it touches the sample tube bulb.

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



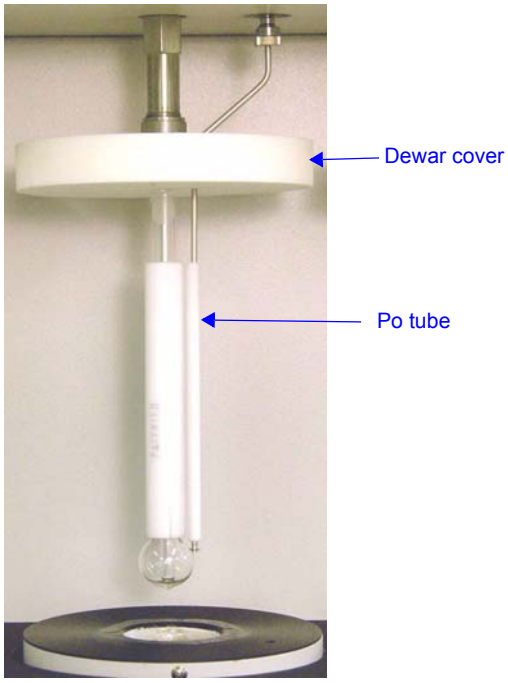
If you are using a TranSeal™, refer to the instructions in the TranSeal kit for information on sample tube installation.

9. Attach the sample tube to the analysis port, pushing it fully up. Turn the connector nut clockwise to hand-tighten.



10. If you wish to degas a sample on the sample port, proceed to **"Degassing Samples on Analysis Ports" on page 3-45**.

If not, place the sample tube Dewar cover over the sample tube stem just above the isothermal jacket as shown in the following illustration. Make sure the Po tube is next to the sample tube.



11. Place the safety shield over the sample tube and Dewar.

12. Begin the analysis. Refer to **"Performing an Analysis" on page 3-51**.

Degassing Samples on Analysis Ports

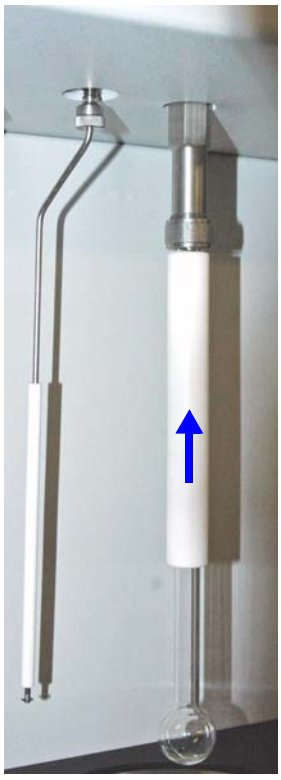
The characterization of microporous materials may be significantly improved by starting the analysis with the sample pre-evacuated. The additional time required for this user-interaction is often offset by improved analyses and faster run time. To avoid exposure to contaminants, it is often best to degas a microporous sample on the same port that will be used for analysis.

Side-lacing heating mantles, which are provided with the micropore option, are required for this type of degassing. For mantle part numbers, refer to **"ORDERING INFORMATION" on page 10-1**.

You may start one degas operation at a time. After you start a degas operation on one sample port, you may start one on another sample port. You may repeat this process for up to six ports. One degas operation starts and completes before the next is started.

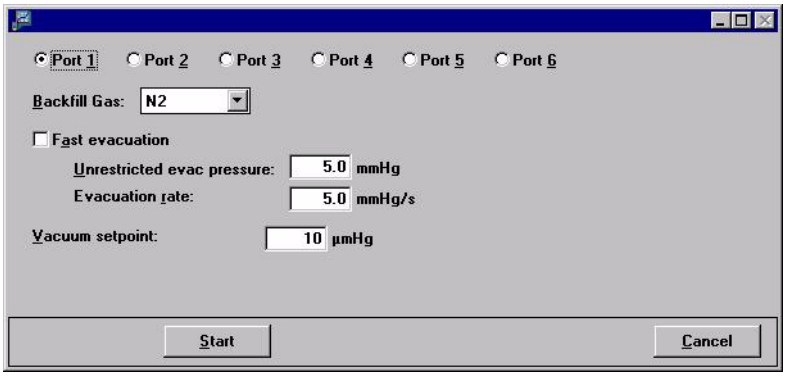
To degas a sample on an analysis port:

1. With the sample tube mounted in the sample port, position the Po tube so that it is away from the sample tube.
2. Slide the isothermal jacket up to the top of the sample tube.



The isothermal jacket fits tightly on the sample tube to ensure thermal stability. Use care when sliding it up or down.

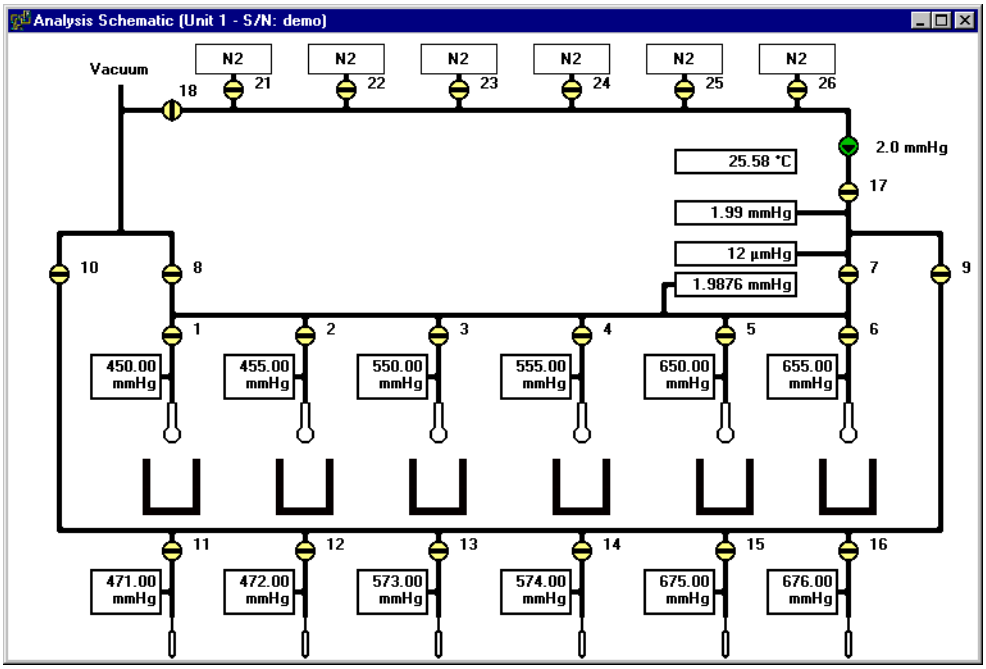
3. Select **Unit [n] > Evacuate Analysis Port**. The Evacuate Analysis Port dialog is displayed.



4. Select the port to which the sample tube is attached.

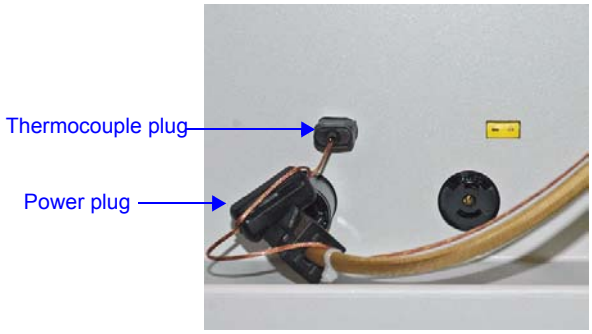
5. Select the evacuation parameters, then click **Start**.

6. Select **Unit [n] > Show Analysis Schematic**, then select **Unit [n] > Enable Manual Control**. The Manual Control screen is displayed.

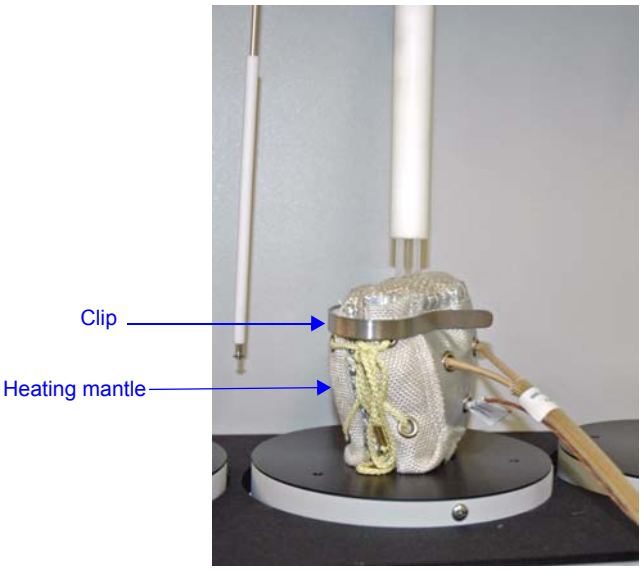


When the evacuation is finished, make sure valve 8 is left open ( ).

7. Insert the heating mantle thermocouple plug into the thermocouple connector on the degas portion of the analyzer below the sample port. Insert the heating mantle power plug into power connector below the thermocouple connector and twist to lock securely.

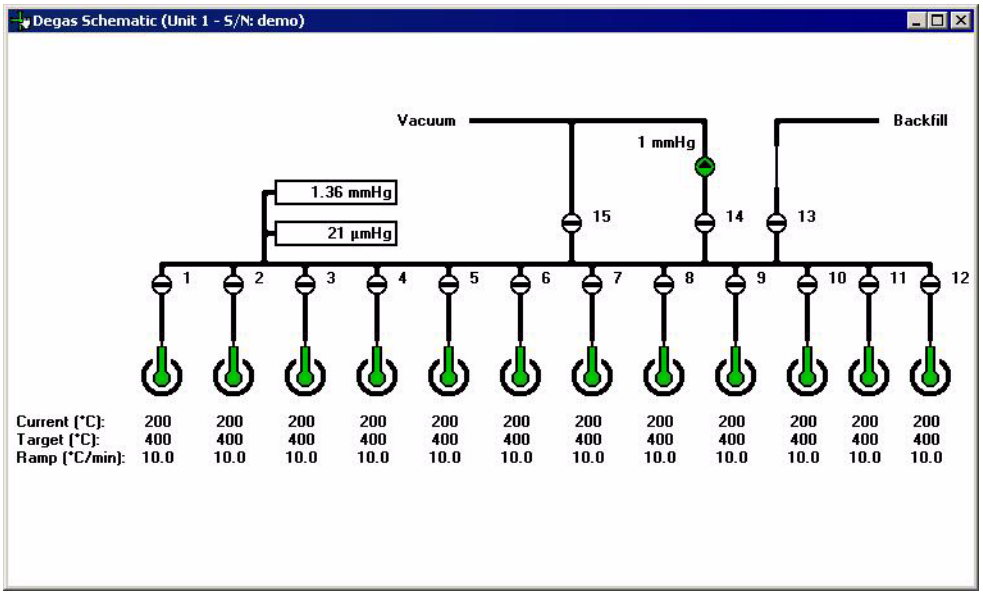


8. Loosen the lace on the heating mantle.
9. Slide the heating mantle around the sample tube, then tighten the lace.
10. Secure the heating mantle with a mantle clip.

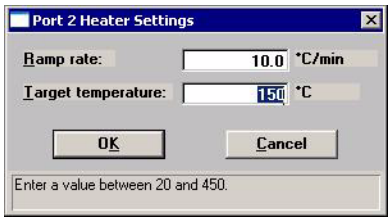


11. Gently heat the sample:

    a. Select **Unit [n] > Show Degas Schematic** to display the Degas Schematic.



    b. Right-click on the degas port to which the heating mantle power plug and thermocouple are connected, then select **Set**.

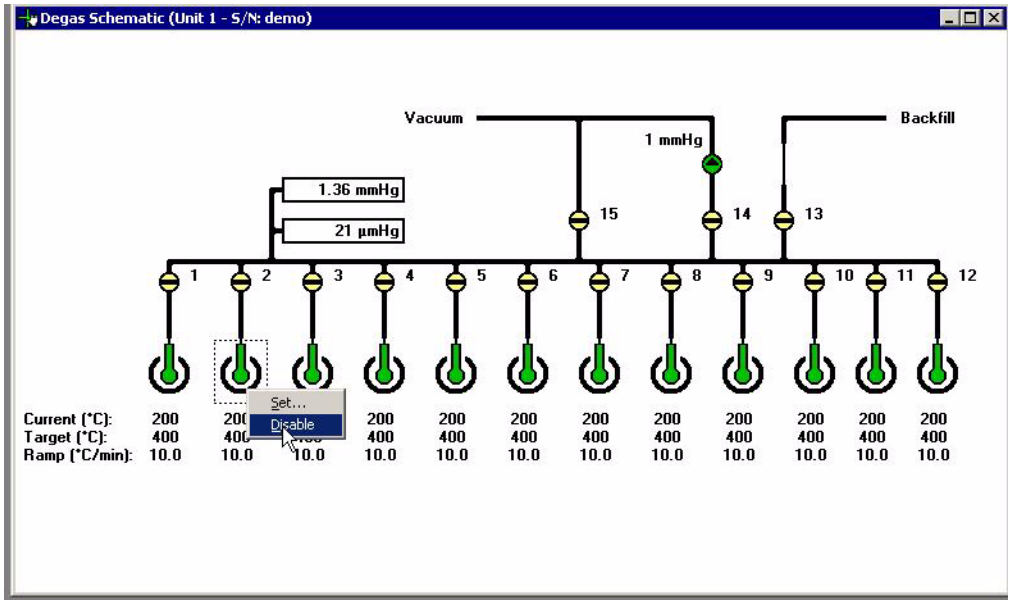


    c. Enter 10 °C/min. for **Ramp rate**.

    d. Enter 150 °C for **Target temperature**.

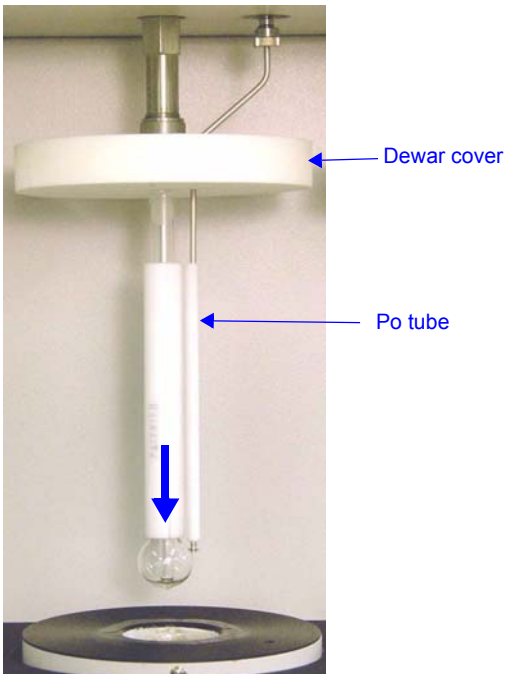
12. Allow the sample to degas for a minimum of one hour. (Additional time may be required to fully degas the sample.)

13. Right-click on the degas port sample tube, then select **Disable**.



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

14. When the heating mantle is cool enough to handle, remove it from the sample tube.
15. Allow the sample to cool to room temperature.
16. Slide the isothermal jacket down.
17. Place the sample tube Dewar cover over the sample tube stem just above the isothermal jacket as shown in the following illustration. Make sure the Po tube is next to the sample tube.



18. Place the safety shield over the sample tube and Dewar.

19. Begin the analysis. Refer to **"Performing an Analysis" on page 3-51**.

Performing an Analysis

Analyses may be performed in one of these modes:

- Standard
- Krypton
- High Throughput
- Micropore

In **Standard** mode, the system performs nitrogen, or similar, gas analyses at 77 K. All analyses must use the same gas. Samples can be removed and added to any of the six ports without disturbing the analyses being performed on other ports.

In **Krypton** mode, up to five Krypton analyses are started together. Port 6 is used to store Krypton for dosing. The data collection is done sequentially — one analysis starts and completes before the next is started.

In **High Throughput** mode, up to six nitrogen, or similar, gas analyses are started together. All analyses must use the same analysis gas and the same Psat gas, which may be different from the analysis gas. Once a set is started, no other samples can be started until the set is complete. The data collection is done in parallel. This mode also supports measured freespace.

In **Micropore** mode, up to six analyses are started together. Once a set is started, no other samples can be started until the set is complete. For micropore analyses, side-lacing heating mantles with longer cables are provided to allow in-place degassing on sample ports.

After the sample has been degassed and transferred to an analysis port, you may begin an analysis.

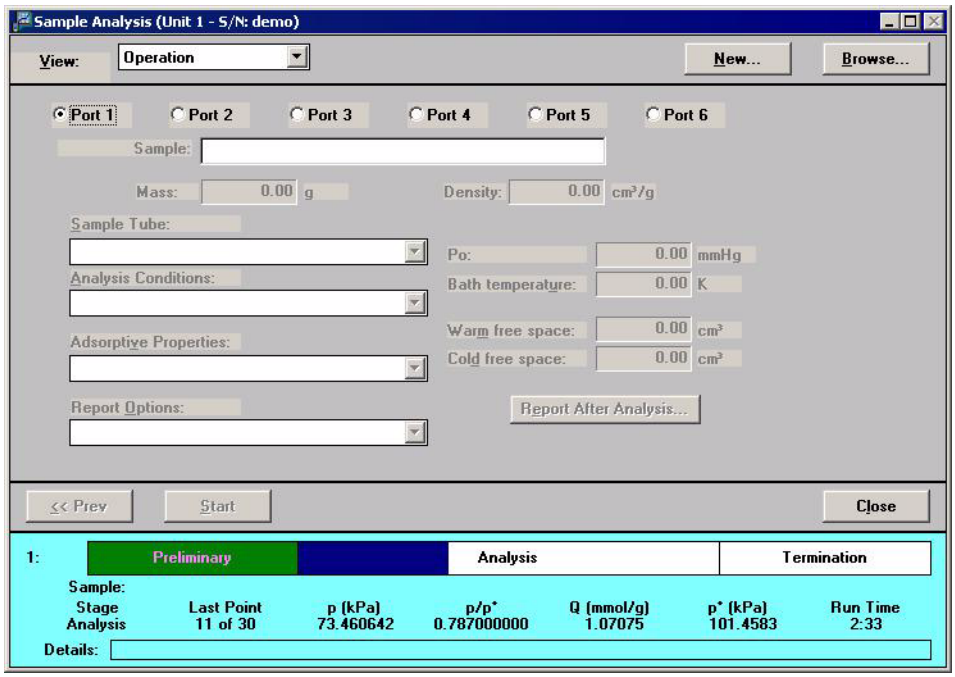
Before beginning an analysis, make sure the tank pressure for the gas regulator is at least 200 psig. Pressures less than 200 psig may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.

Ensure that the analysis gas and the Psat gas specified in the sample file match the system configuration gases; if they do not, correct the sample information file or correct the unit configuration gas. You can check the system configuration gas by selecting **Unit > Unit Configuration** (refer to **"Unit Configuration" on page 5-38**).

Standard Analysis

In standard analyses, the six ports operate independently. You can add and remove samples to and from ports without disturbing the operations on other ports. The only restriction is that you must use the same analysis gas (and the same Psat gas if saturation pressure is being measured) for all ports.

1. Select **Unit** > **Start Analysis**. The Sample Analysis dialog is displayed.

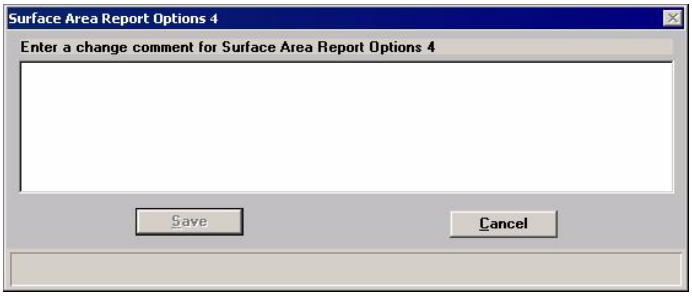


2. Select or create a sample file (refer to **Selecting Files for Degassing or Analyzing Samples**).
3. The name of the sample file or template you selected in step 2 is shown in the **Sample** field. You may edit it if desired.
4. The names of the parameter files in the sample file are displayed. You may select different parameter files from the drop-down lists if desired.
5. Verify the **Mass, Density,** and **Bath temperature**. Make changes if necessary.
6. Click **Report after Analysis** and choose whether to generate a report after analysis. If you select to generate a report, enter the destination. (Refer to **"Report Settings" on page 2-22**).

7. Click **Start** to begin the analysis. The login screen is displayed.

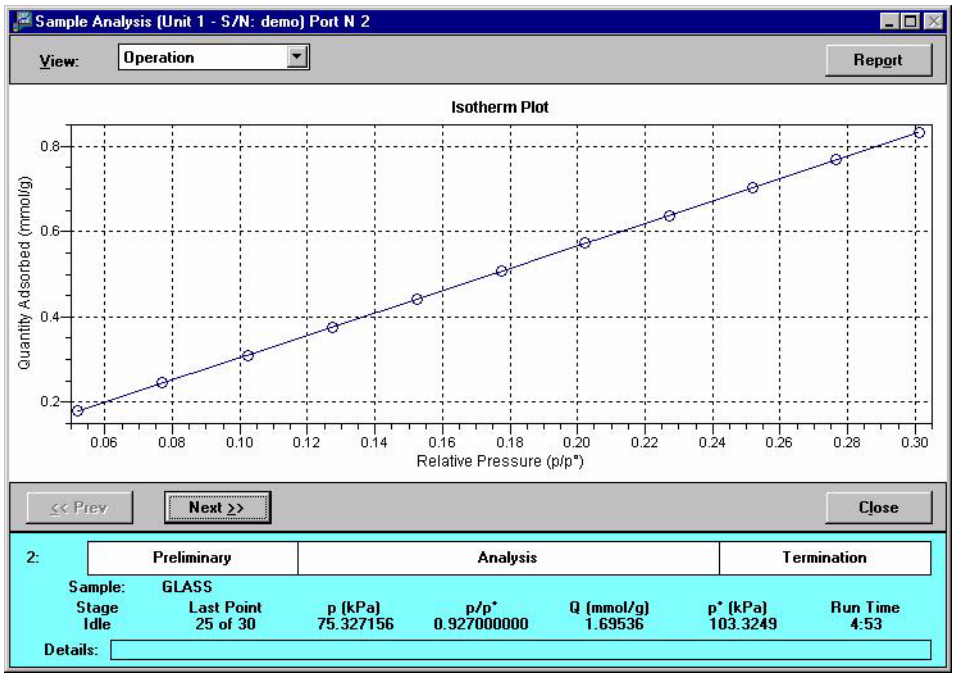
8. Enter your password, then click **OK**.

9. If you edited the sample file, the comment dialog is displayed.



Enter comments about the sample information file, then click **Save**.

10. The Sample Analysis screen is displayed. As data are collected, a graph is drawn in the window.



11. When the analysis is complete, the **Start** button changes to **Next**.

12. Click **Close** to exit or **Next** to perform another analysis. If you click **Next**, the first view of the Analysis dialog is displayed.

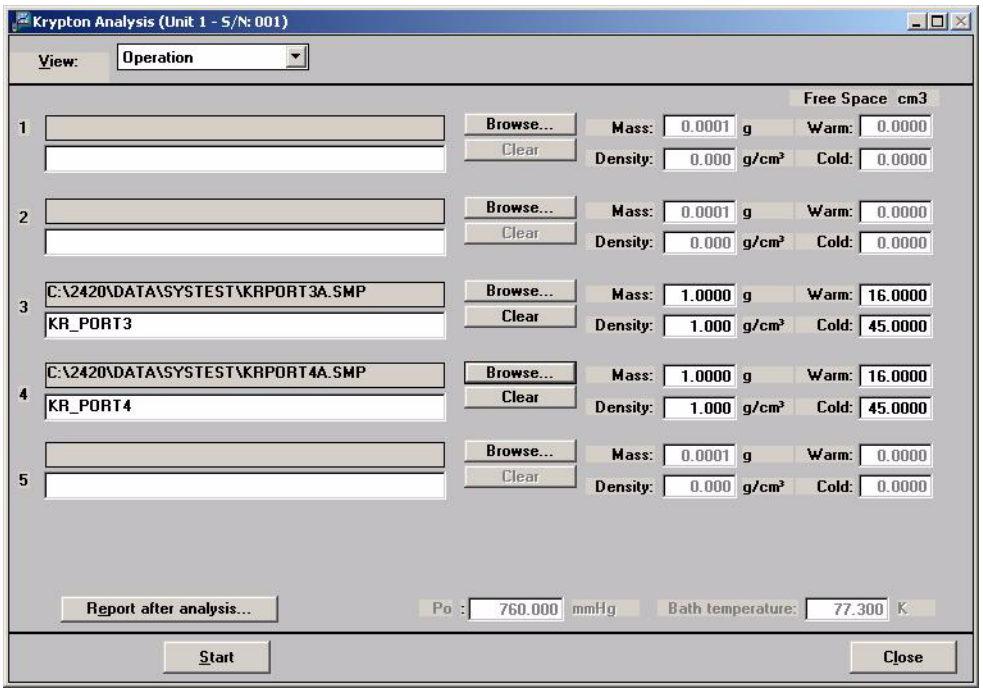
Krypton Analysis

Krypton analyses are available only if the Krypton option is installed on the analyzer.

You can perform from one to five Krypton analyses using ports 1 through 5. Port 6 is reserved for purifying and storing Krypton, which is used for dosing. Once a set of analyses is started, no other samples can be started until the set is complete.

The Dewar below port 6 must be at least 50% full of the analysis bath liquid and an empty sample tube must be installed on port 6 before starting a krypton analysis.

1. Make sure the port 6 Dewar contains analysis bath liquid.
2. Install an empty sample tube on port 6, and place a Dewar cover over the sample tube stem and the Po tube stem.
3. Select **Unit > Start Krypton Analysis**. The Krypton Analysis dialog is displayed.
4. For each port on which you wish to perform an analysis:
   a. Select or create a sample file (refer to **Selecting Files for Degassing or Analyzing Samples**).
   b. The path name and description of the sample file you selected in step 4a. are shown next to the port number. You may edit the description if desired.

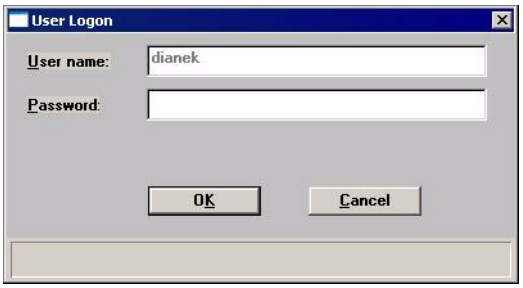


c. You may change the mass, density, and warm and cold free space if necessary.

5. Verify the Po and bath temperature; make changes if required.

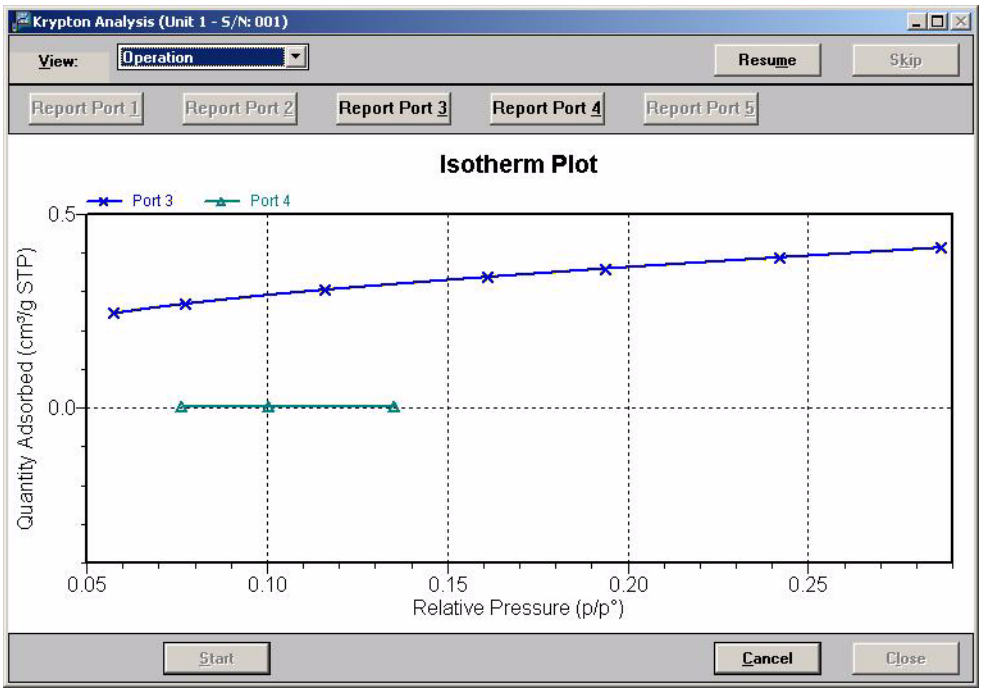
6. Click **Report after Analysis** and choose whether to generate a report after analysis. If you select to generate a report, enter the destination. (Refer to **"Report Settings" on page 2-22**).

7. Click **Start** to begin the analysis. The login screen is displayed.



8. Enter your password, then click **OK**.

9. The Krypton Analysis screen is displayed. As data are collected, a graph is drawn in the window.



10. If you wish to view an analysis report, click the appropriate **Report Port** button.

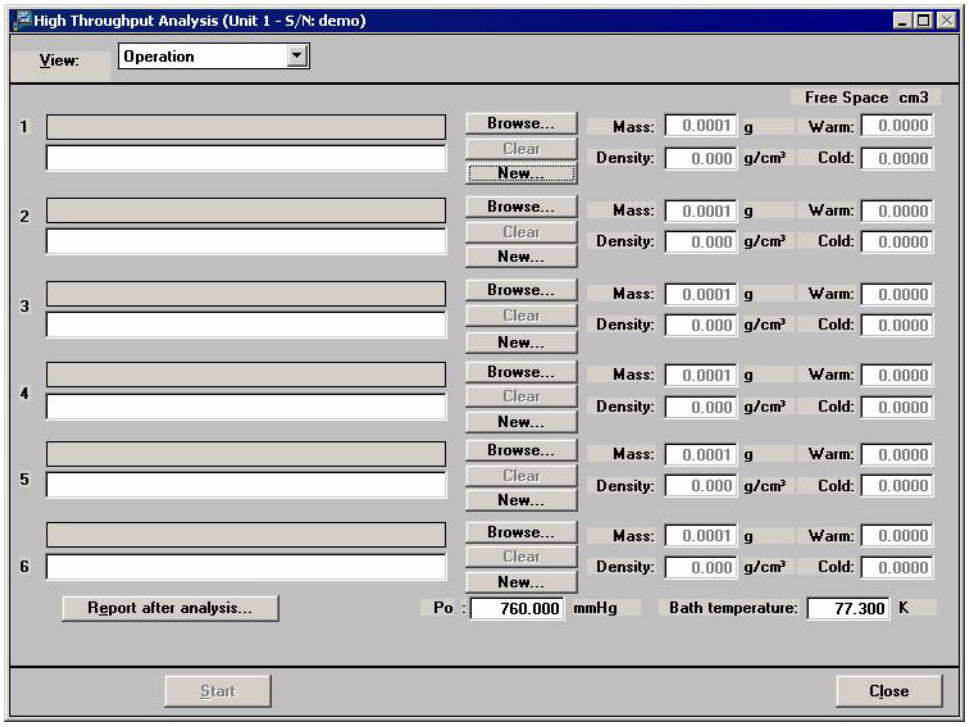
11. When the analysis is complete, the **Start** button changes to **Next**.

12. Click **Close** to exit or **Next** to perform another analysis. If you click **Next**, the first view of the Analysis dialog is displayed.

High Throughput Analysis

You can perform from one to six analyses simultaneously. Once a set of analyses is started, no other samples can be started until the set is complete.

1. Select **Unit > Start High Throughput Analysis**. The High Throughput Analysis dialog is displayed.



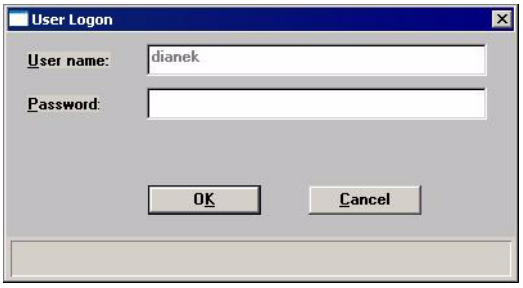
2. For each port on which you want to start an analysis:

   a. Select or create a sample file. Refer to **Selecting Files for Degassing or Analyzing Samples**.

   b. You may change the mass, density, and warm and cold free space if necessary.

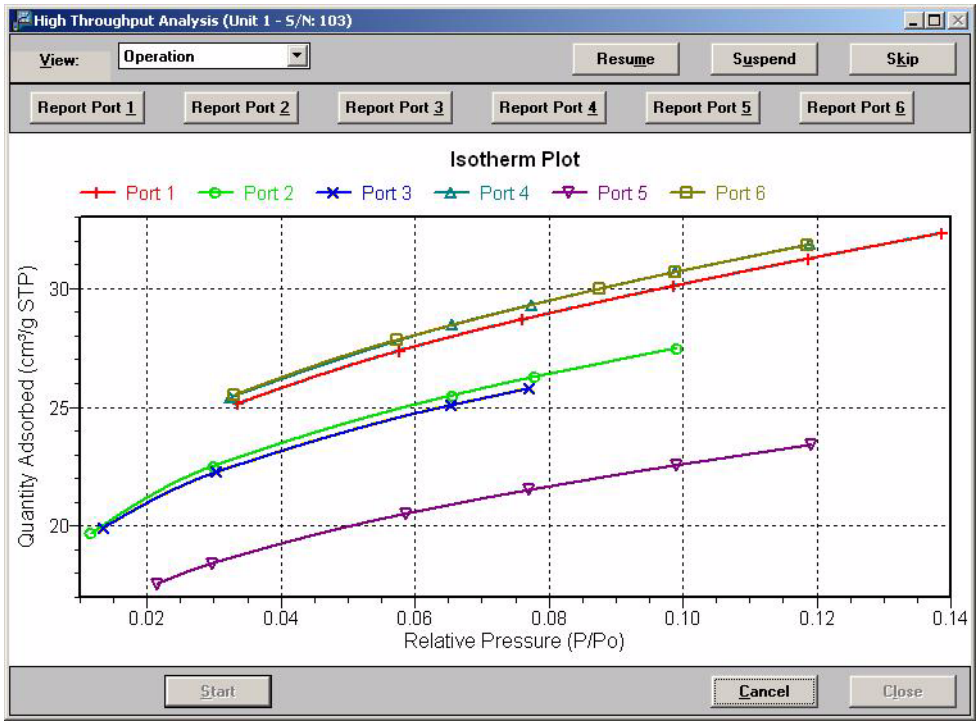
3. Click **Report after Analysis** and choose whether to generate a report after analysis. If you select to generate a report, enter the destination. (Refer to **"Report Settings" on page 2-22**).

4. Click **Start** to begin the analysis. The login screen is displayed.



5. Enter your password, then click **OK**.

6. The High Throughput Analysis screen is displayed. As data are collected, a graph is drawn in the window.



7. If you wish to view an analysis report, click the appropriate **Report Port** button.

8. When the analysis is complete, the **Start** button changes to **Next**.

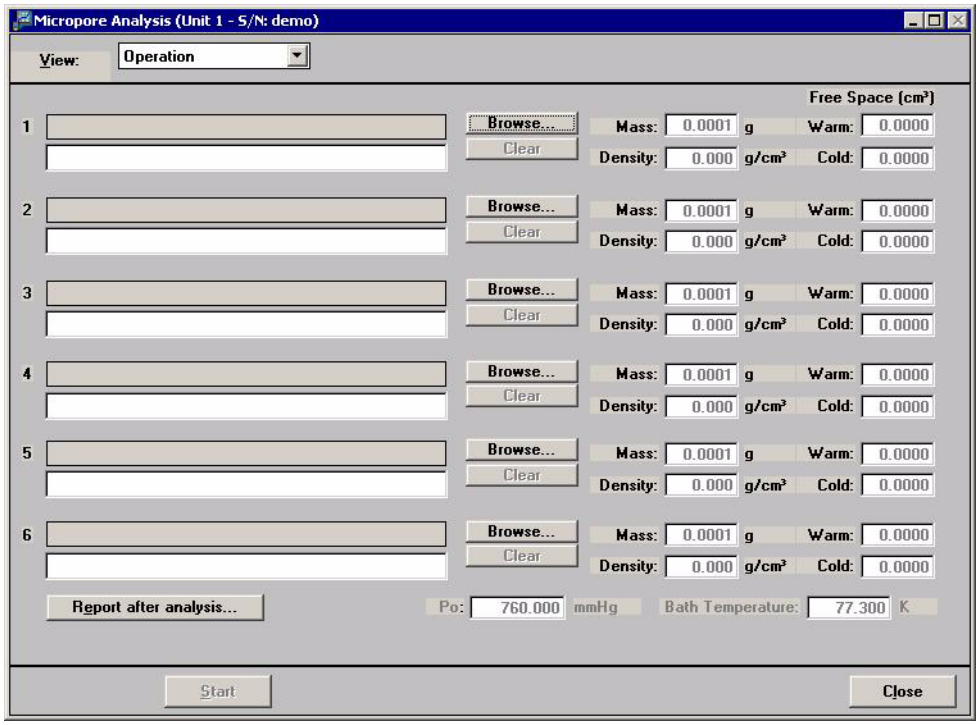
9. Click **Close** to exit or **Next** to perform another analysis. If you click **Next**, the first view of the Analysis dialog is displayed.

Micropore Analysis

Micropore analyses are available only if the Micropore option is installed on the analyzer.

You can perform from one to six analyses simultaneously. Once a set of analyses is started, no other samples can be started until the set is complete.

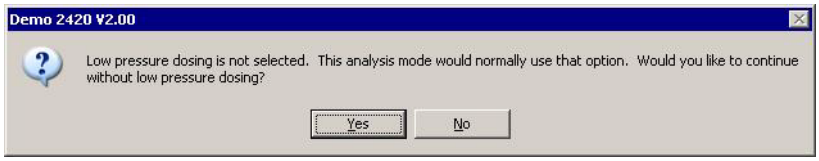
1. Select **Unit > Start Micropore Analysis**. The Micropore Analysis dialog is displayed.



2. For each port on which you want to start an analysis:

   a. Click **Browse** and select a sample file. Refer to **Selecting Files for Degassing or Analyzing Samples**.

      If you select a sample file that does not have low dosing enabled, the following message is displayed.

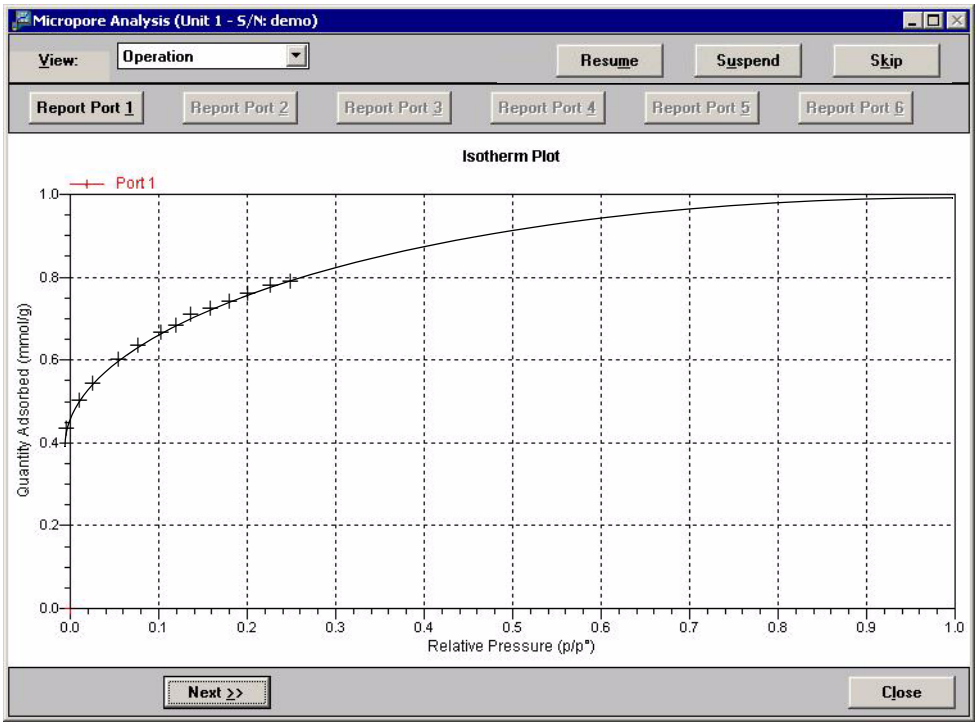


      Select **Yes** to continue without low dosing or **No** if you wish to change the sample file.

   b. You may change the mass, density, and warm and cold free space if necessary.

3. If you wish to print a report after analysis, click **Report after analysis** (refer to **"Report Settings" on page 2-22**).

4. Click **Start** to begin the analysis; as data are collected, a graph is drawn in the window.



5. If you wish to view an analysis report, click the appropriate **Report Port** button.

6. When the analysis is complete, the **Start** button changes to **Next**.

7. Click **Close** to exit or **Next** to perform another analysis. If you click **Next**, the first view of the Analysis dialog is displayed.

Printing File Contents

A Developer can print the contents of:

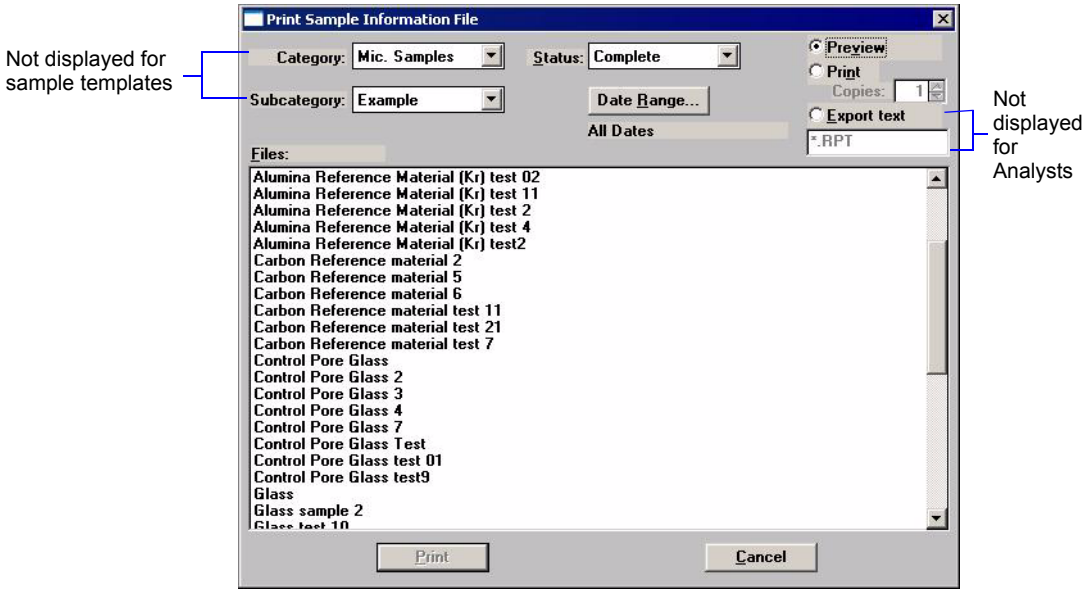
- sample files or templates
- parameter files or templates

An Analyst can print the contents of:

- sample files or templates
- parameter templates (but not parameter files)

Print Sample Files or Templates

1. Select **File** > **Print > Sample Information** or **Sample Template**. A dialog similar to this one is displayed.



2. From the **Files** list, select the desired sample file or template file. If you plan to print multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Category, Subcategory (sample information files only), Status drop-down list and/or the Date Range push buttons to limit the files displayed in the Files list. Refer to "File Selection" on page 2-19.

3. Select the destination:
   - Click **Preview** to display file contents on the screen.
   - Click **Print** to send file contents to your default printer.

If you selected **Print**, use the up and down arrows to select a number or enter a number in the field. You may print from 1 to 4 copies.

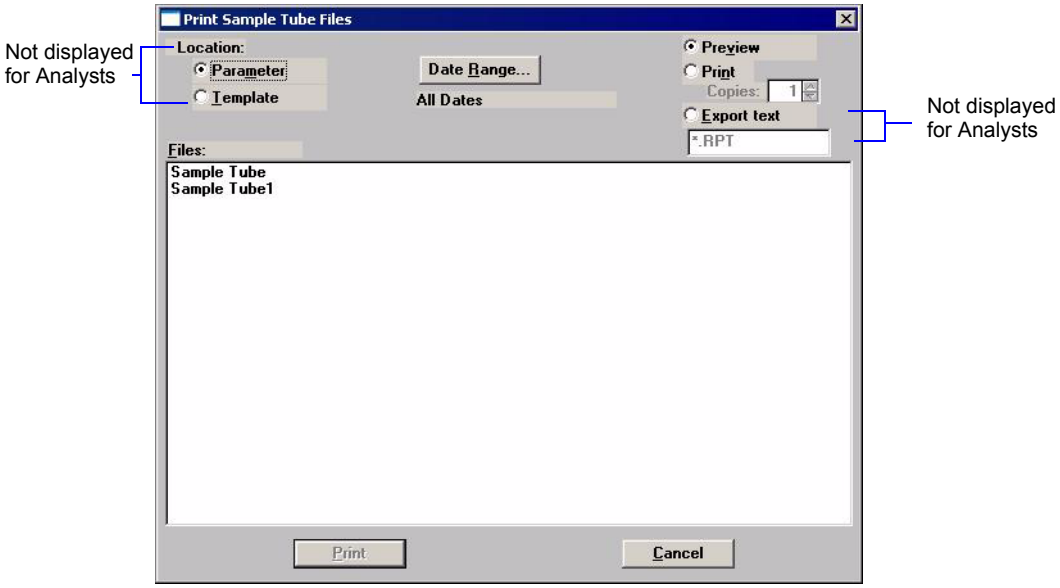
- Click **Export text** to save the file contents in a text file. This option is displayed only if a Developer is logged in.

  Enter a file name in the field below the radio button.The default name is the name of the file with the .RPT extension. The file will be saved to the **Export** directory.

4. Click **Print**; the file(s) is sent to the specified destination.

Print Parameter Files or Templates

1. Select **File** > **Print > (parameter file name)**. For example: **File** > **Print > Sample Tube**. A dialog similar to this one is displayed.



2. For Developers only: Click **Parameter** to list parameter files or **Template** to list parameter templates.

3. From the **Files** list, select the desired sample or template file. If you plan to print multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Date Range button to limit the files displayed in the Files list. Refer to "Selecting a Range of Dates" on page 2-21.

4. Select the destination:

   - Click **Preview** to display file contents on the screen.

   - Click **Print** to send file contents to your default printer.

If you selected **Print**, use the up and down arrows to select a number or enter a number in the field. You may print from 1 to 4 copies.

- Click **Export text** to save the file contents in a text file. This option is displayed only if a Developer is logged in.

  Enter a file name in the field below the radio button.The default name is the name of the file with the .RPT extension. The file will be saved to the **Export** directory.

5. Click **Print**; the file(s) is sent to the specified destination.

Listing File Statistics

A Developer can list file statistics for:

- sample files or templates
- parameter files or templates

An Analyst can list file statistics for:

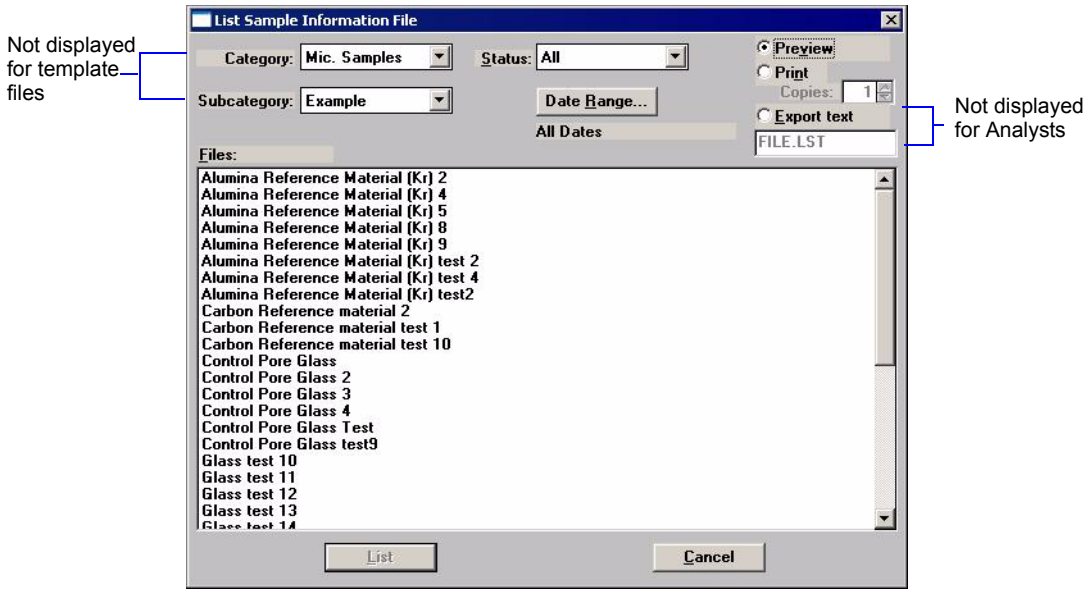
- sample files or templates
- parameter templates (but not parameter files)

The information displayed in the list includes:

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

List Sample File or Template Statistics

1. Select **File** > **List > Sample Information** or **Sample Template**. A dialog similar to this one is displayed



2. From the **Files** list, select the desired sample file or template file. If you want to list multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Category, Subcategory (sample information files only), Status drop-down list and/or the Date Range push buttons to limit the files displayed in the Files list. Refer to "File Selection" on page 2-19.

3. Select the destination:

   - Click **Preview** to display file statistics on the screen.
   - Click **Print** to send file statistics to your default printer.

     If you selected **Print**, use the up and down arrows to select a number or enter a number in the field. You may print from 1 to 4 copies.

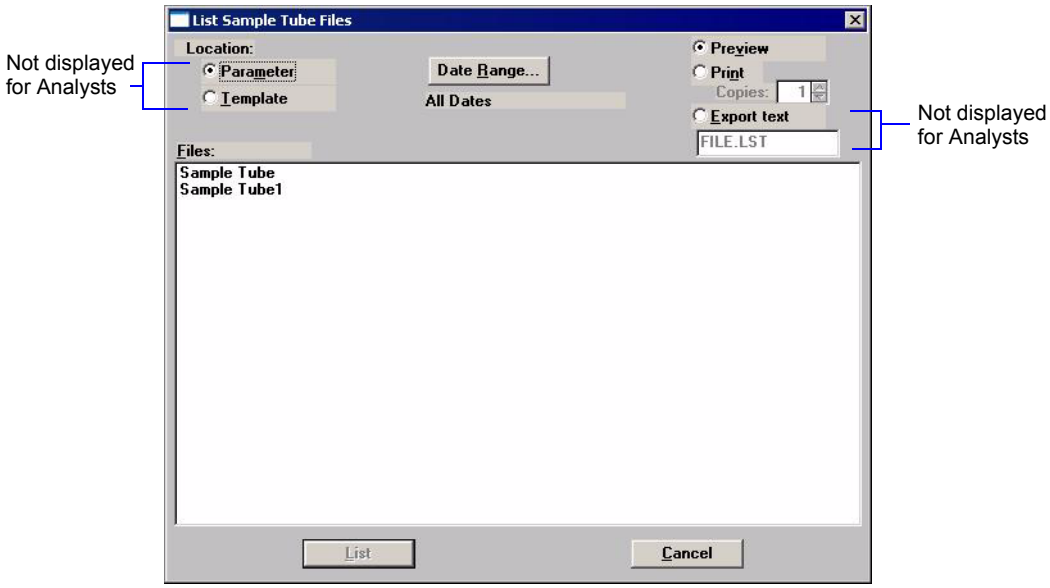
   - Click **Export text** to save the file statistics a in a text file. This option is displayed only if a Developer is logged in.

     Enter a file name in the field below the radio button.The default name is the name of the file with an .LST extension. The file will be saved to the **Export** directory.

4. Click **List**; the file statistics are sent to the specified destination.

List Parameter File or Template Statistics

1. Select **File** > **List> (parameter file name)**. For example: **File** > **List > Sample Tube**. A dialog similar to this one is displayed.



2. For Developers only: click **Parameter** to list parameter files or **Template** to list parameter templates.

3. From the **Files** list, select the desired parameter file or template file. If you want to list multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Date Range button to limit the files displayed in the Files list. Refer to "Selecting a Range of Dates" on page 2-21.

4. Select the destination:

   - Click **Preview** to display file statistics on the screen.
   - Click **Print** to send file statistics to your default printer.

     If you selected **Print**, use the up and down arrows to select a number or enter a number in the field. You may print from 1 to 4 copies.

   - Click **Export text** to save the file statistics a in a text file. This option is displayed only if a Developer is logged in.

     Enter a file name in the field below the radio button.The default name is the name of the file with an .LST extension. The file will be saved to the **Export** directory.

5. Click **List**; the file statistics are sent to the specified destination.

Importing Sample and Parameter Files

An Analyst is not authorized to import sample or parameter files.

The Import function allows you to import files which have been created on a computer other than the one controlling the analyzer and have them display in the **Files** list window.

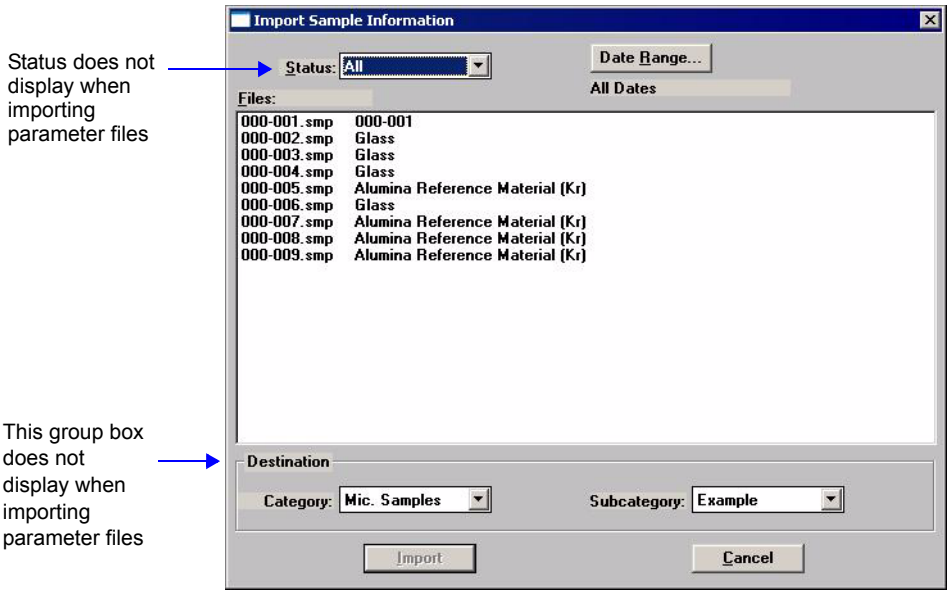
Review the Micromeritics PROGRAM License Agreement for restrictions on the use of another copy.

Perform the following steps to import a file:

1. Copy the file(s) you wish to import to the **Import** directory. Files must be copied into this directory; otherwise they will not display in the **Files** list in the Import File dialog.

2. Select **File** > **Import > (file type)**.

   For example, **File > Import > Sample Information**. A dialog similar to the following is displayed.



3. From the **Files** list, choose the desired file. If you want to import multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Status drop-down list and/or the Date Range buttons to limit the files displayed in the Files list. Refer to "File Selection" on page 2-19.

4. Select the directory and subdirectory into which the file will be imported. If you are importing a parameter file, the **Category** and **Subcategory** fields do not display. Parameter files are copied automatically into the directory assigned for parameter files (**param**).

5. Click **Import** to import the file(s). A confirmation message is displayed:

6. Click **OK.**

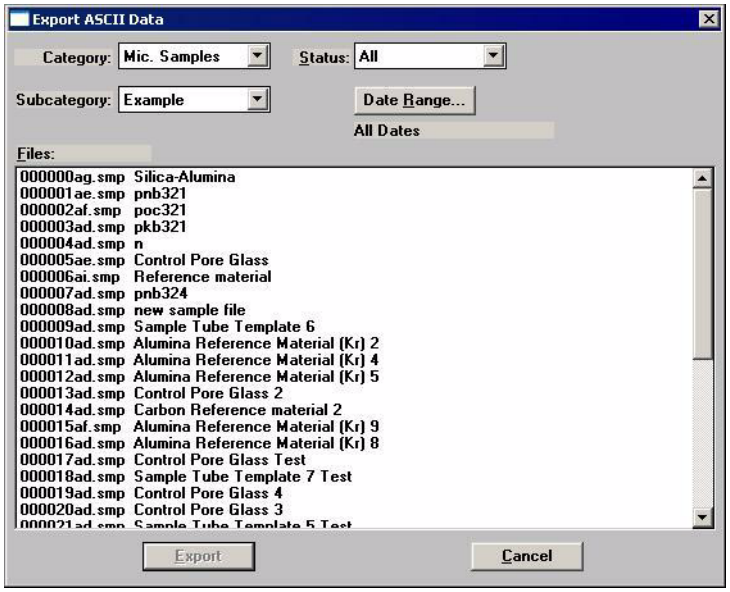
Exporting Sample Data

An Analyst is not authorized to export files.

The Export option on the File menu allows you to:

- export an entire sample file to the **Export** directory. For example, you may wish to use this option to transfer a sample file to another computer.
- copy the isotherm data in a sample information file and export it as ASCII text. If saved to a **File**, the data can be imported into applications, such as spreadsheets. The output file consists of four columns containing the absolute pressure, relative pressure, specific volume adsorbed, and the elapsed time.

1. Select **File** > **Export > Sample information** or **ASCII**. A dialog similar to this one is displayed.



2. From the **Files** list, choose the desired file. If you want to export multiple files, hold down **Ctrl** while clicking on the desired files.

You can use the Status drop-down list and/or the Date Range button to limit the files displayed in the Files list. Refer to "File Selection" on page 2-19.

3. Click **Export**. The files are sent to the Export directory. Sample files have the extension **.SMP**; isotherm data text files have the extension **.ASC**.

Generating Graph Overlays

Use graph overlays when you wish to compare graphically results for multiple samples or multiple graphs for one sample. Graphical lines are differentiated by symbols and colors.

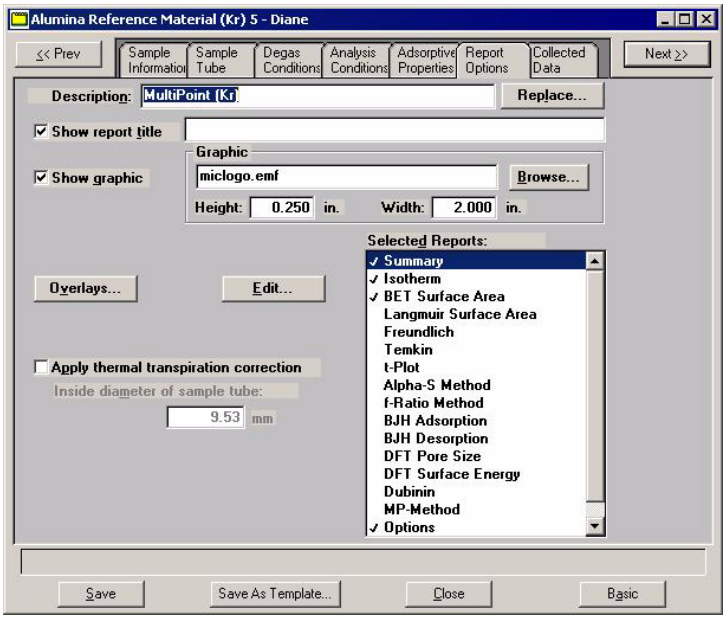
Graph overlays can be generated in two ways:

- **Multiple Sample Overlays**
  Overlay results for up to eight samples on top of a previously selected sample.
- **Multiple Graph Overlays**
  Overlay two different types of graphs from one sample. The multiple graph overlay capability exists for BJH Adsorption or Desorption, Horvath-Kawazoe*, and MP-Method.

*Available only if the micropore option is installed.

Multiple Samples Overlay

1. Select **File > Open > Sample Information** to display the Open Sample Information File dialog.
2. Select a sample on which to overlay graphs of other samples, then click **Open**; the Sample Information dialog is displayed.
3. For Analysts only, click the **Advanced** button on the Sample Information dialog.
4. Click the Report Options tab to display the Report Options dialog.

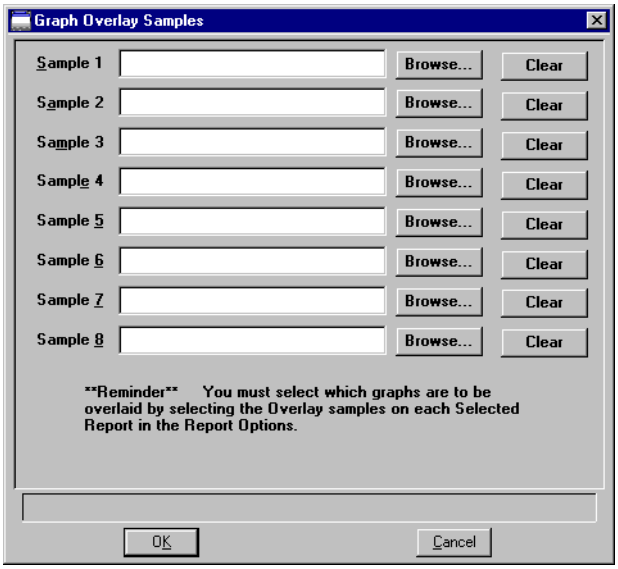


5. Make your report selections (or deselect any you do not wish to generate).

6. Choose the type of report for which you wish to generate overlays; perform the steps below:

If overlaying this type of report...	**Then...**
Alpha-S Isotherm BET/Langmuir surface area F-ratio Freundlich Temkin t-Plot	Select the report for which you want an overlay, then click **Edit**. A report options dialog for that report is displayed.
	Select **Overlay Samples** and do any other editing you wish.
	Click **OK**; the main Report Options dialog reappears.
Dubinin	Select Dubinin, then click **Edit**; a Report Options dialog box for Dubinin is displayed.
	Select the desired graph, then click **Edit**. An options dialog box for that graph is displayed (for example, dV/dw Pore Volume).
	Select **Overlay Samples** and do any other editing you wish.
	Click **OK**; the options dialog box for Dubinin reappears.
	Click **OK**; the main Report Options dialog box reappears.
BJH Adsorption BJH Desorption DFT Pore Size DFT Surface Energy Horvath-Kawazoe MP-Method	Select the report for which you want an overlay, then click **Edit**. A Report Options dialog for that report is displayed.
	Select the desired report, then click **Edit**. An options dialog for that graph is displayed.
	Click on the down-arrow at the **Overlay** field and select **Samples**; do any other editing you wish.
	Click **OK**; the options dialog for the selected report reappears. Do any other editing you wish.
	Click **OK**; the main Report Options dialog reappears.

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



8. Click **Browse** to the right of the **Sample 1** field; the Plot Overlay Sample Selection dialog is displayed.
9. Choose a sample file, then click **Open**. You may choose up to eight files in this manner.
10. After selecting the desired number of sample files, click **OK**; the main Report Options dialog reappears.
11. Click **Save** if you wish to save your Selected Reports list in the sample information file.

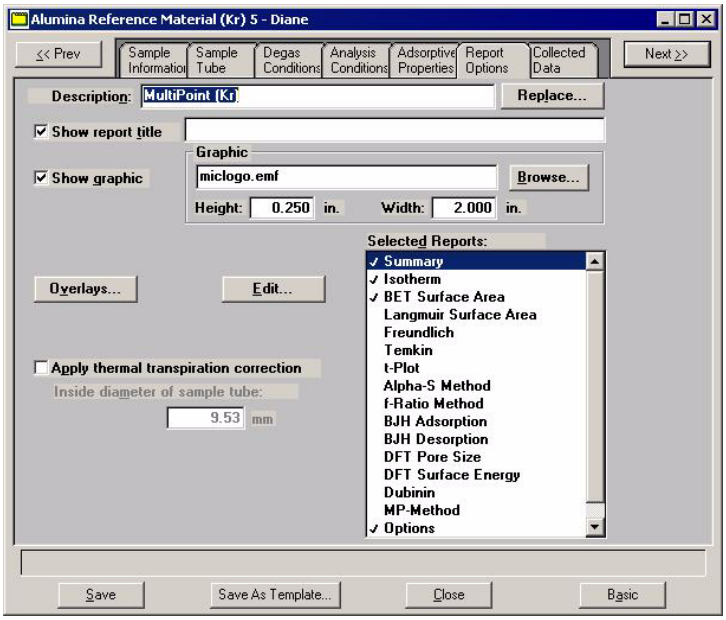
    If you click **Save**, the Comments window is displayed. Enter comments about your edits, then click **Save.**
12. Select **Reports > Start Report**. The Start Report screen is displayed with the name of the sample information file you edited highlighted.
13. Click **Report**; the Select Reports dialog is displayed.
14. Ensure that the desired graph is selected (preceded with a check mark), then click **OK**.

Multiple Graphs Overlay

Multiple graph overlays can be generated for:

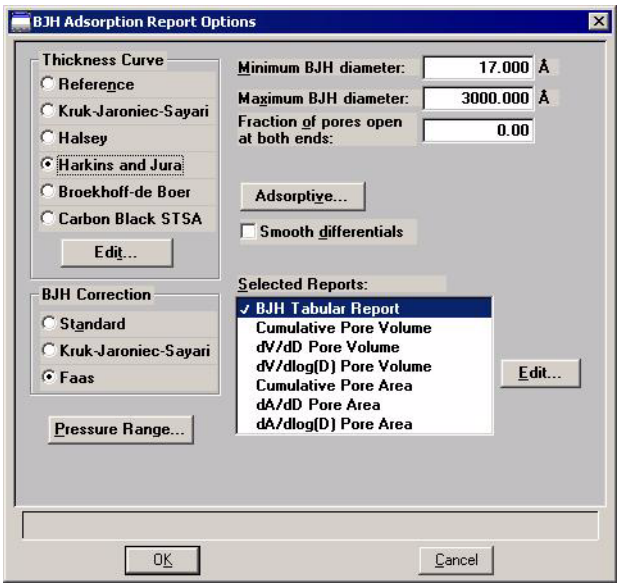
- BJH Adsorption
- BJH Desorption
- DFT Pore Size
- DFT Surface Energy
- Horvath-Kawazoe
- MP-Method

1. Select **File > Open > Sample Information** to display the Open Sample Information File dialog.
2. Select a sample on which to overlay graphs of other samples, then click **Open**; the Sample Information dialog is displayed.
3. For Analysts only, click the **Advanced** button on the Sample Information dialog.
4. Click the Report Options tab to display the Report Options dialog.

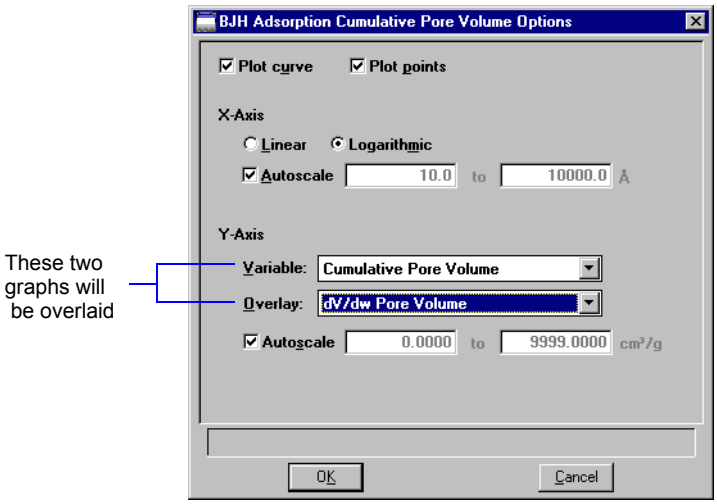


5. Make your report selections (or deselect any you do not wish to generate).

6. From the **Selected Reports** list, select the report for which you want multiple graph overlays, then select **Edit**; a Report Options dialog for that report (or graph) is displayed (this example shows BJH Adsorption).



7. Select a report type, then click **Edit**; an options dialog for that specific item is displayed (this example shows Cumulative Pore Volume).



8. Click the down arrow at the **Overlay** field and select a graph type from the list. This selection will be overlaid on the graph selected in the **Variable** field.

9. Select **OK** to return to the BJH Adsorption Report Options dialog; do any editing you wish.

10. Click **OK** to return to the main Report Options dialog.

11. Click **Save** if you wish to save your Selected Reports list. (Even if you don't save, all of the options you selected are included as your reports are generated.)

If you click **Save**, the Comments window is displayed. Enter comments about your edits, then click **Save.**

12. Select **Reports** >**Start Report**; the Start Report screen is displayed with the name of your primary file highlighted.
13. Click **Report**; the Select Reports dialog is displayed.
14. Ensure that the desired graph is selected (preceded with a check mark), then click **OK**.

4 FILE MENU

Description	4-3
New	4-5
Open	4-91
Save	4-95
Save As	4-96
Save As Template	4-98
Save All	4-98
Close	4-98
Close All	4-99
Print	4-99
List	4-101
Import	4-102
Export	4-104
Convert	4-105
Log In	4-106
Log Out	4-106
Exit	4-107

FILE MENU

The File menu contains commands which allow you to manage sample and parameter files.

Description

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

New Allows you to create a sample information or parameter file.

Open Opens an existing sample information or parameter file.

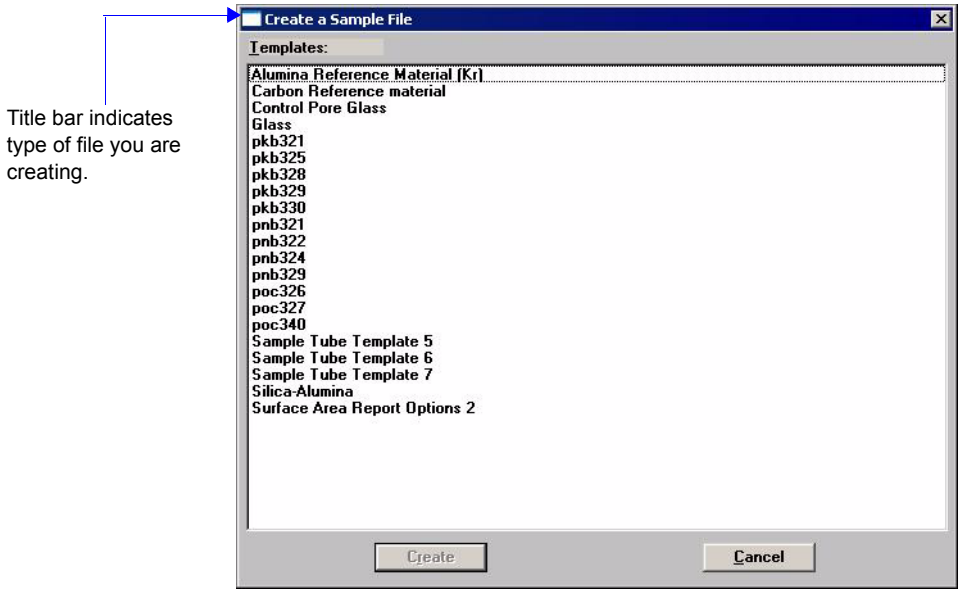
Save Saves the file in the active window.

Save As Saves the file in the active window as a different name. You also can use this option to save a subset of the sample file as a parameter file.

Save As Template	Allows you to save the values from the open file as a template for a sample information or parameter file.
Save All	Saves all open files.
Close	Closes the file in the active window.
Close All	Closes all open files.
Print	Prints the contents of a sample or parameter file.
List	Generates a list of certain information for sample or parameter files.
Import	Allows you to import a sample information or parameter file into the assigned application directory.
Export	Allows you to export the data from a sample information file.
Convert	Allows you to convert a sample file in the MICMOS 2400 or 2405 format to a format compatible with the ASAP 2420 Confirm program.
Log in	Allows you to log into the application.
Log out	Allows you to log out of the application.
Exit	Exits the analysis program.

New

New allows you to create a sample information or parameter file. Regardless of the type of file you are creating, a dialog allowing you to choose a template is displayed.



A Developer can create sample information and parameter files. An Analyst can create a sample information file using predefined parameter files chosen from drop-down lists.

After you choose a template, click **Create** to display a dialog for the associated file.

When you create a new file, you must enter a unique identifier for the file. You will not be able to save the file if the ID you enter already exists in the system.

Sample Information

Sample information files contain information used to control the analysis. Therefore, an analysis cannot proceed until it has been assigned a sample information file. A sample information file consists of the following:

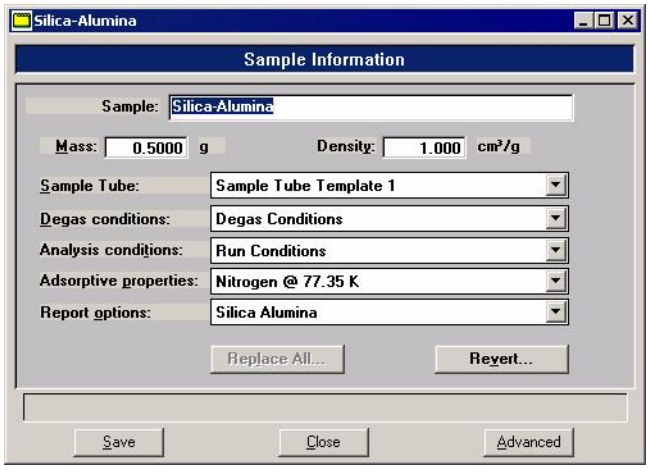
- sample identification
- sample tube criteria
- degas conditions
- analysis conditions
- adsorptive properties
- report options
- collected/entered data (after an analysis has been completed)

Parts of the sample information file can also exist as *parameter* files which are separate from the sample information file itself. Having these files exist independently allows you to use them as many times as you wish. For example, if you typically use the same analysis conditions for many of your analyses, you can create an analysis conditions file containing those conditions. Then when you create your sample file, select that file for your analysis conditions.

A sample information file can be created by an Analyst or a Developer. A Developer can create a sample file in its entirety, specifying values for all parameters; all fields on the dialogs are enabled for input from a Developer. An Analyst can create a sample file by selecting predefined parameter files from drop-down lists.

Analyst

The Sample Information dialog displays in this manner for an Analyst.



Sample

This field defaults to the description of the template. You must enter a new description or edit the existing one; the ASAP 2420 Confirm program does not allow duplicate descriptions. The description you enter in this field appears in the **Files** list window on the Open Sample Information dialog box.

Mass

This field defaults to the value specified in the template. Enter the value recorded for *Mass of Sample After Degas* from the Sample Data Worksheet.

The value in this field is used in report calculations; therefore, it should be exact. To ensure accuracy, reweigh the sample after analysis. Enter the post-analysis value if different from the *After Degas* value before generating reports.

Density

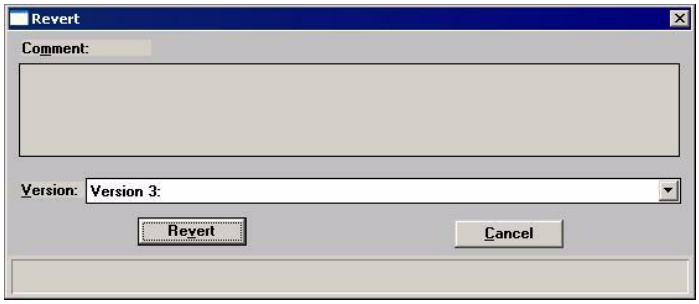
If you plan to use a calculated free space, enter the sample's density in this field. A value in this field is ignored for measured or entered free spaces.

Sample Tube
Degas conditions
Analysis conditions
Adsorptive properties
Report options

These fields contain the descriptions of the current parameter files. Click on the down arrow to the right of the fields to choose different files. These drop-down lists contain predefined files created by a Developer. You can view the values contained in the files by clicking **Advanced**; however, you cannot edit them. You can edit details of some reports by clicking **Edit**.

Revert

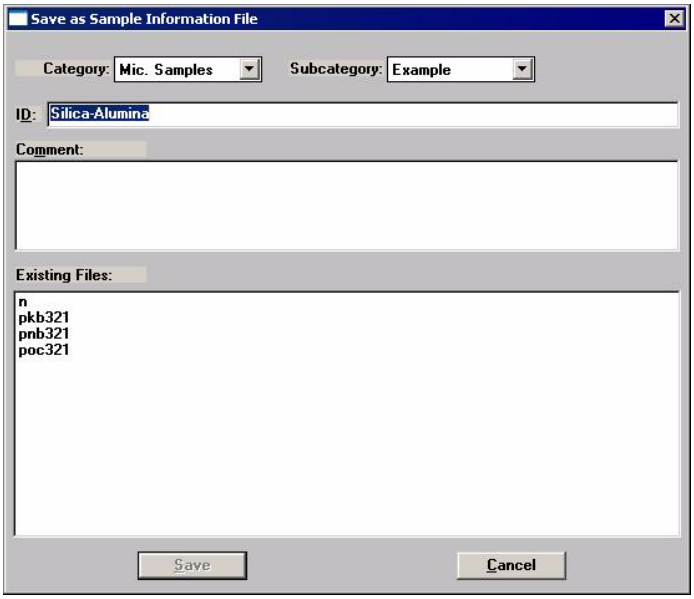
Displays the Revert dialog allowing you to choose a previous version (if any exist) of the sample file.



This dialog lists previous versions of the current file and the comment entered for each one; it does not list the current version. You can use the drop-down list to choose other versions; each version's comments will be listed in the Comment window.

Save

Displays the Save As Sample Information File dialog.



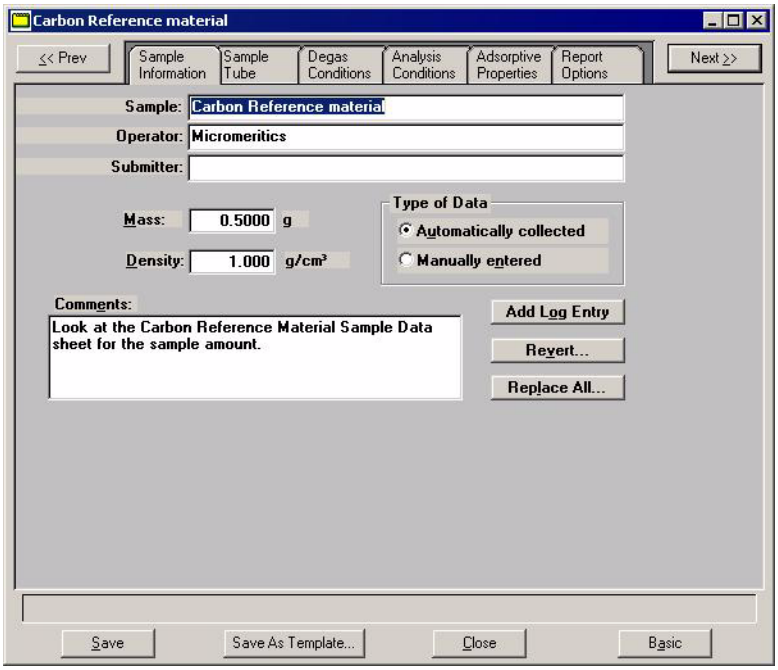
Category
Subcategory

Choose the directory in which to save the file.

Comment	Enter information about the file. This is a required field.
Save	Saves the file. If **Save** does not become enabled after an entry is made in the Comments window, a file with the same ID currently exists in the selected directory. You must choose a different directory or edit the identification of the current file. The ASAP 2420 Confirm program does not allow duplicate descriptions.
Close	Closes the dialog box. If the file contains unsaved changes, you are prompted to save before the dialog box closes.
Advanced	Displays the sample information dialog in the Developer's format. This format allows you to view details of the sample file and allows some fields to be edited. Refer to the next section for a description of the fields for the Developer's format.

Developer

The Sample Information dialog displays in this manner for a Developer.



Some fields on the Developer's view of the Sample Information dialog are also enabled for an Analyst when **Advanced** is clicked from the Analyst Sample Information dialog.

Sample — Displays the description for the current sample template. You may enter a new description or add to the existing one.

Operator
Submitter — Enter the name of the operator (Analyst) who will be using this file to perform the analysis and the name of the person (or department) submitting the sample for analysis.

Mass — This field defaults to the value specified in the template. Enter the value recorded for *Mass of Sample After Degas* from the Sample Data Worksheet.

The value in this field is used in report calculations; therefore, it should be exact. To ensure accuracy, reweigh the sample after analysis. Enter the post-analysis value if different from the *After Degas* value before generating reports.

Density

If you plan to use a calculated free space, enter the sample's density in this field. A value in this field is ignored for measured or entered free spaces.

Type of Data

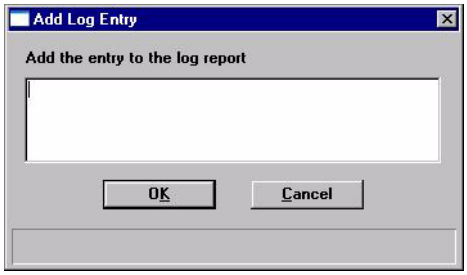
Choose whether you wish to enter the data or have it collected automatically by the analysis program. If you choose Manually entered, an "Entered" tab is added so that you may enter the desired data. You cannot enter data if you are creating the file on a computer that is not attached to the analyzer.

Comments

Allows you to enter comments about the sample or its analysis conditions. Anything you enter in this window is printed in the header of the report.

Add Log Entry

Displays the Add Log Entry dialog.

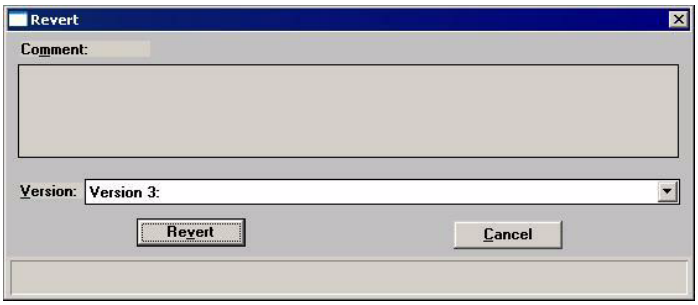


This dialog enables you to enter text to be included in the log report.

Enter text, then click **OK** to close the dialog.

Revert

Displays the Revert dialog allowing you to choose a previous version of the sample file.



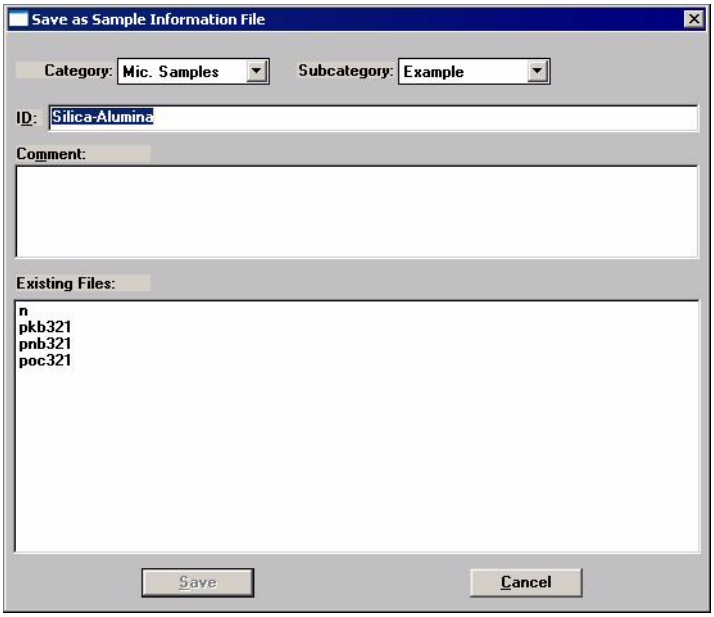
This dialog will list previous versions of the current file and the comment entered for each one; it does not list the current version. You can use the drop-down list to choose other versions; each version's comments will be listed in the Comment window.

Replace All

Click this push button to replace all parameters of the current sample file with those from another one.

Save

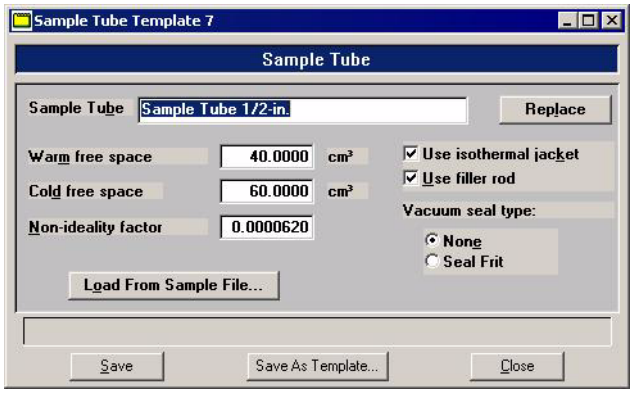
Displays the Save as Sample Information File dialog



Category **Subcategory**	Choose the directory in which to save the file.
Comment	Enter information about the file. This is a required field.
Save	Saves the file. If **Save** does not become enabled after an entry is made in the Comments window, a file with the same ID currently exists in the selected directory. You must choose a different directory or edit the identification of the current file. The ASAP 2420 Confirm program does not allow duplicate descriptions.
Save as Template	Enables you to save all parameters of the current file as a sample information file template.
Close	Closes the dialog box. If the file contains unsaved changes, you are prompted to save before the dialog box closes.
Basic	Displays the sample information dialog in the Analyst format (refer to **"Analyst" on page 4-6**).

Sample Tube

The fields on this dialog are used for storing sample tube information. Before creating a sample tube file, you should perform a blank analysis (no sample) using the sample tube.



Sample Tube — Displays a description of the current file. If this is a new file, this field displays the template description; this description must be edited before you can save it as a file.

Warm free space — Displays the warm free space for the current file.

Cold free space — Displays the cold free space for the current file.

Non-ideality factor — Displays the non-ideality factor determined during the blank analysis.

Use isothermal jacket — Select this option if an isothermal jacket is to be used on the sample tube.

Use filler rod — Select this option if a filler rod is to be used in the sample tube.

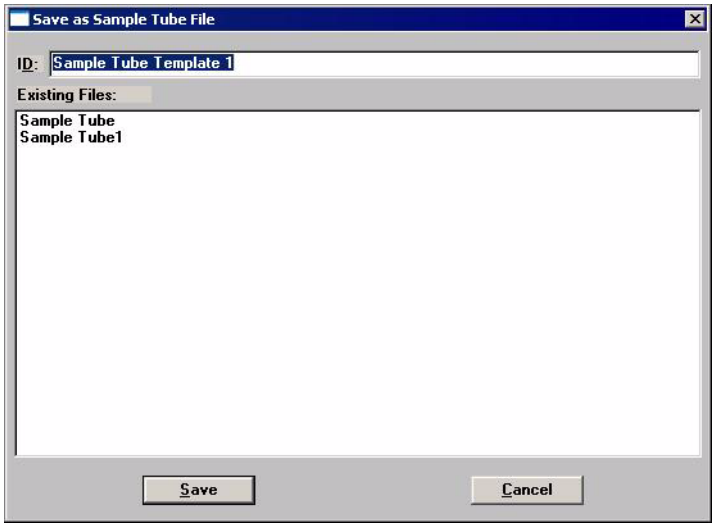
Vacuum seal type — Select **Seal Frit** if the sample tube will have a seal frit. If not, leave **None** selected.

Load From Sample File

Click this push button to choose the sample file that was used in the blank analysis. The values for the warm and cold free spaces, and the non-ideality factor will be copied into the sample tube file.

Save

Displays the Save as Sample Tube File dialog with the description you entered on the previous screen in the ID field.



Click **Save**.

Save as Template

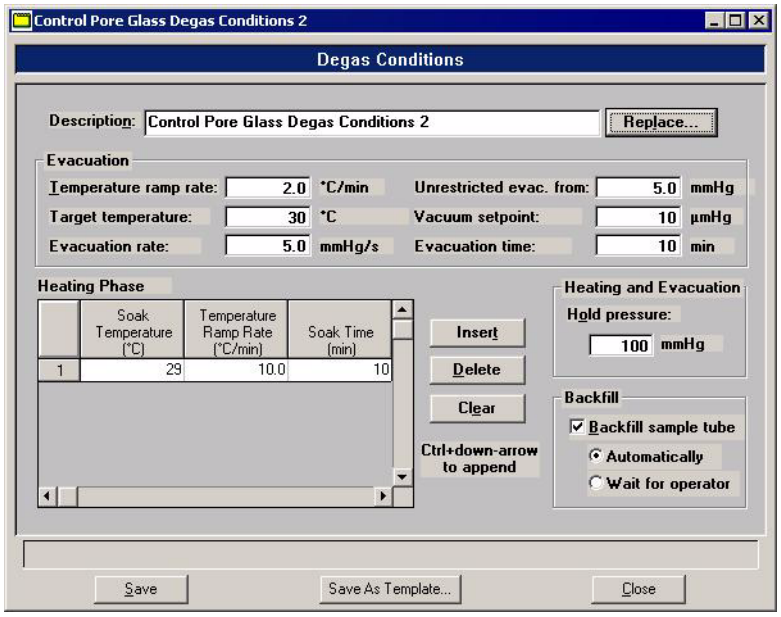
Enables you to save all parameters of the current file as a sample tube file template.

Close

Closes the dialog. If the file contains unsaved changes, you are prompted to save before the dialog box closes.

Degas Conditions

The fields on this dialog are used for specifying degas conditions.



Description

Displays a description of the current Degas Conditions template. Enter a new description or add to the existing one if desired.

Be sure to use an intuitive name so that it can be easily recognized when needed.

Replace

Use this push button to replace the current degas conditions with those from an existing Degas Conditions file. The Open Degas Conditions File dialog is displayed, allowing you to choose a file.

Evacuation Phase group box

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	Enables you to specify an evacuation rate for initial evacuation.
Unrestricted evac. from	Enables you to specify a pressure at which unrestricted sample evacuation will begin.
Vacuum setpoint	Enables you to specify the vacuum level to be achieved before evacuation begins.
Evacuation time	Enables you to specify how long the sample is to be evacuated prior to the second stage of heating.
Heating Phase group box	The options in this group box allow you to specify conditions for the heating phase of the degassing operation.
Soak Temperature	Enables you to specify a temperature at which the sample is to be held during degassing.
Temperature Ramp Rate	Enables you to specify the rate at which the temperature will change after evacuation while advancing to the hold temperature.
Soak Time	Enables 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.

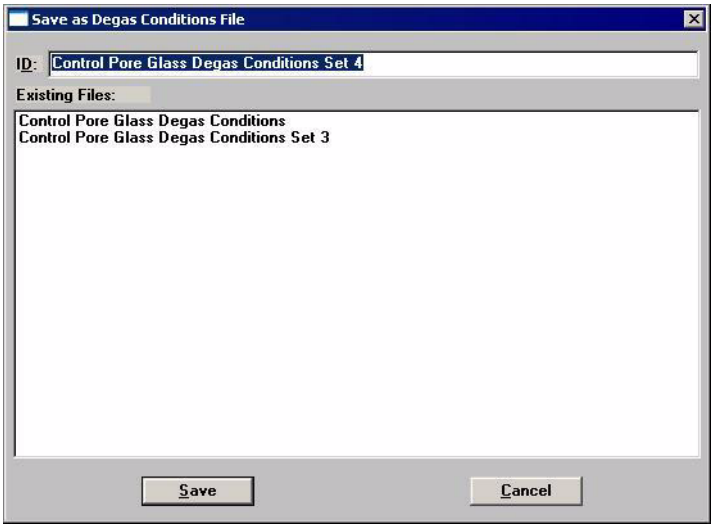
Backfill Sample Tube

Select this option to have the sample tube backfilled.

If you select this option, select whether to have the sample tube backfilled automatically or have the system wait until backfill is requested by the operator.

Save

Displays the Save as Degas Conditions File dialog with the description you entered on the previous screen in the **ID** field.



Click **Save**.

Save as Template

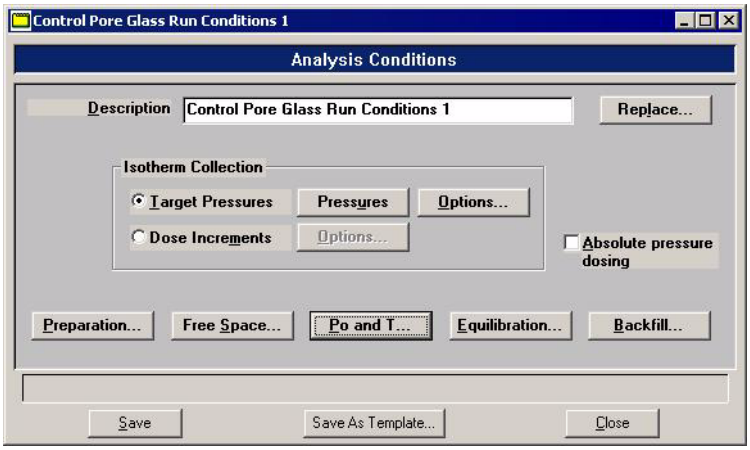
Enables you to save the parameters of the current file as a Degas Conditions file template.

Close

Closes the dialog. If the file contains unsaved changes, you are prompted to save before the dialog box closes.

Analysis Conditions

This dialog allows you to specify the analysis conditions for your sample. An analysis conditions file can be created as an independent parameter file or as part of the sample information file.



Description

Displays a description of the current Analysis Conditions template. Enter a new description or add to the existing one if desired.

Be sure to use an intuitive name (perhaps one that characterizes the analysis conditions) so that it can be easily recognized when needed.

Replace

Click this push button to choose an existing Analysis Conditions file containing parameters you wish to copy and use in the current file. After the values have been copied into the file, you can edit them as desired. It does not affect the values of the file from which they were copied.

Absolute pressure dosing

Select this option to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature.

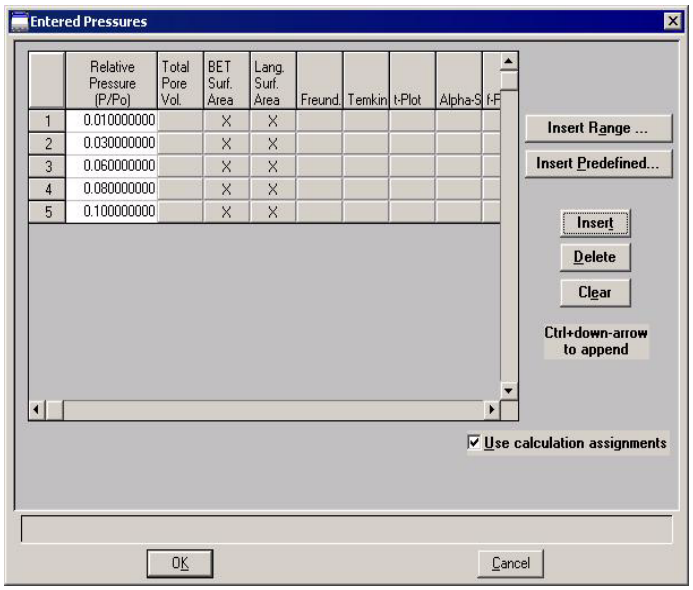
Isotherm Collection

The choices in this group box allow you to specify how you wish to have isotherms collected.

Target Pressures

Select this option to have isotherms collected by specifying the target pressures. When you select this option, **Pressures** and **Options** become enabled.

Pressures



Edit the existing pressure table or clear the table and enter a new pressure table.

A pressure table is a table of relative pressure points (and possibly calculation assignments) at which data (isotherms) are to be collected. The relative pressures may span the entire range of 0.00000001 to 1.0 P/Po. There must be one adsorption branch followed optionally by one desorption branch.

The pressure table for automatically collected data includes the pressure points for data collection and, when **Use calculation assignments** is selected, identifies calculations through which the data are processed. The system processes data for the following reports:

- Bet Surface Area
- Langmuir Surface Area
- t-Plot
- Alpha-S
- f-Ratio
- BJH Adsorption and Desorption
- DFT Pore Size and Surface Energy

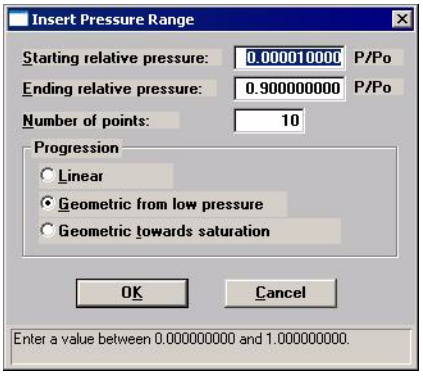
Pressures
(continued)

- Dubinin
- MP- Method
- Total Pore Volume

Several analysis conditions files containing complete pressure tables are included with the ASAP 2420 Confirm software.

Insert Range

Displays the Insert Pressure Range dialog box.



This dialog allows you to specify the starting pressure, the ending pressure, the number of points to insert within the specified range, and whether you wish to have linear or geometric progression.

Linear

Inserts evenly spaced points into your table.

Geometric from low pressure

Inserts geometrically spaced points from the low pressure range. For example, to insert 5 points with a 0.01 starting pressure and a 0.16 ending pressure, the following points are inserted into the table:

0.01
0.02
0.04
0.08
0.16

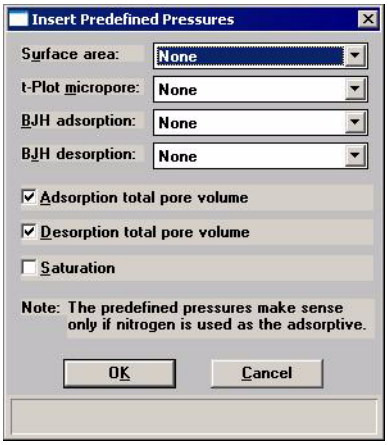
Geometric towards saturation

Inserts geometrically spaced points towards the saturation pressure. For example, to insert 5 points with a 0.84 starting pressure and a 0.99 ending pressure, the following points are inserted into the table:

0.84
0.92
0.96
0.98
0.99

Insert predefined

Displays the Insert Predefined Pressures dialog.



Choose predefined pressure points for surface area, t-Plot micropore, and/or BJH adsorption/desorption.

Click on the down arrow for each field to choose the desired set of points. You can also specify Adsorption/ Desorption total pore volume and Saturation.

Insert

Inserts a row into the table above the selected line.

Delete

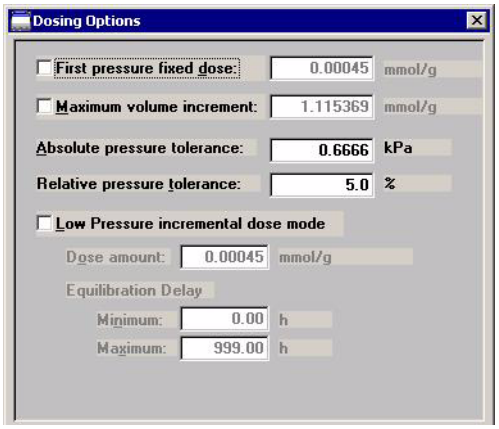
Deletes the selected row.

Clear

Removes all but one required entry from the table. The rows do not have to be selected. A warning message appears requesting confirmation before the table is cleared.

Options

Allows you to specify target pressure options; the Dosing Options dialog is displayed.



First Pressure Fixed Dose

The most frequent use of this option is for performing a standard nitrogen analysis of mesoporous materials such as catalysts. This option can shorten the time required to reach the first point in the pressure table if the first pressure table point is low and the expected gas uptake of the sample is high.

The sample is dosed repeatedly at low pressures with a specified amount of gas until the first pressure point is reached. This initial dosing quickly meets the adsorptive demand of the sample.

The first point on the pressure table is the threshold value, triggering the transition from the **Fixed Dose Mode** to Pressure Table Mode. When the first pressure table value is reached, Fixed Dose Mode is disabled, and points are equilibrated and recorded in accordance with the specified pressure table.

In the field adjacent to this option, enter the amount of gas that is to be added to the sample for each dose cycle.

Maximum volume increment

Select this option to determine when additional data points are collected between target pressures in regions of adsorption. When the maximum increment has been adsorbed since the last collected data point, another point is equilibrated and collected.

During desorption, this field is treated as a maximum volume "decrement" value.

Absolute and Relative pressure tolerance

The values entered 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 lower, and at higher pressures the absolute tolerance value is lower. The lesser of these two criteria is used to determine the tolerance.

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 lower 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.

Normally, surface area measurement points are widely spaced, and the resulting measurement is not very sensitive to the precise location of points so that wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis.

Low pressure incremental dose mode

Choose this option when you are performing an analysis of microporous materials. At low pressures on Type 1 isotherms, the pressure points are very closely spaced, making a useful pressure table difficult to define. Choosing this mode enables you to measure equilibrium points at approximately equal intervals on the quantity adsorbed axis. Each dose is fully equilibrated and recorded as a data point.

In this mode, the sample is successively dosed with a specified amount of gas until the first pressure point is reached. The first point is the threshold value, triggering the transition from Incremental Dose Mode to Pressure Table Mode. When the first pressure table value is reached, Incremental Dose Mode is disabled, and points are recorded in accordance with the specified pressure table. 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 field is disabled if **First Pressure Fixed Dose Mode** is selected.

Dose amount

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

Enabled when **Low pressure incremental dose mode** is selected.

Equilibration delay

Enabled when **Low pressure incremental dose mode** is selected.

Minimum: Prevents premature equilibration caused by reduced percentage sensitivity to pressure changes at the lowest pressures**.**

Maximum: Prevents the effects of long term temperature or pressure drift, which may cause the instrument to wait an excessive length of time for equilibration.

Dose Increments

In this form of dosing, there is no predefined pressure table. The sample is dosed repeatedly with a fixed amount of gas and isotherm points are collected after equilibrating each dose. Each entry in the table defines how much to dose for a segment of the isotherm.

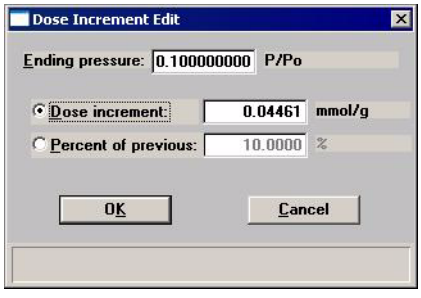
Options

Insert or edit target ranges for up to 10 dose increments.

The current specified dose increments are displayed.The ending relative pressure and the dose amount (entered or as the fraction of the previous volume adsorbed) for each target range are shown.

Insert
Edit

Displays the Dose Increment Edit dialog.



Insert
Edit

Ending Pressure:
Enter the desired ending pressure of this segment.

Dose increment
Select this option to have the sample dosed with a specified amount of gas for each isotherm point taken.

Percent of Previous
Select this option to have the sample dosed with a percentage of the volume adsorbed during the previous interval of the table. For example, if in the previous segment 5 cm^3 STP of gas was adsorbed and you enter 40% in this field, this segment will dose 2 cm^3 STP for each isotherm point taken.

This feature is disabled for the *first* entry of the dose increment table.

Delete

Deletes the selected range.

Clear

Clears the table of all entries except one; one entry is required.

Low pressure incremental dose mode

Enabled only if the MicroPore option is installed. Choose this option when you are performing an analysis of microporous materials. At low pressures on Type 1 isotherms, the pressure points are very closely spaced, making a useful pressure table difficult to define. Choosing this mode enables you to measure equilibrium points at approximately equal intervals on the quantity adsorbed axis. Each dose is fully equilibrated and recorded as a data point.

In this mode, the sample is successively dosed with a specified amount of gas until the first pressure point is reached. The first point is the threshold value, triggering the transition from Incremental Dose Mode to Pressure Table Mode. When the first pressure table value is reached, Incremental Dose Mode is disabled, and points are recorded in accordance with the specified pressure table. 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 field is disabled if **First Pressure Fixed Dose Mode** is selected.

Dose amount

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

Enabled when **Low pressure incremental dose mode** is selected.

Equilibration delay

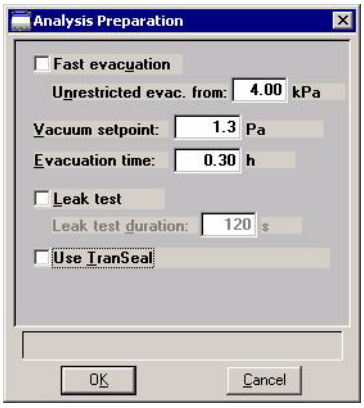
Enabled when **Low pressure incremental dose mode** is selected.

Minimum: Prevents premature equilibration caused by reduced percentage sensitivity to pressure changes at the lowest pressures**.**

Maximum: Prevents the effects of long term temperature or pressure drift, which may cause the instrument to wait an excessive length of time for equilibration.

Preparation

Displays the Analysis Preparation dialog box.



Fast evacuation

Select **Fast evacuation** when analyzing samples (such as pellets) that do not fluidize or shed particles under evacuation.

If you deselect **Fast evacuation, Evacuation rate** and **Unrestricted evac. from** become enabled. Enter the desired evacuation rate and the pressure value at which unrestricted sample evacuation should begin.

Vacuum setpoint

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

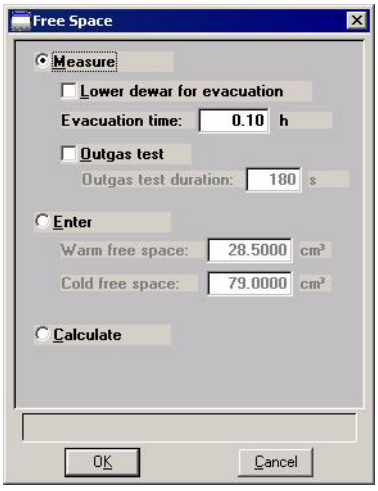
Evacuation time Enables you to enter the length of time for preliminary evacuation, which takes place prior to the free-space measurement.

Leak test Enables the system to 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 0.15 mmHg, the analysis does not proceed and you are notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists.

Leak test duration Enables you to specify how long the pressure is to be monitored.

Use TransSeal Select this option if you are using the TransSeal to transfer the sample from the preparation port to the analysis port under vacuum.

Free Space Allows you to specify free-space measurement conditions; the Free Space dialog is displayed.



Measure

Select this option to have free space measured automatically by the system.

Typically, it is best to measure the free space when analyzing macro- and mesoporous samples, and for routine surface area measurements.

Select **Lower dewar for evacuation** to lower the dewar following free-space measurements.

Evacuation time enables you to specify how long evacuation is to take place following the measured free space.

Select **Outgas test** to check for system leaks or sample outgassing. After free space is measured, the Dewar is lowered and the sample evacuated for 30 minutes. The leak test is performed after evacuation. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the analysis stops and you are notified. Leak testing slightly increases analysis time; however, it prevents the continuation of analysis and collection of erroneous data if a leak occurs.

Outgas test duration indicates the number of seconds the leak test is performed.

Enter

Select this option to enter values for the warm and free spaces. You may need to use this option if the sample retains helium and measurement of very low micron range pressures is required.

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

Warm free space is the sample tube gas capacity measured at room temperature.

Cold free space is the sample tube gas capacity measured with the Dewar raised.

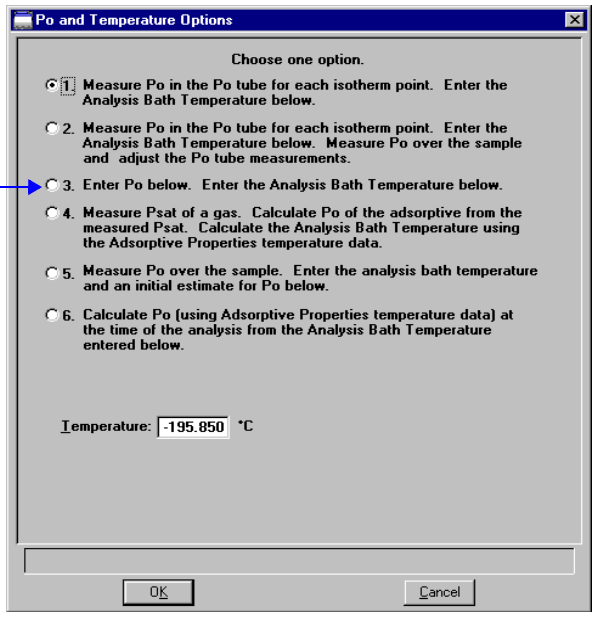
Calculate

Select this option to have the free-space measurement calculated. The free space is calculated using the mass, density, and sample tube parameters entered in the Sample Information and Sample Tube files. Calculated free space measurement takes less time than measured free space, so if you know the mass and density of the sample and the sample tube parameters, you may wish to use *measured* free space.

Po and T

Displays the Po and Temperature Options dialog box, allowing you to choose saturation pressure and analysis bath temperature.

Displays as Analysis Bath Temperature when Absolute dosing is selected (Analysis dialog)



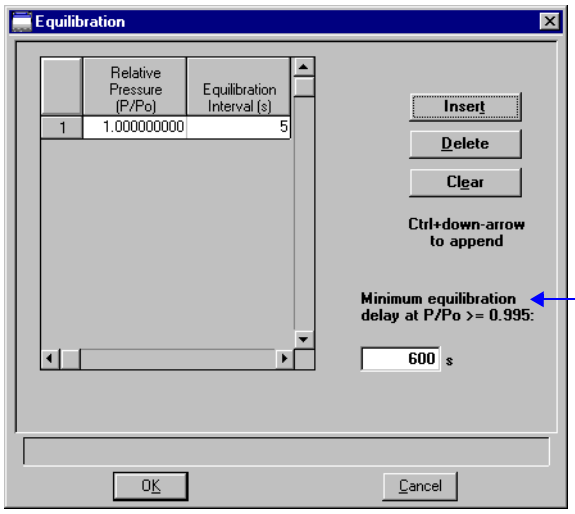
This dialog provides six options for obtaining the saturation pressure (Po) and analysis bath temperature. Clicking on each option prompts you for the entry of related parameters in the lower portion of the dialog box.

Option	Prompts for...
1	the analysis bath temperature
2	the analysis bath temperature

3	a Po; the analysis bath temperature is calculated at the time of analysis an analysis bath temperature only (when Absolute pressure dosing is selected on the Analysis dialog)
4	a Psat gas Click **Psat vs. T** to edit the values of the Psat vs T table if desired. Editing the values in the current table does not affect the ones of the original table.
5	a Po; the analysis bath temperature is calculated at the time of analysis
6	an analysis bath temperature

Equilibration

Displays the Equilibration dialog.



Relative Pressure (P/Po)

The relative pressure for which the equilibration interval will be applied.

Equilibration interval

The number of seconds between successive pressure readings during equilibration. Long equilibration intervals tend to lengthen analyses, however, they do improve data integrity. Short equilibration intervals produce a faster analysis but may reduce the accuracy of data.

Minimum equilibration delay at P/Po> = 0.995

The value entered in this field determines the minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995.

This field does not display if you select Absolute pressure dosing on the analysis conditions dialog.

Insert

Inserts a row into the table above the selected line.

Delete

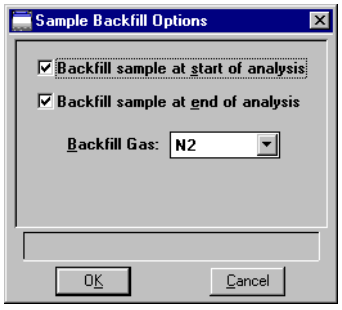
Deletes the selected row.

Clear

Removes all but one required entry from the table. The rows do not have to be selected. A warning message appears requesting confirmation before the table is cleared.

Backfill

Displays the Sample Backfill Options dialog, allowing you to select options for backfilling the sample tube at the start and end of an analysis.



The options on this dialog are not available if the file status displayed in the Open Sample Information File dialog is *Entered*, *Complete*, or *Analyzing*.

Backfill sample at start of analysis

Backfills the sample tube at the beginning of the analysis with the specified backfill gas.

For micropore analyses, it may be best not to select this option. Generally, it is best to manually evacuate the sample and start the analysis under vacuum. This minimizes any stray gas contamination.

Backfill sample at end of analysis

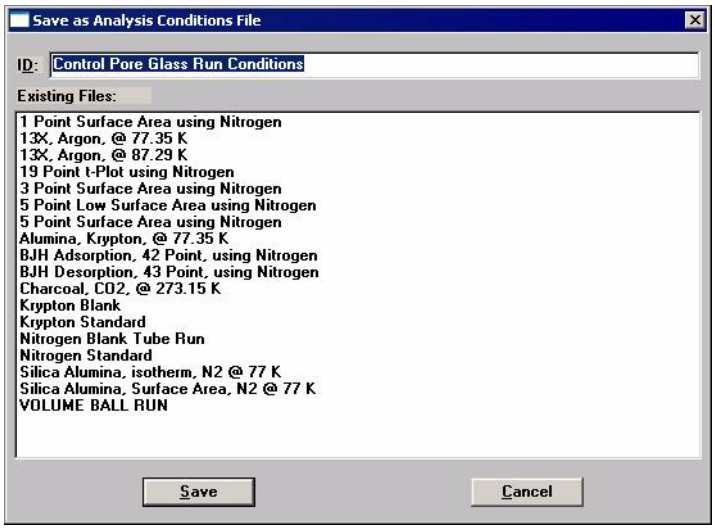
Backfills the sample tube at the end of the analysis with the specified backfill gas. If the sample mass is determined after the analysis, it is best to use nitrogen as the backfill gas.

Backfill Gas

Lists the available backfill gases. The gases in this list are the ones for which Adsorptive Properties files have been created; helium is also included.

Save

Displays the Save as Analysis Conditions File dialog with the description you entered on the previous screen in the *ID* field.



Save as Template

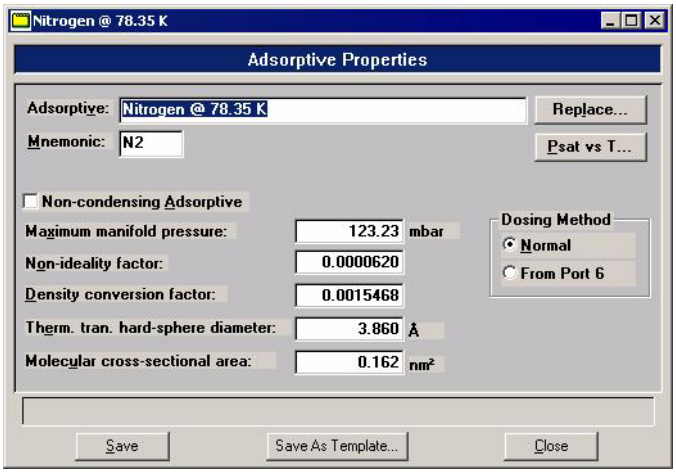
Enables you to save all parameters of the current file as an analysis conditions file template.

Close

Closes the dialog box. If the file contains unsaved changes, you are prompted to save before the dialog box closes.

Adsorptive Properties

This dialog allows you to specify the characteristics of the gases used in the ASAP 2420 system. An Adsorptive Properties file can be created as an independent parameter file or included as part of the sample information file.



Adsorptive — Displays a description of the current file. Enter a new description or edit the existing one.

Replace — Click this push button to choose a file containing parameters you wish to copy and use in the current file. After the values have been copied into the file, you can edit them as desired. It does not affect the values of the file from which they were copied.

Mnemonic — The mnemonic for the adsorptive gas.

Range: 3 alphanumeric

Non-condensing Adsorptive — Select this option if this file is for a non-condensing gas (adsorptive). When you select this option, The **Density conversion factor** field and the **Psat vs. T push** button become disabled.

Maximum manifold pressure — The maximum gas pressure allowed in the manifold.

Non-ideality factor

The factor compensating for the forces of attraction between molecules in a real gas.

Density conversion factor

This value is used to convert the gas volume at STP to liquid volume for the adsorptive. It is the ratio of gas density at STP to liquid density.

Therm. tran. hard-sphere diameter

The thermal transpiration hard-sphere diameter.

Molecular cross-sectional area

The molecular cross-sectional area.

Dosing Method

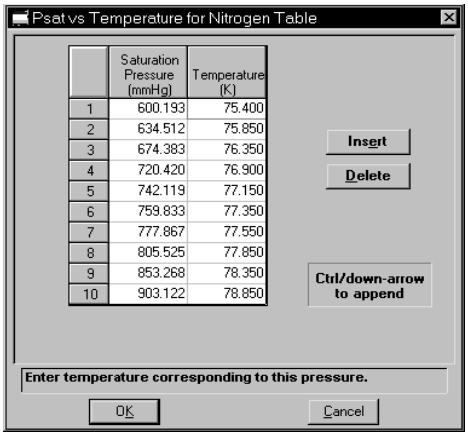
Select the manner in which the sample tube is dosed.

Select **Normal** for nitrogen and nitrogen-like gases.

Select **From Port 6** for krypton analyses.

Psat vs. T

Click to view or edit the Psat vs. Temperature table for the adsorptive gas; this example shows Nitrogen.



Psat vs. T
(continued)

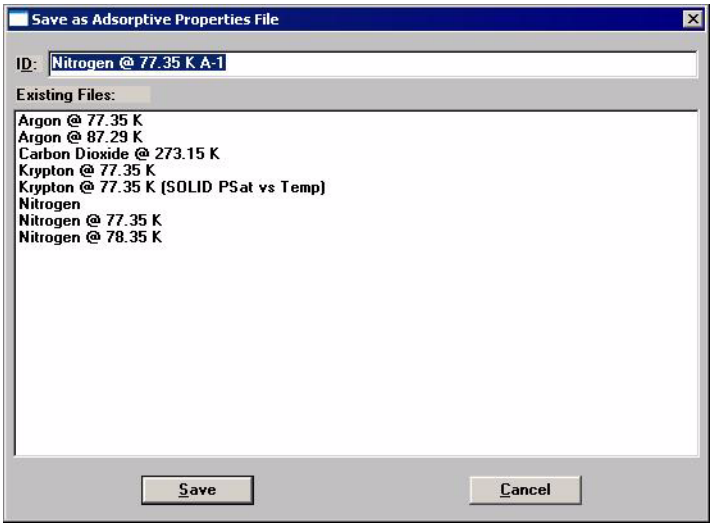
This table contains saturation pressures and their corresponding temperatures. As many as 10 entries may be included; at least two entries are required.

You can edit the table by clicking on the desired saturation pressure or temperature and entering a different value.

Use **Insert** to insert a new row above the selected line; the cursor moves to the new line. Use **Delete** to delete the selected row.

Save

Displays the Save as Adsorptive Properties File dialog.



Click **Save**.

Save as Template

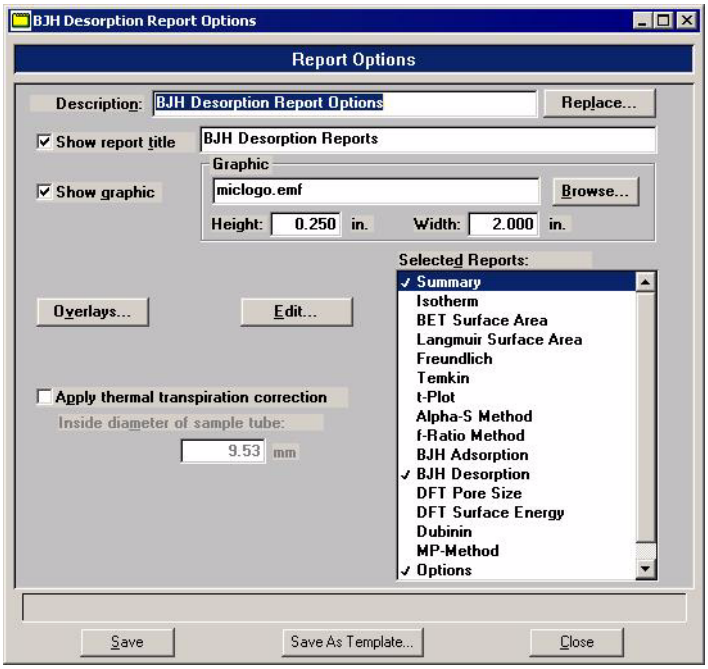
Enables you to save all parameters of the current file as an adsorptive properties file template.

Close

Closes the dialog. If the file contains unsaved changes, you are prompted to save before the dialog box closes.

Report Options

This dialog allows you to specify report options. A report options file can be created as an independent parameter file or included as part of the sample information file.



Description

Displays a description of the current file. Enter a new description or edit the existing one if desired. Be sure to use an intuitive name so that it can be easily recognized when needed.

Replace

Click this push button to choose a file containing parameters you wish to copy and use in the current file. After the values have been copied into the file, you can edit them as desired. It does not affect the values of the file from which they were copied.

Show report title

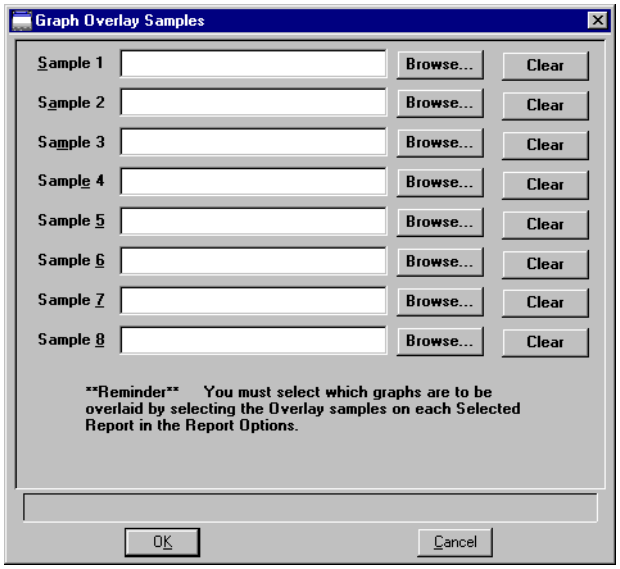
This option allows you to enter a title for your report. If this is a new file, the title you specified as the default is displayed. Accept the default title or enter a different one. Deselect this option to omit the report title.

Show graphic

Select this option to have a graphic display above the report title. The graphic can be in a bitmap (bmp) or an enhanced metafile (emf) format. For example, you may wish to display your company logo. Click **Browse** to choose the graphic, then use the height and width fields to specify the size. The image can also be edited from the report window.

Overlays

Displays the Graph Overlay Samples dialog so that you can choose the sample files containing the data you wish to overlay onto a selected plot.



Click **Browse** to the right of the sample number field to choose the desired files. Use **Clear** to clear a field of its entry. You can select up to eight files.

After choosing the desired files, be sure to edit the type of graph you are overlaying and choose the **Overlay samples** option. Refer to **"Generating Graph Overlays" on page 3-68** for step-by-step instructions on generating overlays.

Edit

Allows you to edit parameters of the selected report. This push button is disabled for the Options and Sample Log Reports.

Apply thermal transpiration correction

Select this option to correct for the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Never use filler rods in the sample tube when applying correction for thermal transpiration.

You should always use thermal transpiration when performing micropore analyses.

Inside diameter of sample tube

Enabled when you select Thermal transpiration correction, so that you may enter the inside diameter of the sample tube.

Selected Reports

Contains a list of available reports. Choose reports by double-clicking on the desired report. Alternatively, you can highlight the desired report and press the **Spacebar**. A report is selected when it is preceded with a check mark. You may *deselect* reports in the same manner.

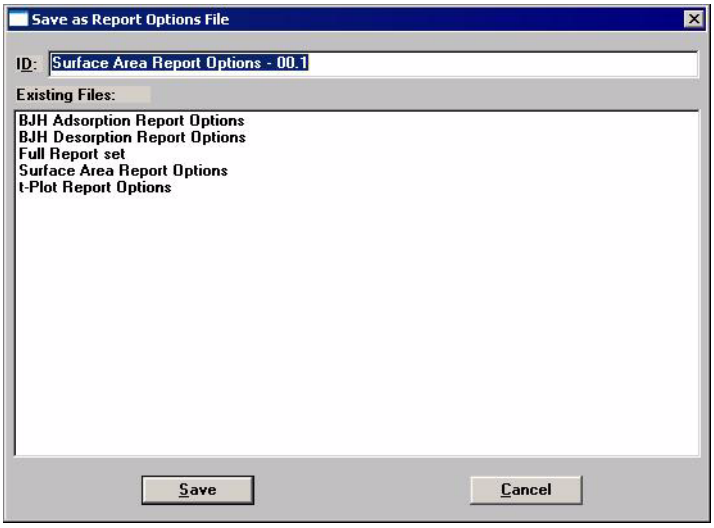
Isotherm
BET Surface Area
Langmuir Surface Area
Freundlich
Temkin
t-Plot
Alpha-S Method
f-Ratio Method
BJH Adsorption
BJH Desorption
Horvath-Kawazoe
DFT Pore Size
DFT Surface Energy
Dubinin
MP-Method
Options
Summary
Sample Log

Editing options for these reports are found in subsequent sections.

You can find printed examples of some reports in **"Report Examples" on page 6-23**.

Save

Displays the Save as Report Options dialog with the identifier you entered on the previous screen in the ID field.



Click **Save**.

Save as Template

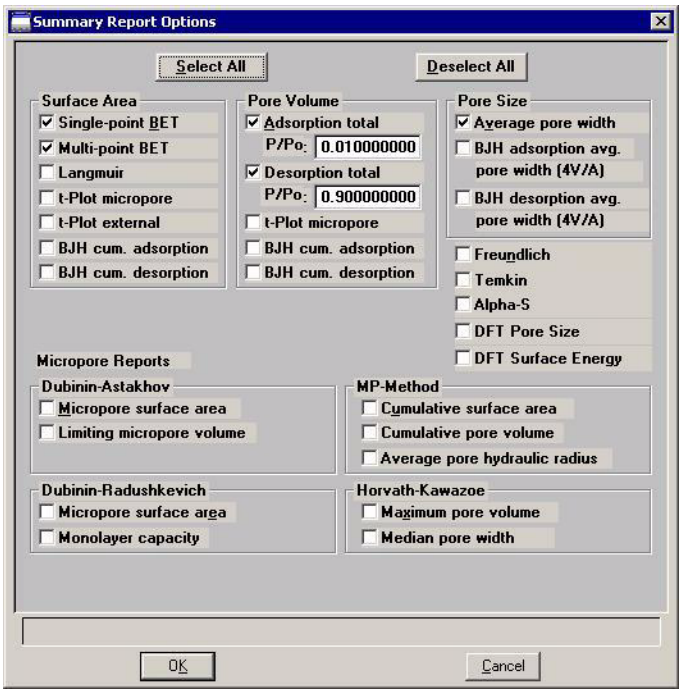
Enables you to save all parameters of the current file as an report options template.

Close

Closes the dialog. If the file contains unsaved changes, you are prompted to save before the dialog box closes.

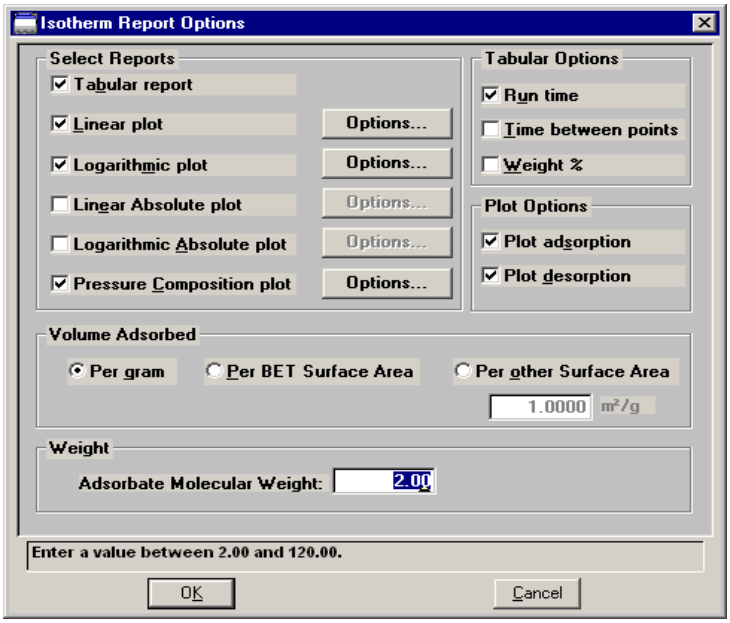
Summary Report

The Summary report provides a condensed listing of analysis statistics and data results. The Summary Report Options dialog allows you to choose the type of information to include in the report.



Isotherm Report Options

The isotherm report indicates adsorption (up to saturation pressure) and desorption (down from saturation pressure) of a gas by a solid held at constant temperature.



Select Reports

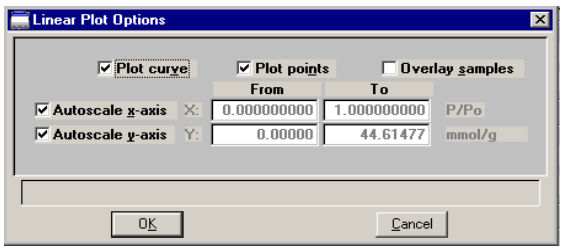
Lists the formats of reports offered.

Choices: *Tabular Report, Linear plot, Logarithmic plot, Linear Absolute plot, Logarithmic Absolute plot, Pressure Composition plot*

When you select *Pressure Composition plot*, the **Adsorbate Molecular Weigh**t field is enabled so that you can enter a value for the adsorbate. This plot is useful for plotting pressure as a function of Weight % adsorbed; for example, H_2 adsorbed on carbon.

Options

Displays the related Plot Options dialog; this example shows the dialog for the Linear plot.



All plot dialogs contain the same options. You can plot the graphs as a curve, points, or both.

Options
(continued)

Select **Overlay samples** to overlay data from the current plot with data from other samples. The other sample files are selected by clicking **Overlays** on the Report Options dialog.

Autoscale options enable you 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.

If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to specify a range. Data collected outside the specified range 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 fields show the relative pressure.The Y-axis fields show the quantity of gas adsorbed.

Tabular Options

Enables you to have **Run Time** and/or **Time Between Points** reported. Run time reports the time elapsed from the beginning of the analysis to the finish. Time between points reports the time elapsed between each point.

Also enables you to report **Weight %**.

Plot Options

Enables you to choose the type of isotherm you wish to plot. You can plot the adsorption and/or the desorption isotherm.

Volume Adsorbed

Enables you to choose the manner in which the volume adsorbed is reported. These data are reported by default as **Per Gram** (cm^3/g). However, you can choose to report data **Per BET Surface Area** (cm^3/m^2) or **Per Other Surface Area** (m^2/g). If the latter field is selected, a field is enabled allowing you to enter a value.

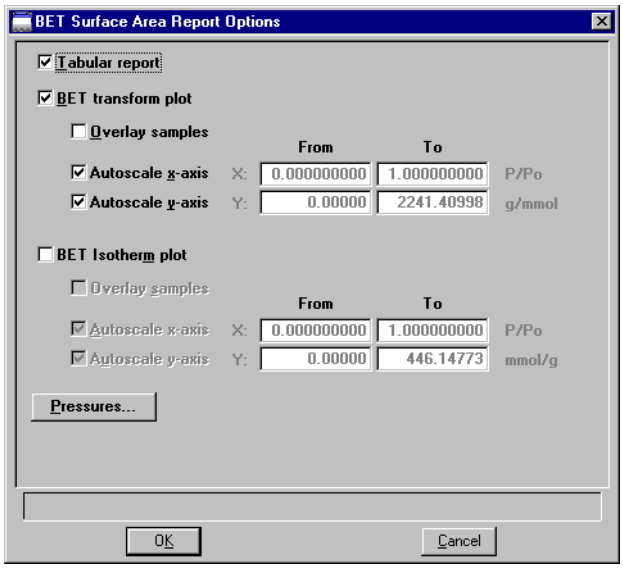
Absolute Molecular Weight

Enables you to enter the molecular weight of the adsorbate.

This field is enabled when you choose *Pressure Composition plot* under Select Reports or when you choose *Weight* under Tabular Options.

BET/Langmuir Surface Area Report Options

- The BET calculation obtains the sample surface area value by determining the monolayer volume of adsorbed gas from the isotherm data.
- The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer.



The Langmuir and BET Surface Area dialogs include the same fields; the operating instructions for both are the same

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

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

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

Overlay samples — Allows you to overlay data of the selected type from the current plot with data from other samples. The other samples are selected by clicking **Overlays** on the main Report Options screen.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axes scaled automatically.

Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points. If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

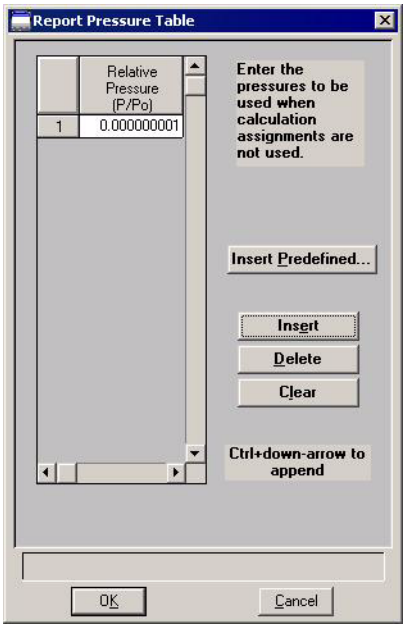
The values entered in the **To** field must be greater than the value entered in the **From** field.

The X-Axis Range fields show the relative pressure.

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

Pressures

Displays the Report Pressure Table dialog so that you may edit or enter relative pressure points.



Pressures
(continued)

- If **Use calculation assignments** is *not* selected on the collected/Entered dialog, the isotherm is interpolated to these pressure points, and those interpolated values are used in the BET calculations.
- If **Use calculation assignments** *is* selected, collected data are used.

Insert predefined

Displays the Surface Area Report Pressure Selection dialog, allowing you to select predefined points.

Choices: None, 1 Point, 3 Point, 5 Point, 5 Point Low Pressure

Insert

Inserts a row into the table above the selected line.

Delete

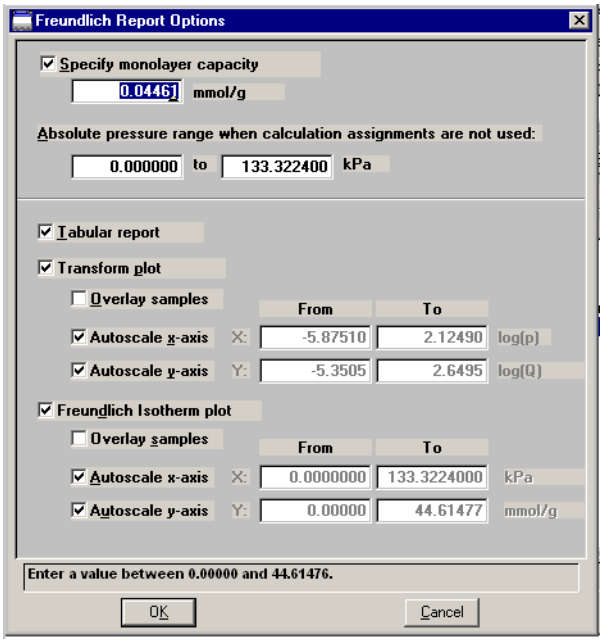
Deletes the selected row.

Clear

Removes all but one entry from the table; one entry is required. The rows do not have to be selected.

A warning message requesting confirmation is displayed before the table is cleared.

Freundlich Isotherm



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

Absolute pressure range — Allows you to enter a pressure range when calculation assignments are not requested.

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

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

Freundlich Isotherm plot — Overlays the Freundlich isotherm with the analysis data.

Overlay samples — Choose this option to overlay Freundlich isotherm data from the current file with the same type of data from other samples (files). The desired files are chosen by clicking **Overlays** on the Report Options dialog.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axis scaled automatically.

Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points.

If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

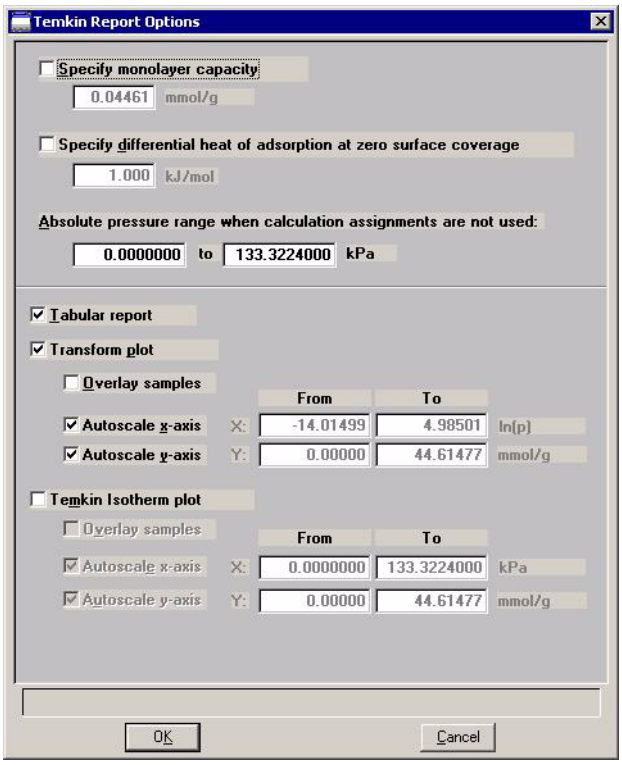
The X-axis shows the log of absolute pressure and the Y-axis shows the quantity of gas adsorbed.

From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

Temkin Isotherm

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 of Temkin constants.

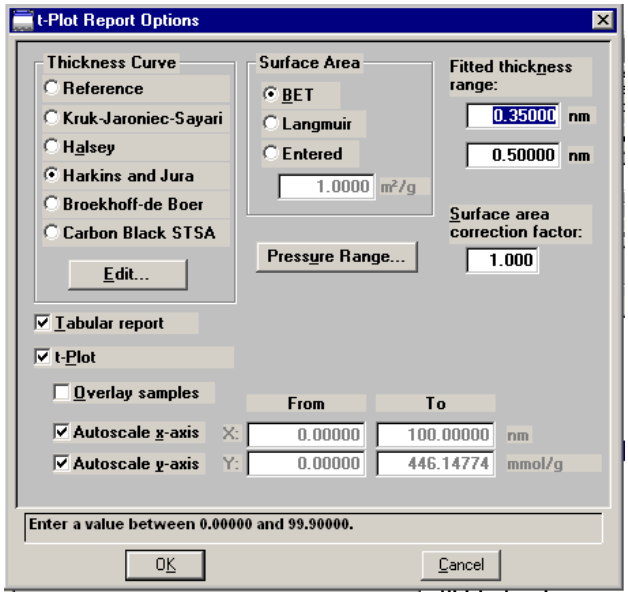
Absolute pressure range — Allows you to enter a pressure range when calculation assignments are not requested.

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

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 Freundlich isotherm data from the current file with the same type of data from other samples (files). The desired files are chosen by selecting **Overlays** on the Report Options dialog.
Autoscale x-axis **Autoscale y-axis**	Select these options to have the X- and/or Y-axis scaled automatically. Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points. The X-axis shows the natural log of absolute pressure and the Y-axis shows the quantity of gas adsorbed. If you choose not to autoscale data, the From and To fields are enabled, allowing you to enter the ranges.
From/To fields	Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

t-Plot Report Options

The t-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area, the area external to micropores, is directly determined and often proves to be a valuable way of characterizing complex mixed materials.



Thickness Curve

Presents the type of thickness curves available.

- Reference
- Kruk-Jaroniec-Sayari
- Halsey
- Harkins and Jura
- Broekhoff-de Boer
- Carbon Black STSA (ASTM D-6556-0lA)

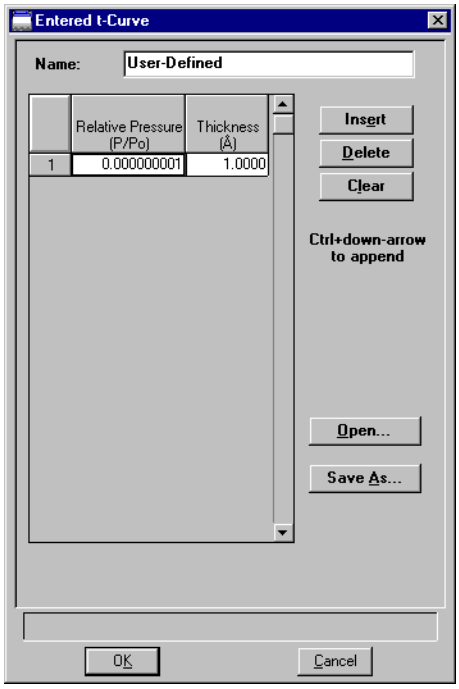
You can also apply the Frenkel-Halsey-Hill thickness curve using the Halsey option by entering the appropriate values in the equation. Use **Edit** to edit the values in the equation.

Edit

Displays the equation for the type of thickness curve selected so that you may view or edit the values.

Reference

Displays the Entered t-Curve dialog allowing you to define a t-curve by entering the relative pressure and thickness values.



This table can also be created in another application if desired and imported into this dialog using **Open**. When creating the table in another application, the file must be saved as ASCII text with a. **THK** extension. Use a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space (or a tab).

Insert
Inserts a row above the selected row. A row cannot be inserted if the value in the selected row is at its lowest value; values must be strictly increasing. For example, in the table shown above, the default value is 0.000000001. Therefore you must use **Ctrl + Down Arrow** to insert rows.

Delete
Deletes the selected row.

Clear
Clears the table of all but one entry, one entry is required.

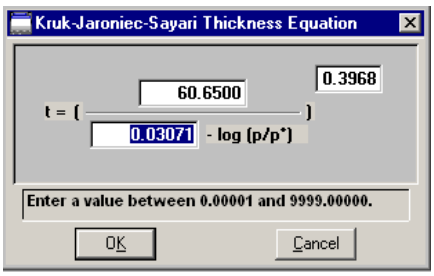
Reference
(continued)

Open
Enables you to import the values from an existing t-curve.

Save As
Enables you to save the current table of values under a file name.

Kruk-Jaroniec-Sayari

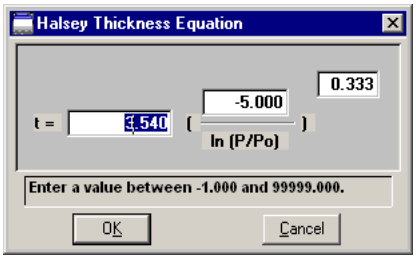
Displays the Kruk-Jaroniec-Sayari Thickness Equation dialog.



You can edit the values for the numerator, the first element of the denominator, and the exponent.The range for a selected field is shown in the information bar at the bottom of the dialog.

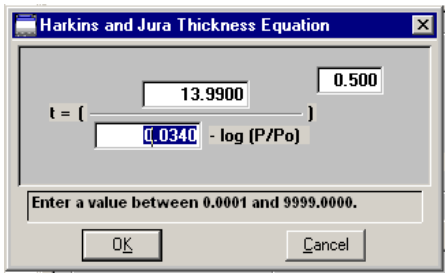
Halsey

Displays the Halsey Thickness Equation dialog.



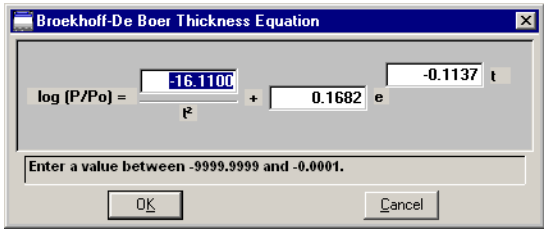
You can edit the values for the multiplier, numerator, and exponent.The range for a selected field is shown in the information bar at the bottom of the dialog.

Harkins and Jura Displays the Harkins and Jura Thickness Equation dialog.



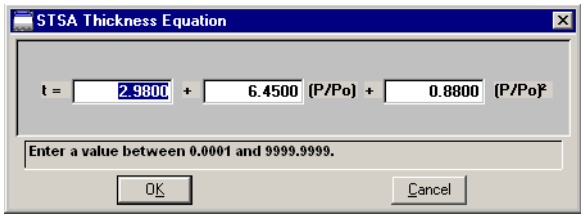
You can edit the values for the numerator, first element of the denominator, and exponent. The range for a selected field is shown in the information bar at the bottom of the dialog.

Broekhoff-de Boer Displays the Broekhoff-de Boer Thickness equation dialog.



You can edit the values for the multiplier, numerator, and exponent. The range for a selected field is shown in the information bar at the bottom of the dialog.

Carbon Black STSA Displays the STSA Thickness Equation dialog.

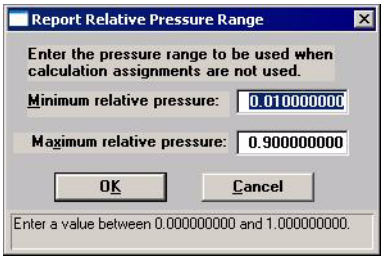


All coefficients can be edited. The range for a selected field is shown in the information bar at the bottom of the dialog.

Surface Area group box Allows you to choose the surface area value used for thickness calculations. You can use BET (most commonly used), Langmuir, or enter a surface area value.

Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.



- If **Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.
- If **Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Fitted Thickness Range

Provides two fields, allowing you to enter the minimum and maximum thicknesses you wish to include in the thickness curve.

Surface area correction factor

This value corrects for surface areas that are not smooth and brings the values for BET surface area and micropore surface area into accordance. For most samples, the default value of 1.000 is adequate

Tabular report

Select this option to generate a table of collected data.

t-Plot

Select this option to generate a graphical representation of the collected data.

Overlay samples

Allows you to overlay data from the current sample file with data from other sample files. The other sample files are chosen by clicking **Overlays** on the main Report Options screen.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axes scaled automatically.

Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points.

If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

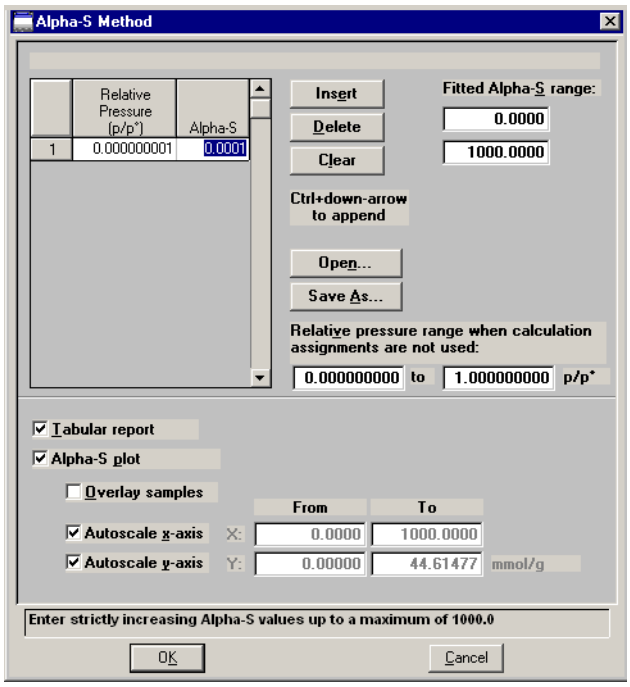
From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the relative pressure.

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

Alpha-S Plot



The Alpha-S plot converts the standard adsorption isotherm into a dimensionless isotherm using the quantity adsorbed at a relative pressure of 0.4.

Table

Provides two columns for entering the relative pressure (first column) and the alpha-s values (second column). This table can also be created in another application if desired and imported into this dialog using **Open**. When creating the table in another application, the file must be saved as ASCII text with an ALS extension. Use a two-column format with the relative pressures in the first column and the alpha-s values in the second column. Columns must be separated by a space (or a tab).

Insert

Inserts a row above the selected row. A row cannot be inserted if the value in the selected row is at its lowest value; values must be strictly increasing. For example, in the table shown above, the default value is 0.000000001. Therefore you must use **Ctrl + Down Arrow** to insert rows.

Delete

Deletes the selected row.

Clear	Clears the table of all but one entry, one entry is required.
Open	Allows you to import the values from an existing alpha-s curve (ALS) into the table. One predefined curve is shipped with the analysis program. After the values are copied into the table, you may edit them if desired. Editing these values will not affect the file from which they were copied.
Save As	Enables you to save the current table of values under a file name.
Fitted alpha-S Range	Provides two fields for entering the minimum and maximum relative pressures from which the fit will be determined.
Relative pressure range	Provides two fields for entering a pressure range when calculation assignments are not used.
Tabular report	Select this option to generate collected data in a tabular manner.
Alpha-S plot	Select this option to generate collected data graphically.
Overlay samples	Allows you to overlay data from the current sample file with data from other sample file(s). The other sample file(s) are chosen by clicking **Overlays** on the main Report Options screen.
Autoscale x-axis **Autoscale y-axis**	Select these options to have the X- and/or Y-axes scaled automatically. Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points. If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.

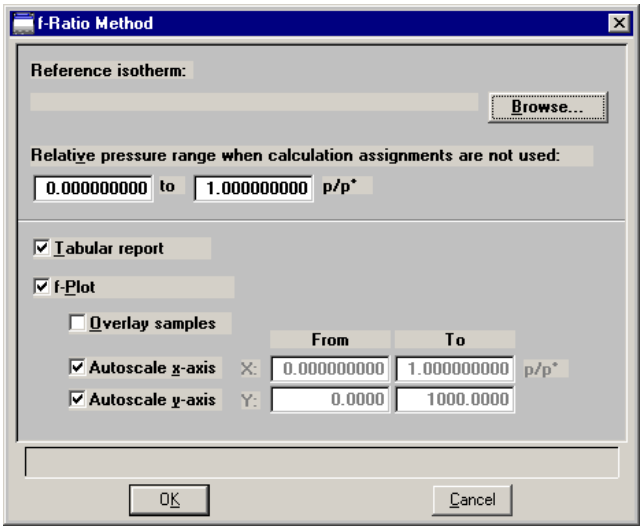
From/To fields

Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

The X-Axis Range fields show the relative pressure.

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

f-Ratio Plot



The f-Ratio report allows you to take the measured isotherm and normalize it using a reference isotherm.

Reference Isotherm

Displays the sample file you choose as a reference. You should always choose a file containing an isotherm measured from a non-porous sample of the same material as the current sample.

Browse

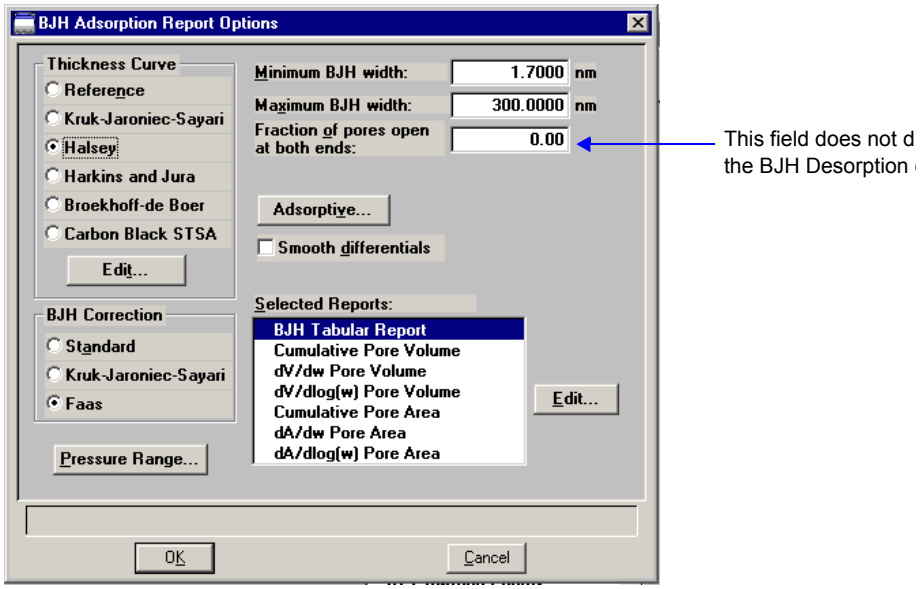
Click to choose the sample file you wish to use as a reference for the isotherm.

Relative pressure range

Allows you to enter a pressure range when calculations assignments are not used.

Tabular report	Choose this option to have a tabular report generated The tabular report contains four columns: relative pressure, quantity adsorbed for the isotherm of interest, quantity adsorbed for the reference isotherm, and the ratio of the quantities adsorbed.
f-Plot	Choose this option to have the data plotted as a graph.
Overlay samples	Allows you to overlay data from the current sample file with data from other sample files. The other sample files are chosen by clicking **Overlays** on the main Report Options screen.
Autoscale x-axis **Autoscale y-axis**	Select these options to have the X- and/or Y-axes scaled automatically. Both X- and Y-axes begin at zero; the system uses the highest values collected during analysis as the ending points. If you choose not to autoscale data, the **From** and **To** fields are enabled, allowing you to enter the ranges.
From/To fields	Enabled when you choose not to autoscale data (deselect the Autoscale option), allowing you to specify the beginning and ending ranges of the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

BJH Adsorption/Desorption Report Options



The BJH Adsorption and Desorption dialogs include the same fields; the operating instructions for both are the same.

The BJH calculation determines the mesopore volume/area distribution which accounts for both the change in adsorbate layer thickness and the liquid condensed in pore cores. You can generate BJH reports from adsorption and desorption data

Thickness Curve Type

Presents the type of thickness curves available.

- Reference
- Kruk-Jaroniec-Sayari
- Halsey
- Harkins and Jura
- Broekhoff-de Boer
- Carbon Black STSA (ASTM D-6556-0lA)

You can also apply the Frenkel-Halsey-Hill thickness curve using the Halsey option by entering the appropriate values in the equation. Use **Edit** to edit the values in the equation. Halsey is the default thickness curve and typically is used for BJH calculations.

If you select a thickness curve which is not a good match for the sample being analyzed, an incomplete pore distribution may be generated.

Edit

Displays the equation for the type of thickness curve selected.

Refer to t-Plot Report Options beginning on page **4-52** for information on the equations for thickness curves.

Minimum/Maximum BJH diameter

Provides fields for entering the minimum and maximum size pore to be included in BJH reports. The size can be reported in width (default), radius, or diameter. Select **Options** > **Units** to specify desired unit.

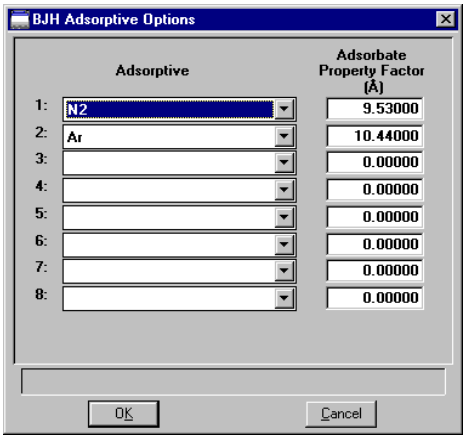
Fraction of pores open at both ends
(not displayed on desorption dialog)

During calculations, the software assumes that all pores are closed at one end. Sometimes a percentage of pores may be open at both ends, causing disagreement in the adsorption and desorption data or in the values for total volume and total BJH pore volume. Enter a value in this field to compensate for this error.

Due to normal hysteresis, data may disagree even after applying this corrective factor.

Adsorptive

Displays the BJH Adsorptive Options dialog, allowing you to specify up to eight adsorptive/adsorbate property factor combinations.



Adsorptive
(continued)

The recommended adsorptives and their values are shown. You may specify up to six additional adsorptive/adsorbate property factor combinations.

The adsorbate property factor is the combination of constants in the numerator of the BJH equation which is specific to the type of gas used. Adsorbate property factors may be specified in angstroms or nanometers. Select **Options** > **Units** to specify desired units.

Smooth differentials

Smooths all of the differential calculations, eliminating variations in the differential computation caused by noise in the input data.

BJH Correction

Enables you to choose the type of correction to apply to calculations. The type you choose will be displayed in the report header.

Standard

Uses original BJH models.

Kruk-Jaroniec-Sayari

Good for reference thickness curves.

Faas

Good for statistical thickness curves.

Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- **If Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.

- **If Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Selected Reports

Lists the available BJH reports.

- Tabular Report
- Cumulative Pore Volume
- dV/d* Pore Volume
- dV/dlog(*) Pore Volume
- Cumulative Pore Area
- dA/d* Pore Area
- dA/dlog(*) Pore Area

* = width, radius, or diameter

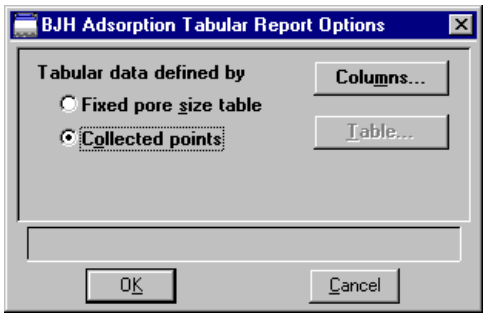
Choose a report by double-clicking on the report name or click the report name and press **Spacebar**. A report is selected when it is preceded with a check mark.

Edit

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

Tabular Report

The BJH Adsorption/Desorption tabular report allows you to specify the method of data reduction.



You can measure pore width, diameter, or radius; pore size can be expressed in angstroms or nanometers. Select Options > Units to specify desired units.

Fixed pore size table

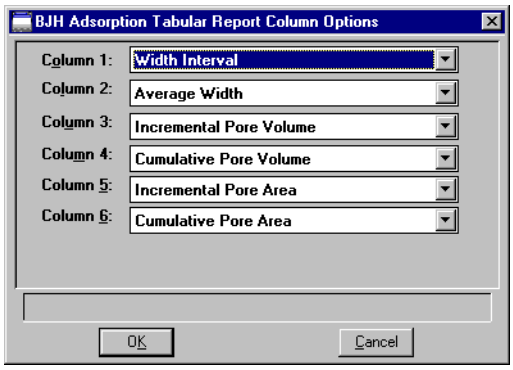
Select this option to specify exact pore sizes for which volume or area data are reported. Only the pore sizes within the specified range appear on the report. Click **Table** to enter (or edit) a fixed pore size table.

Collected points

Select this option to include the values collected by the system in the report.

Columns

Displays the BJH Adsorption (or Desorption) Tabular Report Column Options dialog, allowing you to select up to six columns of data for the tabular report.



The default column title appears next to the column number. Each column includes a pull-down list of the data types to include in the report; the pore width, radius, or diameter can be measured (select **Options > Units** to specify desired units.).

Table

Enabled when **Fixed pore size table** is selected for tabular data. Displays the BJH Adsorption (or Desorption) Fixed Pore Size Table dialog, allowing you to enter or edit a fixed pore size table

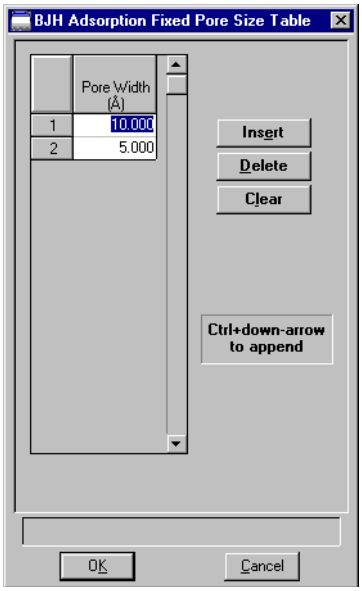


Table
(continued)

The table must contain a minimum of two points and may include as many as one thousand. Points must be strictly decreasing.

Insert
Inserts a row into the pressure table. The row is inserted above the selected row and the cursor moves to the new row.

Delete
Deletes the selected row from the pressure table.

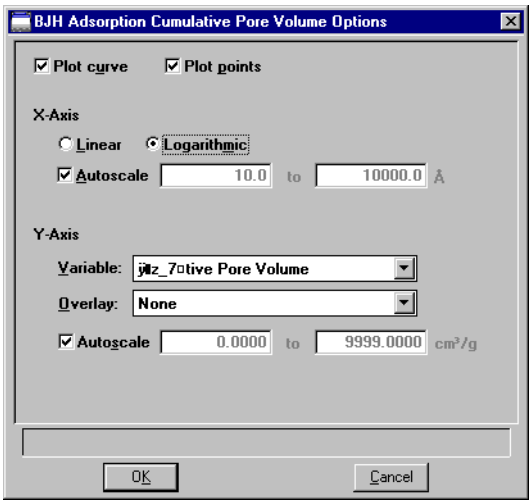
Clear
Removes all entries (except the two required ones) from the table. A warning message requests confirmation before the table is cleared.

Plot Options

Selecting a BJH plot option from the BJH Adsorption or Desorption Report Options dialog enables you to specify the plotting method used for your report and to customize the plot.

You can measure pore width, diameter, or radius; pore size can be expressed in angstroms or nanometers. Select Options > Units to specify desired units.

When you edit a BJH Adsorption or Desorption plot, a dialog similar to the following is displayed.



Plot Options Allows you to have data plotted as a curve, points, or both.

X-Axis Lists the scale options for the x-axis

Linear/Logarithmic Choose whether you wish to have the X-axis on a logarithmic or linear scale. Unless you are performing micropore analyses, use the logarithmic option.

Autoscale Choose this option to have the X-axis scaled automatically. Linear X-axes begin at zero, and logarithmic X-axes begin at an appropriate value. The system uses the highest values collected during analysis as the ending points.

If you deselect this option, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside the specified ranges are not included in the plot.

X-axis range fields show pore radius or diameter in angstroms or nanometers.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-axis Lists the options for the Y-axis.

Variable Click on the down-arrow to choose a variable for the Y-axis.

Overlay Displays a list of overlay choices. You can choose a different type of plot to overlay with the current plot. Or, you can choose **Samples** to overlay the current type of plot with the same type from other sample files. If you choose **Samples**, you select the other files using **Overlays** on the Report Options dialog.

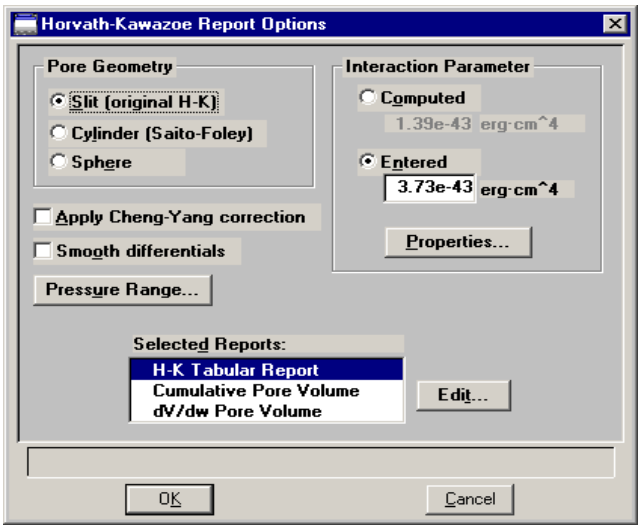
Autoscale

Choose this option to have the Y-axis scaled automatically. Y-axes begin at zero. The system uses the highest values collected during analysis as the ending points.

If you deselect this option, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside the specified ranges are not included in the plot.

Y-axis range fields show the quantity of gas adsorbed. Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Horvath-Kawazoe Report Options



Pore Geometry

Select the option in this group box which best represents the physical geometry of the micropores in the sample material. Refer to **Appendix C** for additional information on calculations used for these parameters.

Apply Cheng/Yang correction

Allows you to apply the Cheng/Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation (see Appendix C).

Smooth differentials

Smooths the differential calculations, eliminating variations in the differential computation caused by noise in the input data.

Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

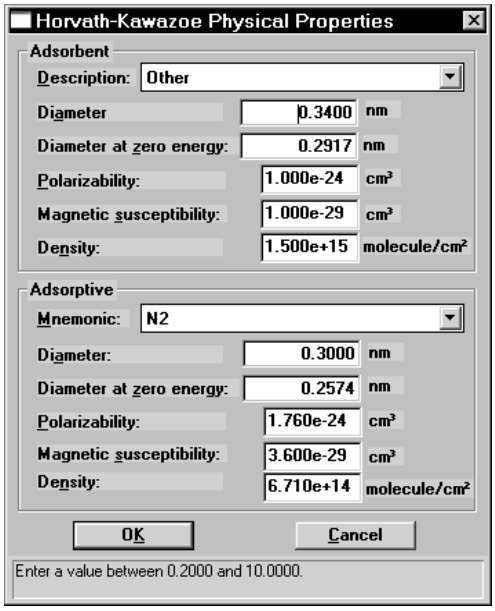
- If **Use calculation assignments** is not selected on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.
- If **Use calculation assignments** is selected, collected data points which are assigned to this report type are used.

Interaction Parameter Select one of the options in this group box to determine which interaction parameter is used during report generation.

Computed The interaction parameter is calculated using the parameters on the Horvath-Kawazoe Physical Properties dialog. You can click **Properties** to view or edit these parameters. Each time you change one of the parameters, the interaction parameter is recalculated. The formula for computing the interaction parameter is given in Appendix C.

Entered The value you enter in the adjacent field will be used.

Properties Use this push button to view or edit the constants (used during report generation) describing the physical properties of the adsorbent and adsorptive; the Horvath-Kawazoe Physical Properties dialog box is displayed.



Adsorbent Group Box

Contains the parameters for the sample. If you select **Computed** for the interaction parameter, all fields are enabled and can be edited if desired. If you select **Entered**, only the values in the **Diameter** and **Diameter at zero energy** fields may be edited.

Description
The name of the adsorbent used in the analysis.

Click on the down arrow to make a new selection. Each time you change the selection, the values for that adsorbent are displayed in the appropriate fields.

Diameter
The diameter of the sample atom.

Diameter at zero energy
The diameter of an atom at zero interaction energy, $(2/5)^{1/6}$ x diameter.

Polarizability
The polarizability of the adsorbent.

Magnetic susceptibility
The magnetic susceptibility of the adsorbent.

Density
The density per unit area of the adsorbent.

Adsorptive Group Box

Contains the parameters for the adsorptives (provided with the software and/or user-defined). If you select **Computed** for the interaction parameter, all fields are enabled and can be edited if desired. If you select **Entered**, only the values in the **Diameter** and **Diameter at zero energy** fields may be edited.

Mnemonic
The mnemonic name of the adsorptive gas in use.

Click on the down arrow to make a new selection. Each time you change the selection, the values for that adsorptive display in the appropriate fields. If no parameters have been defined, the default values are displayed.

Diameter
The diameter of the adsorptive atom.

Adsorptive Group Box *(continued)*

Diameter at zero energy
The diameter of an atom at zero interaction energy, $(2/5)^{1/6}$ x diameter.

Polarizability
The polarizability of the adsorptive.

Magnetic susceptibility
The magnetic susceptibility of the adsorptive.

Density
The density per unit area of the adsorptive.

Selected Reports

Lists the available Horvath-Kawazoe 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 mark.
The following reports are available:

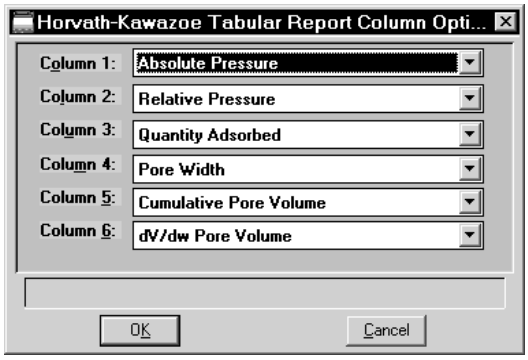
- Tabular Report
- Cumulative Pore Volume
- dV/dw Pore Volume

Edit

Allows you to edit the selected report.

Tabular Report Options

Displays the Horvath-Kawazoe Tabular Report Column Options dialog so that you can select the type of data to display in each column.

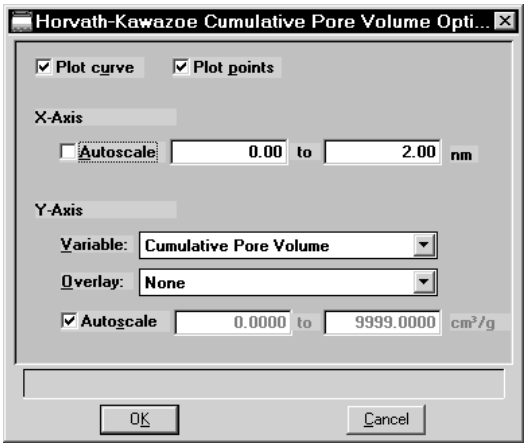


The default column title displays next to the column number. Each column includes a drop-down list of the types of data to include in the report.

Plot Options

Selecting a Horvath-Kawazoe plot option from the Horvath-Kawazoe Report Options dialog enables you to specify the plotting method used for your report and to customize the plot. Plots for Cumulative Pore Volume and dV/dw Pore Volume are available.

When you select either of these plots from the Horvath-Kawazoe Report Options dialog, a dialog like the following is displayed.



Plot Options — Lists the data presentation styles available; you must choose to plot a curve, points, or both.

X-axis options — Choose **Autoscale** to have the x-axis scaled automatically. The X-axis begins at zero and the system uses the highest value collected during analysis as the ending point.

If you deselect **Autoscale**, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

X-axis range fields show pore radius or diameter in angstroms or nanometers.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-axis options

Click on the down-arrow at the Variable field to choose a variable for the Y-axis.

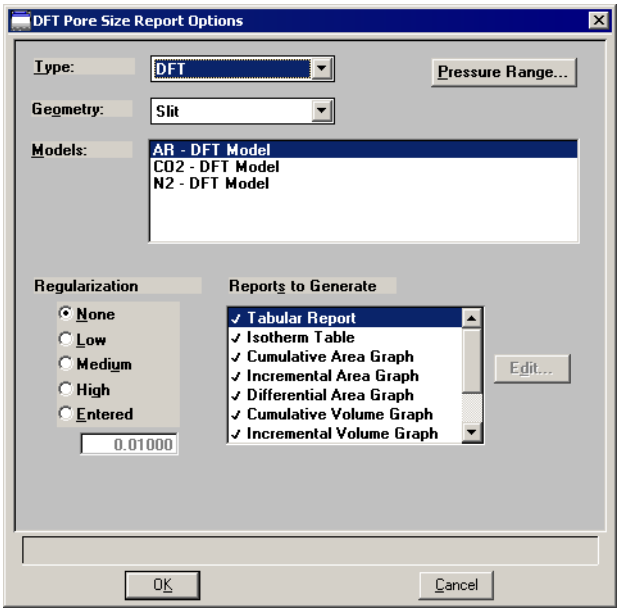
Click on the down-arrow at the **Overlay** field to overlay the plot with data from other samples.

Choose **Autoscale** to have the y-axis scaled automatically. Y-axes begin at zero. The system uses the highest value collected during analysis as the ending point.

If you deselect **Autoscale**, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

Y-axis range fields show the quantity of gas adsorbed. Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

DFT Pore Size



Type

Drop-down list containing the types of models available: DFT or Classical.

DFT models are based on the density functional theory. Classical models are based on the Kelvin equation and thickness for determining the pore size distribution. Refer to **Appendix F** for a discussion on models.

Geometry

Drop-down list containing pore shapes available; *Slit* or *Cylinder*.

Models

Lists the models that meet the criteria specified and which match the adsorbate and temperature of the sample data. The models display in alphabetical order with the first one automatically selected; you may select any one desired. If the list is empty, there were no models that meet the selected criteria.

Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- If **Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.
- If **Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Regularization

These choices enable you to choose the extent of smoothing you wish to have applied to your data, or your can choose **None**.

If you choose **Entered**, its field is enabled allowing you to enter a number giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.

Reports to Generate

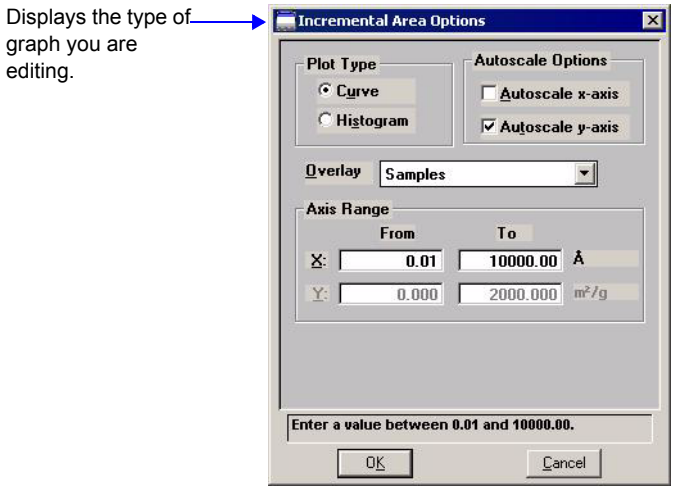
Displays the types of reports available.

- Tabular report
- Isotherm table
- Cumulative area graph
- Incremental area graph
- Differential area graph
- Cumulative volume graph
- Incremental volume graph
- Differential volume graph
- Log goodness of fit graph
- Goodness of fit graph

Graph details can be edited by selecting the **Edit** push button.

Edit

Use this push button to edit details of a selected graph; a dialog similar to the following is displayed.



This push button is disabled for tables and Goodness of Fit graphs.

Plot Type

Enables you to choose the manner in which you wish to display plotted data.

Autoscale Options

Select these options to have the axes scaled automatically. If you deselect one or both of these options, you must enter a range in the related **Axis Range** fields which become enabled automatically.

Overlay

This drop-down list contains the type of overlays available for the current graph.

Choose **Samples** to overlay the same type of graph as the current one from other data reductions. Then select **Overlays** on the Report Options dialog to choose your files.

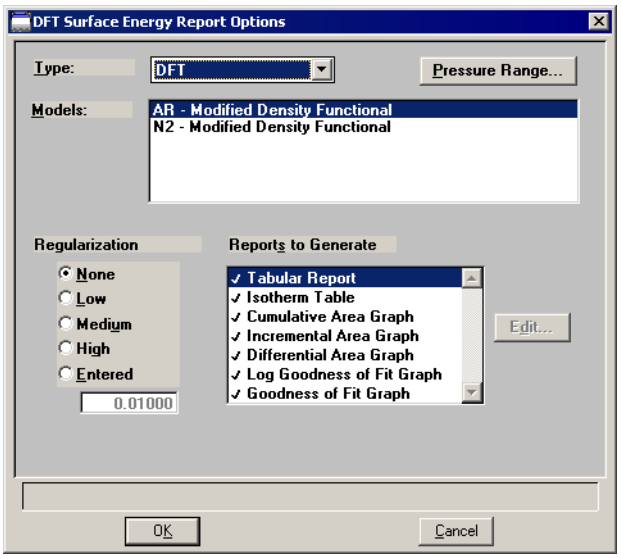
Axis Range

The fields in this group box become enabled when you deselect Autoscale, allowing you to specify beginning and ending values for the X- and/or Y-axis. Data collected outside these ranges are not included in the plot.

Valid ranges for a selected field are displayed in the information bar across the lower portion of the dialog.

The X-axis displays the energy and the Y-axis displays the area.

DFT Surface Energy



The fields on the Surface Energy dialog are identical to those on the DFT Pore Size dialog with the following exceptions:

Geometry drop-down list — Is not applicable to the Surface Energy report and, therefore, does not display on this dialog.

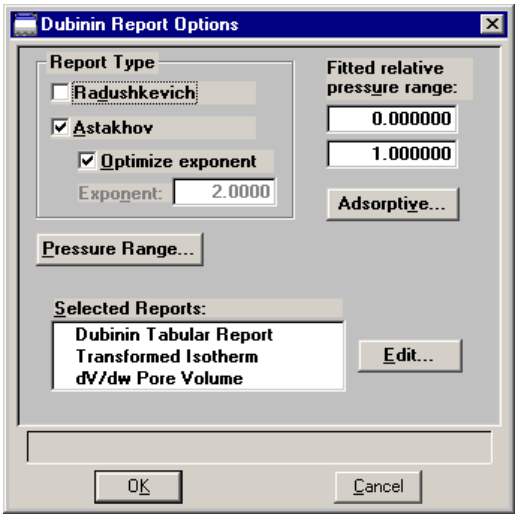
Reports to Generate — Displays the types of reports available.

- Tabular report
- Isotherm table
- Cumulative area graph
- Incremental area graph
- Differential area graph
- Log goodness of fit graph
- Goodness of fit graph

Graph details can be edited by selecting the **Edit** push button.

Refer to the previous section for the DFT Pore Size report beginning on page **4-75** for information on the fields contained on this dialog.

Dubinin Report Options



Report Type group box

Allows you to specify the type of report you want. At least one type of report must be selected.

Select **Radushkevich** to generate the Radushkevich report.

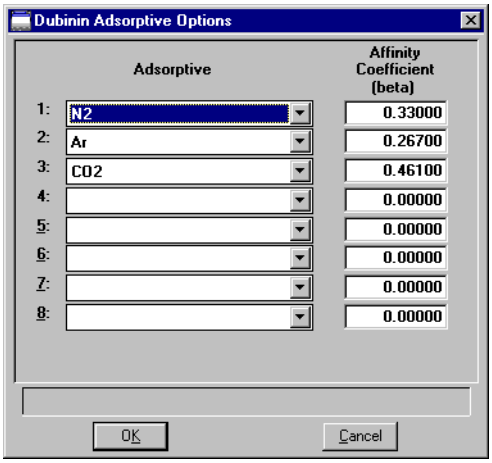
Select **Astakhov** to generate the Astakhov report. With this choice, you may select **Optimize Astakhov exponent**. If you do not select this option, the **Exponent value** field is enabled so that you can enter a value.

Fitted relative pressure range

Allows you to specify minimum and maximum limits on relative pressures included in the line fit. Data collected outside these limits are not included.

Adsorptive

Displays the Dubinin Adsorptive Options dialog allowing you to specify up to 8 Adsorptive/Affinity Coefficient (beta) combinations.



Pressure Range

Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.

- **If Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.
- **If Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Selected Reports

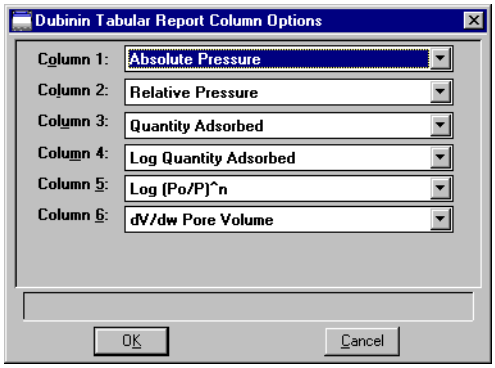
Lists the available Dubinin reports. Choose a report by double-clicking on the report name or highlighting the report name and pressing the **Spacebar**. A report is selected when it is preceded with a check mark.

The following reports are available:

Tabular Report
Transformed Isotherm
dV/dw Pore Volume (Astakhov only)

Tabular Report

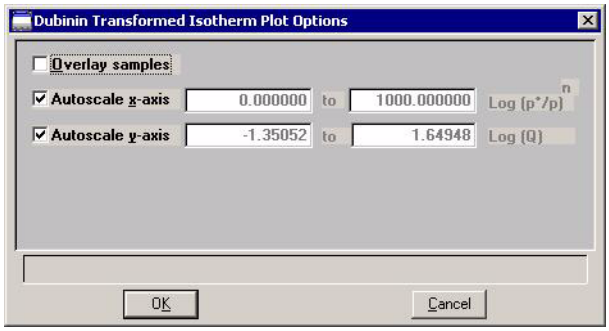
The Tabular report enables you to customize your report with one to six columns of data for Astakhov reports and one to five columns of data for Radushkevich reports. The Dubinin Tabular Report Column Options dialog is displayed.



The default column title appears next to the column number. Each column includes a drop-down list of the data types to include in the report.

Transformed Isotherm

The Transformed Isotherm report allows you to restrict the line fit to a portion of the isotherm. The Dubinin Transformed Isotherm Plot Options dialog is displayed.



Overlay Samples — Select this option to overlay the current plot with data from other samples. Then click **Overlays** on the Report Options dialog to choose the sample files.

Autoscale x-axis
Autoscale y-axis — Select these options to have the X- and/or Y-axes automatically scaled. Both X- and Y-axes begin at zero, and the system uses the highest values collected during analysis as the ending points for axes ranges.

Autoscale x-axis
Autoscale y-axis
(continued)

If you choose not to autoscale data for either (or both) axis, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

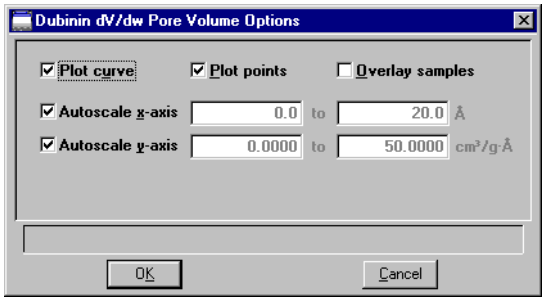
The X-Axis Range fields show the quantity of gas adsorbed at standard temperature and pressure.
The Y-Axis Range fields show the log of relative pressure.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Pore Volume

When you select the dV/dw Pore Volume plot from the Dubinin Report Options dialog, the Dubinin dV/dw Pore Volume Options dialog is displayed.

The Pore Volume report is unavailable if you choose Radushkevich as the type of report.



Plot options

You can plot data with a curve, points, or both.

Overlay Samples

Select this option to overlay data from other samples. Then click **Overlays** on the Report Options dialog to choose the files.

Autoscale x-axis
Autoscale y-axis

Select these options to have the X- and/or Y-axes automatically scaled. Both X- and Y-axes begin at zero, and the system uses the highest values collected during analysis as the ending points for axes ranges.

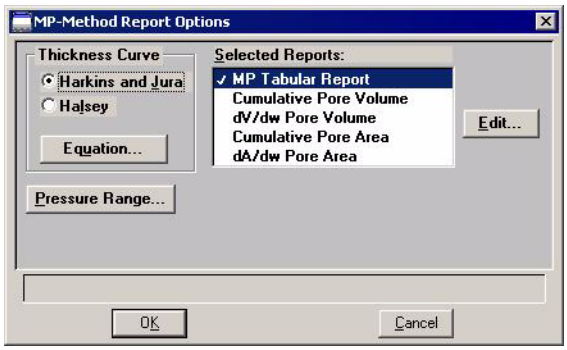
If you choose not to autoscale data for either (or both) axis, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

The X-axis Range fields show pore width, radius, or diameter in angstroms or nanometers.

The Y-axis Range fields show the quantity of gas adsorbed.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

MP-Method Report Options



Thickness Curve group box

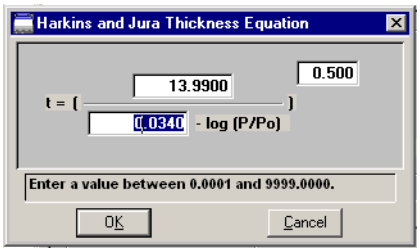
Select the type of thickness curve from this group box. You must choose either the Halsey equation or the Harkins and Jura equation. Use **Equation** to edit the values in the equation.

Equation

Displays the equation for the type of thickness curve selected.

Harkins and Jura

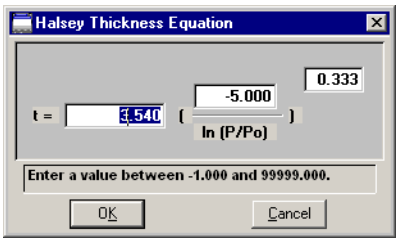
Displays the Harkins and Jura Thickness Equation dialog.



You can edit the values for the numerator, first element of the denominator, and exponent. The range for a selected field is shown in the information bar at the bottom of the dialog.

Halsey

Displays the Halsey Thickness Equation dialog.



You can edit the values for the multiplier, numerator, and exponent.The range for a selected field is shown in the information bar at the bottom of the dialog.

Pressure Range

Displays the Report Relative Pressure Range dialog so that you can specify minimum and maximum relative pressures to use with this report.

- **If Use calculation assignments is not selected** on the Collected/Entered dialog, all of the nonoutlier points of the collected data within the specified range are used for calculating the data for this report.
- **If Use calculation assignments is selected**, collected data points which are assigned to this report type are used.

Selected Reports

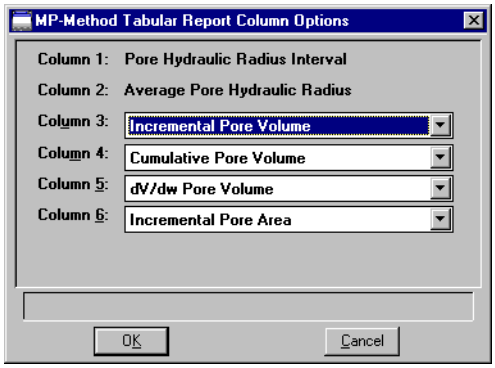
Lists the available MP-Method reports. Choose a report by double-clicking on the report name or by highlighting the report name and pressing the **Spacebar**. A report is selected when it is preceded with a check mark.

The following reports are available:

- MP Tabular Report
- Cumulative Pore Volume
- dV/dw Pore Volume
- Cumulative Pore Area
- dA/dw Pore Area

MP Tabular Report

The Tabular report enables you to customize your report with one to six columns of data. (The data types in the first and second columns cannot be changed.) The MP-Method Tabular Report Column Options dialog box is displayed.

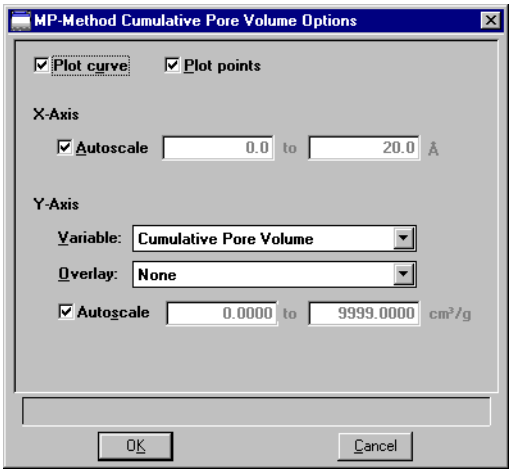


The default column title appears next to the column number for columns three through six. Each of these columns includes a drop-down list of the data types to include in the report. Columns 1 and 2 are fixed and cannot be edited.

The MP-Method reports hydraulic radius only. If you select Pore size in diameter on the Units dialog, the MP-Method still reports pore size in radius.

Pore Volume/Pore Area Plot

MP-Method plot options enable you to specify the plotting method used for your report and to customize the plot. A dialog similar to the following is displayed; the fields on all dialogs are the same.



Plot options

You can plot data with a curve, points, or both.

X-axis options

Choose **Autoscale** to have the X-axis scaled automatically. The X-axis begins at zero and the system uses the highest value collected during analysis as the ending point.

If you deselect **Autoscale**, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

X-axis range fields show pore radius or diameter in angstroms or nanometers.

Data ranges are displayed in the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-axis options

Allows you to choose Y-axis options.

Variable

Click on the down-arrow to choose a variable for the Y-axis.

Overlay

You can use the choices in this drop-down list to overlay plots. You can choose a different type of plot for the current sample to overlay with the listed **Variable**, or you can choose **Samples** to overlay the **Variable** plot with the same type of plot from other samples.

Autoscale

Choose this option to have the Y-axis scaled automatically. Y-axes begin at zero. The system uses the highest value collected during analysis as the ending point.

If you deselect **Autoscale**, the adjacent fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not included in the plot.

Y-axis range fields show the quantity of gas adsorbed Data ranges are displayed in the information bar across the bottom of the dialog box when the cursor is in a numerical data entry field.

Options Report

The Options report is a subset of the contents report; it provides pertinent information for the following:

- Degas conditions
- Adsorptive properties
- Analysis conditions
- Sample tube criteria
- Free space
- Po and temperature
- Equilibration
- Isotherm collection

Sample Log Report

The Sample Log report displays the following:

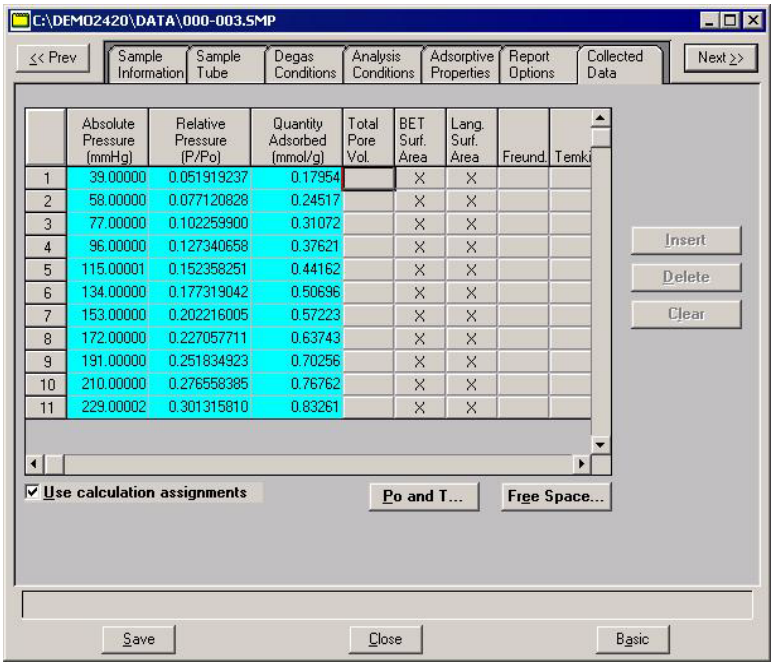
- Manual control operations performed during analysis
- Information entered using **Add Log Entry** on the sample file editor
- Warnings and/or errors which occurred during analysis

The Sample Log report is a new feature with the ASAP 2420 program. Therefore, if you request a Sample Log report on a file that was used with the ASAP 2400 or 2405 program, no information will be available.

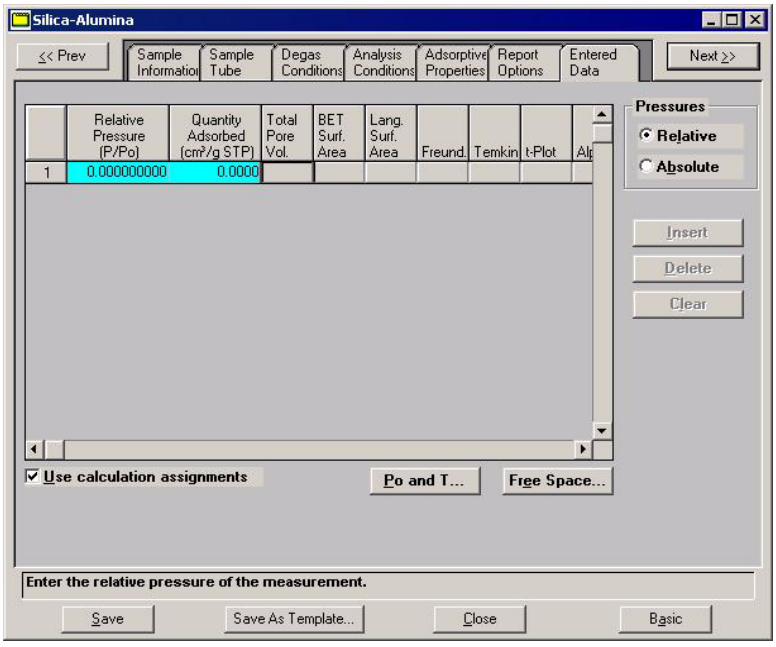
Collected or Entered Data

If you choose **Automatically collected** as the type of data in the Sample Information dialog, a **Collected Data** tab is added when analysis is complete. The Collected Data dialog will contain the data points collected during analysis and the specified calculation assignments.

This example shows the Collected Data dialog from a completed sample information file.



If you choose **Manually entered** as the type of data in the Sample Information dialog, an **Entered** tab is added allowing you to enter the data.



Pressure table

For **collected data**, columns for the following are displayed:

- absolute pressure
- relative pressure
- quantity adsorbed
- outliers (if Use calculation assignments is not selected)
- calculation assignments for each requested report option (if Use calculation assignments is selected)

For **entered data**, columns for the following are displayed:

- absolute or relative pressure (depending on the selection in the Pressures group box)
- quantity adsorbed

Pressures

This buttons appear on the dialog for Entered data only. Select either *Relative* or *Absolute* pressure.

Insert

Enabled for entered data; inserts a row into the pressure table.

Delete

Enabled for entered data; deletes the selected row.

Clear

Enabled for entered data; clears the entire table of all entries except one (one is required).

Use calculation assignments

If selected, allows you to assign the points to be collected for each report type.

If deselected, each report (with the exception of Langmuir and BET) uses a range of pressures as selected in Report Options. The Langmuir and BET reports interpolate to entered relative pressures on Report Options.The outlier points can be selected so that they will not be reported.

Po and T

Displays the Po and T dialog, allowing you to edit the saturation pressure and temperature. Only the applicable options are enabled.

Free Space

Displays the Free Space dialog, allowing you to edit the cold and warm free spaces. This dialog also includes the plotted isotherm. The isotherm is redrawn each time you edit the values in either field.

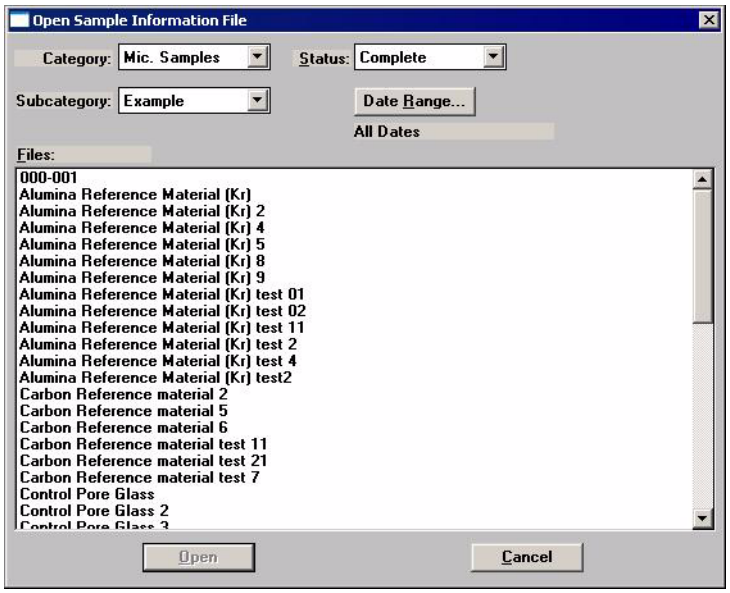
Also available for the isotherm is a shortcut menu containing options allowing you to edit the curve and axes. You also can zoom in for finer detail. The options available are explained in Chapter 6.

Open

Open allows you to open an existing sample or parameter file, as well as a sample or parameter template.

Sample Information File

The following dialog displays for the Analyst and Developer when **Open** > **Sample information** is selected from the File menu.



A sample file can be edited by a Developer or an Analyst. The changes can also be saved. If you choose to save the changes, a dialog prompting for a reason is displayed. After you enter a reason in the **Comments** field, **Save** is enabled so that you can complete the save operation.

These changes are saved as a version of the sample file, becoming part of the sample file history. All versions of the sample file (of the Status selected) can be viewed when you click **Revert** on the Sample file dialog box.

Category/Subcategory

These drop-down lists contain existing archive locations (directories) in which files have been stored.

Many laboratories use the Year/Month system. A Developer can edit these labels using the **Edit Labels** command on the Options menu. Refer to page **7-5**.

Status

This drop-down list allows you to choose the types of sample files to display in the **Files** list. All files of the type you choose, within the range of dates, and in the current directory are displayed. Refer to **"Selecting a File Status" on page 2-20**.

Date Range

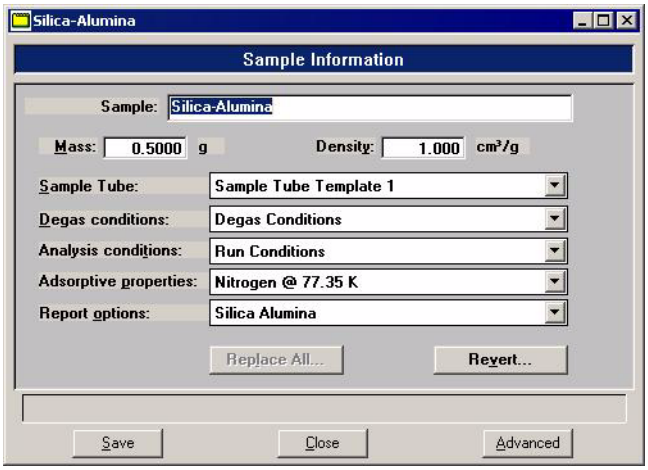
Allows you to choose the dates of the files displayed in the Files list. Refer to **"Selecting a Range of Dates" on page 2-21**.

Files list

Lists the files of the type selected in the **Status** list within the selected directory and range of dates.

Analyst

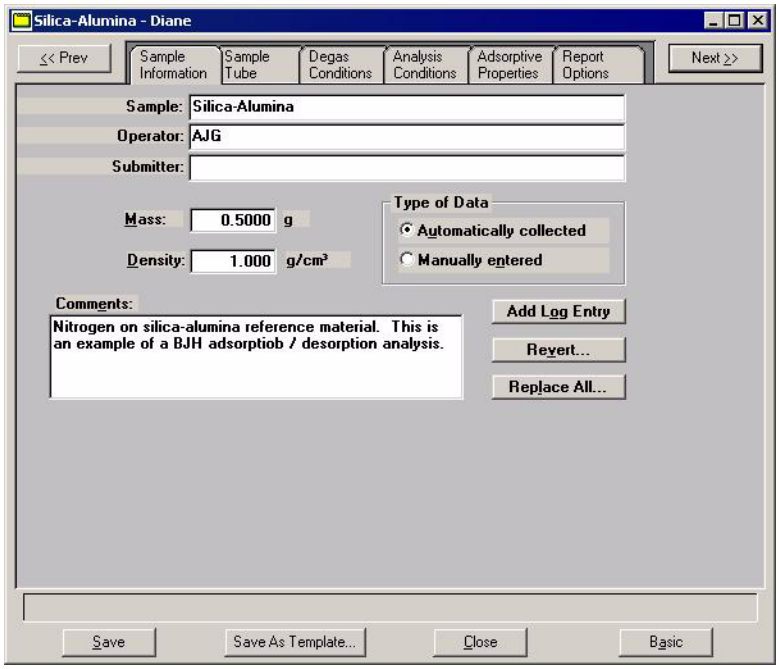
When an Analyst opens a sample information file, the dialog displays in this manner:



If desired, an Analyst can click **Advanced** to access a Developer's view of the dialog. This view allows an Analyst to review the parameters used in the analysis. An Analyst can edit certain parameters of the sample file; the fields for those parameters are enabled. Report options can also be edited and reports generated.

Developer

When a Developer opens a sample information file, the dialog displays in this manner:



A Developer can view and edit all parameters of the file, and edit and generate reports.

A Developer's view of the dialog includes a Collected Data tab which allows you to view the points collected during analysis. The **Save As Template** button allows you to save the values of the open file as a Sample File template.

Templates

Sample and Parameter templates can be opened and their contents viewed by a Developer or an Analyst. You cannot make changes to an existing template. If you wish to make changes, a Developer will have to create a new template. An Analyst is not authorized to create templates.

Parameter Files

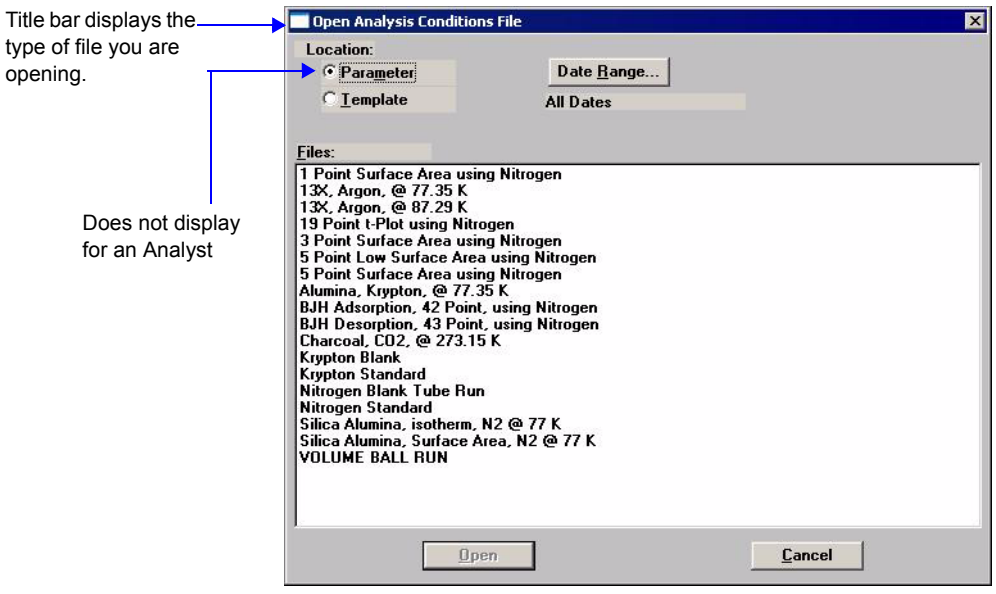
A Developer can:

- open sample files and templates
- open parameter files and templates
- edit sample and parameter files

An Analyst can:

- open sample files and templates
- open parameter templates (but not parameter files)
- edit portions of a sample file

When you select **File** > **Open > (file or template name)**, an Open (parameter) dialog is displayed.



Location

Displays only when a Developer is logged in.

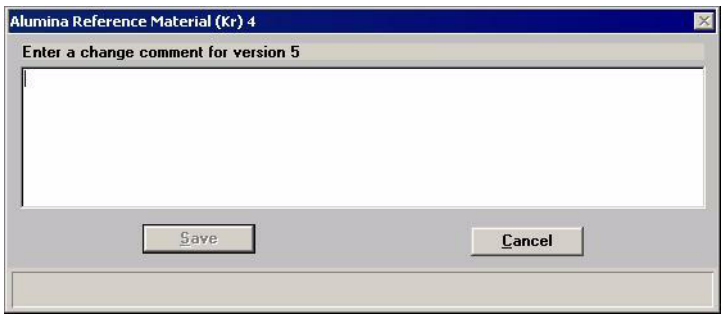
Allows you to open a parameter file or a template for a parameter file. You can open a template and view the details but you cannot edit the values. If you wish to make changes, you will have to create a new template. You can edit and save changes in a parameter file.

Date Range

Allows you to choose a range of dates in which to display files in the **Files** window. Refer **"Selecting a Range of Dates" on page 2-21**.

Save

Save enables you to save any changes you have made to the file in the active window. When you click **Save**, the comment window is displayed.

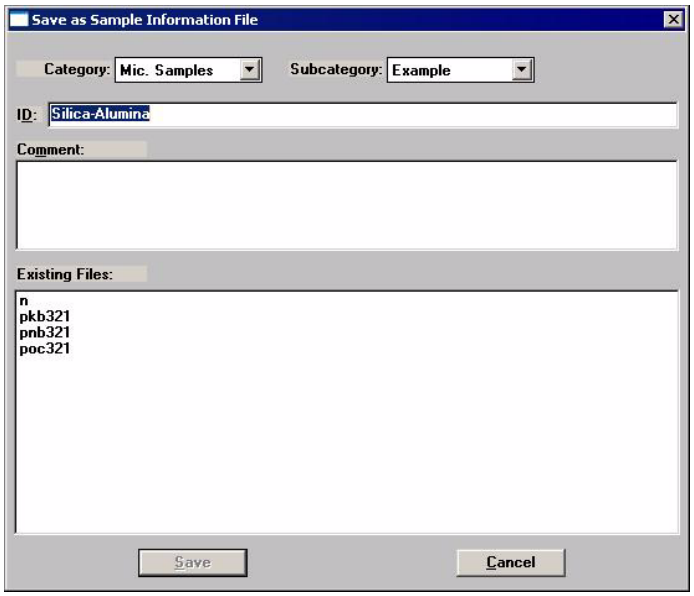


Enter an explanation for the change, then click **Save**.

Save As

Save As is unavailable for an Analyst; a Developer can use this function to:

- Save a sample file in the active window as another sample file with a different name. When you select this option, this type of dialog is displayed.

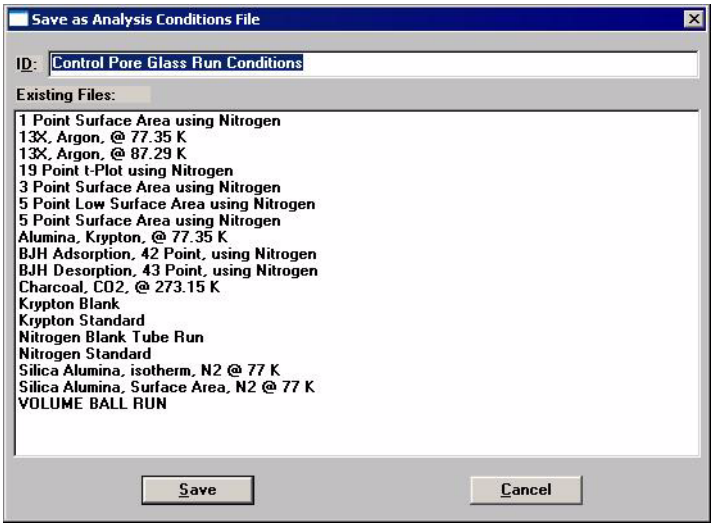


  Enter a description in the **ID** field. You must also enter a comment in the **Comment** window to enable the **Save** push button.

  If the **Save** push button does not become enabled, a file with the same description already exists. The ASAP 2420 Confirm program does not allow duplicate descriptions.

- save a parameter file in the active window with a different name. The parameter file must be of the same type; for example, you can save an Analysis conditions file only as another Analysis conditions file.

When you save as a parameter file, this type of dialog is displayed.



Enter a description in the **ID** field. If the **Save** push button does not become enabled, a file with the same description already exists. The Confirm program does not allow duplicate file descriptions. Edit the description to enable the **Save** push button.

- save a subset (parameter) of the sample file in the active window as a standalone parameter file. For example, select Analysis Conditions from the Save As menu to create a standalone parameter file of the analysis conditions portion of the active sample file.

  When you save a subset of a sample as a parameter file, the same type of dialog as shown above is displayed.

- save as an ASCII file the relative pressures and corresponding thicknesses (t-Curve) from the collected data. These data are derived by dividing the condensed volume of adsorptive by the selected surface area. The density conversion factor in the adsorptive properties file is used to convert quantity adsorbed to volume of condensed adsorptive.

- save as an ASCII file the relative pressures and resulting quantities adsorbed (Alpha-S) from the collected data. These data are derived by dividing the isotherm by the quantity adsorbed at 0.4 relative pressure.

Save As Template

An Analyst is not authorized to create sample and parameter file templates.

Save As Template is unavailable for an Analyst; a Developer can use this function to:

- save the values of the sample file in the active window as a sample template
- save a subset (parameter) of the sample file in the active window as a parameter file template.
- save the values of a parameter file in the active window as a parameter file template.

Save All

Save All enables you to save all open files under their current names. This option provides a faster way to save *all* open files at one time and avoids having to perform a **Save** operation on each individual file.

A dialog prompting for a reason for the current change version of each file is displayed.

Close

Close enables you to close the file in the active window. If the file contains changes that have not been saved, the following message is displayed:

(file name) has been changed. Save changes before closing?

Yes **No** **Cancel**

Click **Yes**; a dialog prompting for a reason for the change is displayed before the file closes.

Click **No** to close the file without saving the changes.

Click **Cancel** to return to the active file.

Close All

Close All enables you to close *all* open files under their current names. The following message displays for every file containing changes that have not been saved:

(file name) has been changed. Save changes before closing?

Yes **No** **Cancel**

Click **Yes**; a dialog prompting for a reason for the change is displayed before the file closes.

Click **No** to close the file without saving the changes.

Click **Cancel** to return to the active file.

Print

Print enables you to print the contents of a sample or parameter file or template.

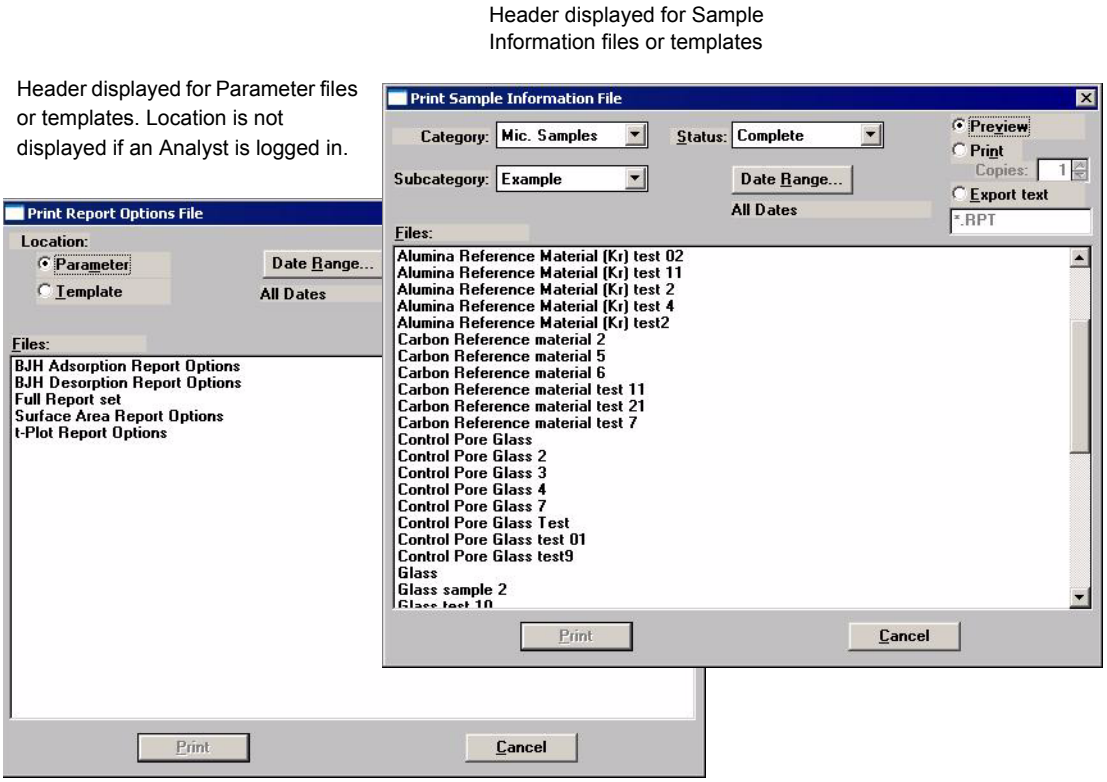
A Developer can print the contents of:

- sample files or templates
- parameter files or templates

An Analyst can print the contents of:

- sample files or templates
- parameter templates (but not parameter files)

Regardless of the type of file you choose, the dialogs are similar; the options presented in the header vary slightly.



Category/Subcategory — These drop-down lists display only when you select to print the contents of a sample file. These lists contain the existing directories in which files are stored.

Status — This drop-down list displays only when you select to print the contents of a sample file. It enables you to select the types of files displayed in the **Files** list. Refer to **"Selecting a File Status" on page 2-20**.

Location — Displays for parameter templates (or files) only when a Developer is logged in. Enables you to list either parameter files or parameter templates in the **Files** list.

Date Range — Allows you to specify a range of dates for the files you wish to display in the **Files** list. Refer to **"Selecting a Range of Dates" on page 2-21**.

Preview **Print** **Export**	Allows you to select a print destination. Refer to **"Report Settings" on page 2-22**.
Files list	Displays the files or templates of the type selected in the **Status** list within the selected directory and range of dates. You may print the contents of multiple files by holding down **Ctrl** while selecting files.

List

A Developer can list file statistics for:

- sample files or templates
- parameter files or templates

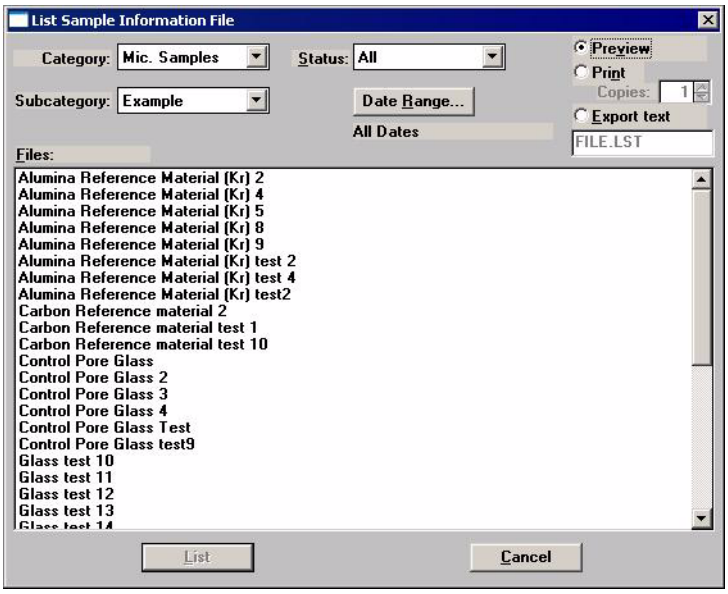
An Analyst can list file statistics for:

- sample files or templates
- parameter templates (but not parameter files)

The information displayed in the list includes:

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

Regardless of the type of file you choose, the dialogs are similar; the options presented in the header vary slightly. The types of dialogs displayed are identical to the ones displayed for the **Print** function; you simply obtain different types of information. This example shows a dialog for sample files.



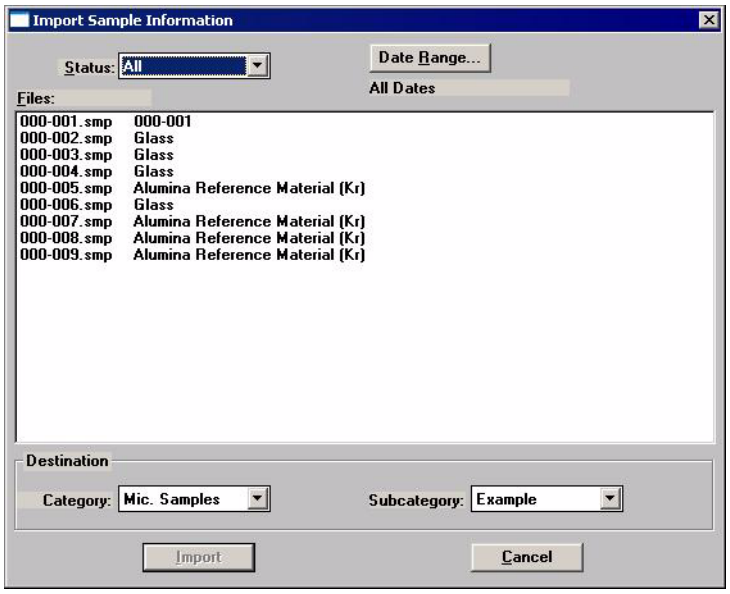
Refer to the previous section, **Print**, for an explanation of the fields on this dialog.
You may request a list of multiple files by holding down **Ctrl** while selecting files. If no files are selected, a list is generated for *all* files.

Import

An Analyst is not authorized to import sample and parameter files.

A Developer can use this option to import files that have been created external to the Confirm software and copied into the Import directory. If the files are not copied into the Import directory, they will not display in the Files window.

Use Windows Explorer to copy the files into the Import directory.



Category
Subcategory

Use these drop-down lists to choose the directory into which you wish to import the file. These drop-down lists do not display when importing parameter files. Parameter files are imported into the **param** directory.

Status
Date Range

Use these two options to limit the files displayed in the **Files** window. The **Status** drop-down list does not display when importing parameter files.

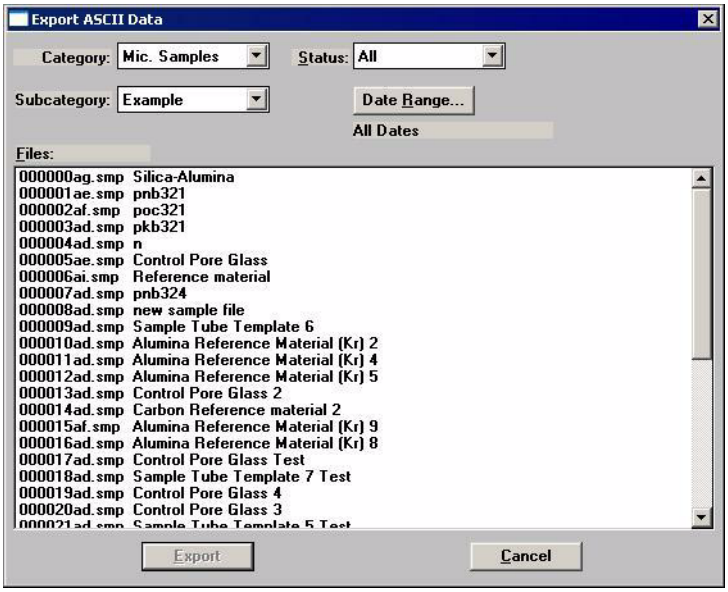
Export

An Analyst is not authorized to export sample files.

A Developer can use the Export option to:

- export the entire sample file to the Export directory. For example, you may wish to use this option to transfer a sample file to another computer.
- copy the isotherm data in a sample information file and export it as ASCII text. When exported as ASCII text, isotherm data can be imported into other applications (such as spreadsheets) accepting ASCII format. The data are exported in a comma-delimited format.

You may select multiple files by holding down **Ctrl** and clicking the left mouse button once on each file.

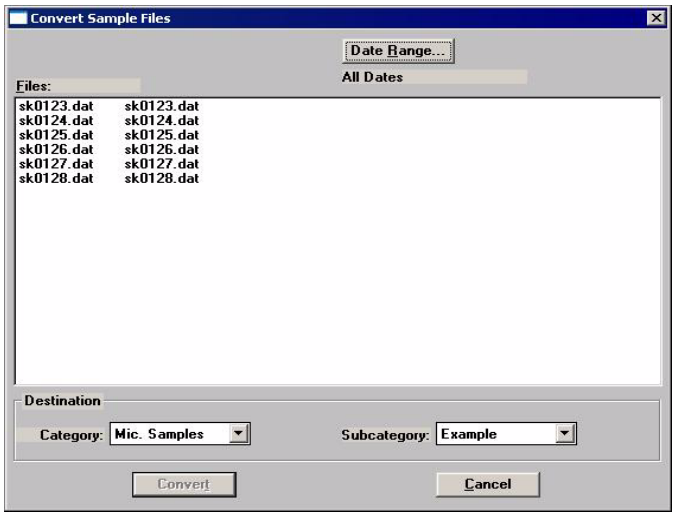


All fields on this dialog function in the same manner as the Print dialog. Refer to the **Print** section beginning on page **4-99** if you need information on these fields.

Convert

An Analyst is not authorized to convert sample files.

Convert allows a Developer to convert sample information files created using the ASAP 2400 or 2405 to files compatible with the ASAP 2420 Confirm program. The files to be converted must be copied into the Import directory so they will display in the Convert dialog.



Date Range

Use this two options to limit the files displayed in the **Files** window.

Category
Subcategory

Use these drop-down lists to choose the directory into which you wish to import the file.

These drop-down lists do not display when converting parameter files. Parameter files are converted and sent to the **param** directory.

Log In

Log in enables you to log into the application; the User Logon dialog is displayed.

Enter your user name and password, then click **OK**.

User name — Typically in this field, you should enter your Windows User name, unless your Administrator has specified a different user name.

Password — Enter your password. If you enter your password incorrectly a specified number of times (your Administrator determines the number when setting up system options), the application will lock and you will not be allowed to proceed. Contact the Administrator to unlock the application and assign a new password.

The first time you log into the application after the Confirm software is installed, you will be prompted to change your password to one of personal preference. You must enter at least six characters.

Change password — Select this option to change your password after you have logged in; the Set password dialog allowing you to change your password is displayed.

Log Out

Select this option to log out of the system. If files are open and contain changes that have not been saved, you are prompted to save or discard them before log out is completed.

Exit

Exit enables you to exit (close) the ASAP 2420 Confirm program.

- If a window containing a modified file is open, the following message is displayed:

  (file name) has been changed. Save changes before closing?

  Yes No Cancel

  Click **Yes**; a dialog prompting for a reason for the change is displayed before the file closes.

  Click **No** to close the file without saving the changes.

  Click **Cancel** to return to the active file.

- If an analysis is in progress, the following message is displayed:

  2459- An analyzer is busy. A delay in restarting this application could result in loss of new data. Continue with program exit?

  Yes No

  Click **Yes** to exit the analysis program; the analysis will continue until completion; analysis data are collected and stored in the analyzer's memory.

Although data are stored in the analyzer when you exit the program during analysis, they are not saved in the file until the program is restarted. At that time the data are saved automatically. If a power failure occurs in the interim and you do not have an Uninterruptible Power Supply (UPS), loss of data will result.

  Click **No** to allow the analysis program to remain active and the analysis to finish.

- If a report is in progress, you are cautioned that reports are being generated, and given the opportunity to either cancel the print job or allow the reports to print before exiting.

5 UNIT MENU

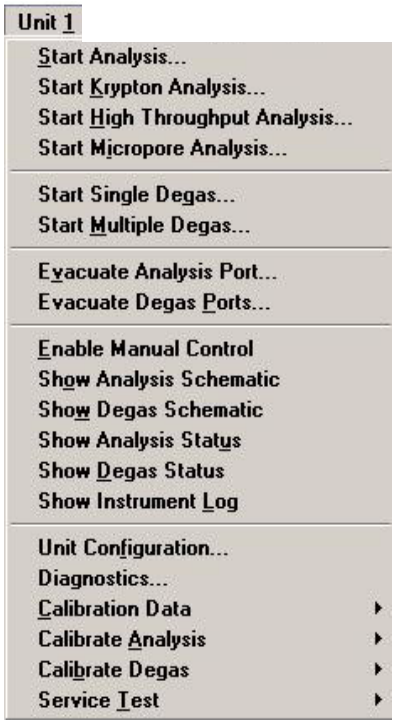
Description	5-3
Start Analysis	5-5
Start Krypton Analysis	5-9
Start High Throughput Analysis	5-13
Start Micropore Analysis	5-17
Start Single Degas	5-21
Start Multiple Degas	5-22
Evacuate Analysis Port	5-24
Evacuate Degas Port	5-25
Enable Manual Control	5-26
Show Analysis Schematic	5-31
Show Degas Schematic	5-32
Show Analysis Status	5-33
Show Degas Status	5-34
Show Instrument Log	5-36
Unit Configuration	5-38
Diagnostics	5-40
Calibration Procedures and Service Tests	5-44

UNIT MENU

The Unit menu contains the operations that can be performed with the ASAP 2420 analyzer. The main menu contains 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



Following are brief descriptions of the Unit menu options. Detailed descriptions are found in subsequent sections.

Start Analysis Starts an analysis on a sample in the selected port.

Start Krypton Analysis Starts a krypton analysis on the samples in the selected ports (up to five).

Start High Throughput Analysis	Starts an analysis on the samples in the selected ports (up to six).
Start Micropore Analysis	Starts a micropore analysis on the samples in the selected ports (up to six)
Start Single Degas	Allows you to start the degassing operation on one sample.
Start Multiple Degas	Allows you to start the degassing operation on up to 12 samples.
Evacuate Analysis Port	Allows you to manually evacuate an analysis port.
Evacuate Degas Ports	Allows you to manually evacuate degas ports.
Enable Manual Control	Allows you to control the system manually.
Show Analysis Schematic	Displays a schematic of the ASAP 2420 analysis system.
Show Degas Schematic	Displays a schematic of the ASAP 2420 degas system.
Show Analysis Status	Displays a window showing the status of current analyses.
Show Degas Status	Displays a window showing the status of current degas operations.
Show Instrument Log	Displays a log of recent analyses, calibrations, and error messages.
Unit Configuration	Displays the configuration of the analyzer.
Diagnostics	Enables you to perform predefined user diagnostics (at the request of a Micromeritics service representative).
Calibration Data	Enables calibration data to be entered. This option is enabled only with the direction of a Micromeritics service representative.
Calibrate Analysis	Enables calibration procedures to be performed on certain analysis system components. This option is enabled only with the direction of a Micromeritics service representative.
Calibrate Degas	Enables calibration procedures to be performed on certain degas system components. This option is enabled only with the direction of a Micromeritics service representative.

Service Test	Enables certain troubleshooting procedures to be performed. This option is enabled only with the direction of a Micromeritics service representative.

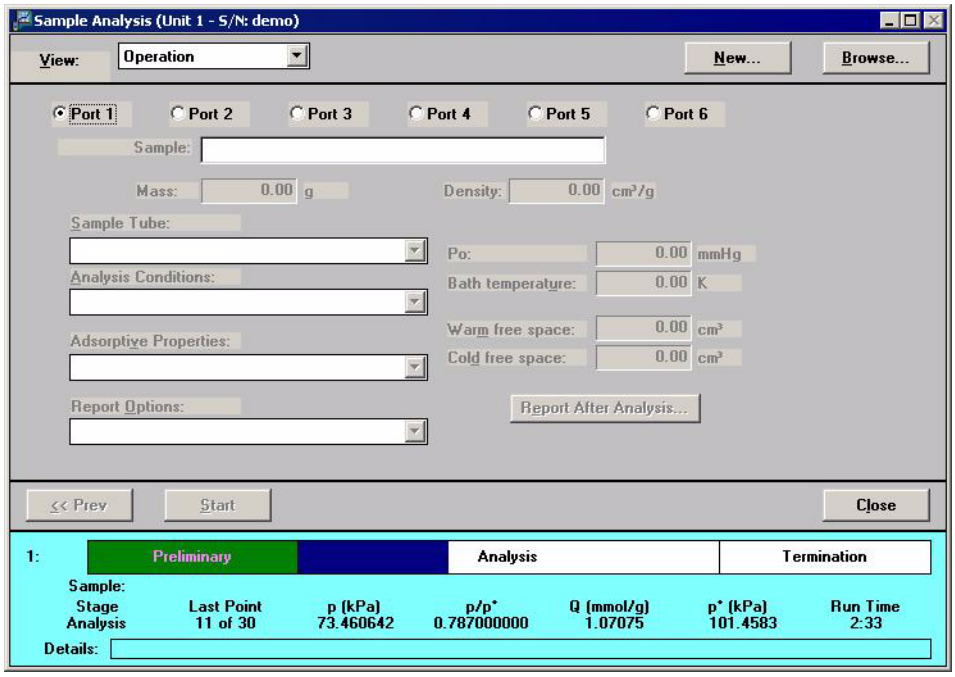
Start Analysis

This option allows you to start an analysis using nitrogen or a similar gas. Use the following guidelines for performing standard analyses:

- Standard analyses cannot be performed if a high throughput or krypton analysis is in progress.
- One analysis can be started at a time.
- A sample can be added to any idle port and an analysis started without disturbing the analyses being performed on other ports.
- Samples can be removed from any of the six ports without disturbing the analyses being performed on other ports.
- All analyses must use the same gas.
- A manual evacuation can be performed on any idle port.

When you select this option from the Unit menu, the Select a File for analysis dialog is displayed. This allows you to select a sample file for your analysis or to create a new one. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

After you have designated a sample file, the Sample Analysis dialog is 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 analysis system schematic
- the instrument log
- a graph of the analysis in progress

New

Displays the Create a File for Analysis dialog allowing you to create a sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Browse

Displays the Select a File for Analysis dialog allowing you to select an existing sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Port

Allows you to select the port to be used for the analysis. Only one port may be selected at a time.

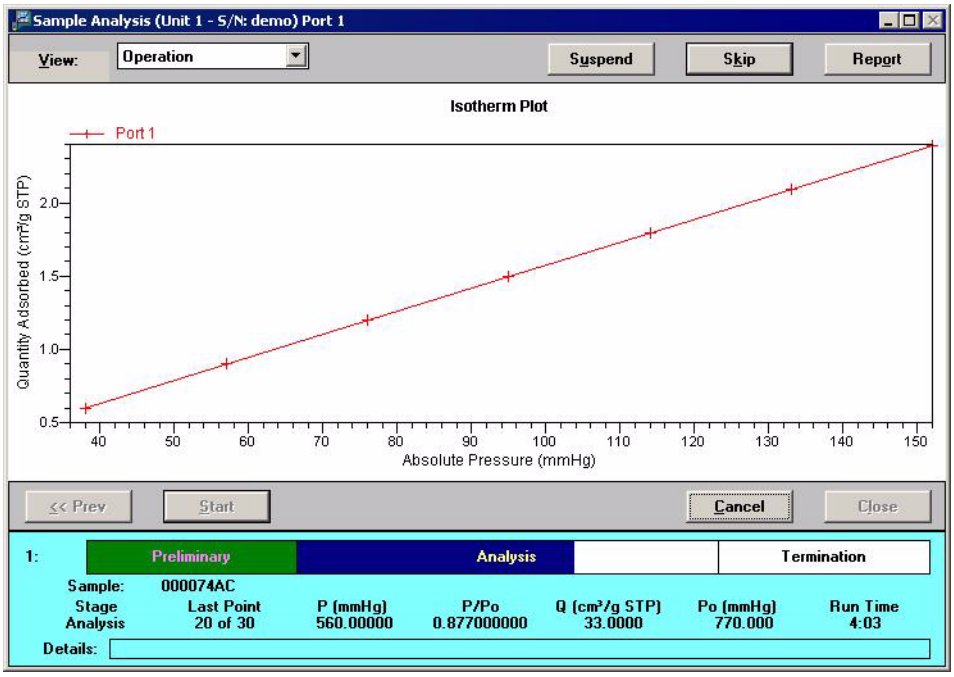
Sample	Displays the sample's file name.
Mass	Displays the mass entered in the sample information file. You may edit it if required.
Density	Displays the density entered in the sample information file. You may edit it if required.
Sample Tube **Analysis Conditions** **Adsorptive Properties** **Report Options**	These fields display the names of the parameter files in the current sample file. If this is a new sample file, the fields contains the files you specified as defaults. The drop-down lists contain predefined parameter files. You may choose a different file from a list if desired.
Po	Enabled if you have chosen to enter the Po measurement. Enter (or confirm) the saturation pressure of the adsorptive, or edit the current one if desired.
Bath temperature	Enabled if you have chosen to enter the bath temperature (Po and T option on the Analysis Conditions dialog). Enter (or confirm) the temperature for the analysis bath.
Warm Free Space	Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the warm free space value.
Cold Free Space	Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the cold free space value.
Report After Analysis	Displays the Report Settings dialog so that you may specify report output options. (Refer to **"Report Settings" on page 2-22**).

Start

Begins the analysis. When you click **Start**, a Logon screen is displayed. Enter you password and click **OK**.

If you made changes to the sample file, a Comments window is displayed. Enter your information about the file, then click **OK**.

The Sample Analysis screen is displayed.



Suspend

Suspends the analysis.

Skip

Skips the current step of the analysis.

Use the Skip function with caution; the ASAP 2420 performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.

Resume

Resumes a suspended analysis.

Cancel

Cancels the analysis.

Report

Displays a report of the data collected during analysis.

Next When the analysis is complete, the **Start** button changes to **Next**. The **Next** button returns you to the first analysis dialog, enabling you to start another analysis.

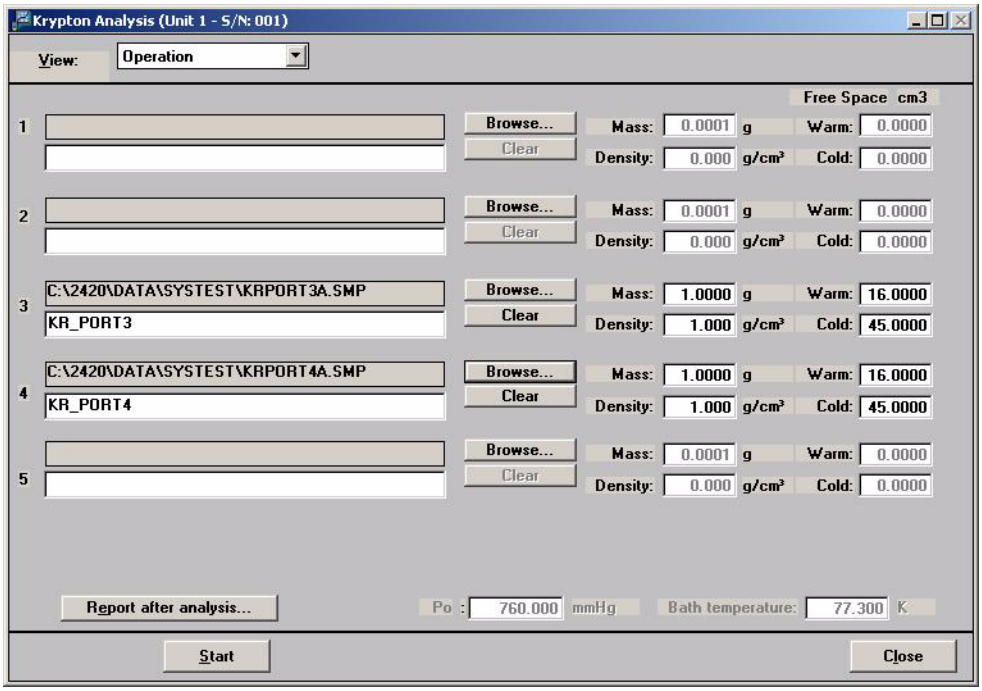
Close Enabled at the completion of analysis. Closes the dialog.

Start Krypton Analysis

When you select this option from the Unit menu, the Krypton Analysis dialog is displayed.

Use the following guidelines for performing krypton analyses:

- All ports must be idle in order to start an analysis.
- All analyses must use krypton gas.
- From one to five analyses can be started at the same time using ports 1 through 5. Port 6 is reserved for dosing.
- The sample file used for an analysis must specify the Dosing Method: **From Port 6** in the Adsorptive Properties.
- Samples cannot be removed from or added to ports until the full set of analyses has completed.



View

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

- the current operation
- the instrument schematic
- the instrument log
- the status of the analyses on all active ports

New

Displays the Create a File for Analysis dialog allowing you to create a sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Browse

Displays the Select a File for Analysis dialog allowing you to select an existing sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Clear

Removes the information entered for the associated port enabling you to select a different sample file.

Mass

Displays the mass entered in the sample information file. You may edit it if required.

Density

Displays the density entered in the sample information file. You may edit it if required.

Warm

Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the warm free space value.

Cold

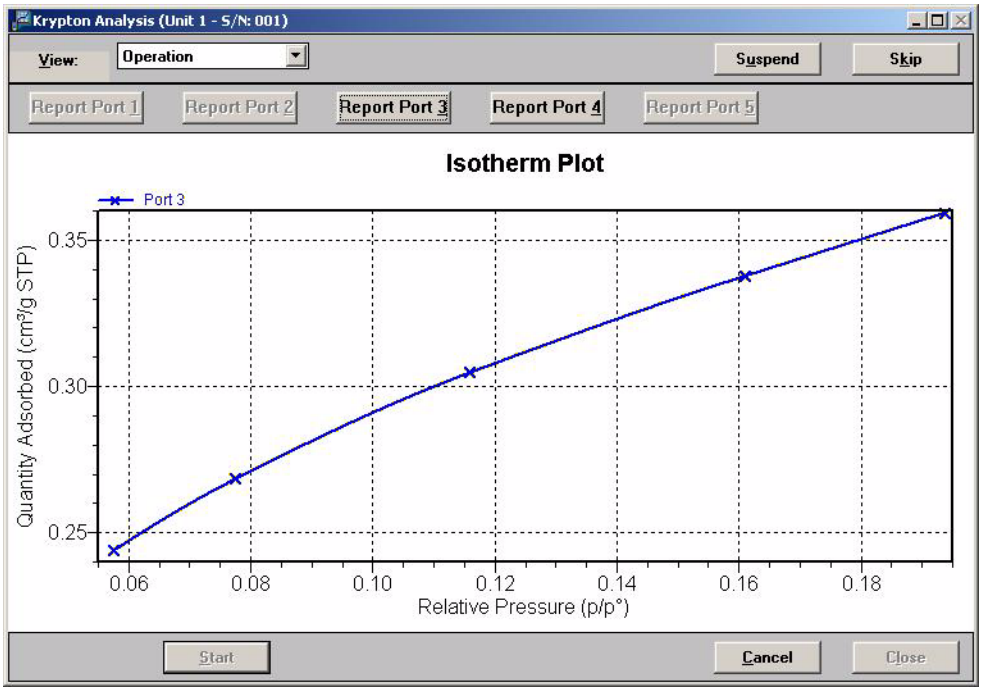
Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the cold free space value.

Report After Analysis Displays the Report Settings dialog so that you may specify report output options. (Refer to **"Report Settings" on page 2-22**).

Po Enabled if you have chosen to enter the Po measurement. Enter (or confirm) the saturation pressure of the adsorptive, or edit the current one if desired.

Bath temperature Enabled if you have chosen to enter the bath temperature (Po and T option on the Analysis Conditions dialog). Enter (or confirm) the temperature for the analysis bath.

Start Begins the analyses on the specified ports. When you click **Start**, a Logon screen is displayed. Enter you password and click **OK**.



Suspend Suspends the analyses of all samples.

Skip Skips the current step on all analyses.

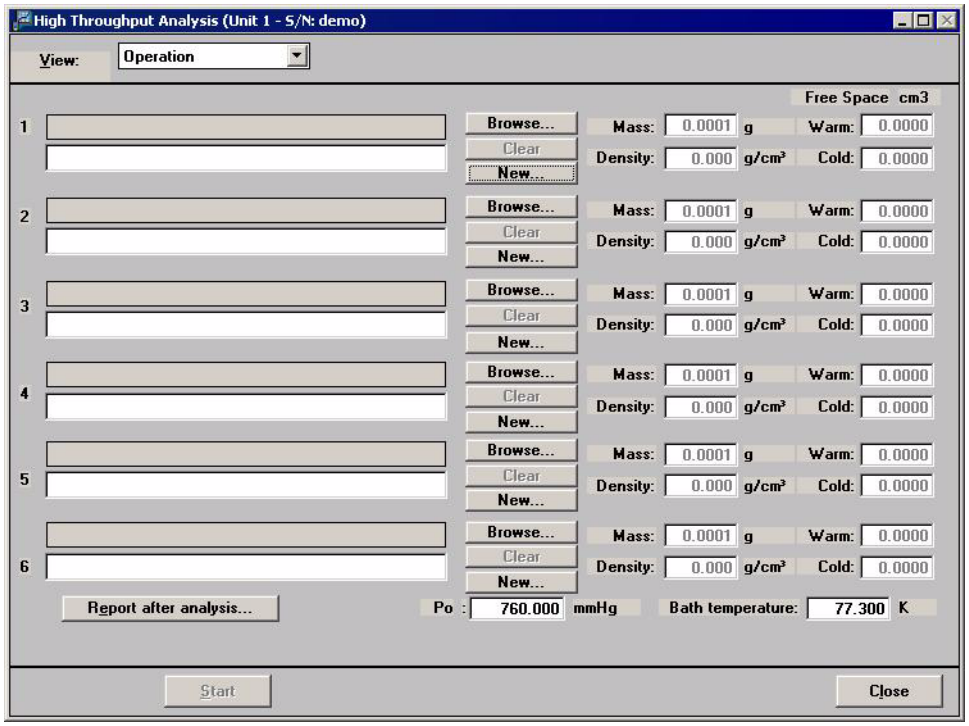
Use the Skip function with caution; the ASAP 2420 performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.

Resume	Resumes suspended analyses.
Report Port (1 - 5)	Displays a report for the sample on the selected port.
Cancel	Cancels the analyses.
Next	When the analysis is complete, the **Start** button changes to **Next**. The **Next** button returns you to the first analysis dialog, enabling you to start another analysis.
Close	Enabled at the completion of analysis. Closes the dialog.

Start High Throughput Analysis

When you select this option, the High Throughput Analysis dialog is displayed. This dialog enables you to start up to six analyses at the same time. Use the following guidelines for performing high throughput analyses:

- All ports must be idle in order to start an analysis.
- All analyses must use the same analysis gas.
- All analyses that measure Psat must use the same Psat gas (which may be different from the analysis gas)
- From one to six analyses can be started at the same time.
- Samples cannot be removed from or added to ports until the full set of analyses has completed.



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

- the current operation
- the instrument schematic
- the instrument log
- the status of the analyses on all active ports.

For ports 1 through 6, you may use the following commands:

New Displays the Create a File for Analysis dialog allowing you to create a sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Browse Displays the Select a File for Analysis dialog allowing you to select an existing sample file for the current analysis. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**).

Clear Removes the information entered for the associated port enabling you to select a different sample file.

Mass Displays the mass entered in the sample information file. You may edit it if required.

Density Displays the density entered in the sample information file. You may edit it if required.

Warm Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the warm free space value.

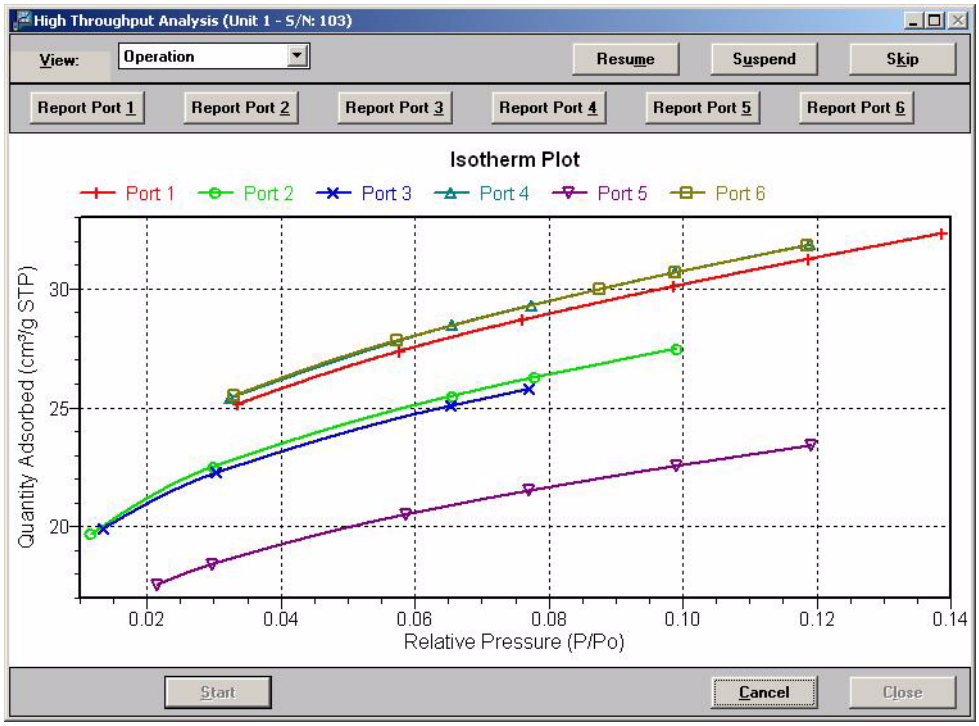
Cold Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the cold free space value.

Report After Analysis Displays the Report Settings dialog so that you may specify report output options. (Refer to **"Report Settings" on page 2-22**).

Po Enabled if you have chosen to enter the Po measurement. Enter (or confirm) the saturation pressure of the adsorptive, or edit the current one if desired.

Bath temperature Enabled if you have chosen to enter the bath temperature (Po and T option on the Analysis Conditions dialog). Enter (or confirm) the temperature for the analysis bath.

Start Begins the analyses on the specified ports.



Resume Resumes suspended analyses.

Suspend Suspends the analyses of all samples.

Skip Skips the current step on all analysis.

Use the Skip function with caution; the ASAP 2420 performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.

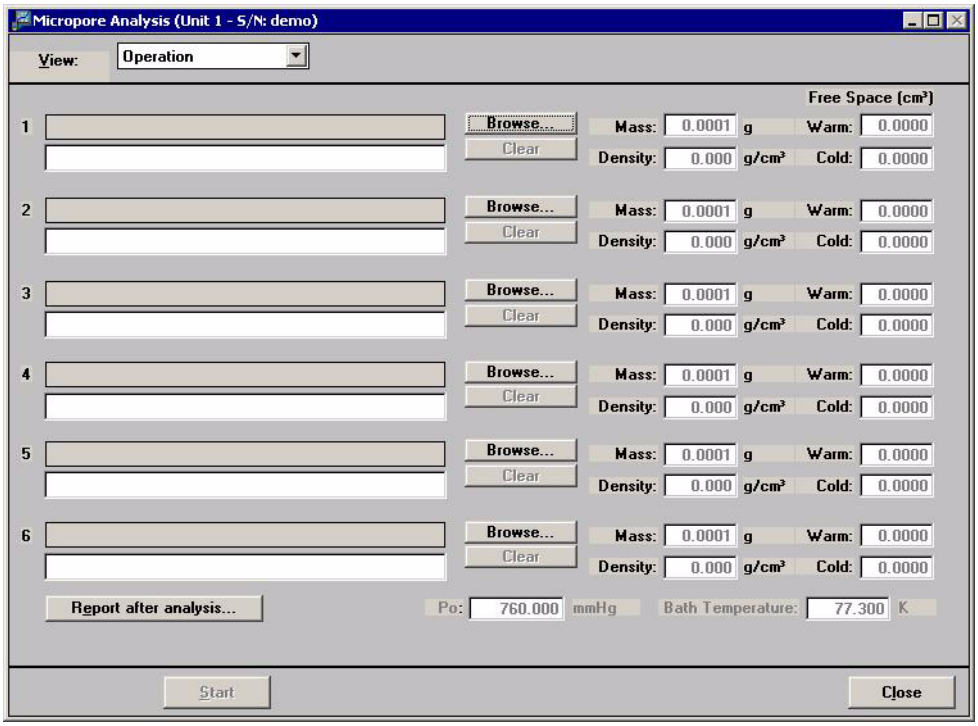
Report Port (1 - 6)	Displays a report for the sample on the selected port.
Cancel	Cancels the analyses.
Next	When the analysis is complete, the **Start** button changes to **Next**. The **Next** button returns you to the first analysis dialog, enabling you to start another analysis.
Close	Enabled at the completion of analysis. Closes the dialog.

Start Micropore Analysis

When you select this option, the Micropore Analysis dialog is displayed. This dialog enables you to start up to six analyses at the same time. Use the following guidelines for performing micropore analyses:

- All ports must be idle in order to start an analysis.
- All analyses must use the same analysis gas.
- All analyses that measure Psat must use the same Psat gas (which may be different from the analysis gas).
- The sample dosing method should be **Normal**.
- From one to six analyses can be started at the same time.

Samples cannot be removed from or added to ports until the full set of analyses has completed.



View

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

- the instrument schematic (refer to **"Show Analysis Schematic" on page 5-31**)
- the instrument log (refer to **"Show Instrument Log" on page 5-36**)
- the status of the analyses on all active ports (refer to **"Show Analysis Status" on page 5-33**)
- a graph of the analysis in progress

Browse

Displays the Select a File for Analysis dialog allowing you to select or create a sample file for the associated port (refer to **"Selecting or Creating a File for Analysis" on page 3-45**).

When you select a file, its path name and description are displayed.

Clear

Removes the information entered for the associated port enabling you to select a different sample file.

Mass

Displays the mass entered in the sample information file. You may edit it if required.

Density

Displays the density entered in the sample information file. You may edit it if required.

Warm

Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the warm free space value.

Cold

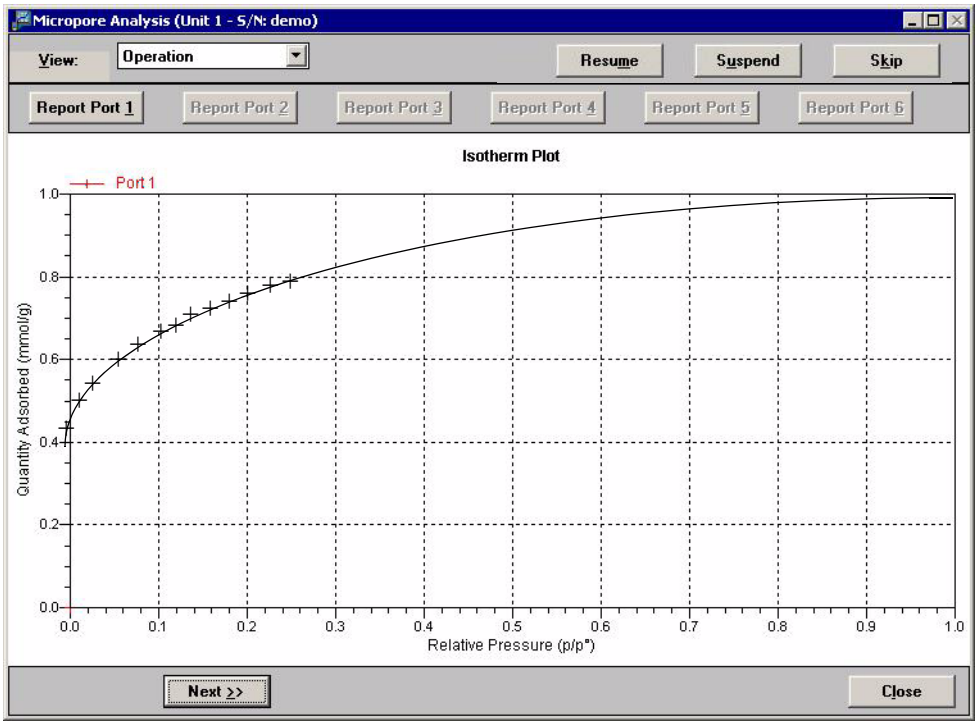
Enabled if you have chosen to enter the free space value (Free Space option on the Analysis Conditions dialog). Enter (or confirm) the cold free space value.

Report After Analysis Displays the Report Settings dialog so that you may specify report output options. (Refer to **"Report Settings" on page 2-22**).

Po Enabled if you have chosen to enter the Po measurement. Enter (or confirm) the saturation pressure of the adsorptive, or edit the current one if desired.

Bath temperature Enabled if you have chosen to enter the bath temperature (Po and T option on the Analysis Conditions dialog). Enter (or confirm) the temperature for the analysis bath.

Start Begins the analyses on the specified ports.



Resume Resumes suspended analyses.

Suspend Suspends the analyses of all samples.

Skip Skips the current step on all analyses.

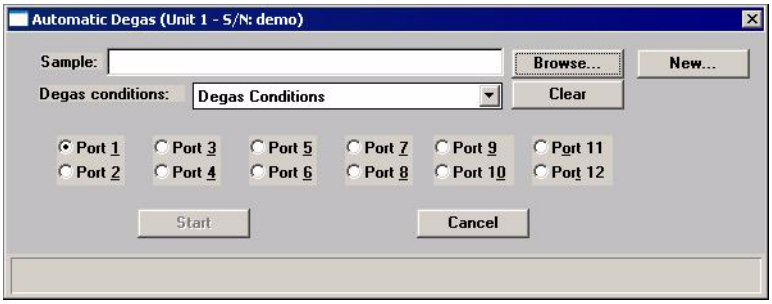
Use the Skip function with caution; the ASAP 2420 performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury.

Report Port (1 - 6)	Displays a report for the sample on the selected port.
Cancel	Cancels the analyses.
Next	When the analysis is complete, the **Start** button changes to **Next**. The **Next** button returns you to the first analysis dialog, enabling you to start another analysis.
Close	Enabled at the completion of analysis. Closes the dialog.

Start Single Degas

This option allows you to start a degas operation on one sample. This approach is useful for degassing samples without using predefined sample information files. You can, for example, create a default degas conditions file and use it to perform multiple degas operations, creating new sample files as you proceed. Use these guidelines for performing a single degas:

- One degas operation can be started at a time.
- A sample can be added to any idle port and a degas operation started. When you add a sample, the degas operation in progress will be suspended until the sample reaches the target temperature, then degassing on all ports will resume.
- Samples can be removed from any of the 12 ports without disturbing the degas operations being performed on other ports.
- A manual evacuation can be performed on any idle degas port.



Browse — Displays the Select a File for Analysis dialog allowing you to select an existing sample file. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**.)

New — Displays the Create a File for Analysis dialog allowing you to create a sample file. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31.**)

Degas Conditions — Displays the Degas Conditions file designated as the default. After you choose a sample file, this field displays the file associated with the sample file.

You can choose a different Degas Conditions file from the drop-down list if desired. If you choose a different file, the values in the current file will be overwritten with the values of the new one.

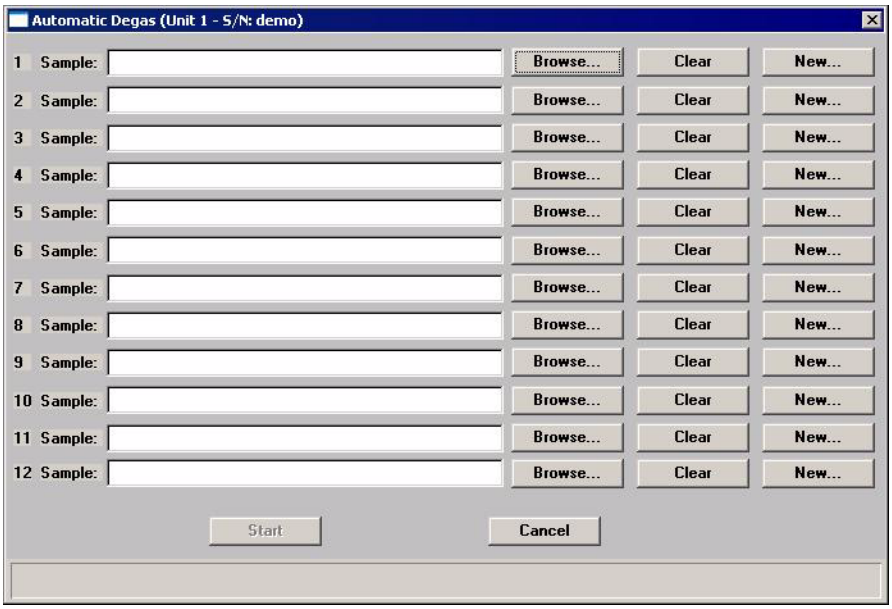
Port (1 through 12) — Enables you to choose the port on which to degas the sample.

Clear	Clears the sample file from the port assignment. Click **Browse** to choose a different file.
Start	Starts the degassing operation.
Cancel	Closes the dialog, canceling the degassing operation.

Start Multiple Degas

This option enables you to degas up to 12 samples at the same time. Use these guidelines for performing multiple degas operations:

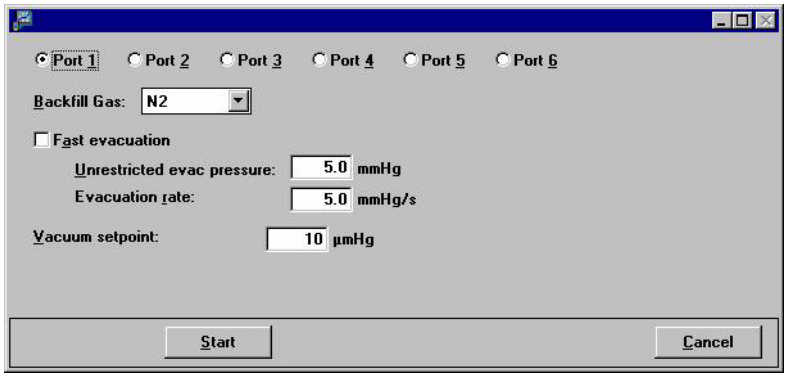
- Up to 12 degas operations can be started at a time.
- A sample can be added to any idle port and a degas operation started. When you add a sample, the degas operation in progress will be suspended until the sample reaches the target temperature, then degassing on all ports will resume.
- Samples can be removed from any of the 12 ports without disturbing the degas operations being performed on other ports.
- A manual evacuation can be performed on any idle degas port.



Browse	Enables you to select an existing sample file. The Select a File for Analysis dialog is displayed. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31**.)
New	Displays the Create a File for Analysis dialog allowing you to create a sample file. (Refer to **"Selecting Files for Degassing or Analyzing Samples" on page 3-31.**)
Clear	Clears the **Sample** field and enables you to select another sample file.
Start	Starts the degassing operation.
Cancel	Closes the dialog, canceling the degassing operation.

Evacuate Analysis Port

This option enables you to manually evacuate a single analysis port. Select **Unit > Evacuate Analysis Port**.



This option is not available if the system is performing a High Throughput or Krypton analysis.

Ports 1 through 6 — Click the port you wish to evacuate.

Backfill Gas — Select the backfill gas to be used from the drop-down list.

Fast evacuation — Select **Fast evacuation** when analyzing samples (such as pellets) that do not fluidize or shed particles under evacuation.

Unrestricted evac pressure
Evacuation rate — If you deselect **Fast evacuation, Unrestricted evac. pressure** and **Evacuation rate** become enabled. Enter the pressure value at which unrestricted sample evacuation should begin and the evacuation rate.

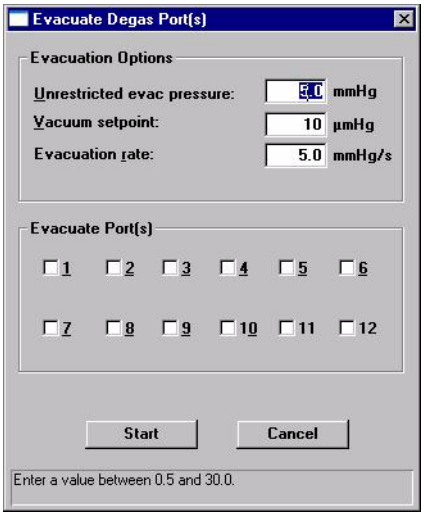
Vacuum setpoint — Allows you to specify the vacuum level to be achieved before evacuation begins. When this level is reached, the evacuation begins and continues for the amount of time specified in the Analysis Conditions dialog (refer to **"Evacuation time" on page 4-28**).

Start — Starts the evacuation.

Cancel — Cancels the evacuation.

Evacuate Degas Port

This option enables you to manually evacuate up to 12 degas ports. Select **Unit > Evacuate Degas Port**.



Ports 1 through 12 — Click the ports you wish to evacuate.

Unrestricted evac pressure — Enter the pressure value at which unrestricted sample evacuation should begin.

Vacuum setpoint — Allows you to specify the vacuum level to be achieved before evacuation begins. When this level is reached, the evacuation begins and continues for the amount of time specified in the Degas Conditions dialog (refer to **"Evacuation time" on page 4-17**

Evacuation rate — Enter the evacuation rate.

Start — Starts the evacuation.

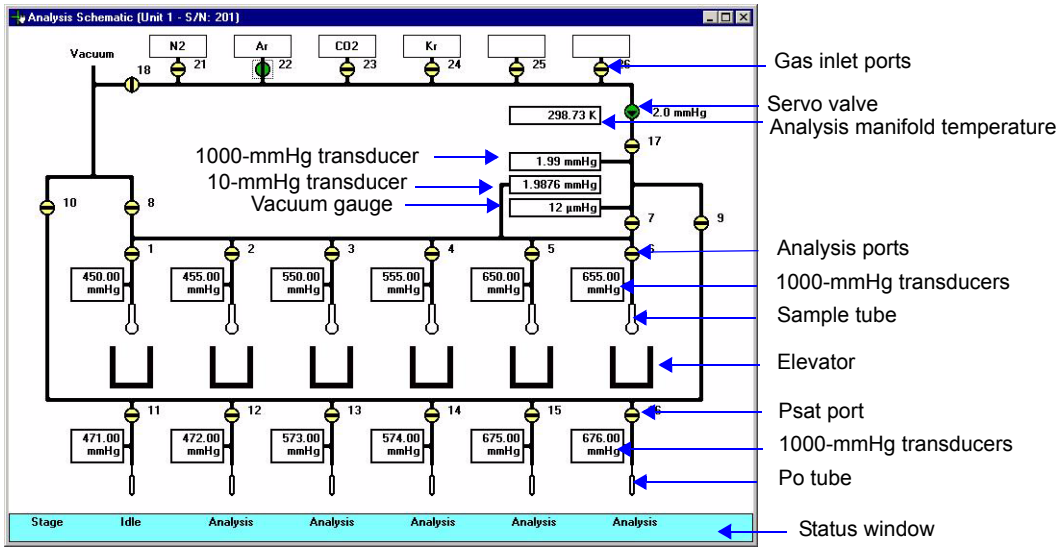
Cancel — Cancels the evacuation.

Enable Manual Control

Select this option to control certain components of your analysis or degas system manually.

Analysis System

If the analysis schematic is not displayed, select **Unit > Show Analysis Schematic**, then select **Enable Manual Control**.



When manual control is enabled, the valve symbols change color on the computer screen to indicate their status.

Green = open

Yellow = closed

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 desired component.

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

- right-click on the valve or elevator symbol to 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 the table below.

Actions: Open, Close, Pulse

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

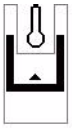
Valve	Description
1 through 6	Sample ports
7	Lower manifold isolation
8	Sample ports unrestricted vacuum
9	Po ports access
10	Po ports unrestricted vacuum
11 through 16	Po ports
17	Upper manifold isolation
18	Gas inlets unrestricted vacuum
21 through 26	Gas inlets
Unmarked	Servo

Transducers

Each sample port and Psat port contains a 1000-mmHg transducer. The transducer readings display next to the ports.

Dewar positions

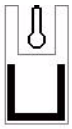
The Dewar can be raised, lowered, or stopped.



Indicates the Dewar is being raised; note the position of the arrow in the center of the Dewar symbol.

Indicates the Dewar is being lowered; note the position of the arrow in the center of the Dewar symbol.

Indicates the Dewar is stopped in the raised position.

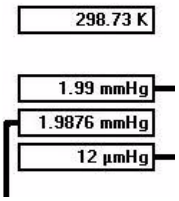


Indicates the Dewar is stopped in the lowered position.

The remainder of the components are displayed for informational purposes only; they cannot be controlled manually.

Represents the sample tube.

Represents the Psat tube.



This group of rectangles on the left side of the schematic displays the temperature, the transducer readings, and the vacuum gauge pressure.

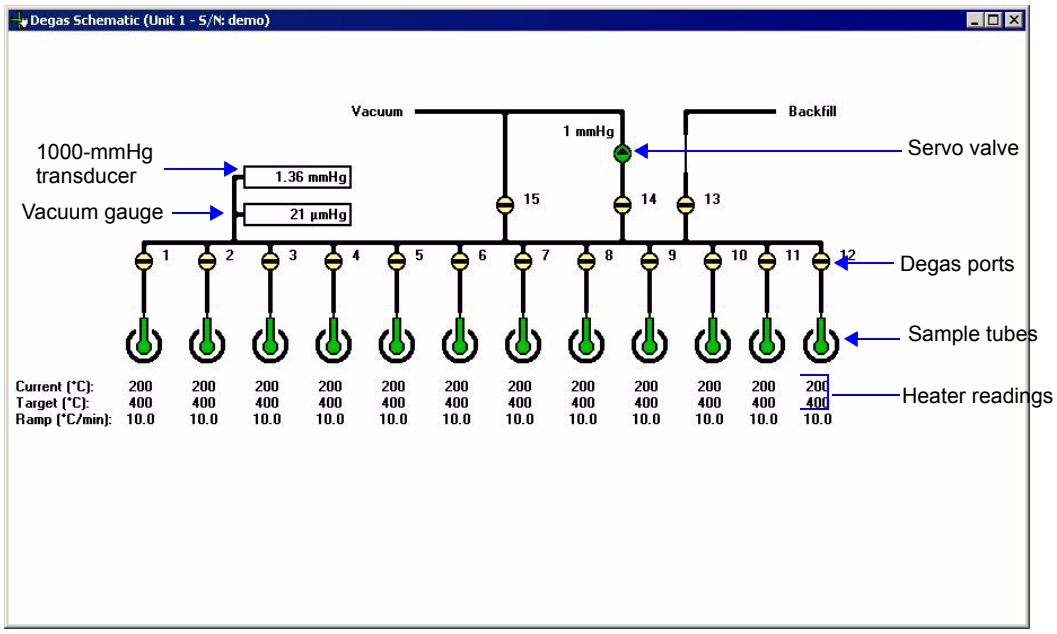
The 1000-mmHg transducer is present in all ASAP 2420 systems. The 10-mmHg transducer is shown only on units containing the Krypton option.

Status window

This is a two-line status window enabling you to view the status of the analysis on each port. The top line displays the state of the analysis, or **Idle** if an analysis is not in progress. The second line displays the point being collected during data collection.

Degas System

If the degas schematic is not displayed, select **Unit > Show Degas Schematic**, then select **Enable Manual Control**.

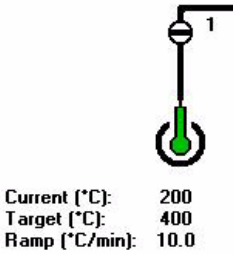


When manual control is enabled, the valve symbols change color on the monitor screen to indicate their status.

Green = open Yellow = closed

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 desired component.

Valve	Description
1 through 12 13 14 15	Sample ports Backfill Servo vacuum Unrestricted vacuum

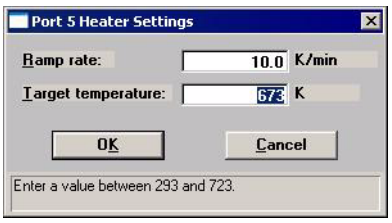


Represents the sample tube.The following are displayed below the sample tube:

- Current temperature
- Target temperature
- Ramp Rate

You can set the target temperature or disable the heater for a port as follows:

To enter the ramp rate and target temperature, right-click on the appropriate sample tube and select **Set**. The Heater Settings dialog is displayed.

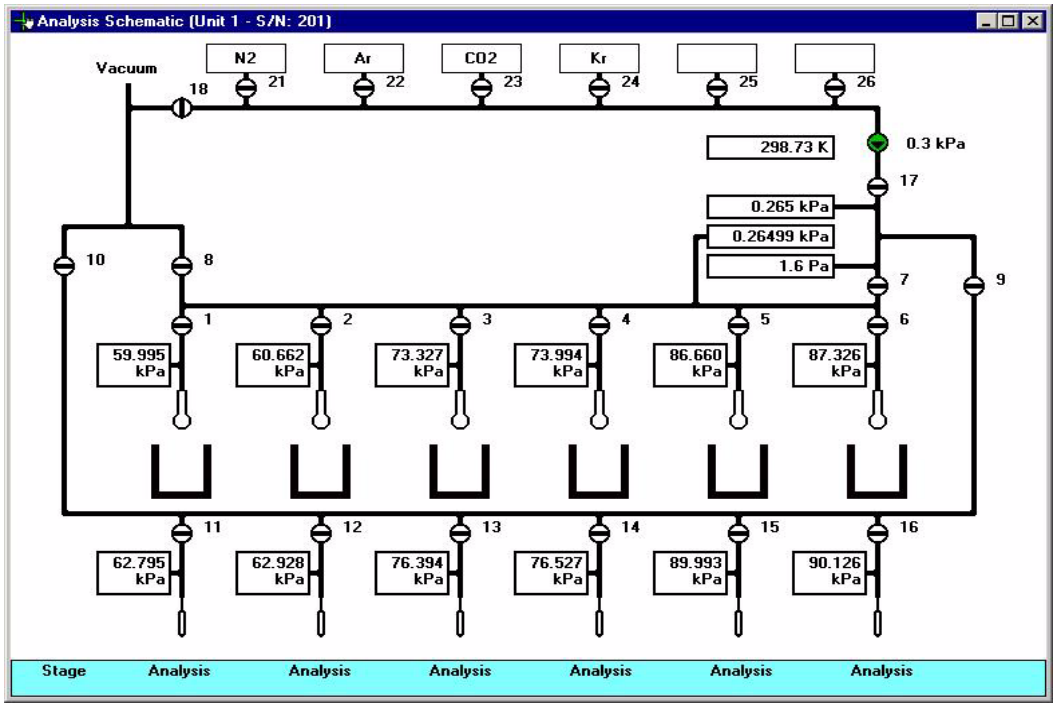


Enter the ramp rate and target temperature, then click **OK** to close the dialog.

To disable the heater for a port, right-click on the appropriate sample tube and select **Disable**. To enable a disabled heater, enter a new target temperature.

Show Analysis Schematic

Select this option to display a schematic of the ASAP 2420 analysis system. The schematic is a graphical representation of the analysis system including system valves, analysis stations, and Dewar positions. The valves and elevators can be controlled from the schematic when Manual control is enabled. Refer to **"Analysis System" on page 5-26** for an explanation of the components displayed on the analysis schematic.



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

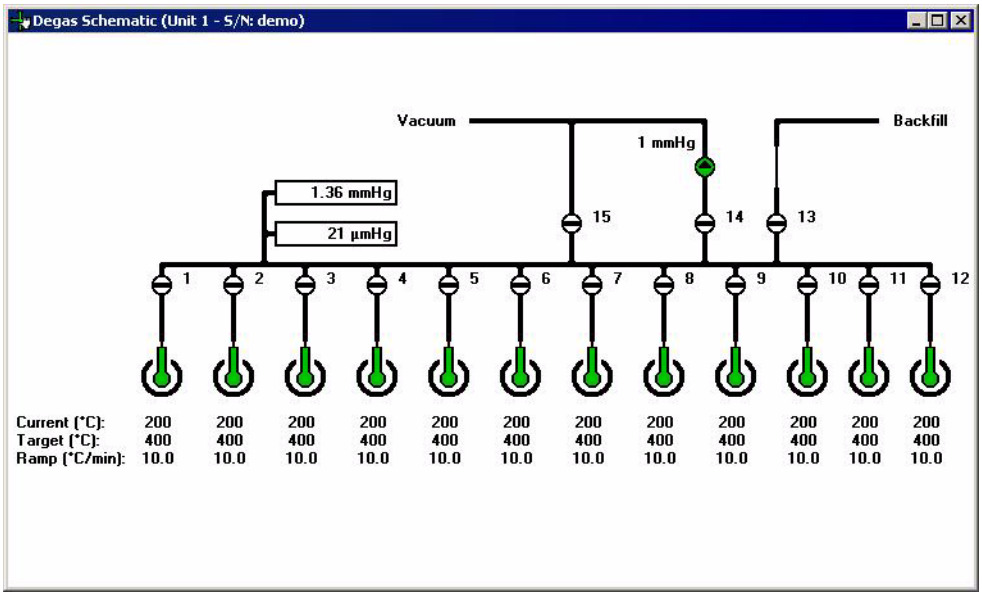
Green = open

White = closed

If you wish to change the state of a valve, you must enable manual control.

Show Degas Schematic

Select this option to display a schematic of the ASAP 2420 degas system. The schematic is a graphical representation of the plumbing system including degas valves and preparation ports. The valves and port heaters can be controlled from the schematic when Manual control is enabled. Refer to **"Degas System" on page 5-29** for an explanation of the components displayed on degas schematic



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

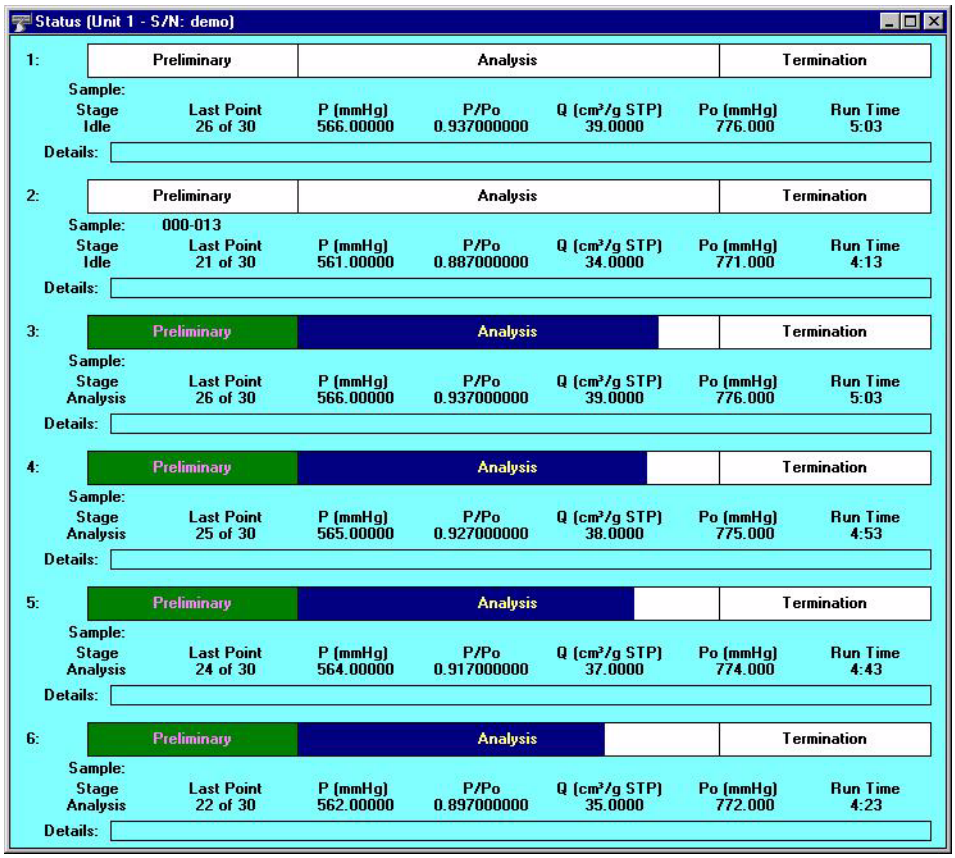
Green = open

White = closed

If you wish to change the state of a valve, you must enable manual control.

Show Analysis Status

The Show Analysis Status option enables you to view the status of all active analysis ports.



The following information is displayed for each active port.

Analysis status bar

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

- **Preliminary**: warm and cold free spaces are determined; displays green during progression
- **Analysis**: data are collected; displays blue during progression
- **Termination**: the Dewar lowers, and the sample tube is warmed and backfilled; displays gray 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 name

Stage: Stage of the analysis (as in status bar)

Last Point: Last point and number of points requested

P: Absolute pressure of last point

P/Po: Relative pressure of last point

Q(cm^3/g STP): Volume adsorbed

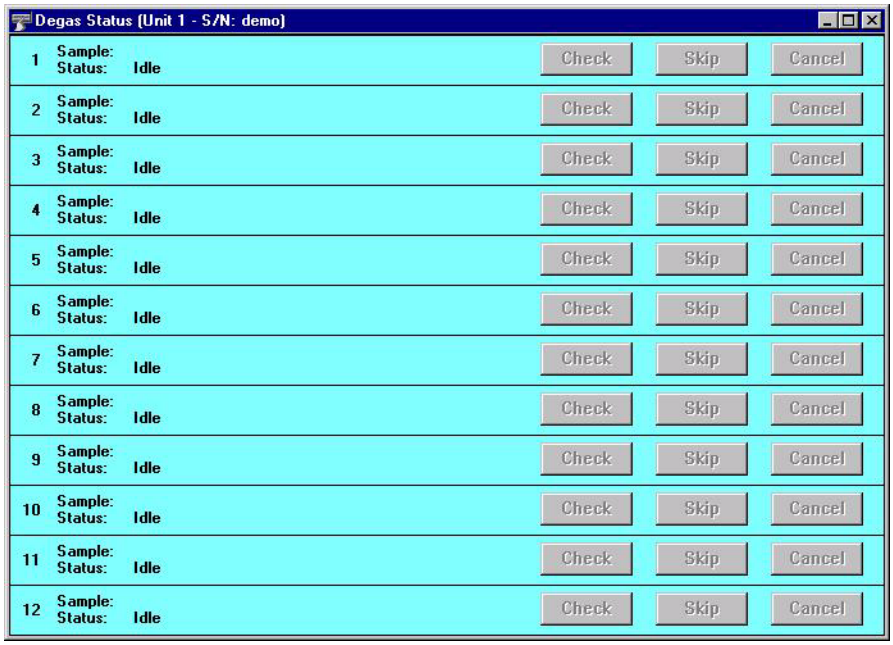
Po: Absolute pressure of last measured or calculated saturation pressure (Po)

Run Time: Elapsed time since the start of analysis

Step details Provides details of the current step of the analysis.

Show Degas Status

The Show Degas Status option enables you to view the status of all active degas ports.



Sample Status

Shows the status of the degas operation on each port.

Check

Allows you to check the outgassing rate of the samples on the selected ports; the following actions occur:

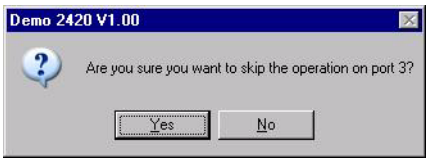
- current degassing step is suspended.
- vacuum valves are closed and the vacuum level on the selected ports is monitored.
- the Status window indicates that the degassing operation is being checked and displays the outgassing rate as it becomes available.

During the degas check, this push button changes to **Continue**. When **Continue** is clicked, the valves open and the degassing operation resumes.

If the outgassing rate indicated that the sample has been freed of contaminants (minimal pressure increases), you can click **Skip** to advance to the next state of the degassing operation. For example, if you check degassing after the setpoint is attained, **Skip** advances you to the ramping stage.

Skip

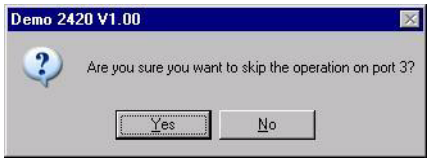
Enables you to skip the current degas operation. When you click **Skip**, the following dialog is displayed.



Click **Yes** to skip the operation or **No** to continue the operation.

Cancel

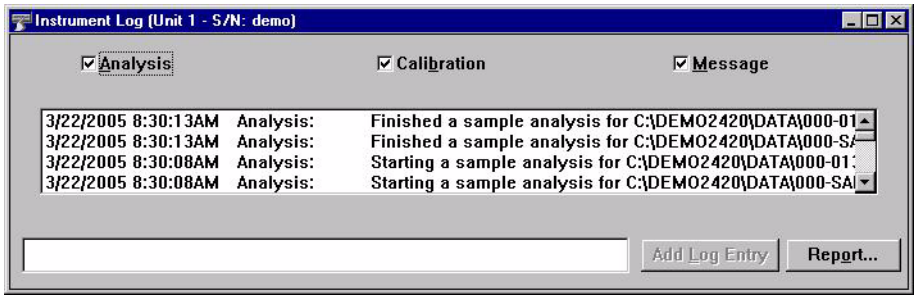
Cancels the current degas operation. When you click **Cancel**, the following dialog is displayed.



Click **Yes** to cancel the operation or **No** to continue the operation.

Show Instrument Log

Displays a log of recent analyses, calibrations, and errors or messages.



By default, this information is logged for a 60-day period for analyses and messages, and a 90-day period for calibrations.You may change the time for which this information is retained in the Unit section of the WIN2420.INI file. Simply replace the default value with the desired number.

Analysis
Calibration
Messages

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

Add Log Entry
(not enabled for Analysts)

This field is enabled for Developers only.

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 push button allows you to enter the operation; the push button is enabled when you make an entry in the field, allowing you to add the entry.

Report

Displays the Log Report Settings dialog allowing you to generate the log contents to a specified destination.

Use the **Start Date** field to specify the date at which to start the printout. This date does not limit the entries that display in the window, it only limits the entries printed on the report.

You can specify a date using one of the following methods:

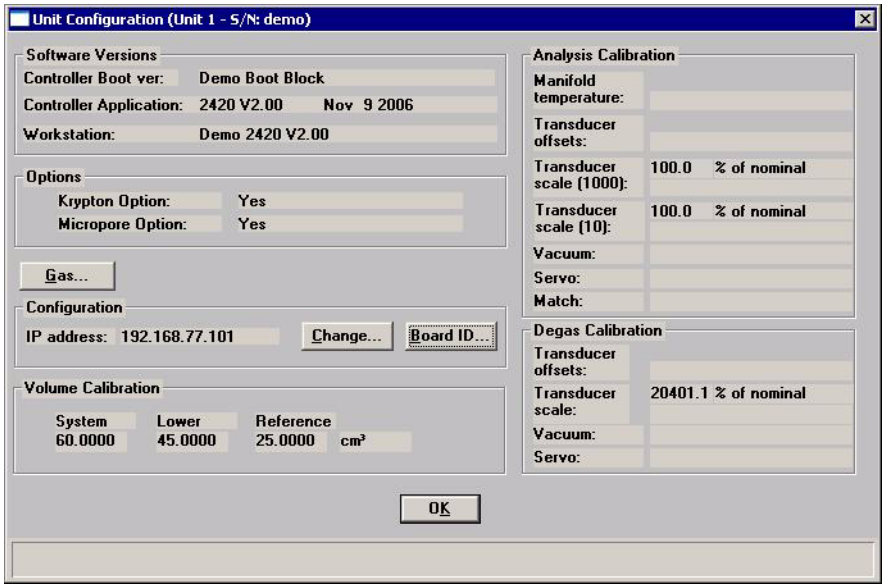
- Highlight the field (or press **F2** to clear the field) and type in the desired date.
- Press **F2** to clear the field, then **F3** to insert the current date.
- Double-click in the field (or press **F4**) to display a calendar to choose a date.

Enter the number of copies desired in the **Copies** field; you may print up to 4. This field is disabled if you are printing to a File or to the Screen.

Choose the report destination from the drop-down list in the **Destination** field. If you choose File, the **File name** field is enabled, allowing you to enter a name for the printed file.

Unit Configuration

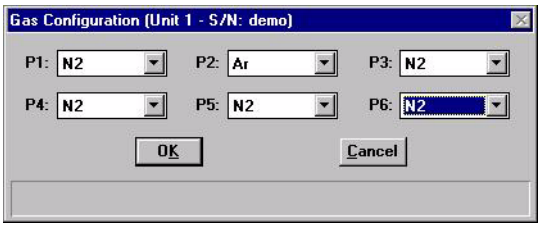
Select this option to view the current calibration settings, the date on which calibration was performed, and the software and hardware configuration of your system.



Software Versions The software versions of the Controller and ASAP 2420 System software.

Options The options installed in the ASAP 2420 system (for example, Krypton).

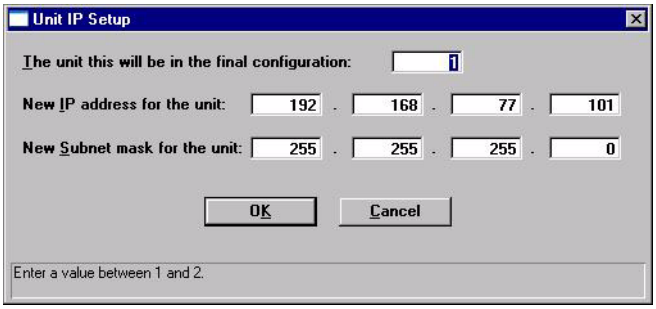
Gas Displays the Gas Configuration dialog.



This dialog allows you to choose the gases attached to each analysis port of the analyzer.

Configuration

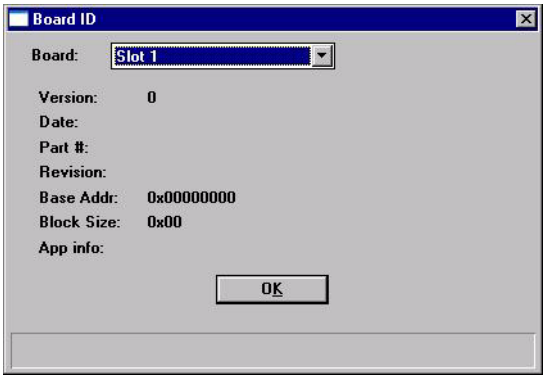
Displays the IP address of the unit. The **Change** button enables you to change the IP address. When you click **Change**, the following dialog is displayed.



Enter the IP address, then click **OK**.

Board ID

Displays the Board ID dialog so that you can view information about the board contained in the requested slot of the card cage in the instrument.



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.

Analysis Calibration

Displays analysis calibration data.

Degas Calibration

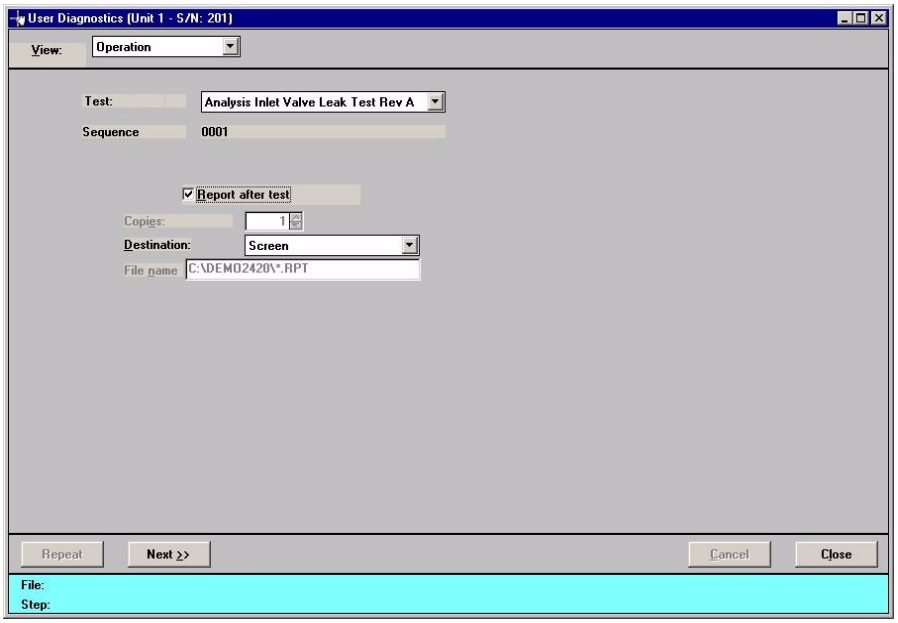
Displays degas calibration data.

Diagnostics

This option enables you to perform diagnostic tests which your service representative may request to resolve a problem. The data generated from these tests may be insignificant to you as a user, but are very helpful to your service representative. Ultimately, the results obtained from these tests in many situations can eliminate costly downtime, as well as on-site repair costs.

Each test generates a file to the **2420cfr\Service\userdiag** directory, assuming you used the default directory. Your service representative will request that you E-mail this file to him so that he may examine the data. If E-mail is unavailable, you may print the report and fax it. Your service representative will provide you with applicable destination information.

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



View

Allows you to view the current operation, the instrument schematic, or the instrument log in the User Diagnostics dialog. **Operation** is the appropriate choice for this test.

Test

Contains a list of the diagnostic tests. Click the down arrow to the right of the field to choose the test.

Analysis Inlet Valve Leak Test Rev A
Tests the analysis inlet valve for leaks; this test should be performed before any others.

Leak Test All Manifolds Rev. –
Tests for leaks in the manifolds.

SVC: Analysis Servo & Transducer Rev. B
Tests the servo operation at various pressures.

SVC: Analysis Vacuum Gauge Test Rev. B
Tests the analysis vacuum gauge.

SVC: Analysis Sample + Po Xdcr Test Rev. A
Tests the accuracy of the sample and Po transducers at various pressures.

SVC: Degas Servo Evacuation Test Rev. B
Tests the accuracy of the degas servo at various pressures.

SVC: Degas Vacuum Gauge Test Rev. A
Tests the degas vacuum gauge.

SVC: Degas Transducer Test Rev. A
Tests the linearity of the degas transducer.

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 will be 0001.SVT. Be sure to make a note of this number so you will know which file to send.

Report after test

Select this option to have a report generated automatically after the test. Refer to **"Report Settings" on page 2-22** for an explanation of output options.

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

Cancel

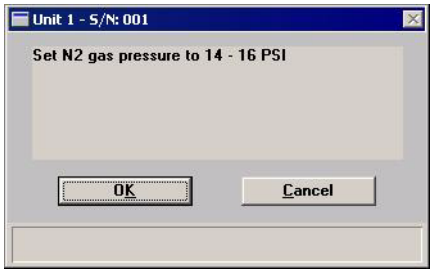
Cancels the test and closes the dialog.

Next

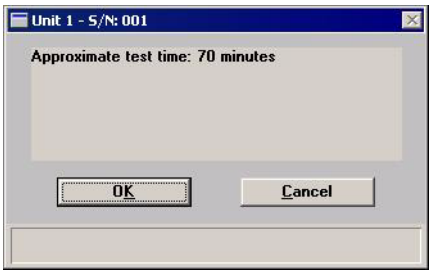
Begins the test; displays the next view of the User Diagnostics test dialog. While this dialog is displayed, you will receive a series of messages. Two types of messages may display:

Next
(continued)

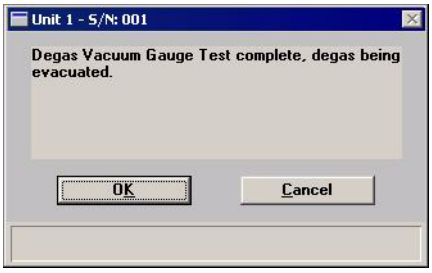
Task message: this type of message will instruct you to perform a task. After you have completed the task, click **OK** to close the dialog. This is an example of a Task message:



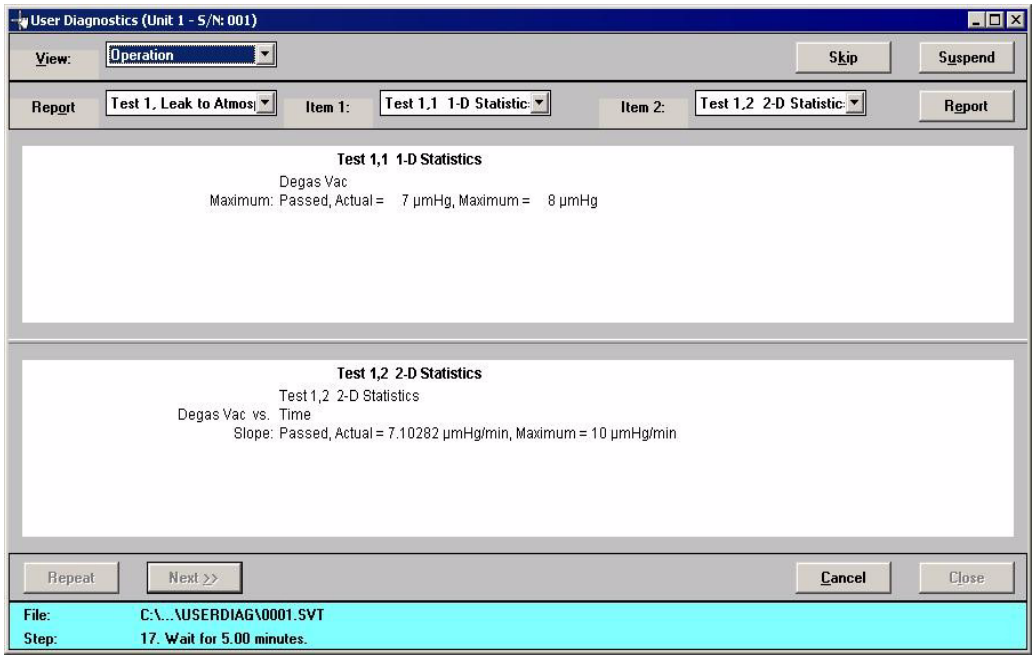
Informational message: this type of message simply provides you with information. After you have read the information, click **OK** to close the dialog. This is an example of an Informational message:



At the end of the test, you will receive a message indicating completion of the test similar to this one:



Simply click **OK** to close the dialog; all valves will close automatically.



Report (drop-down list) Contains a list of predefined reports.

Item [n[These drop-down lists contain components of the selected report. Item 1 data display in the upper pane and Item 2 data display in the lower pane.

Report (pushbutton) Enables you to generate a report.

Cancel Cancels the test.

The following push buttons become enabled when the test is complete.

Repeat Repeats the current test, using the same test number and writing over the data. Typically, this function will not be used.

Next Returns you to the first view of the User Diagnostics window allowing you to perform other diagnostics.

Close Closes the dialog.

Calibration Procedures and Service Tests

Various calibration procedures and service tests are included in the ASAP 2420 operating program. These procedures and 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.

These procedures are disabled (grayed out) on the Unit menu and can be enabled and performed only with the assistance of a trained Micromeritics service representative:

- Calibration Data
- Calibrate Analysis
- Calibrate Degas
- Service Test

6 REPORTS MENU

Description	6-3
Start Report	6-4
Close Reports	6-5
Open Report	6-5
SPC Report Options	6-6
Regression Report	6-7
Control Chart	6-11
Printed Reports	6-14
Report Examples	6-23

REPORTS MENU

This chapter describes the commands on the Reports menu; it also contains examples of reports.

Reports can be generated for data:

- collected on a sample that has completed analysis
- collected on a sample that is currently being analyzed (includes only the information collected up to the time of the report)
- that is manually entered

Description

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

Start Report — Allows you to generate a report on a completed sample analysis or on the data collected thus far for an analysis in progress.

Close Reports — Closes all open report windows.

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

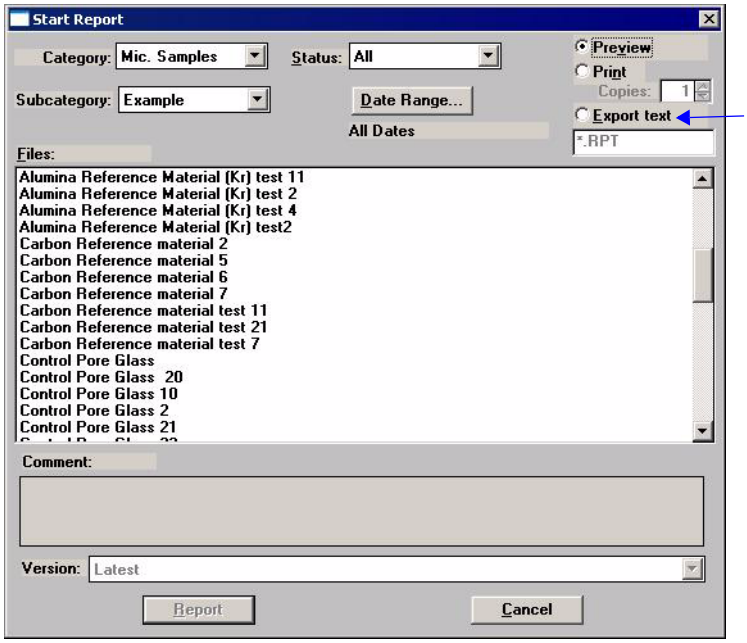
SPC Report Options — Allows you to specify the sample data to be included in SPC reports.

Regression Report — Allows you to generate a regression report.

Control Chart — Allows you to generate a control chart report.

Start Report

Select this option to generate a report on a sample analysis. Select **Start report** from the **Reports** menu to display the Start Report dialog.



Does not display if an Analyst is logged in.

Category
SubCategory

Enables you to change the files shown in the files list by selecting another directory. Refer to **"File Selection" on page 2-19**.

Status

Enables you to limit the files shown in the files list to those of a selected status. Refer to **"Selecting a File Status" on page 2-20**.

Date Range

Enables you to limit the files shown in the files list to those created in a specified date range. Refer to **"Selecting a Range of Dates" on page 2-21**.

Report Destination

Enables you to specify the report destination. The choices are: *Preview*, *Print*, and (for Developers only, *Export Text*).

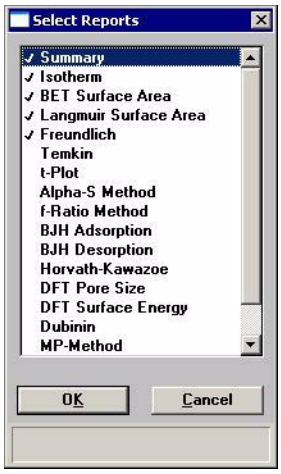
Refer to **"Report Settings" on page 2-22**.

Version

Enables you to select the version of the sample files you wish to generate a report for.

Report

Displays the Select Reports dialog.



Double-click on the report(s) you wish to select (or deselect); alternatively, you can highlight the report and press **Spacebar**. A check mark is placed before each selected report. 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 specified in each sample file will be generated.

Close Reports

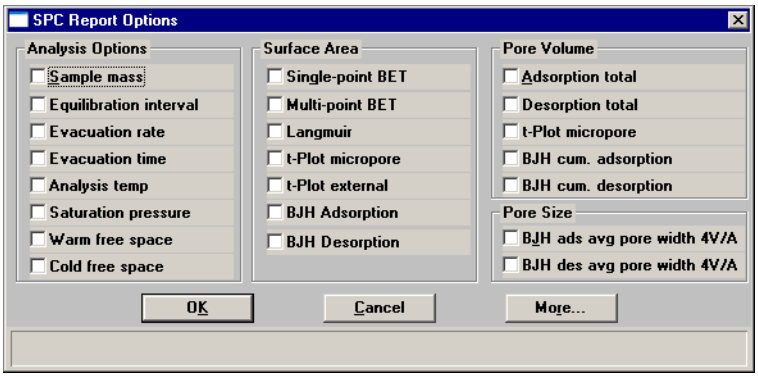
This option enables you to close all open report windows at one time. This avoids having to select close on each report window.

Open Report

Enables you to open a report that you saved from the report window.

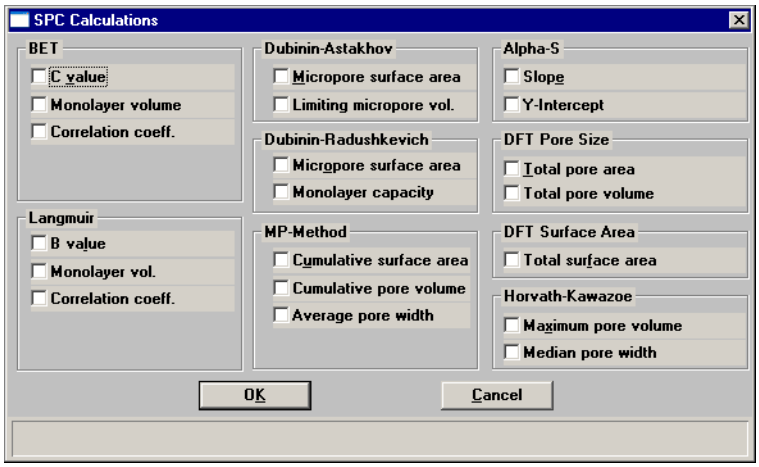
SPC Report Options

When you select SPC Report Options, the SPC Report Options dialog is displayed.



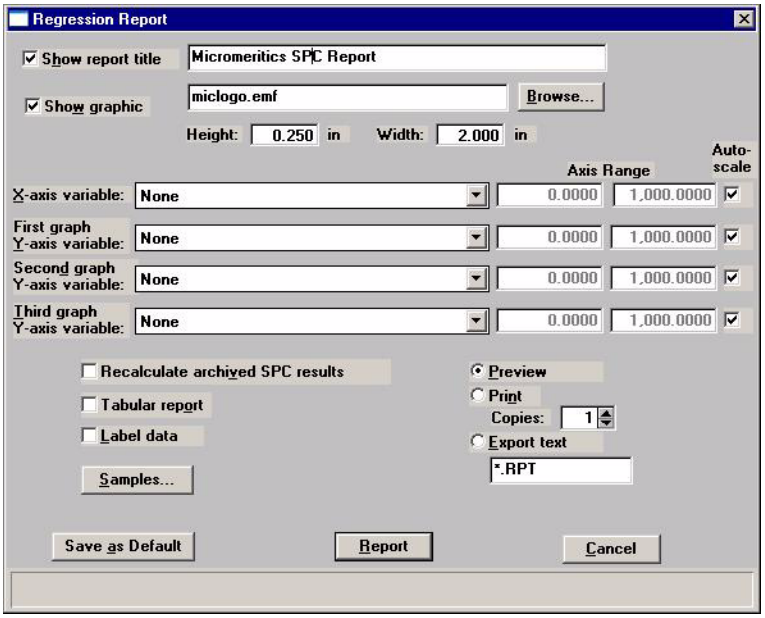
The SPC Report Options dialog lists the variables that are most frequently used for SPC reporting. You can check as many as desired. All variables selected are computed for *each* sample file used in an SPC report.

Click **More** for additional SPC calculations for the detailed reports.



Regression Report

Select this option to generate a regression report. The regression report is used to determine the interdependency between two variables. Up to three dependent variables (Y-axis) may be plotted against a single independent variable (X-axis). The degree of correlation between the variables also is reported. The graphs for the regression report are scaled so that all three fit on a single page. If you choose less than three, the graphs are scaled to fill most of the page.



Show report title

Select this option to have a title display on your report. Accept the default or enter a new title. You can enter up to 40 alphanumeric characters.

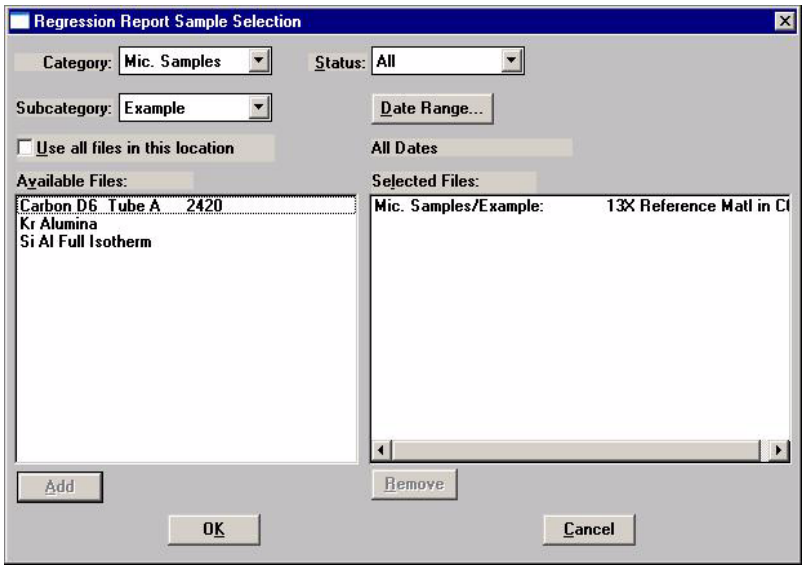
Deselect this option to omit the report title.

Show graphic

Select this option to have a graphic display above your report title. For example, you may wish to display your company logo. The graphic must be a bitmap (bmp) or enhanced metafile (emf).

Click **Browse** to choose the file, then use the **Height** and **Width** fields to specify a size. This image can be edited in the report window (when printed to the screen), or removed if desired.

X- and Y-Axes Variable fields	Allows you to designate the X- and Y-axes variables. Click on the down arrow to display a list of variables. The variables in this list are the ones you specified in the **SPC report options**. With this option, you can plot the regression of up to three Y-axis variables against the X-axis variable. The X-axis specifies the independent variable for the regression, while the Y-axes provide the dependent variables.
Axis Range	Enables you to specify the beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot. These fields are disabled if you choose **Autoscale**.
Autoscale	Allows you to have the X- and/or Y-axes scaled automatically. When scaled automatically, both axes begin at zero. The analysis program uses the highest values collected during analysis as the ending points.
Recalculate archived SPC results	Select this option to have archived SPC values recalculated. This ensures that any changes made to the SPC Report Options are included in the new report; however, it does lengthen the time required to generate the report.
Tabular report	Enables you to generate tabular, as well as graphical, data of the included samples. A tabular report contains the numeric values contributed by each sample.
Label data	Allows you to label the points on the plot to correspond with the values in the sample files.
Samples	Displays the Regression Report Sample Selection dialog, allowing you to choose the sample files you wish to have reported.



Category
SubCategory

Enables you to change the files shown in the files list by selecting another directory. Refer to **"File Selection" on page 2-19**.

Status

Enables you to limit the files shown in the files list to those of a selected status. Refer to **"Selecting a File Status" on page 2-20**.

Date Range

Enables you to limit the files shown in the files list to those created in a specified date range. Refer to **"Selecting a Range of Dates" on page 2-21**.

Use all files in this directory

Select this option to include all files from the selected directory in the report.

Add

Moves the selected file in the **Available Files** pane to the **Selected Files** pane. Alternatively, you can simply double-click on the desired file(s). You can select multiple files by holding down **Ctrl** while making your selections. You can include up to 200 sample files.

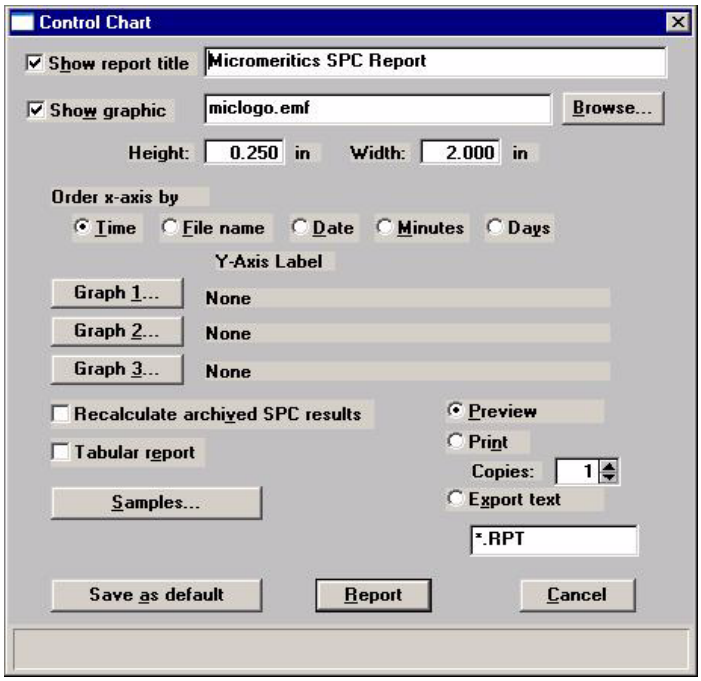
Remove

Removes the selected file from the **Selected Files** pane and places it back into the **Available Files** pane.

Preview **Print** **Export text**	Enables you to specify the report destination. Refer to **"Report Settings" on page 2-22**.
Save As Default	Saves the current definition of the report as the default.
Report	Generates the report.

Control Chart

This option enables you to generate a control chart report which plots the changes in a statistic.



Show report title

Select this option to have a title display on your report. Accept the default or enter a new title. You can enter up to 40 alphanumeric characters.

Deselect this option to omit the report title.

Show graphic

Select this option to have a graphic display above your report title. For example, you may wish to display your company logo. The graphic must be a bitmap (bmp) or enhanced metafile (emf).

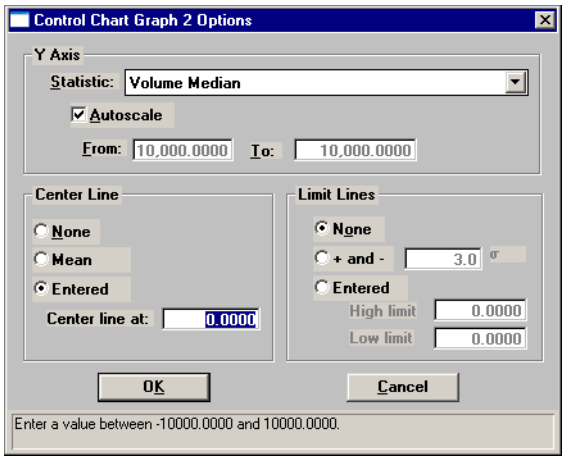
Click **Browse** to choose the file, then use the **Height** and **Width** fields to specify a size. This image can be edited in the report window (when printed to the screen), or removed if desired.

X-axis Order By

Enables you to choose the order in which X-axis statistics are placed. You can have them placed by **Time**, **File name**, **Date**, **Minutes**, or **Days**.

Graph [n]

Displays the Control Chart Graph [n] Options dialog, allowing you to define the Y-axis of each graph.



Statistic

This drop-down list displays the SPC variables selected on the SPC Report Options dialog. The variable you choose will be plotted against time.

Autoscale

Allows you to have the Y-axis scaled automatically. If you wish to specify a range, deselect this option and enter a range in the **From** and **To** fields.

Center Line

Displays placement options for the variable's optional value. Choose *Entered* to specify placement of the line.

Limit Lines

Displays the options available for limiting lines. You can have the lines placed at some multiple of the standard deviation or at specified positions (*Entered*).

When you select *Entered*, the **High limit** and **Low limit** fields are enabled, allowing you to enter appropriate values.

Tabular report

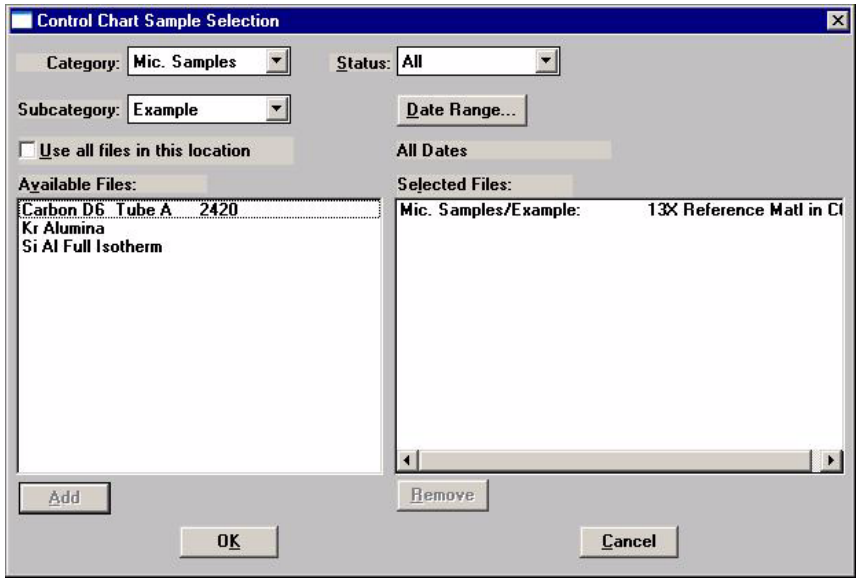
Allows you to generate tabular, as well as graphical, data of the included samples. A tabular report contains the numeric values contributed by each sample.

Recalculate archived SPC results

Select this option to have archived SPC values recalculated. This ensures that any changes made to the SPC Report Options are included in the new report. It also lengthens the time required to generate the report.

Samples

Displays the Control Chart Sample Selection dialog, allowing you to choose the sample files on which you wish to report.



This dialog functions in the same manner as the Regression Report Sample Selection Dialog explained on page **6-8**.

Preview
Print
Export text

Enables you to specify the report destination.

Refer to **"Report Settings" on page 2-22**.

Save As Default

Saves the current definition of the report as the default.

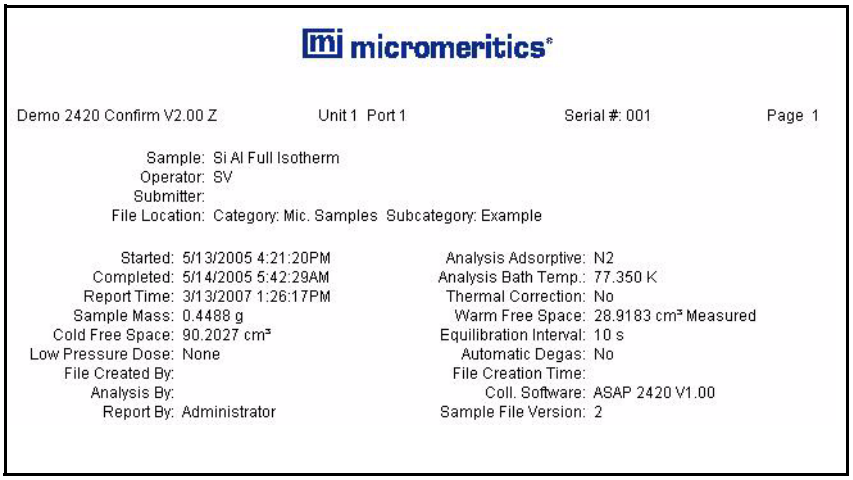
Report

Generates the report.

Printed Reports

Header

All printed reports (either to the screen or to a printer) contain a header displaying file statistics, as shown in the following example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Demo 2420 Confirm V2.00 Z Unit 1 Port 1 Serial #: 001 Page 1

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location: Category: Mic. Samples Subcategory: Example

Started:	5/13/2005 4:21:20PM	Analysis Adsorptive:	N2
Completed:	5/14/2005 5:42:29AM	Analysis Bath Temp.:	77.350 K
Report Time:	3/13/2007 1:26:17PM	Thermal Correction:	No
Sample Mass:	0.4488 g	Warm Free Space:	28.9183 cm³ Measured
Cold Free Space:	90.2027 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Automatic Degas:	No
File Created By:		File Creation Time:	
Analysis By:		Coll. Software:	ASAP 2420 V1.00
Report By:	Administrator	Sample File Version:	2

The report header may contain a graphic (such as company logo) and/or title if specified by the user. The header also contains:

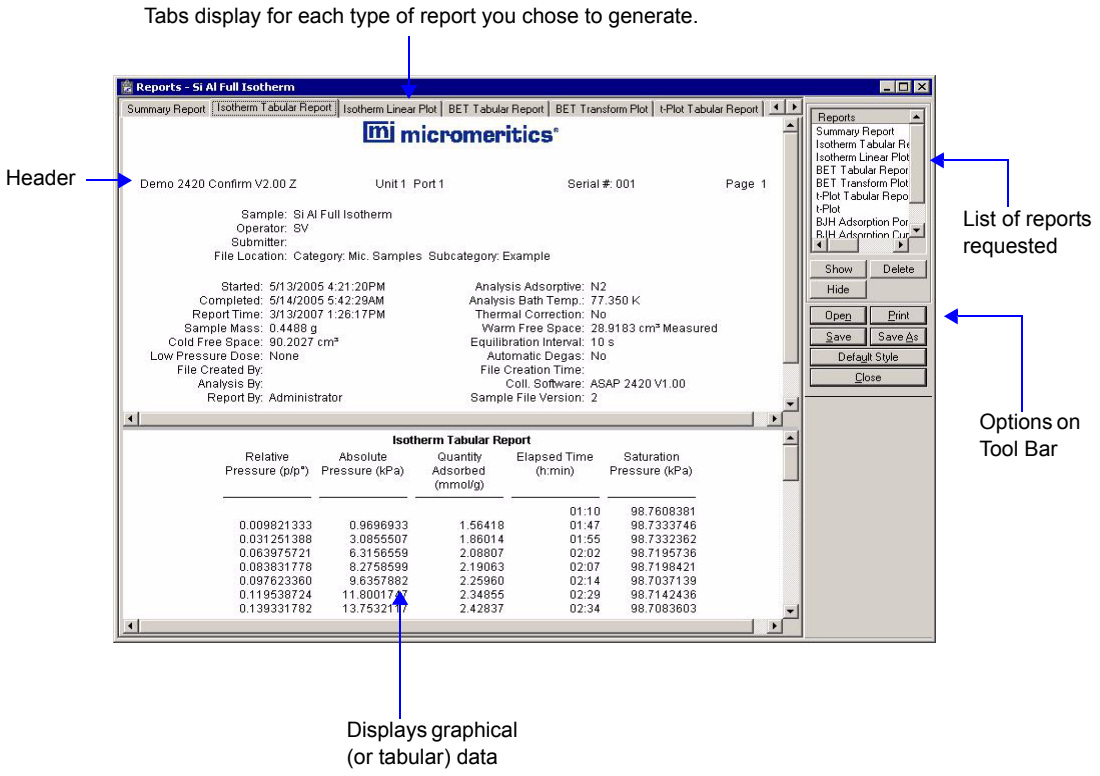
- The measurement principle and calculation method used in compiling the report.
- Sample and instrument statistics such as date and time of analysis, analysis conditions, and background data for tabular and graphical reports.
- Notes of any changes to the sample file which occur after analysis (excluded in summary reports).

Onscreen Reports

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

- a tool bar
- shortcut menus
- zoom feature
- axis cross-hairs

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.

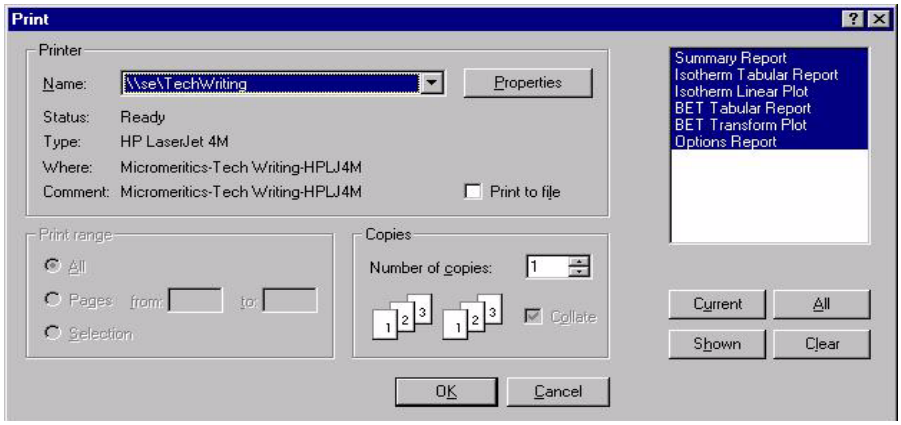
Show Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible.

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 from the report window. The report's associated tab is also removed.

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 which reports to print, push buttons are provided beneath the report window; or you can make your selection by clicking on the desired reports.

Current selects the report displayed in the report window.

Shown selects only the shown reports; any non-highlighted 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. The file will be given an **rep** extension. If you wish to specify a name and/or specific reports to save, use the **Save As** push button.

Save As

Saves all or specified reports from the currently open file. The push buttons displayed on this dialog perform in the same manner as the print dialog (explained above).

Reports can be saved in three different formats:

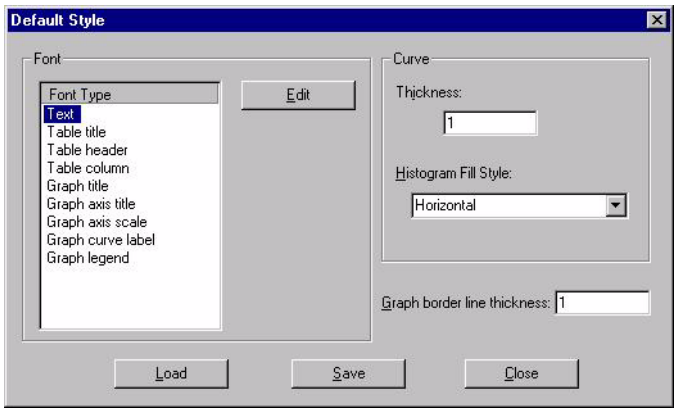
Report system (.rep)*: Saved in a format which allows you to reopen the file using the push button on the Report window tool bar.

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 desired element and click **Edit**; a font dialog is displayed so that you can specify the desired font and attributes.

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.
Close	Closes the dialog and applies the changes. If you clicked **Save**, the changes become the defaults. If you did not click **Save**, the changes apply to the current report only.

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 the columns that have not been hidden, enabling you to select the one you wish to hide.
Show column	Displays a list of all hidden columns, enabling you to select the one you wish 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 function is useful for returning to a full view after having zoomed in.

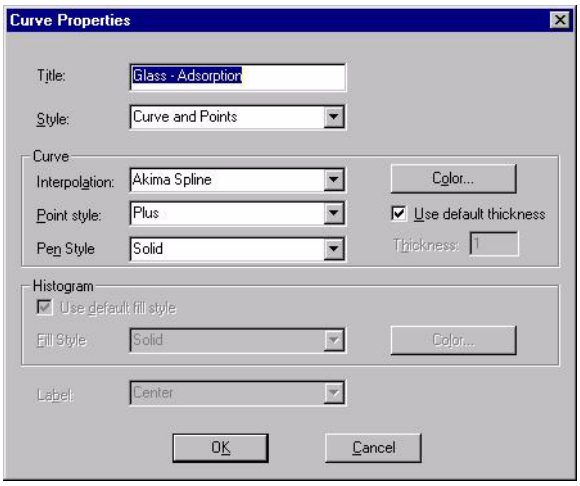
Redraw Sets axis boundaries to its original view. You can also use this function to remove cross-hairs.

Show curve Shows any curves that have been hidden. This option is disabled (grayed) if no curves have been hidden.

Hide curve Hides (removes from view) any 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. You can edit the title if desired.

Style

This drop-down list contains the styles in which collected data can be displayed.

Choices: Curve, Histogram, Points, Curve and Points

Curve group box

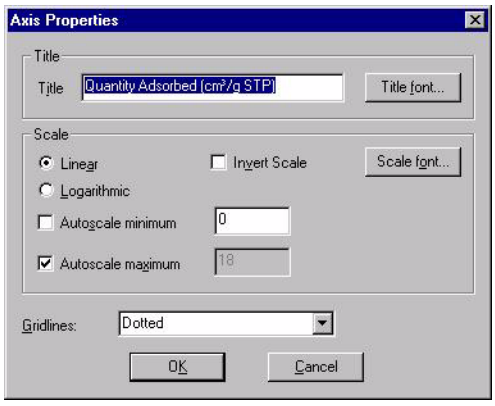
Contains options for curves and points. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color. The options in this group box are disabled if Histogram is chosen in the Style drop-down list.

Histogram group box

The options in this group box are enabled when you choose Histogram as the style for collected data. The choices in the drop-down list allow you to choose the type of fill for the Histogram, then you can click **Color** to select the fill color.

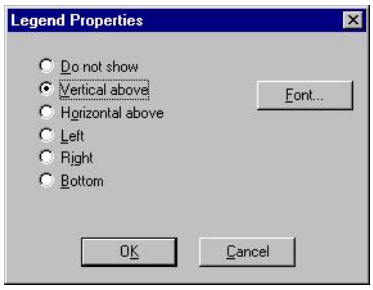
Edit axis

Displays the Axis Properties dialog, allowing you to edit axis properties.



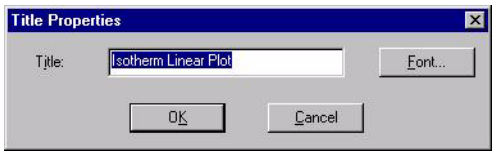
Edit legend

Displays the Legend Properties dialog, allowing you to edit the placement of the legend. You can also edit the font if desired.



Edit title

Displays the Title Properties dialog, allowing you to edit the current graph's title and font.



Copy as metafile

Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

Copy as text

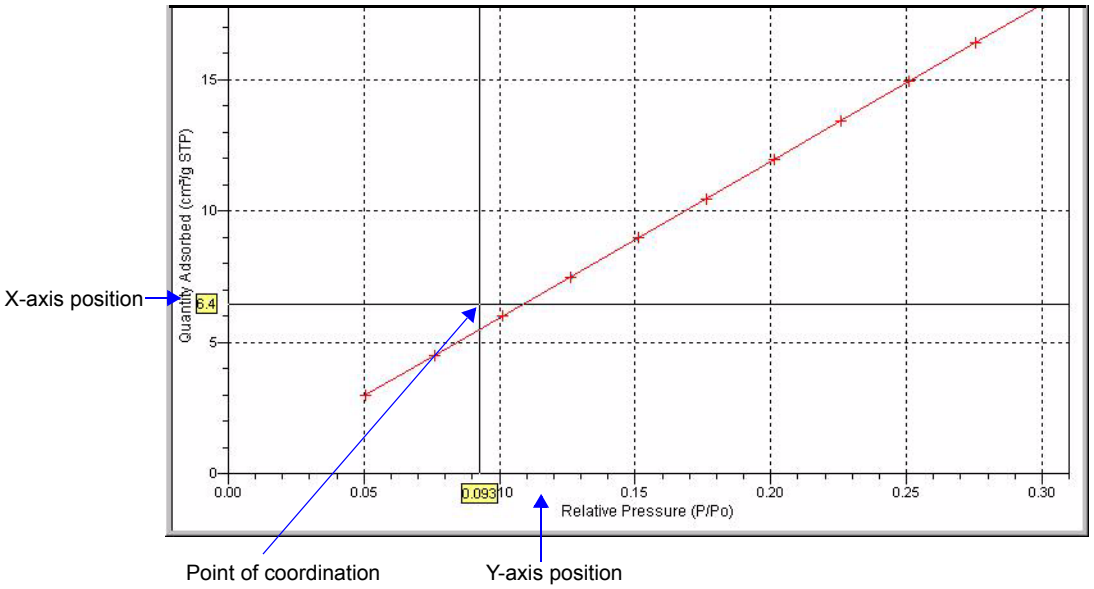
Copies the data used to generate the graph as a series of tab-delimited columns of text.

Zoom Feature

A zoom feature is included with the report system so that you can zoom in to examine fine details. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a box) across the area you wish to view; then release the button. The enlarged area immediately fills the graph area. Right-click in the graph area and choose **Autoscale** from the shortcut menu to return to the normal view.

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 desired area of the graph.



To remove cross-hair lines and return to the normal view, right-click in the graph area and choose **Autoscale** or **Redraw** from the shortcut menu. Alternatively, you can click outside of the graph area.

Report Examples

The remainder of this chapter contains samples of some of the reports that can be generated by the ASAP 2420 Program. Most of the reports can be varied through entries on the report options screens.

Summary Report

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ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 1

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2

Summary Report

Surface Area

Single point surface area at p/p° = 0.199440139: 207.4253 m²/g

BET Surface Area: 214.8064 m²/g

t-Plot Micropore Area: 17.8892 m²/g

t-Plot External Surface Area: 196.9172 m²/g

BJH Adsorption cumulative surface area of pores between 17.000 Å and 3000.000 Å width: 235.048 m²/g

BJH Desorption cumulative surface area of pores between 17.000 Å and 3000.000 Å width: 294.0750 m²/g

Pore Volume

Single point adsorption total pore volume of pores less than 678.748 Å width at p/p° = 0.970845743: 0.591128 cm³/g

t-Plot micropore volume: 0.006126 cm³/g

BJH Adsorption cumulative volume of pores between 17.000 Å and 3000.000 Å width: 0.602834 cm³/g

BJH Desorption cumulative volume of pores between 17.000 Å and 3000.000 Å width: 0.602275 cm³/g

Pore Size

Adsorption average pore width (4V/A by BET): 110.0765 Å

BJH Adsorption average pore width (4V/A): 102.589 Å

BJH Desorption average pore width (4V/A): 81.921 Å

Isotherm Tabular Report

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ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 2

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2

Isotherm Tabular Report

Relative Pressure (p/p°)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm³/g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
			01:10	740.767029
0.009821333	7.273296	35.0596	01:47	740.561035
0.031251388	23.143528	41.6929	01:55	740.559998
0.063975721	47.371304	46.8006	02:02	740.457520
0.083831778	62.074039	49.0982	02:07	740.459534
0.097623360	72.274338	50.6430	02:14	740.338562
0.119538724	88.508568	52.6347	02:29	740.417542
0.139331782	103.157547	54.4218	02:34	740.373413
0.159275382	117.939911	56.1533	02:39	740.477966
0.179339804	132.792587	57.8457	02:43	740.452393
0.199440139	147.679398	59.5195	02:49	740.469788
0.244485099	181.058899	63.2427	02:54	740.572327
0.300088255	222.227203	67.9350	03:01	740.539490
0.351194417	260.048126	72.5227	03:07	740.467712
0.397428153	294.249725	77.0333	03:14	740.384705
0.446843769	330.798737	82.3558	03:22	740.300659
0.495903120	367.073700	88.3952	03:30	740.212524
0.545068877	403.512054	95.4910	03:39	740.295532
0.601443665	445.258392	105.8225	03:55	740.316040
0.643425805	476.339111	115.7954	04:07	740.317078
0.694475443	514.024536	132.7941	04:23	740.162292
0.750453643	555.508240	164.3134	04:52	740.229919
0.793592023	587.400696	210.2867	05:21	740.179688
0.813675751	602.410583	242.8590	05:46	740.357056
0.843925714	624.702515	302.8170	06:22	740.234009
0.873312000	646.319214	342.2581	06:52	740.078247
0.894852183	662.047791	354.9357	07:07	739.840393
0.923313742	683.124695	366.8412	07:23	739.861938
0.953308457	705.127075	377.1246	07:38	739.663086
0.970845743	718.046021	382.1620	07:47	739.608765
0.977999548	723.356079	383.8543	07:52	739.628235
0.988348899	730.975281	385.9007	07:58	739.592346
0.993743344	735.043396	386.8768	08:02	739.671265
0.973494862	720.053223	385.9677	08:05	739.657959
0.939081215	694.570007	383.5833	08:11	739.627197
0.913527446	675.618225	381.3738	08:16	739.570801
0.900212206	665.789124	379.9591	08:21	739.591309
0.877004835	648.631470	376.8491	08:28	739.598511
0.854330480	631.809875	372.3142	08:37	739.538025
0.830424484	614.075134	365.7459	08:47	739.471375
0.806468393	596.340454	356.6952	09:00	739.446777
0.749751161	554.132874	234.1025	10:21	739.089050

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ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 3

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2

Isotherm Tabular Report

Relative Pressure (p/p°)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm³/g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
0.705809373	521.674805	157.8970	11:07	739.115723
0.648049073	478.890228	121.9325	11:35	738.972168
0.606266563	447.968750	108.3844	11:52	738.897339
0.555561657	410.615234	97.5302	12:04	739.099304
0.492429196	363.942474	87.7061	12:17	739.075745
0.439131978	324.503174	81.0359	12:28	738.965027
0.404117748	298.661194	77.1729	12:35	739.044983
0.352580177	260.553802	72.0455	12:43	738.991638
0.301908818	223.130997	67.4427	12:50	739.067505
0.251732667	186.081497	63.1567	12:56	739.202820
0.202578204	149.738281	59.0576	13:02	739.162842
0.142650294	105.451157	53.9680	13:09	739.228455

Isotherm Linear Plot

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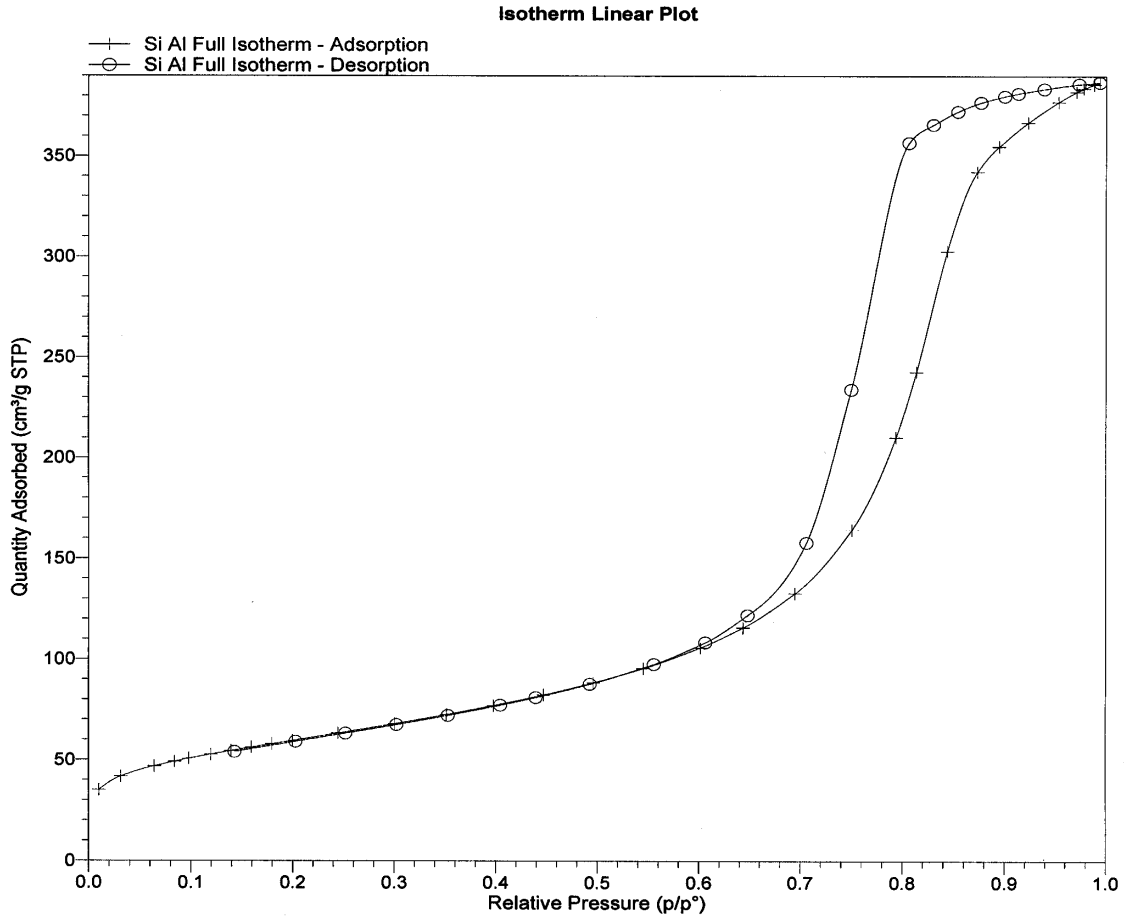
ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 4

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2



Bet Surface Area

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ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 5

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2

BET Surface Area Report

BET Surface Area: 214.8064 ± 0.1916 m²/g
Slope: 0.020089 ± 0.000018 g/cm³ STP
Y-Intercept: 0.000176 ± 0.000002 g/cm³ STP
C: 114.858651
Qm: 49.3445 cm³/g STP
Correlation Coefficient: 0.9999976
Molecular Cross-Sectional Area: 0.1620 nm²

Relative Pressure (p/p°)	Quantity Adsorbed (cm³/g STP)	1/[Q(p°/p - 1)]
0.063975721	46.8006	0.001460
0.083831778	49.0982	0.001864
0.097623360	50.6430	0.002136
0.119538724	52.6347	0.002579
0.139331782	54.4218	0.002975
0.159275382	56.1533	0.003374
0.179339804	57.8457	0.003778
0.199440139	59.5195	0.004186

Bet Surface Area Plot

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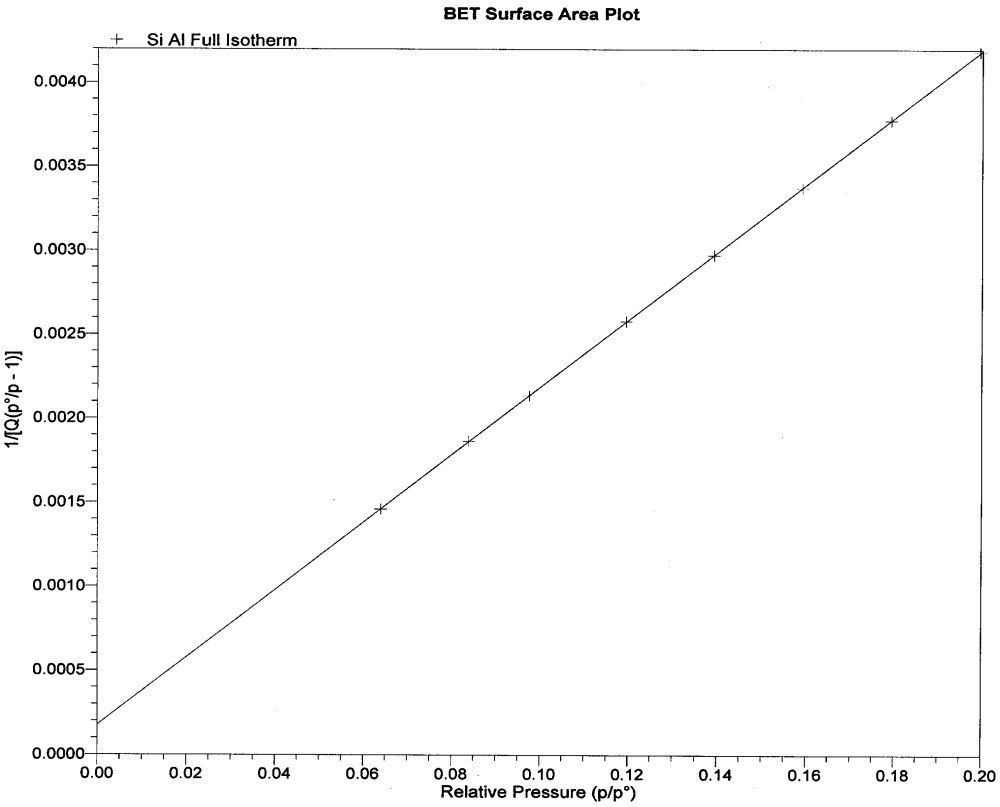
Unit 1 Port 1 Serial #: 001 Page 6

Si Al Full Isotherm

Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2



t-Plot Report

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ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 7

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2

t-Plot Report

Micropore Volume: 0.006126 cm³/g
Micropore Area: 17.8892 m²/g
External Surface Area: 196.9172 m²/g
Slope: 12.730616 ± 0.045700 cm³/g·Å STP
Y-Intercept: 3.960189 ± 0.185495 cm³/g STP
Correlation Coefficient: 0.999961
Surface Area Correction Factor: 1.000
Density Conversion Factor: 0.0015468
Total Surface Area (BET): 214.8064 m²/g
Thickness Range: 3.5000 Å to 5.0000 Å
Thickness Equation: Harkins and Jura

$$t = [\ 13.99 / (\ 0.034 - \log(p/p^\circ)\ )\ ]\ ^\wedge\ 0.5$$

Relative Pressure (p/p°)	Statistical Thickness (Å)	Quantity Adsorbed (cm³/g STP)
0.009821333	2.6176	35.0596
0.031251388	3.0149	41.6929
0.063975721	3.3753	46.8006
0.083831778	3.5492	49.0982
0.097623360	3.6599	50.6430
0.119538724	3.8244	52.6347
0.139331782	3.9648	54.4218
0.159275382	4.1010	56.1533
0.179339804	4.2342	57.8457
0.199440139	4.3652	59.5195
0.244485099	4.6545	63.2427
0.300088255	5.0128	67.9350
0.351194417	5.3518	72.5227
0.397428153	5.6727	77.0333
0.446843769	6.0371	82.3558
0.495903120	6.4278	88.3952
0.545068877	6.8569	95.4910
0.601443665	7.4098	105.8225
0.643425805	7.8765	115.7954

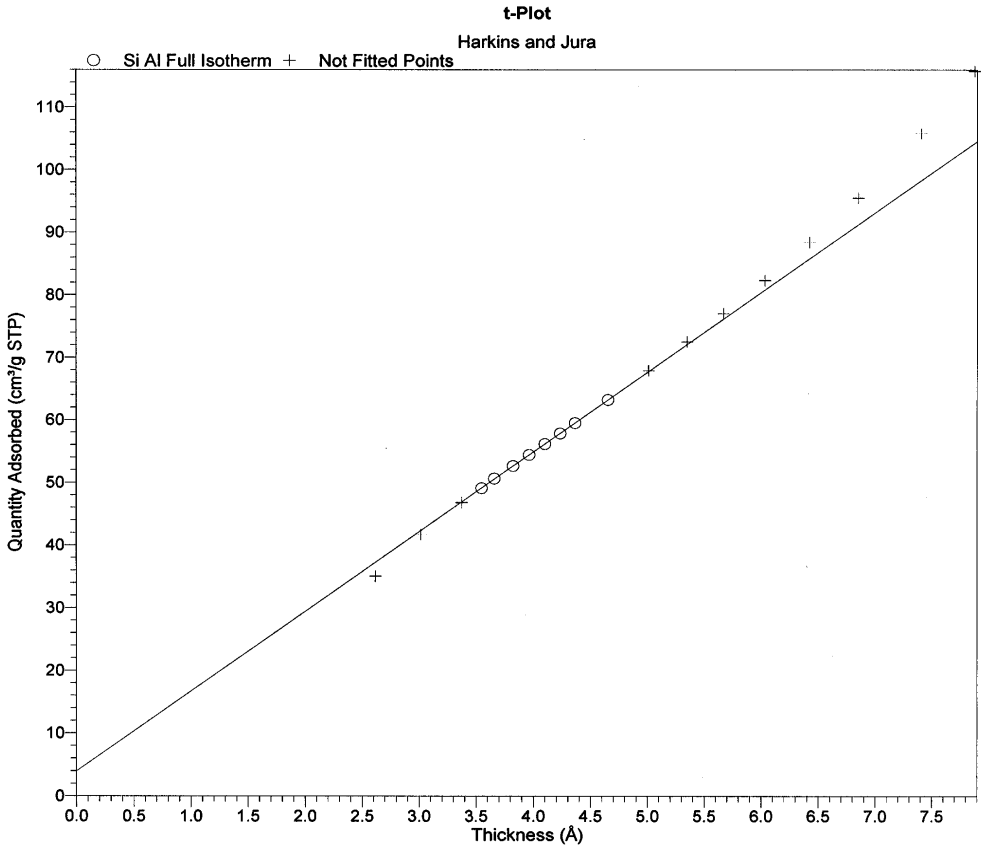
t-Plot

micromeritics

ASAP 2420 Confirm V1.00 Unit 1 Port 1 Serial #: 001 Page 8

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started:	5/13/2005 4:21:20PM	Analysis Adsorptive:	N2
Completed:	5/14/2005 5:42:29AM	Analysis Bath Temp.:	77.350 K
Report Time:	10/4/2005 1:54:54PM	Thermal Correction:	No
Sample Mass:	0.4488 g	Warm Free Space:	28.9183 cm³ Measured
Cold Free Space:	90.2027 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Automatic Degas:	No
File Created By:		File Creation Time:	
Analysis By:		Coll. Software:	ASAP 2420 V1.00
Report By:	gea	Sample File Version:	2



BJH Adsorption Pore Distribution

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Unit 1 Port 1 Serial #: 001 Page 9

ASAP 2420 Confirm

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location: Category: Mic. Samples Subcategory: Example

Started:	5/13/2005 4:21:20PM	Analysis Adsorptive:	N2
Completed:	5/14/2005 5:42:29AM	Analysis Bath Temp.:	77.350 K
Report Time:	11/10/2005 11:11:36AM	Thermal Correction:	No
Sample Mass:	0.4488 g	Warm Free Space:	28.9183 cm³ Measured
Cold Free Space:	90.2027 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Automatic Degas:	No
File Created By:		File Creation Time:	
Analysis By:		Coll. Software:	ASAP 2420 V1.00
Report By:	Diane	Sample File Version:	1

BJH Adsorption Pore Distribution Report

Faas Correction
Harkins and Jura
t = [13.99 / (0.034 - log(P/Po))] ^ 0.5

Diameter Range: 17.000 Å to 3000.000 Å
Adsorbate Property Factor: 9.53000 Å
Density Conversion Factor: 0.0015468
Fraction of Pores Open at Both Ends: 0.00

Pore Diameter Range (Å)	Average Diameter (Å)	Incremental Pore Volume (cm³/g)	Cumulative Pore Volume (cm³/g)	Incremental Pore Area (m²/g)	Cumulative Pore Area (m²/g)
3075.7 - 1664.0	1978.8	0.001576	0.001576	0.032	0.032
1664.0 - 892.4	1060.3	0.003400	0.004976	0.128	0.160
892.4 - 678.6	755.9	0.002873	0.007849	0.152	0.312
678.6 - 430.4	499.3	0.008869	0.016717	0.710	1.023
430.4 - 267.3	310.7	0.019112	0.035829	2.461	3.483
267.3 - 197.5	221.4	0.023162	0.058991	4.185	7.669
197.5 - 165.1	178.2	0.025648	0.084639	5.758	13.427
165.1 - 135.0	146.8	0.083884	0.168523	22.864	36.290
135.0 - 113.6	122.3	0.132139	0.300662	43.235	79.525
113.6 - 102.7	107.5	0.072017	0.372679	26.788	106.313
102.7 - 85.0	92.0	0.100338	0.473016	43.615	149.928
85.0 - 69.2	75.3	0.063001	0.536018	33.470	183.398
69.2 - 58.8	63.1	0.028654	0.564672	18.174	201.573
58.8 - 52.2	55.0	0.013916	0.578587	10.113	211.686
52.2 - 45.0	48.0	0.011758	0.590345	9.806	221.492
45.0 - 39.9	42.1	0.005884	0.596229	5.593	227.085
39.9 - 35.6	37.5	0.003717	0.599947	3.970	231.055
35.6 - 31.9	33.5	0.002165	0.602112	2.586	233.641
31.9 - 28.8	30.1	0.001075	0.603187	1.427	235.069
28.8 - 25.7	27.0	0.000277	0.603463	0.409	235.478

BJH Adsorption Cumulative Pore Volume

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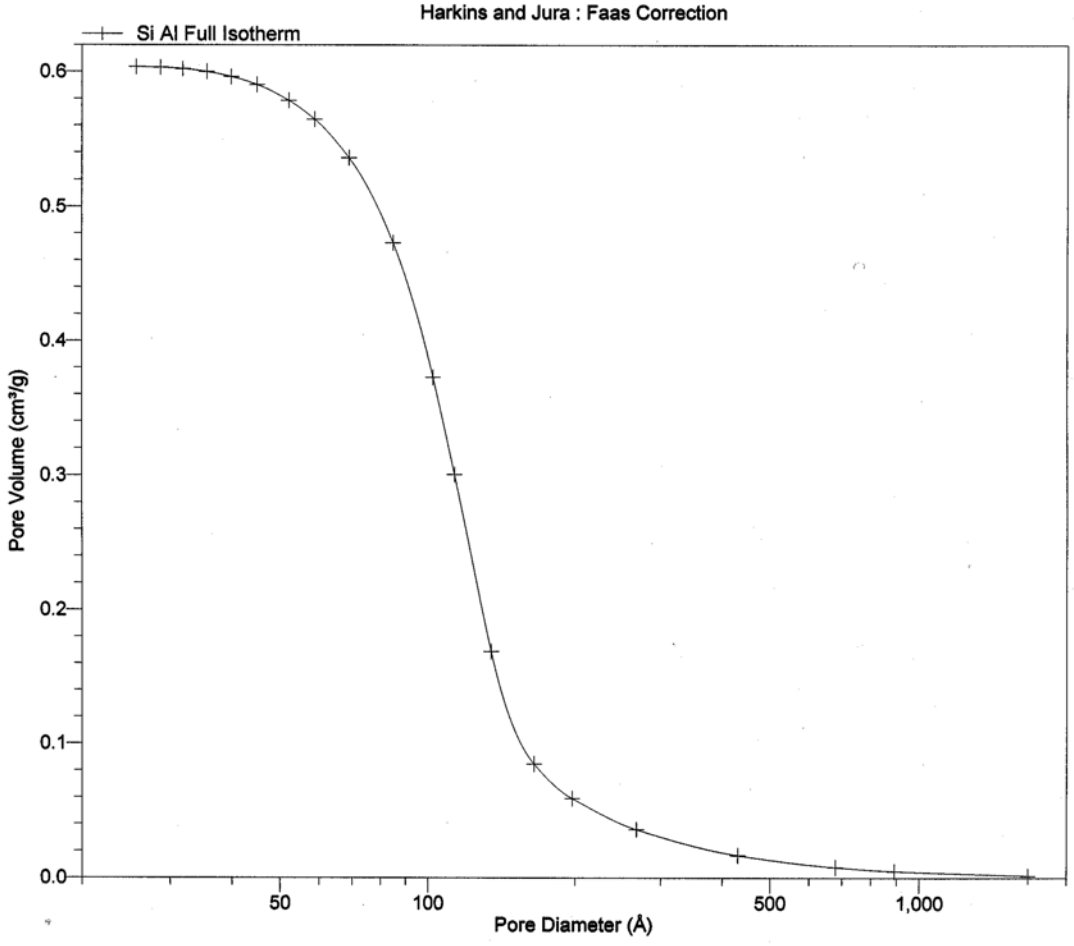
ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 10

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location: Category: Mic. Samples Subcategory: Example

Started:	5/13/2005 4:21:20PM	Analysis Adsorptive:	N2
Completed:	5/14/2005 5:42:29AM	Analysis Bath Temp.:	77.350 K
Report Time:	11/10/2005 11:11:36AM	Thermal Correction:	No
Sample Mass:	0.4488 g	Warm Free Space:	28.9183 cm³ Measured
Cold Free Space:	90.2027 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Automatic Degas:	No
File Created By:		File Creation Time:	
Analysis By:		Coll. Software:	ASAP 2420 V1.00
Report By:	Diane	Sample File Version:	1

BJH Adsorption Cumulative Pore Volume

Harkins and Jura : Faas Correction



BJH Adsorption dV/dlog(D) Pore Volume

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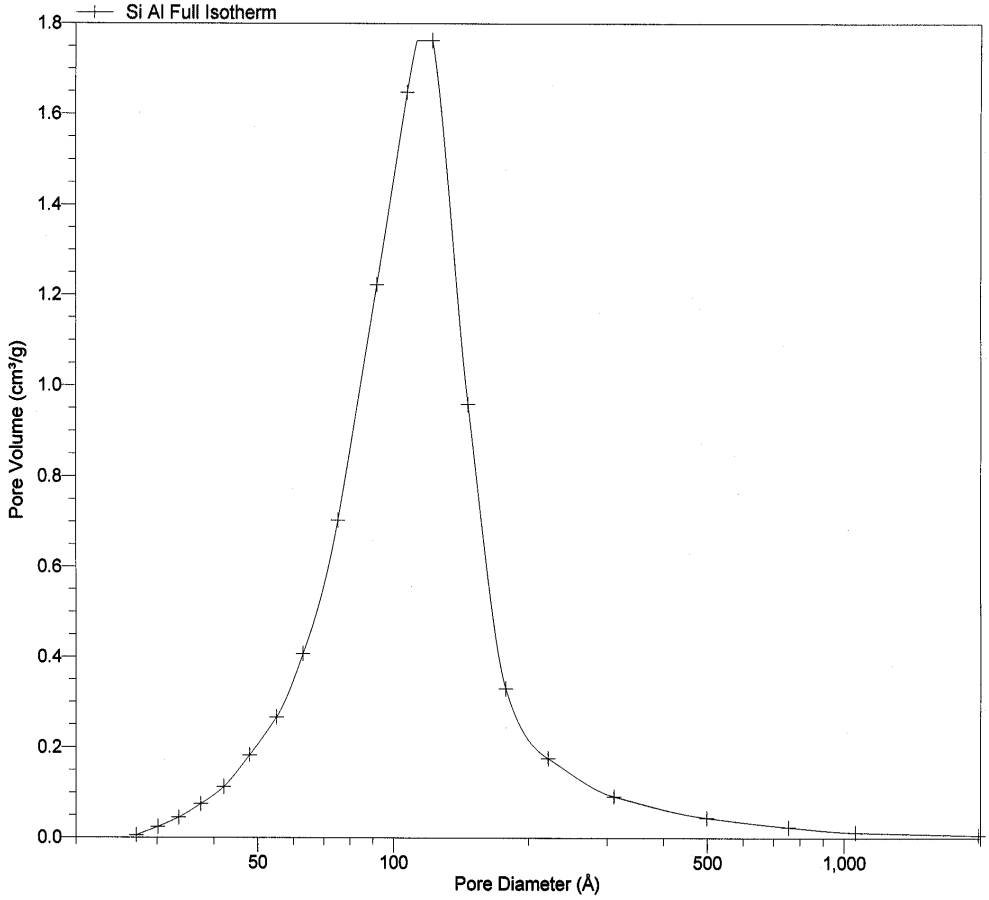
ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 11

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location: Category: Mic. Samples Subcategory: Example

Started:	5/13/2005 4:21:20PM	Analysis Adsorptive:	N2
Completed:	5/14/2005 5:42:29AM	Analysis Bath Temp.:	77.350 K
Report Time:	11/10/2005 11:11:36AM	Thermal Correction:	No
Sample Mass:	0.4488 g	Warm Free Space:	28.9183 cm³ Measured
Cold Free Space:	90.2027 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Automatic Degas:	No
File Created By:		File Creation Time:	
Analysis By:		Coll. Software:	ASAP 2420 V1.00
Report By:	Diane	Sample File Version:	1

BJH Adsorption dV/dlog(D) Pore Volume

Harkins and Jura : Faas Correction



BJH Desorption Pore Distribution

micromeritics®

ASAP 2420 Confirm Unit 1 Port 1 Serial #: 001 Page 12

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location: Category: Mic. Samples Subcategory: Example

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 11/10/2005 11:11:36AM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: Diane

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 1

BJH Desorption Pore Distribution Report

Faas Correction
Harkins and Jura
t = [13.99 / (0.034 - log(P/Po))] ^ 0.5

Diameter Range: 17.000 Å to 3000.000 Å
Adsorbate Property Factor: 9.53000 Å
Density Conversion Factor: 0.0015468
Fraction of Pores Open at Both Ends: 0.00

Pore Diameter Range (Å)	Average Diameter (Å)	Incremental Pore Volume (cm³/g)	Cumulative Pore Volume (cm³/g)	Incremental Pore Area (m²/g)	Cumulative Pore Area (m²/g)
3073.2 - 741.9	863.0	0.001545	0.001545	0.072	0.072
741.9 - 330.8	394.8	0.004292	0.005837	0.435	0.507
330.8 - 235.7	266.8	0.004123	0.009960	0.618	1.125
235.7 - 205.2	218.2	0.002704	0.012664	0.496	1.620
205.2 - 167.4	182.2	0.006104	0.018769	1.340	2.960
167.4 - 141.8	152.3	0.009179	0.027948	2.410	5.371
141.8 - 122.0	130.3	0.013645	0.041593	4.188	9.559
122.0 - 106.9	113.4	0.019273	0.060866	6.798	16.357
106.9 - 82.3	91.2	0.277556	0.338422	121.763	138.120
82.3 - 69.4	74.7	0.170653	0.509075	91.398	229.518
69.4 - 57.2	62.0	0.066457	0.575531	42.870	272.388
57.2 - 50.4	53.3	0.017212	0.592743	12.918	285.307
50.4 - 43.7	46.5	0.007899	0.600642	6.793	292.099
43.7 - 37.1	39.8	0.002260	0.602903	2.272	294.372

BJH Desorption Cumulative Pore Volume

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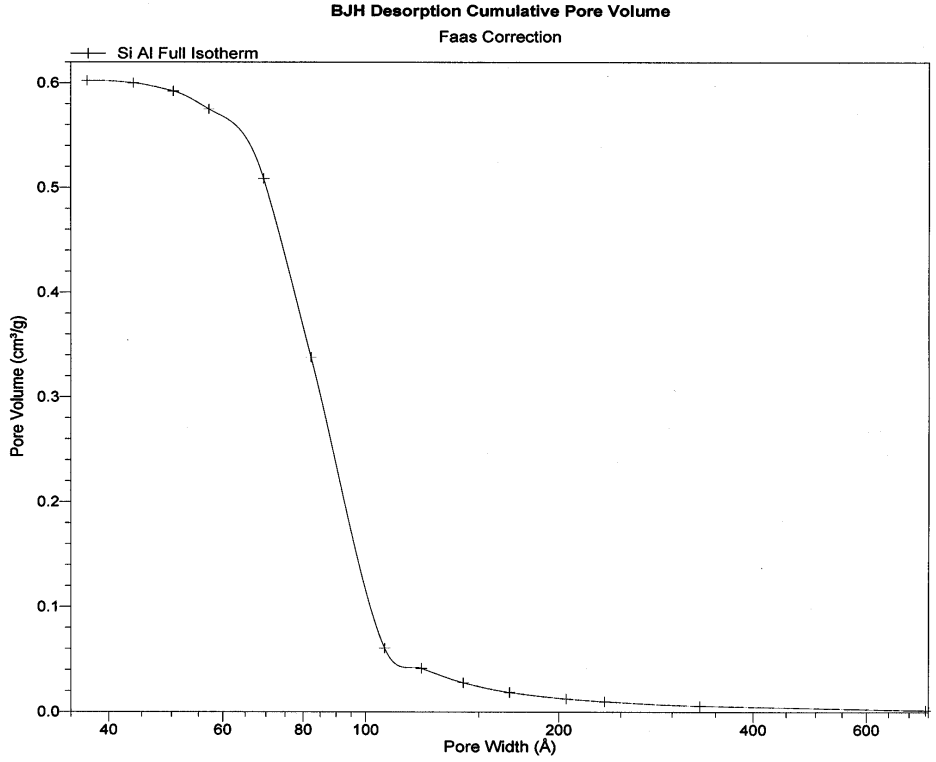
ASAP 2420 Confirm V1.00 Z Unit 1 Port 1 Serial #: 001 Page 13

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File Location:

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 10/4/2005 1:54:54PM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None
File Created By:
Analysis By:
Report By: gea

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No
File Creation Time:
Coll. Software: ASAP 2420 V1.00
Sample File Version: 2



BJH Desorption dV/dlog(D) Pore Volume

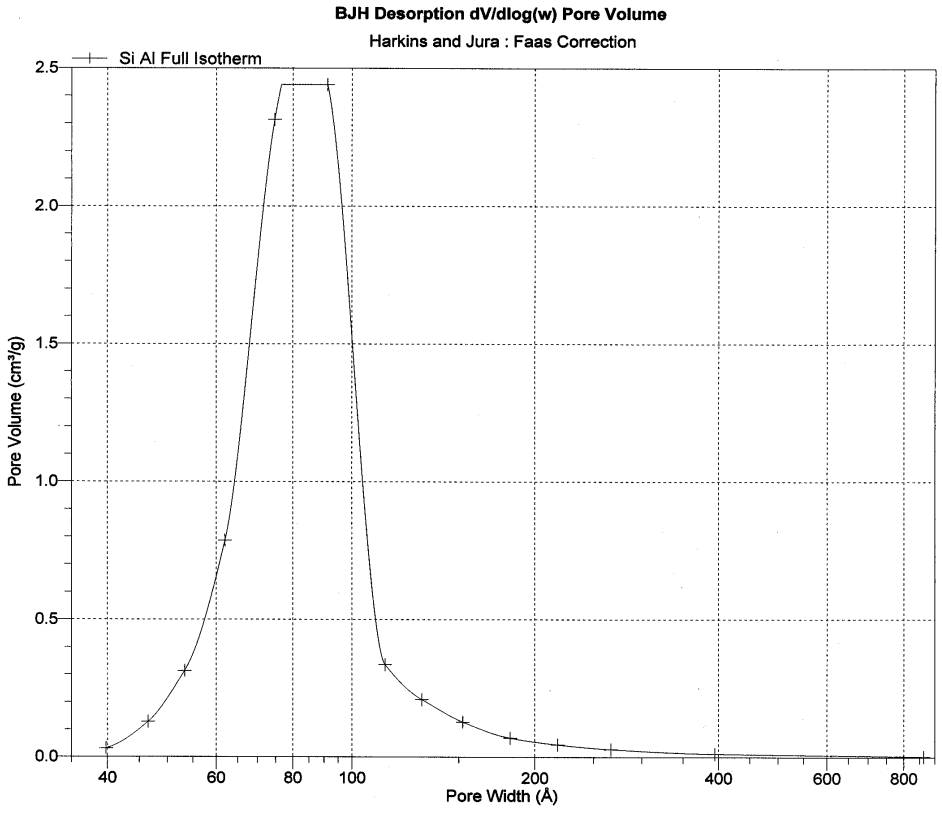
micromeritics®

Demo 2420 V1.01 Z Unit 1 Port 1 Serial #: 001 Page 14

Sample: Si Al Full Isotherm
Operator: SV
Submitter:
File: C:\DEMO2420\DATA\SIAL.SMP

Started: 5/13/2005 4:21:20PM
Completed: 5/14/2005 5:42:29AM
Report Time: 11/9/2005 8:11:06AM
Sample Mass: 0.4488 g
Cold Free Space: 90.2027 cm³
Low Pressure Dose: None

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.350 K
Thermal Correction: No
Warm Free Space: 28.9183 cm³ Measured
Equilibration Interval: 10 s
Automatic Degas: No



7 OPTIONS MENU

Description	7-3
Add Archive Location	7-4
Edit Labels	7-5
Update File List	7-5
Units	7-6
Graph Grid Lines	7-6
Live Graph	7-7
Service Test Mode	7-7

OPTIONS MENU

The choices on the Options menu allow you to configure the system to your laboratory's requirements. With the options on this menu, you can:

- Add directories
- Edit labels for sample files and directories
- Specify data presentation modes

Description

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

The only functions available to an Analyst on the Options menu are the Add Archive Location and Live Graph commands. All other operations are initiated by a Developer.

Add Archive Location Allows you to add a directory.

Edit labels Allows you to specify names for the labels that appear on the sample file editor.

Update file list Allows you to update the cache file for the sample or parameter file directory. This operation has to be initiated by the Administrator.

Units Enables you to choose the way in which data are presented on the screen and in printed reports.

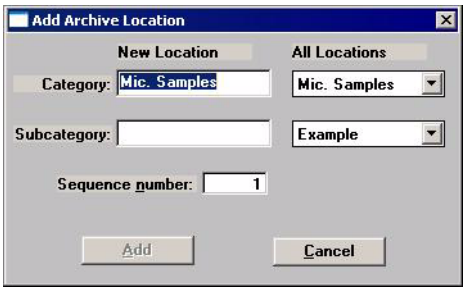
Graph Grid Lines Enables you to choose the type(s) of grid lines to show on your report.

Live Graph Enables you to choose the variable for the X-axis during data collection.

Service Test Mode Enables you to perform certain troubleshooting procedures. under the direction of a Micromeritics service representative.

Add Archive Location

Archive locations (directories) can be added by an Analyst or a Developer; the Add Archive Location dialog box is displayed.



Category
Subcategory

These fields allow you to create new directories and/or subdirectories for the repository.

The drop-down lists in the **All Locations** column contain the existing directories and subdirectories.

The fields in the **New Location** column allow you to enter new directories. **Category** represents the "Directory," and **Subcategory** represents the "subdirectory." You can add a subdirectory to an existing directory by choosing the directory from the drop-down list, then entering the new directory name in the **Subcategory** field.

The labels for the directories can be changed by the Developer (see following section). Many laboratories use the month/year combination for organizing files.

These directories and their contents cannot be seen through use of Windows Explorer; they can only be viewed through the use of the analysis program.

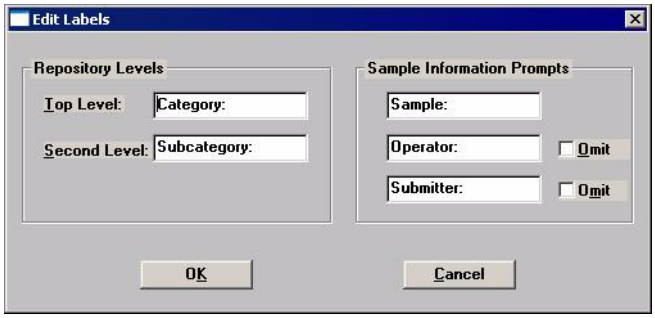
Sequence number Allows you to enter a new sequence number for the directory you create. This number is incrementally sequenced for each sample file you create and displays as part of the identification if the sample template includes a $ symbol.

Add

Enabled when you make an entry in the **Subcategory** field, allowing you to add the new directory and close the dialog box.

Edit Labels

Edit labels allows the Developer to edit the labels that display on the sample file editor; the Edit Labels dialog is displayed.



Repository Levels

The Top and Second Levels represent the directory and sub-directory for the location of sample files.

Sample Information prompts

These are the labels that appear on the sample information dialog. You can edit the labels to display differently if desired; for example, you may wish to use **Material** rather than Sample.

If you do not wish to have the **Operator** and **Submitter** (or other specified names) lines, you can click **Omit** to have them excluded.

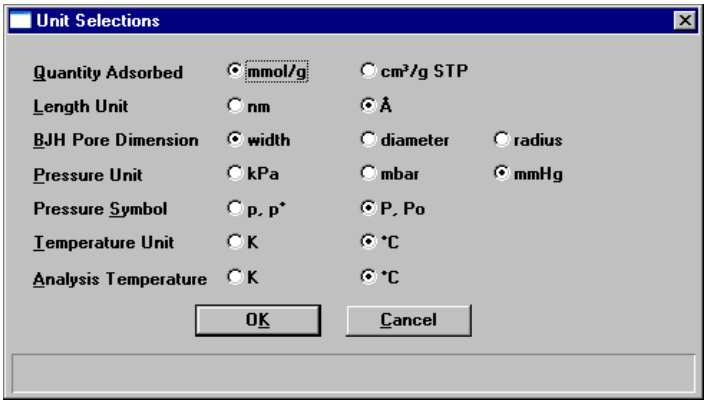
Update File List

This function cannot be performed by an Analyst nor a Developer, unless the Developer has Administrator rights. This is an Administrator function only; details for performing this operation are covered in the Administrator's Guide.

This option allows the directory to be updated when files have been added outside of the application. For example, if you need to restore the directory with a backup file.

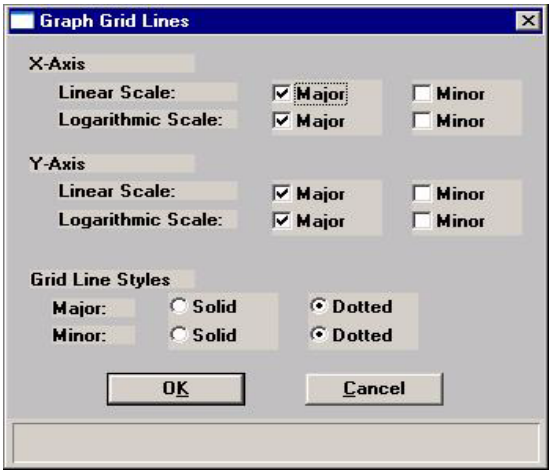
Units

This menu command displays the Units Selections dialog which allows you to choose the manner in which to display data on 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

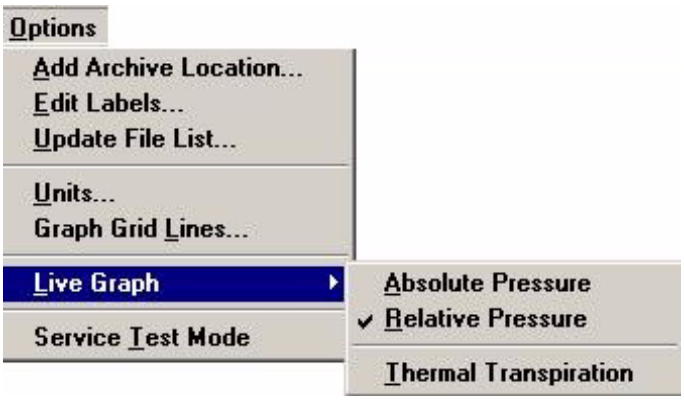
Enables you to choose **Major** and/or **Minor** lines to display in printed reports for the Logarithmic and Linear scales.

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.

Live Graph



This option enables you to choose the variable for the x-axis during data collection. You can also choose to correct for thermal transpiration which should always be selected for micropore analyses (refer to page **4-39** for additional information on thermal transpiration).

Service Test Mode

Various service tests are included in the ASAP 2420 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 box prompting you to enter a password is displayed. This password is coded to change on a regular basis and is known only by your service representative. You will not be able to perform these tests without his guidance.

After Service Test Mode has been enabled, the tests are accessible from the Unit menu.

8 TROUBLESHOOTING AND MAINTENANCE

Troubleshooting	8-3
Preventive Maintenance	8-7
Routine Maintenance	8-33

TROUBLESHOOTING AND MAINTENANCE

The ASAP 2420 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 includes troubleshooting information, recommended preventive maintenance procedures, and routine maintenance procedures.

Troubleshooting

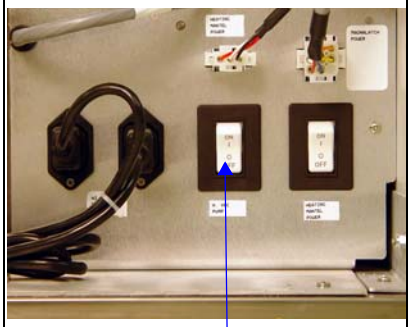
Most operational problems are caused by the following:

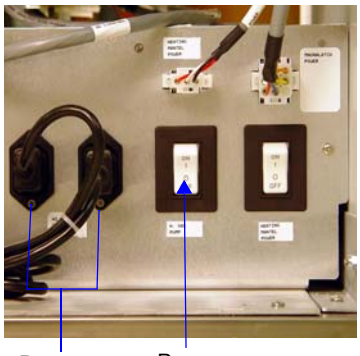
- Leaks (commonly around the sample tube O-ring at the analysis port)
- Sample weighing errors
- Use of too much analysis bath fluid in the Dewar at the start of an analysis
- Entry of incorrect system volume for analysis

Always check these first when expected analysis results are not obtained. Some common operational problems, which are not indicated on the computer screen, and their respective causes and solutions are provided in the following table.

What Happened	Why	What To Do
Analysis Dewar cannot be raised (or lowered).	Elevator that moves Dewar stuck in up position, down position, or somewhere in between.	Check for possible obstruction to elevator movement.
Vacuum pump system makes loud noise continuously.	Sample tube connector nut is loose.	Turn the connector nut clockwise to tighten.

What Happened	Why	What To Do
Vacuum makes loud noise continuously. *(continued)*	Sample tube fitting is loose.	Tighten the fitting securely with a wrench.
	Sample tube O-ring is worn or cracked.	Replace the sample tube O-ring. Refer to **"Replacing a Sample Tube O-ring" on page 8-12**.
	Sample tube is cracked.	Replace with a new sample tube.
	No sample tube is loaded on a selected port. A gas inlet valve is open while the vacuum valve is open.	Make sure the port valve is closed. Install a plug or empty sample tube on the port. Enable manual control, then use the instrument schematic to close the gas inlet valve. Refer to **"Enable Manual Control" on page 5-26**.
Vacuum gauge shows reading above 20 µmHg, even after extended pumping through unrestricted valve with analysis or degas ports closed.	**Oil-based Pump Only:** 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. Refer to **"Inspecting and Changing Vacuum Pump Fluid" on page 8-16**.
	No power to the vacuum pump.	Check the pump power plug, power switch, and line circuit breaker.
	Filter in the port being used is dirty.	Replace the port filter. Refer to **"Replacing the Port Filters" on page 8-9**.

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. *(continued)*	Alumina in the oil vapor trap is holding moisture because it was not sufficiently dried before being added to the alumina trap.	Replace or dry the alumina. Refer to **"Replacing the Alumina in the Oil Vapor Traps" on page 8-20**.
	Oil-Free Pump Only: Filter in the port being used is dirty.	Replace the port filter. Refer to **"Replacing the Port Filters" on page 8-9**.
	High-vacuum pump may have timed out.	Remove the lower front panel from the analyzer. Refer to **"Accessing the Vacuum Pumps" on page 8-14**. Turn the high-vacuum pump power switch off, then back on.  Power switch
	No power to the vacuum pump.	Check the pump power plug, power switch, and line circuit breaker.
	The diaphragm in the pump is worn or damaged.	Contact your Micromeritics Service Representative.

What Happened	Why	What To Do
Analysis valves cannot be operated.	Circuit was opened by the circuit breaker.	Push in the breaker button, located on the side of the instrument near the power entrance. If it will not stay in, contact appropriate service personnel. Breaker button
	Cable from the computer to the analyzer is loose.	Make sure the cable is connected properly to the computer and the analyzer.
High-vacuum pump indicator light does not come on.	No power to the high-vacuum pump.	Remove the lower panel on the front of the analyzer. Refer to **"Accessing the Vacuum Pumps" on page 8-14**. Check the pump power plug. Turn the high-vacuum pump power switch off, then back on.  Power plugs Power switch

Preventive Maintenance

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

Maintenance Required	Frequency
Checking and cleaning analysis port Dewars, page **8-8**	Weekly
Replace port filters, page **8-9**	Every 30 days
Replace sample tube O-ring, page **8-12**	As required or every 3 months
Inspect and change vacuum pump fluid*, page **8-16**	As required or every 3 months
Replace alumina in oil vapor traps*, page **8-20**	As required or every 3 months
Lubricate elevator screw, page **8-25**	As required or every 6 months
Clean the outside of the analyzer, page **8-26**	As required or every 6 months
Check analyzer outgassing rate, page **8-26**	Every 6 months
Test analyzer for leaks, page **D-1**	As required or every 12 months
Replace vacuum pump exhaust filter, page **8-31**	As required or every 12 months
Replace diaphragm in vacuum pump (if an oil-free pump is used)**	Every 12 months

*Oil-sealed vacuum pumps only.

**For oil-free pumps only: After about 12 to 18 months of operation, the diaphragm in the pump will wear out. When the diaphragm wears out, the pump becomes completely inoperable. To prevent any instrument down time due to an inoperable pump, we recommend that you have the diaphragm replaced by a Micromeritics Service Representative every 12 months.

Checking and Cleaning the Analysis Port Dewars

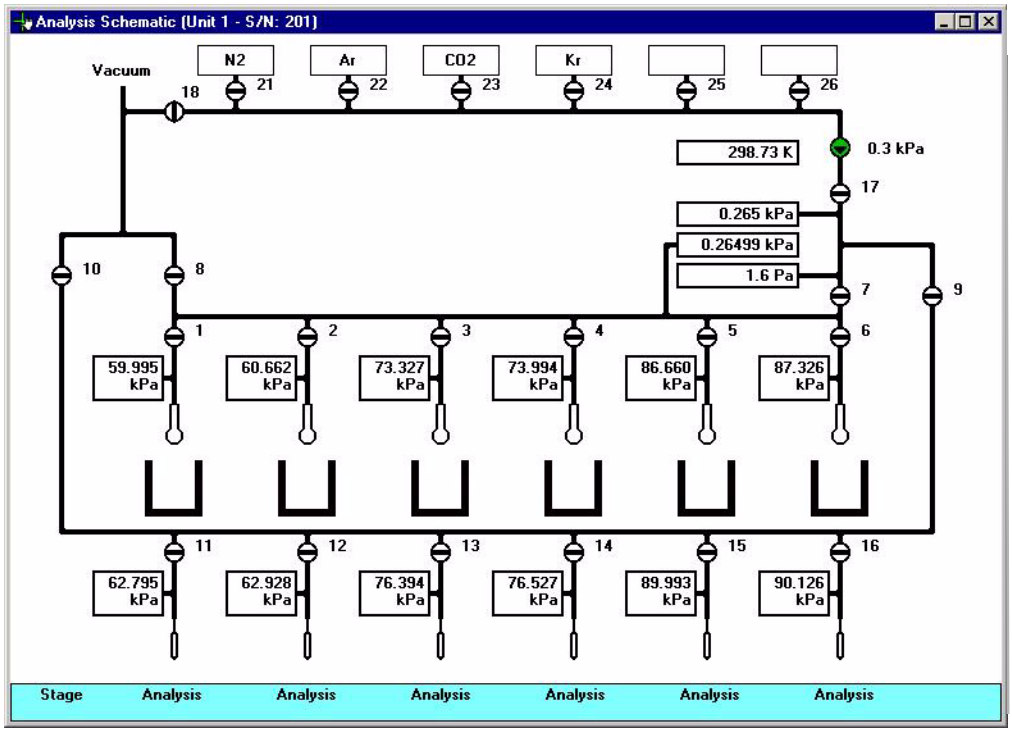
When handling Dewars, be sure to observe the Dewar precautions outlined in "Filling Dewars" on page 3-38.

Ice and suspended frost particles may accumulate in the bottom of an analysis port Dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tube and the bottom of the Dewar, causing the Dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tube. This causes the sample temperature to be slightly higher which, in turn, can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure.

Accumulated ice is likely to melt and form a pool of water in the Dewar if all liquid nitrogen evaporates. The water must be removed; otherwise it will solidify when liquid nitrogen is added and could press on the bottom of the sample tube causing breakage.

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

1. Remove the sample tubes from the analysis ports.
2. Ensure that the Po tubes are out of the way.
3. Display the Analysis schematic and enable manual control (refer to **"Enable Manual Control" on page 5-26**).



4. Raise the Dewar elevators (refer to **"Enable Manual Control" on page 5-26**).
5. Remove the Dewars and pour the liquid nitrogen from the Dewars into an appropriate cryogenic container.

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

6. Rinse the Dewars with warm water to melt any ice accumulation which may remain in the Dewars then dry thoroughly.
7. Replace the Dewars.
8. Lower the Dewar elevators (refer to **"Enable Manual Control" on page 5-26**).
9. Reposition the Psat tubes and tighten the tube fittings.

Replacing the Port Filters

A porous metal filter is located in each analysis and degas port. If a degas port is not plugged with either a sample tube or a port plug, then the filter will absorb atmospheric gases, resulting in extended degassing time for samples on that port.

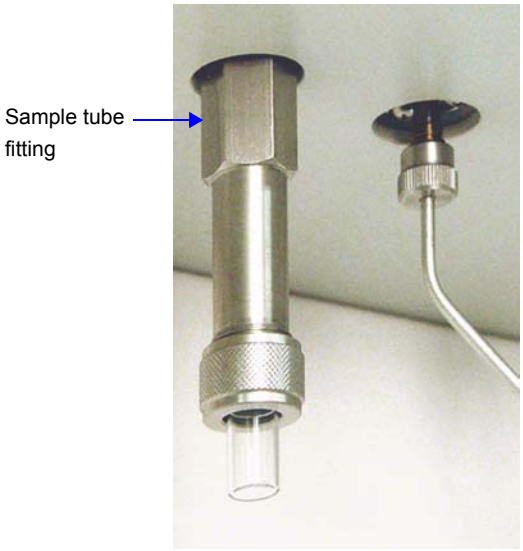
Using a contaminated filter on an analysis port may extend the time required to achieve a vacuum at that port. More importantly, the contaminant may adsorb or desorb during analysis, affecting the analysis results. A contaminated filter on an analysis port may cause a leak test to fail (if the contaminant outgasses) or cause a free space reading to be much lower than normal.

Analysis Ports

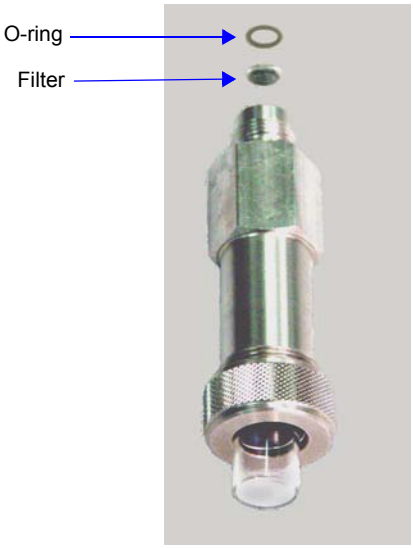
Before removing and installing a port filter, ensure that the port valve is closed. Observe the analysis system schematic to verify valve status.

Place a Dewar cover on the Dewar before replacing port filters.This prevents parts from falling into the Dewar and possibly causing breakage.

1. Using a wrench, remove the sample tube fitting from the analyzer.



2. Remove the filter and O-ring.



3. Replace the filter and the O-ring.

To avoid analysis problems, the new filter and O-ring must be clean. Do not touch them with bare hands.

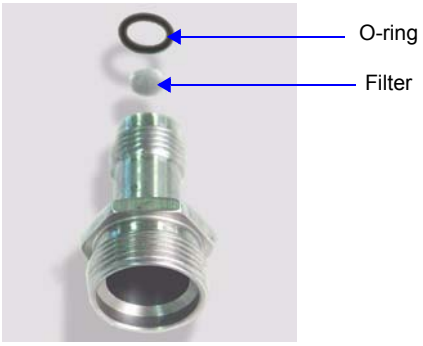
4. Reinstall the analysis port fitting; securely tighten with a wrench to prevent leaks during evacuation.

Degas Ports

Before removing and installing a port filter, ensure that the port valve is closed. Observe the degas system schematic to verify valve status.

1. Using a wrench, remove the degas port fitting.

2. Remove the O-ring and filter.



3. Replace the filter and the O-ring.

To avoid analysis problems, the new filter and O-ring must be clean. Do not touch them with bare hands.

4. Reinstall the degas port fitting; securely tighten with a wrench to prevent leaks during evacuation.

Replacing a Sample Tube O-ring

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced. This procedure applies to both degas and analysis ports.

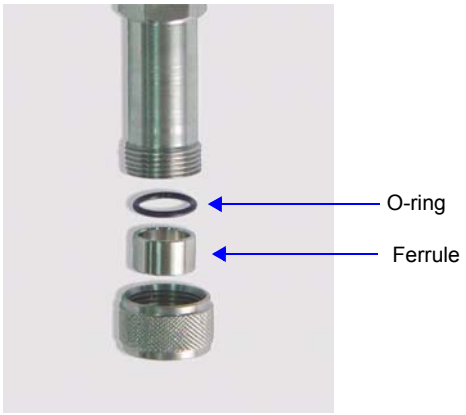
Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the analysis schematic or degas schematic to verify valve status.

1. Rotate the Po tube if necessary to move it away from the sample tube.
2. Holding the sample tube firmly with one hand, loosen and remove the sample tube connector nut by turning it counterclockwise.

Be careful not to let the sample tube connector nut drop onto the sample tube bulb as it may break the tube.

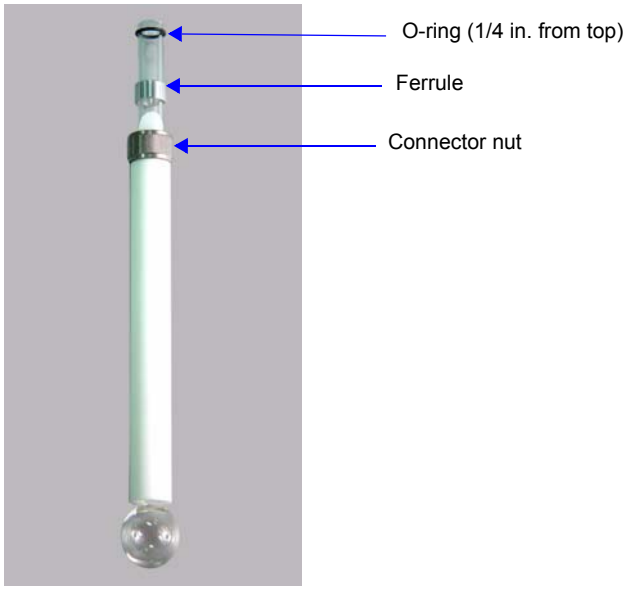
3. Carefully pull the sample tube down until it is free from the port.

4. The connector nut contains a ferrule and an O-ring. Remove the O-ring from the top of the sample tube.



If the O-ring remains inside the sample port, you may use a pair of tweezers or needle-nose pliers to remove it.

5. While holding the connector nut in place, slide a new O-ring over the sample tube, about 1/4-in. (5 mm) from the top of the tube.

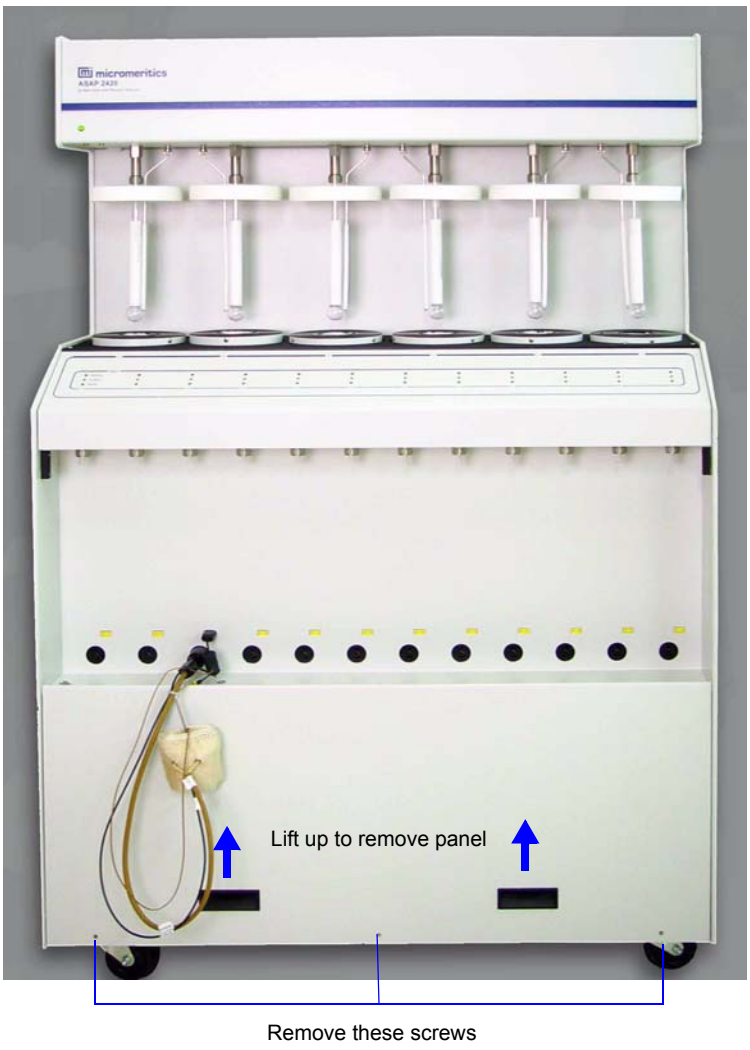


6. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.

7. Turn the connector nut clockwise until it is hand-tight.

Accessing the Vacuum Pumps

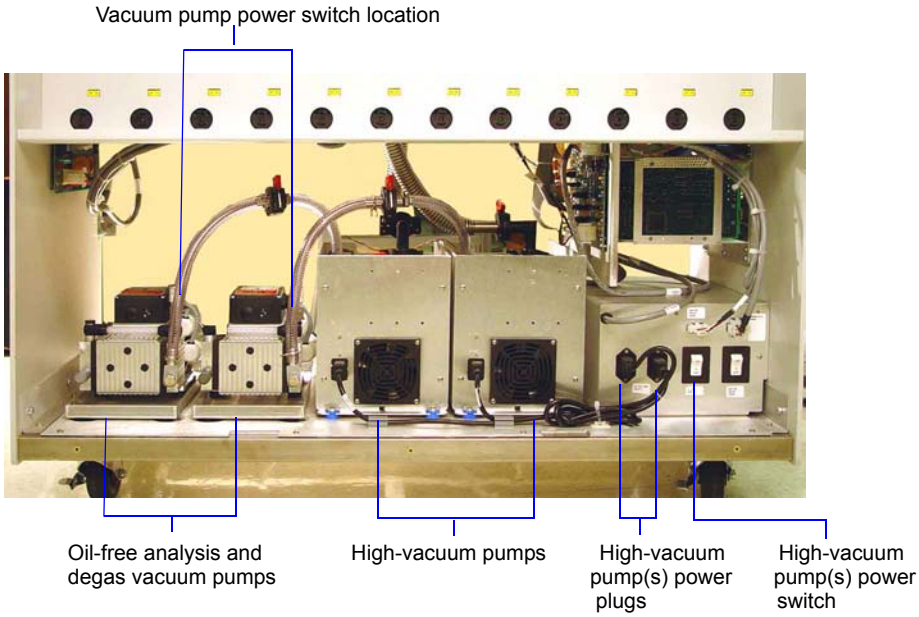
You will need to access the vacuum pumps to perform certain maintenance procedures. The pumps can be accessed by removing the lower front panel of the analyzer.



To remove the panel:

1. Remove the three screws located along the bottom of the panel.
2. Use the hand grips in the front panel to lift the panel up and off of the analyzer.

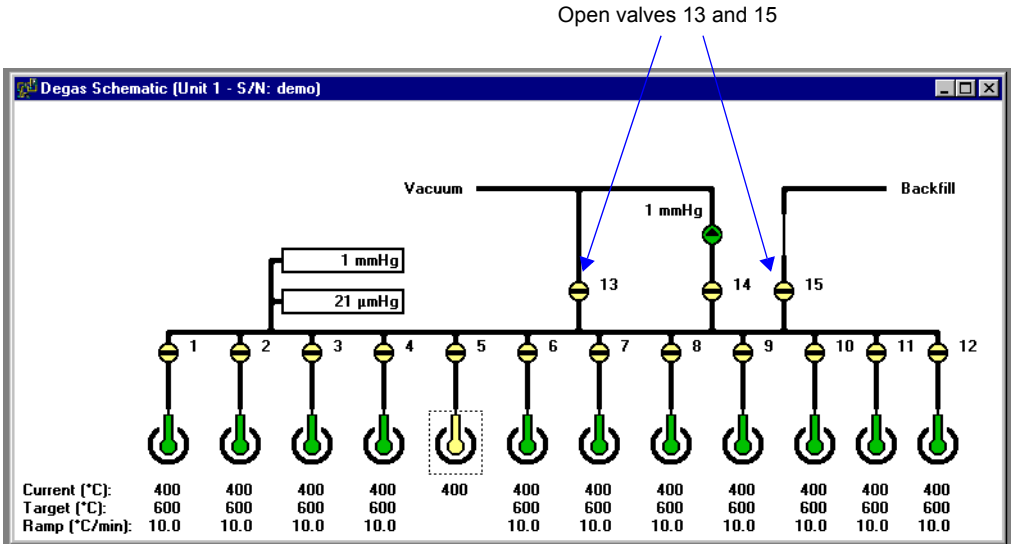
3. The pumps are housed on a shelf on the bottom of the analyzer as shown below.



Your system may contain two oil-based pumps, two oil-free pumps, or one of each type. A high-vacuum pump is installed for each oil-free pump.

The illustration above shows two oil-free pumps and two high-vacuum pumps.

4. If you plan to remove a vacuum pump(s) for servicing, select **Unit > Show Degas Schematic** on the ASAP 2420 main menu, then select **Unit > Enable Manual Mode**.



5. Turn off the vacuum pumps:

- For oil-based pumps: disconnect the power plug from the power source.
- For oil-free pumps: place the On/Off switch on the side of the pump in the **Off** position. Place the high-vacuum pump power switch in the **Off** position.

6. Open valve 13 and valve 15.
7. When the pressure stabilizes around atmospheric pressure, close valve 13, then 15.

   This step fills the vacuum section with the backfill gas before you disconnect a pump.

Inspecting and Changing Vacuum Pump Fluid

This procedure applies to oil-sealed vacuum pumps only.

The fluid in the vacuum pump should be changed every three months or when the efficiency of the vacuum pump declines (requiring increased time to reach vacuum levels). The fluid can first be inspected to determine if a change is necessary.

Use oil supplied by Micromeritics, or refer to the vacuum pump manual for other acceptable oils.

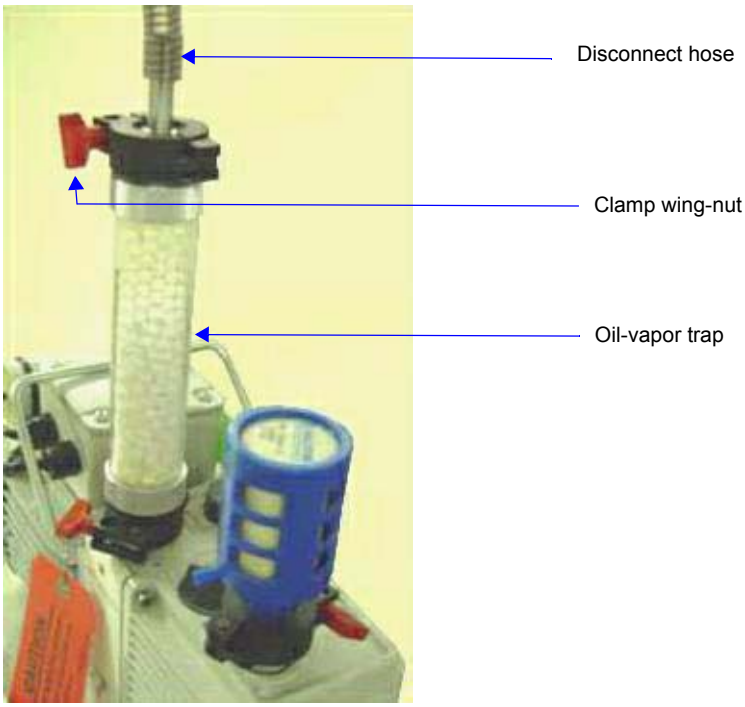
Inspecting Fluid

1. Remove the lower front panel from the analyzer (refer to **"Accessing the Vacuum Pumps" on page 8-14**).
2. View the vacuum pump fluid through the oil level window. Fluid in good condition is clean, clear or light in color, and transparent. If the color of the fluid is darkened, the fluid should be changed.

Changing Fluid

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

1. Remove the lower front panel from the analyzer if it is not already removed. (refer to **"Accessing the Vacuum Pumps" on page 8-14**).
2. Unplug the vacuum pump from the power source.
3. 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.

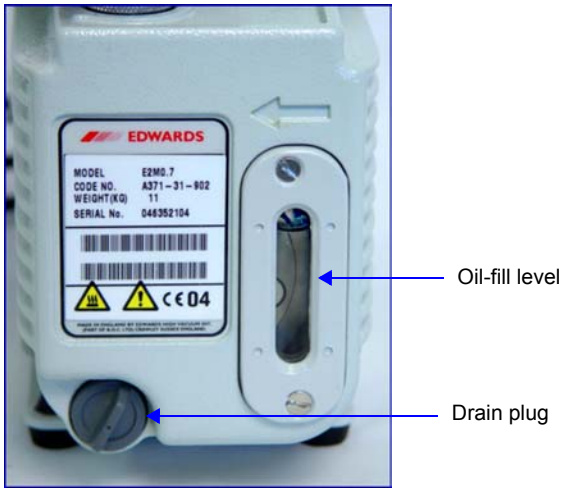


4. Grasp the handle on top of the vacuum pump and lift it out of the analyzer; place the vacuum pump on a work table.

5. Drain the used oil:

   a. Place a waste container under the drain spout.

   b. Remove the plug from the drain spout.



   c. Allow the oil to drain into the waste container.

   d. Replace the drain plug.

6. Remove the oil-fill plug.

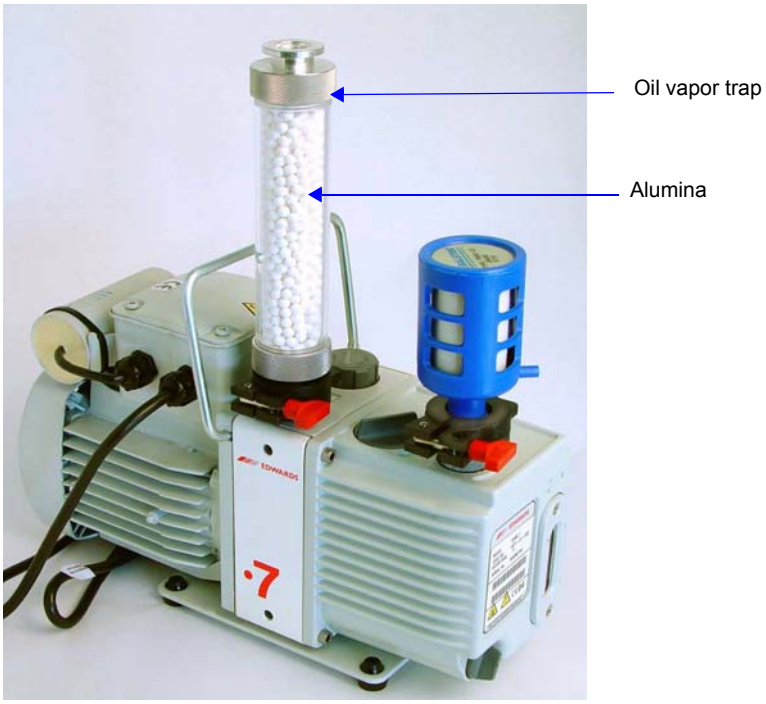
Adding fluid above the midway position on the fluid level indicator may cause fluid to splash into the vacuum hoses and leak from the vacuum pump.

7. Add fresh fluid to the oil-fill port until the level is midway between the two indicator lines in the oil-level window.
8. Check the washer or O-ring used at the oil-filling port; replace if necessary.
9. Insert the oil-fill plug and turn counterclockwise to tighten.
10. If the fluid was badly contaminated (dark brown color), flush the pump with fresh fluid as follows:
    a. Cover the top of the oil vapor trap.
    b. Plug the pump power cord into a power source and run for about 2 minutes.
    c. Unplug the power cord.
    d. Remove the plug from the drain spout.
    e. Allow the oil to drain into the waste container.
    f. Reinstall the drain plug.
    g. Add fresh fluid to the oil-fill port until the level is midway between the two indicator lines.
    h. Reinstall the oil-fill plug.
    i. Repeat steps a through h if necessary.
11. Check the alumina in the oil vapor trap and replace if necessary (refer to **"Replacing the Alumina in the Oil Vapor Traps" on page 8-20**).
12. Place the vacuum pump back onto the drip tray in the analyzer.

13. Reconnect the vacuum pump hose.
14. Reconnect the power cord to the power source.
15. Reinstall the front analyzer panel.
16. The pump must run a few hours (or overnight) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

Replacing the Alumina in the Oil Vapor Traps

This procedure applies to oil-based vacuum pumps only.



The activated alumina in the oil vapor traps becomes saturated during use. The alumina should be replaced if either of the following conditions exist:

- The alumina has been used for a three-month period.
- 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. Prepare fresh alumina as follows:

   a. Heat an oven between 250 °C and 350 °C.

   b. Pour about 180 grams of alumina into a glass or metal container for each trap (approximately 250 mL if a graduated beaker is used). Place the container in the oven.

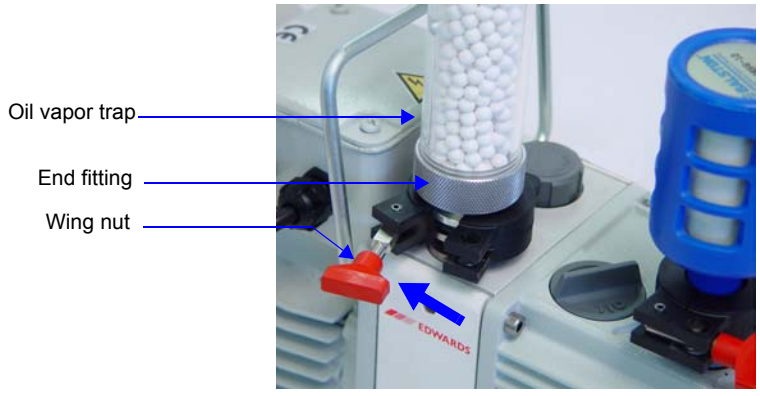
   c. Bake the alumina for two hours; turn off the oven.

   d. Allow the alumina to cool in the oven before pouring it into the traps.

2. Choose one of the following:

   - If the vacuum pump is removed from the analyzer, proceed to step 3.
   - If the vacuum pump is not removed from the analyzer and you wish to remove it, refer to steps 1 through 4 under **"Changing Fluid" on page 8-17**.
   - If the vacuum pump is not removed from the analyzer and you do not wish to remove it, remove the lower front panel of the analyzer (refer to **"Accessing the Vacuum Pumps" on page 8-14**), then unplug the pump from its power source.

3. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Swing the clamp open and remove it.



4. Remove one end fitting from the trap body.
5. Remove the used alumina from the trap body and dispose of it in an appropriate manner.

6. If the trap body interior appears oily or dirty, wash it with isopropyl alcohol or ethyl alcohol and dry it thoroughly.

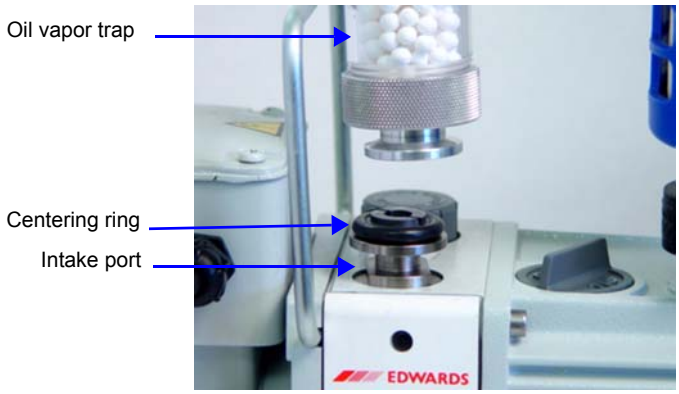
Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.

Ensure adequate ventilation when using solvents for cleaning purposes.

7. Pour the new activated alumina pellets into the trap until they are level with the top of the trap body.

8. Screw the end fitting back onto the trap and tighten securely by hand. Insert the O-ring and screw the end fitting back onto the trap and tighten securely by hand.
9. Make sure the centering ring is in place on the intake port.

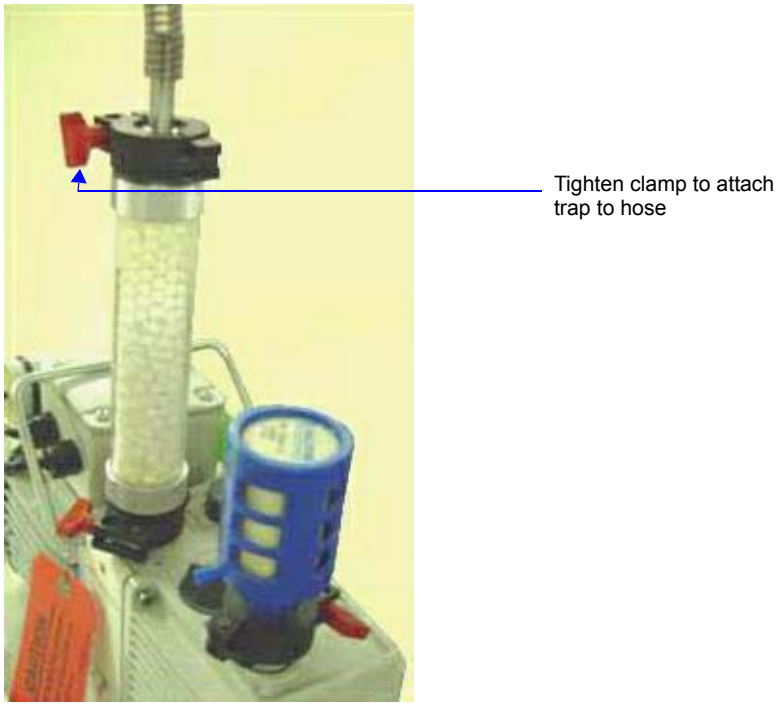
10. Place the trap on the centering ring.



11. 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.

12. If you removed the pump from the analyzer, place it onto the drip tray in the analyzer.

13. Reconnect the hose from the analyzer to the oil vapor trap using the clamp and wing nut as described in step 11 above.

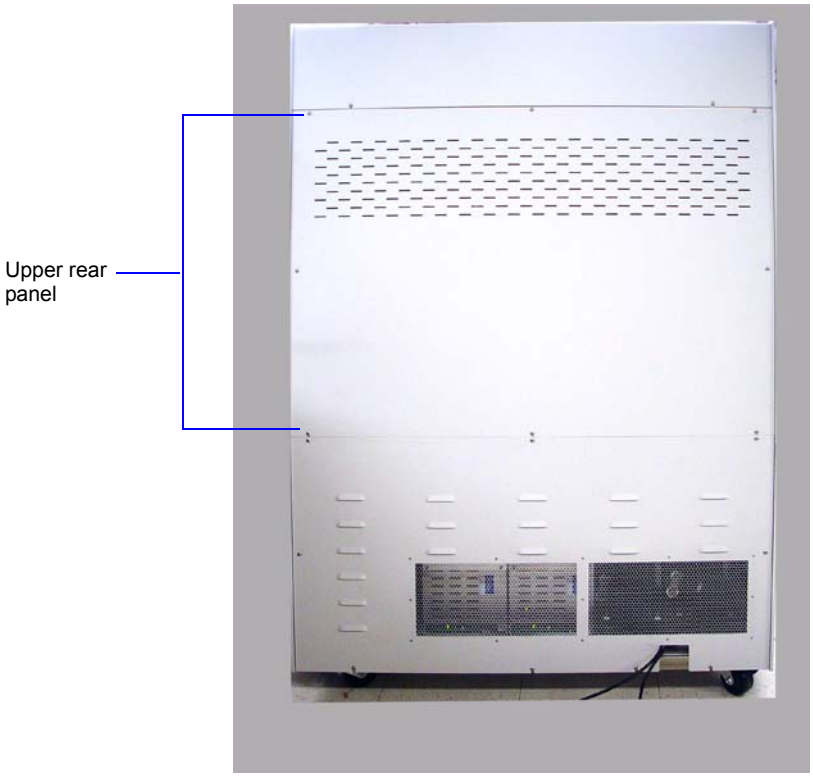


14. Plug the pump power cord into the power source.

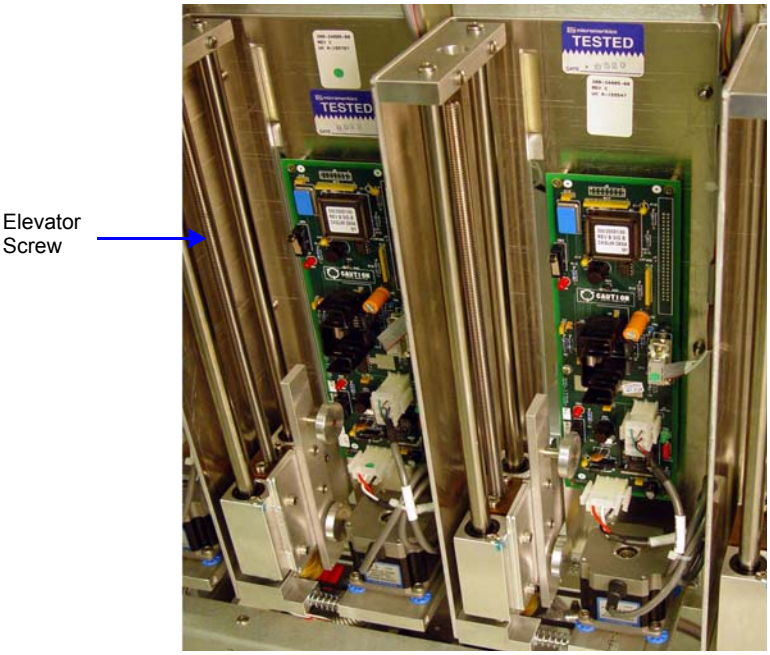
15. Reinstall the front panel on the analyzer.

Lubricating the Elevator Screws

1. Remove the analyzer's upper rear panel by loosening, then removing the retaining screws.



2. Apply a light coat of lithium grease to the six elevator screws.



3. Replace the back panel.

Cleaning the Analyzer

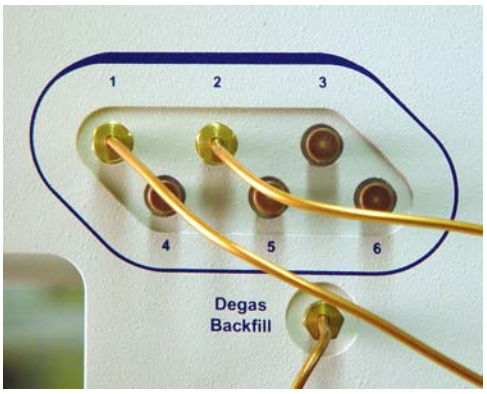
The ASAP 2420 should be cleaned every six months, or sooner, if required.

Clean the outside casing of the analyzer (including the shields) with a clean cloth dampened with isopropyl alcohol (IPA), a mild detergent in water, or a 3% hydrogen peroxide solution. It is not necessary to remove any switches or screws to clean the outside casing.

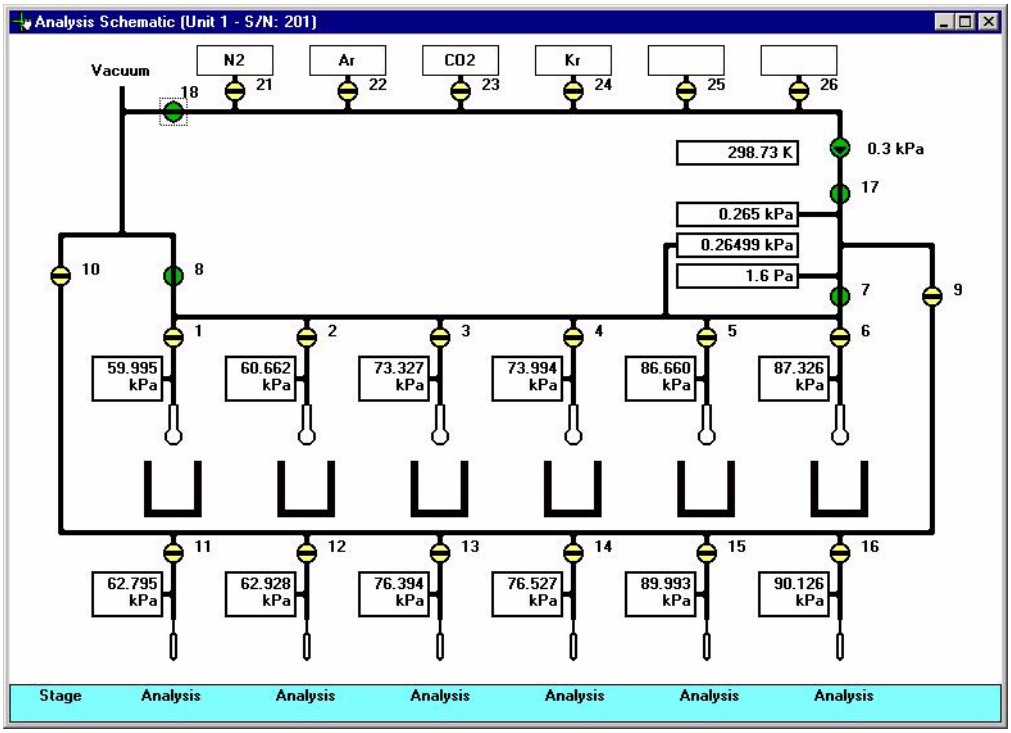
Use IPA or a mild detergent in water to clean the shields. Do not use acetone. Acetone will damage the surface of the shields.

Checking the Analysis Manifold Outgassing Rate

1. Close the supply valves (Nupro valves) on the gas inlet lines.
2. Insert leak tight plugs in unused gas inlet ports.



3. If the analysis system schematic in manual control mode is not displayed on the computer screen, select **Unit > Enable Manual Control**, then **Unit > Show Analysis Schematic**.



4. Open valves 18, 8, 17, and 7.
5. Set the servo valve to dose to 1000 mmHg to ensure that it stays open.
6. Evacuate the inlet ports by opening valves 21 to 26.
7. Evacuate at least 20 minutes. Overnight evacuation is preferable.

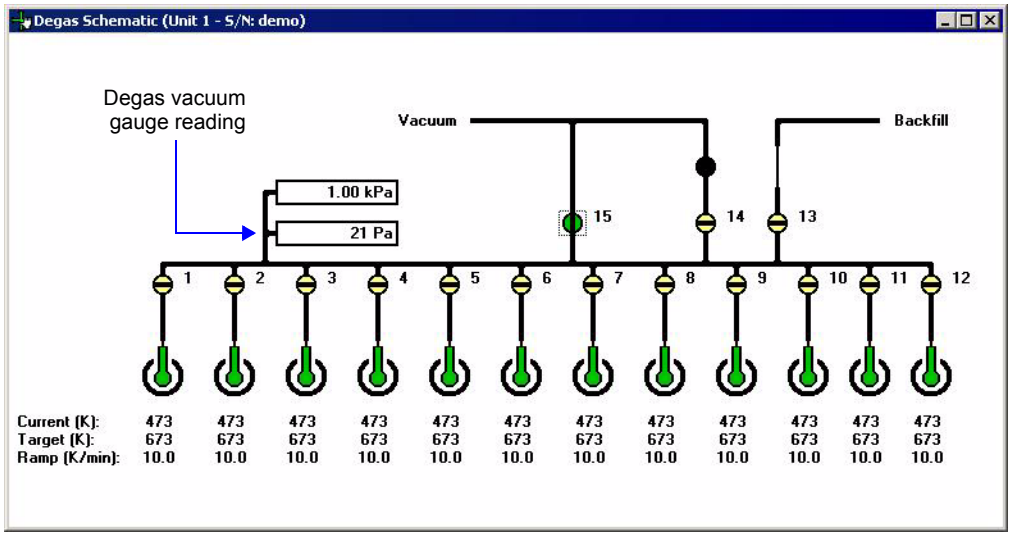
8. Close valves 8 and 18, and record the pressure as the **Initial Reading** for Inlet Ports in the table below.

Test	Initial Reading	3 Min. Reading	Difference	Limits	OK?
Inlet Ports				30 microns (0.3 mmHg)	
Valve 21				2.1 microns (0.0021 mmHg)	
Valve 22				2.1 microns (0.0021 mmHg))	
Valve 23				2.1 microns (0.0021 mmHg)	
Valve 24				2.1 microns (0.0021 mmHg)	
Valve 25				2.1 microns (0.0021 mmHg)	
Valve 26				2.1 microns (0.0021 mmHg)	

9. Wait 3 minutes, then record the pressure as the **3 Minute Reading** in the table above.
10. Subtract the first reading from the second reading and record in the **Difference** column in the table above.
11. If the value in the **Difference** column is at or below the value in the **Limits** column, enter Yes in the **OK?** column. If the Difference value is not below the Limits value, a gas inlet valve, inlet plug, or gas line is leaking from atmosphere.
12. Close all gas inlet manifold valves (21 through 26).
13. Record the pressure as the **Initial Reading**, then begin timing as soon as you complete the next step.
14. Gas inlet valves 21 through 26 must remain closed during this procedure. Pressurize the inlet to valve 21 by opening the supply valve or removing the port plug. This allows gas or air to pressurize the inlet valve above the seat.
15. After 3 minutes, record the pressure as the **3 Minute Reading**. Subtract the first reading from the second and record in the **Difference** column in the table above.
16. Repeat steps 13 through 15 for the inlet valves 22 through 26.

Checking the Degas Manifold Outgassing Rate

1. If the degas system schematic in manual control mode is not displayed on the computer screen, select **Unit > Enable Manual Control**, then **Unit > Show Degas Schematic**.



2. Close all valves except valve 15. Leave valve 15 open to thoroughly evacuate the degas manifold.

3. Evacuate the degas manifold for at least one hour or longer after attaining a pressure below 10 microns.The outgas rate is affected by how recently the manifold was exposed to air, water vapor in the air, and any other gas. Prolonged evacuation, such as overnight, will help to remove such gases and water vapor from the elastomer seals and the surface of the manifold itself.

4. Close valve 15.

5. Remove all of the plugs from the degas ports if present. If the pressure rises, one of the degas stations is leaking. Clear leaks before proceeding to the next step.

6. Record the reading of the degas vacuum gauge in the **Reading 1** row in the table below.

Manifold	Degas Vacuum Gauge Reading
Reading 1	_____
Reading 2	_____
Reading 2 minus Reading 1	=_____
Result divided by 5	_____/5 = _____
Maximum Rate = 10 microns/min. (0.010 mmHg/min.) after overnight evacuation.	

7. Wait 5 minutes.
8. Record the degas vacuum gauge reading in the **Reading 2** row in the table above.
9. Subtract the first reading from the second reading.
10. Divide the result by 5 to obtain the degas manifold outgassing rate in microns/min.

    If the result is at or below the maximum rate shown in the table, no degas valves or manifold components are leaking. If the result is above the maximum rate, a leak is present. The leak may be from the gas inlet valve or it may be across a seal to atmosphere.

Testing the Analyzer for Leaks

Refer to **page D-1** for instructions on testing the ASAP 2420 for leaks.

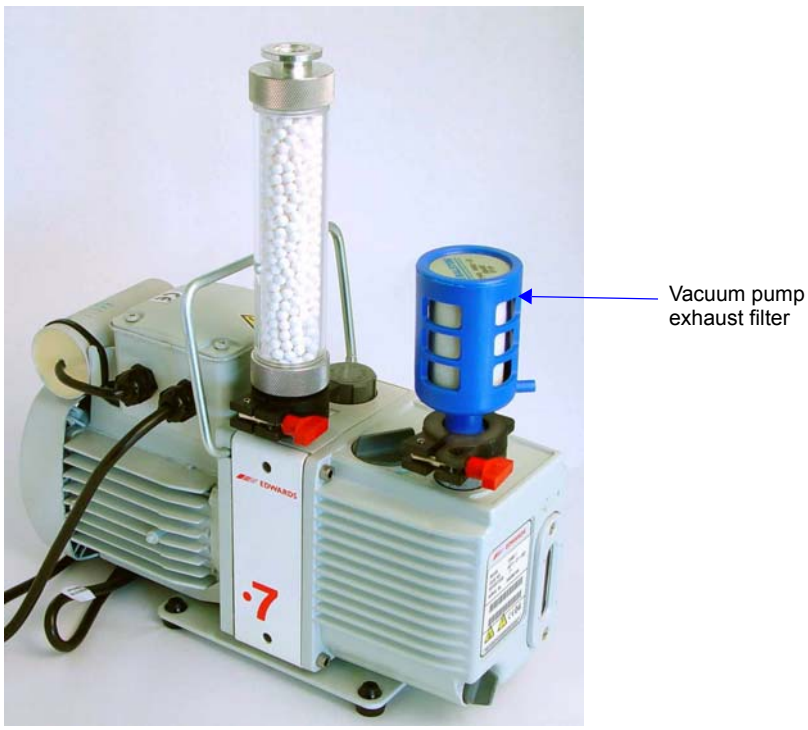
Replacing the Vacuum Pump Exhaust Filter

This procedure applies to oil-based vacuum pumps only.

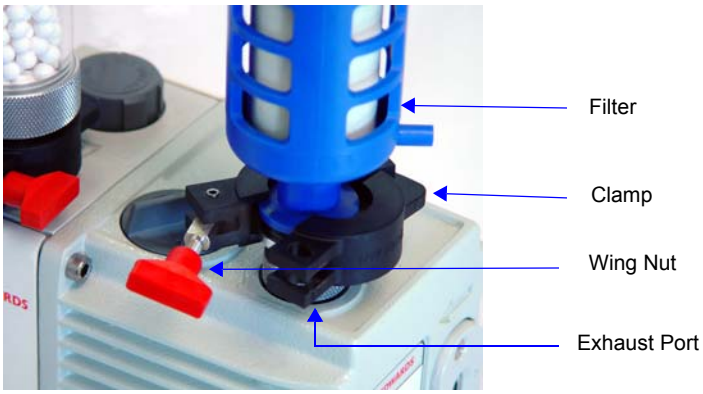
The gases used by the ASAP 2420 are exhausted by vacuum pumps. An exhaust filter is installed on the exhaust port of each oil-sealed vacuum pump. The filter minimizes the release of oil vapor and should be replaced when it becomes so saturated with oil that it is ineffective.

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.

1. Remove the lower front panel from the analyzer (refer to **"Accessing the Vacuum Pumps" on page 8-14**).
2. If you wish to remove the vacuum pump from the analyzer, refer to steps 1 through 4 under **"Changing Fluid" on page 8-17**.



3. Loosen the wing nut of the clamp at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.



4. Discard the oil filter.
5. Retain the centering ring, clamp, and wing nut.
6. Remove the dust cover from the new filter.
7. Make sure the centering ring is on the exhaust port.
8. Place the new filter on the centering ring.
9. 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.
10. If you removed the vacuum pump from the analyzer, place it back onto the drip tray in the analyzer, then reinstall the vacuum pump panel.

Routine Maintenance

The procedures in this section are performed as needed.

Changing Gases

Guidelines for Connecting Gases to the Analyzer

Use these guidelines when installing regulators and gas lines:

- Ensure that all gases needed are available and near the instrument. If the gas bottles are not located near the instrument, you will have to use gas line extenders, which may degrade gas quality and reduce pressure.
- Carefully route the gas lines from the bottle to the analyzer, avoiding overlapping or entangling gas lines. This will make maintenance easier.
- Label both ends of the gas lines before connecting for ease in identification and maintenance.
- Ensure that all gas bottles are closed before connecting them to the analyzer.

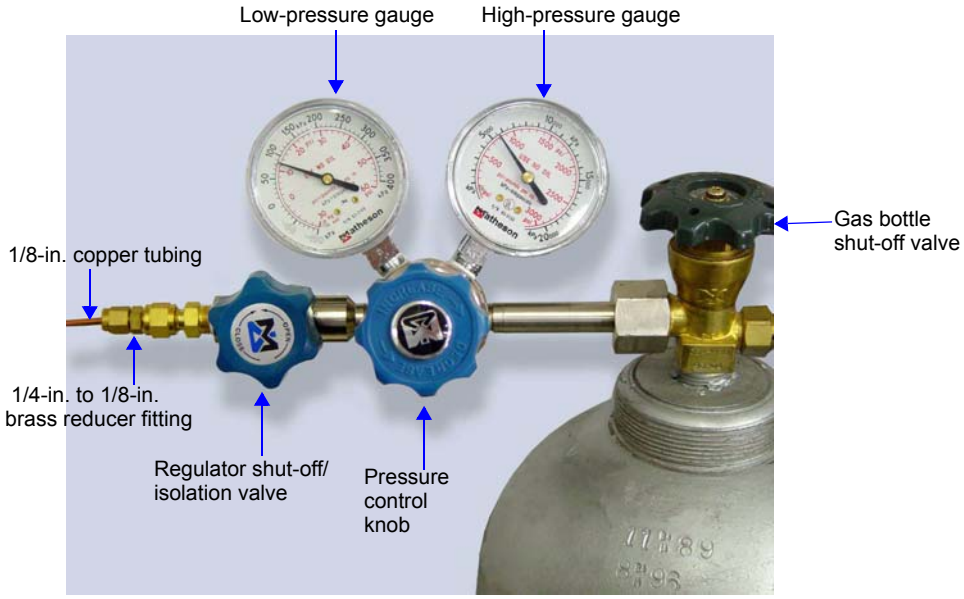
The instructions below describe a typical installation. Some configurations require additional components, such as regulator expansion kits, when one gas source will be used for several operations or when the gas bottle cannot be located close to the analyzer.

Connecting a Gas Regulator to the Gas Supply

Dual-stage regulators must be used for all gas supplies.

1. Attach an appropriate dual-stage regulator to the gas supply bottle or gas line you want to connect and tighten with a wrench. Leave the gas bottle shut-off valve closed until instructed otherwise.

If an expansion kit is required, attach it to the regulator before proceeding. (Refer to the instructions included in the expansion kit.)



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/isolation valve and tighten the valve nut with a 9/16-in. wrench.

Do not overtighten the fittings. Doing so could collapse the copper tubing and cause a leak.

3. Attach the gas inlet line to the regulator, reducer fitting, or regulator expansion and tighten with a wrench.

4. Purge the regulator as follows:

It is important to purge the regulator before proceeding. This will prevent contamination of the analysis gas.

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

b. Turn the pressure regulator control knob fully counterclockwise.

c. Slowly open the gas bottle valve by turning it counterclockwise, then quickly 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, make sure the gas supply equipment is adequately vented to prevent purging the regulator and tubing into the lab atmosphere in the following step.

e. Turn the pressure regulator control knob clockwise until the low pressure gauge indicates 15 psig (100 kPag). Open each regulator shut-off valve by turning it counterclockwise. Open the gas bottle shut-off valve. Flow gas for 10 to 30 seconds, then close each valve. These actions purge the regulator and gas inlet tubing.

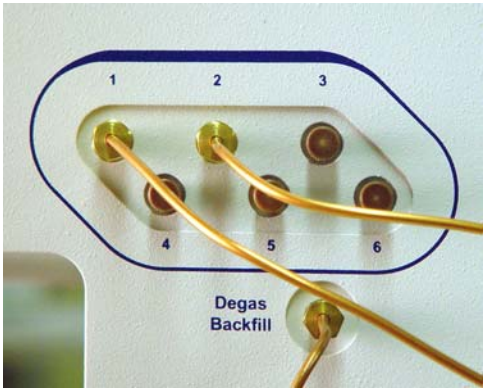
f. Ensure that the gas shut-off bottle valve is completely closed (turn clockwise).

5. Repeat steps 1 through 4 for each gas supply bottle to be attached to the analyzer.

6. Proceed to the next section to connect the copper delivery tubing to the analyzer.

Connecting the Gas Delivery Tubing to the Analyzer

The gas inlet ports are located on the right side panel of the analyzer. The ports are labeled **1** through **6**, and **Degas Backfill** for the backfill gas.



To change the gas attached to one of the ports:

1. Make sure the port valve is closed (refer to **"Enable Manual Control" on page 5-26**).

2. Detach the gas line from the desired port.

3. Attach the new gas line to the port.

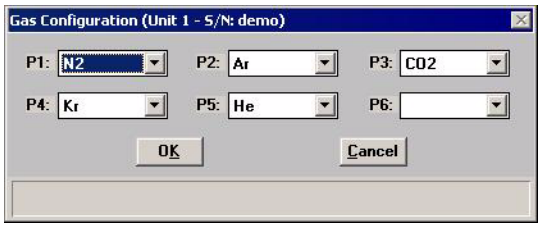
4. Make sure any unused gas inlet ports are plugged.

5. Record the gas installed in the port in the analysis program as described in the following section.

Specifying Gas Ports

When you change the gas attached to a port on the analyzer, you must enter the change in the analysis program as follows.

1. Select **Unit 1 > Unit Configuration** from the main menu. The Unit Configuration dialog is displayed.
2. Click **Gas**. The Gas Configuration dialog is displayed.



3. Click on the down-arrow at the port to which you attached a gas line and choose the appropriate gas.
4. Click **OK** to close the dialog, and then click **OK** again to close the Unit Configuration dialog.

Performing a Reference Material Analysis

When you have doubts about the integrity of analysis data or when you would just like to ensure the highest quality data, perform a reference analysis. Reference Material Kits are available from Micromeritics (refer to chapter 9, **ORDERING INFORMATION**). Each kit contains reference material and an instruction booklet. Follow the instructions in the booklet to perform the analysis and review the results.

9 ORDERING INFORMATION

Analysis System Components and Accessories	9-3
Sample Tubes and Components	9-6

ORDERING INFORMATION

The ASAP 2420 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:

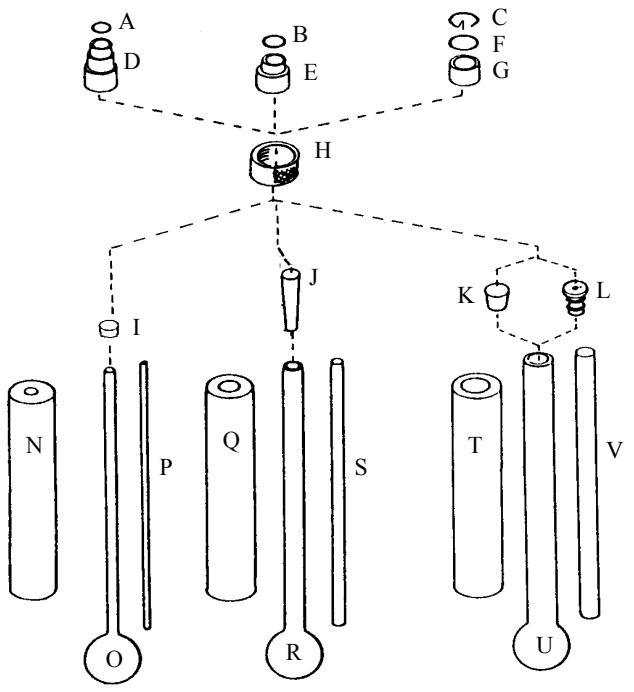
Analysis System Components and Accessories

Part Number	Item and Description
	Accessories
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
201-25830-00	Dewar, 2.5 liter
240-25901-00	Dewar depth gauge
202-31707-00	Dewar cover (used during analyses)
240-25895-00	Dewar insulator (used when Dewar is in the lowered position)
004-25103-00	Ferrule, front, Teflon 1/4 in.
004-25104-00	Ferrule, rear, Nylon 1/4 in.
004-25913-00	Ferrule set, 1/8-in. PEEK, used on gas lines
290-25846-00	Copper tube, for gas inlet, 1/8-in. diameter
004-25549-00	Reducer, 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 assembly, CGA 580, no-pressure relief valve (30 psig for N_2, He, Kr)

Part Number	Item and Description
004-33601-00	Regulator 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)
004-62230-32	Gas pressure regulator, CGA 320 fitting (CO2), 30 psig
003-26044-00	Heating mantle with type K thermocouple, 450 ºC maximum, 24 V
003-26045-00	Heating mantle with type K thermocouple, 450 ºC maximum, 24 V, side-laced with extended leads
230-25808-00	Heating mantle clip
242-42811-01	Operator's manual
004-16821-00	Reference material, high surface area
004-16816-00	Reference material, low surface area (krypton)
004-54609-00	Sample tube brush
240-25853-00	Sample tube funnel
240-14855-00	Sample tube rack
240-32805-00	Sample tube support, assists sample weighing
200-25840-00	Saturation pressure (Po) tube, includes isothermal jacket
004-25006-00	O-ring, size 006, Buna-N, for Po tube
250-25608-00	Valve gasket, Kel-F, for analysis manifold
250-25627-00	Valve plunger, Buna-N seal, for analysis manifold and fast evacuation valve on degas manifold
201-22600-00	Valve spring, for Buna-N plunger, for analysis manifold and fast evacuation valve on degas manifold
003-22617-00	Valve, for analysis gas inlets and degas inlet (yellow top)
003-22626-00	Valve, for degas ports and vacuum valve on inlet manifold (green top)

Part Number	Item and Description
	Vacuum System Components
062-00100-00	Vacuum foreline pump, oil-free, for systems with high-vacuum option, 115/230 VAC
062-00001-11	Vacuum pump, oil-based, with built-in anti-suckback valve, 100/120 VAC; includes hose kit and alumina oil vapor trap
062-00001-23	Vacuum pump, oil-based, with built-in anti-suckback valve, 220/240 VAC; includes hose kit and alumina oil vapor trap
062-33002-00	Activated alumina oil vapor trap, for one vacuum pump
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
	Software
202-33024-00	DataMaster; includes software and operator's manual. This program allows you to generate and manipulate data on a computer other than the one controlling the analyzer.
242-33011-00	Software package, ASAP 2420 Confirm; includes current version of software and operator's manual
	Micropore Upgrade Option
242-33002-00	Upgrade kit; enables you to perform micropore analyses on ASAP 2420 Krypton units.

Sample Tubes and Components



Part Number	Item and Description
	Sample Tubes and Components
004-25466-00 A	O-ring, size 010, for 1/4-in. sample tube
004-25022-00 B	O-ring, size 012, for 3/8-in. sample tube
260-25891-00 C	Opener, seal frit, for 1/2-in. sample tube
240-25803-00 D	Ferrule, 1/4 in.
300-25826-00 E	Ferrule, 3/8 in.
004-25469-00 F	O-ring, size 014, for 1/2-in. sample tube
260-25843-00 G	Ferrule, 1/2 in.
300-25824-00 H	Nut, sample tube
004-32604-00 I	Cap (stopper), for 1/4-in. sample tube
004-32004-00 J	Stopper, for 3/8-in. sample tubes
240-32000-00 K	Stopper, for 1/2-in. sample tube

Part Number	Item and Description
260-25890-00 L	Seal Frit with built-in check valve for air-sensitive samples (for 1/2-in. tubes only)
202-25901-00 N	Isothermal jacket, 1/4 in.
240-61001-00 O	Sample tube, 1/4 in.
240-61014-00 P	Volume displacement insert, 1/4 in.
202-25902-00 Q	Isothermal jacket, 3/8 in.
240-61002-00 R	Sample tube, 3/8 in.
240-61015-00 S	Volume displacement insert, 3/8 in.
202-25903-00 T	Isothermal jacket, 1/2-in. sample tube
240-61003-00 U	Sample tube, 1/2 in.
240-61016-00 V	Volume displacement insert, 1/2 in.

A ASAP SERIES SAMPLE DATA WORKSHEET

micromeritics®

ASAP 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)
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

2200, 2300, and 2400 Series	B-3
2500 Series	B-19
4000 Series	B-28
6100 Series	B-44
6500 Series	B-54

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, 2300, 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.

2204- Communication attempt with <application file name> timed out. Please check application directory for file.

Cause: A secondary software application may have become corrupt and cannot be started.

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.

2331- Logged-in Windows user is not an application user.

Cause: You attempted to log into the application but the user logged into Windows is not an application user.

Action A: Close the application, restart Windows, and log in as a windows user. Then log into the application.

Action B: Contact your Administrator to assign application rights to the current logged-in Windows user.

2333- You did not enter user name or password.

Cause: You attempted to access the application without entering your user name or password.

Action: Enter your Windows OS user name and password in the User Logon dialog box.

2334- Incorrect user name or password.

Cause A: You entered an incorrect user name or password in the User Logon dialog box.

Action A: Reenter your user name and password. Ensure that Caps Lock is not enabled; entries are case-sensitive.

Cause B: You entered your user name but failed to enter your password.

Action B: Reenter your user name and password.

Cause C: You entered your application password in both fields.

Action C: Enter your Windows OS user name in the user name field and your password in the password field.

2335- Attempt of unauthorized access was detected. Please contact administrator.

Cause: You have exceeded the allowed number of log-on attempts.

Action: Contact your Administrator.

2336- This user account is locked. Please contact administrator.

Cause: You entered an incorrect password more than the allowed number of times (by default, this is three), and our account has been locked (disabled).

Action: Contact your Administrator to unlock your account.

2337- The user is not an administrator.

Cause: You attempted to log in and perform a task that only the Administrator is allowed to perform.

Action: Contact your Administrator to complete the task.

2338- Your password has expired or needs to be changed. You will not be able to log in without changing your password.

Cause: The allowed time using a password has expired.

Action: Change your password.

2339- Your password will expire soon. Do you want to change your password?.

Cause: The allowed time for using a password is about to expire.

Action: Change your password.

2340- You must log in as a Windows OS user to change a password.

Cause: You attempted to change your password but you are not logged in as a Windows OS user.

Action: You cannot change your password unless you are logged in as a Windows OS user. Close the application, then restart Windows and log in as a user.

2341- Error writing file or the user is not Windows OS user.

Cause: You attempted to log into the application but have been denied access. Your password file may have become corrupt.

Action: Contact your Administrator.

2342- Please enter a valid password.

Cause: You entered an incorrect password.

Action: Reenter your password in both fields. Ensure that **Caps Lock** is not enabled; entries are case-sensitive.

2343- This password is already in use. Please enter a valid password.

Cause: You attempted to change your password but entered one that has already been used.

Action: Choose a different password.

2344- Application locked.

Cause: Your account has been disabled.

Action: Contact your Administrator.

2345- Attempt of unauthorized access was detected.

Cause: You attempted to log into the application but entered and incorrect login name or password.

Action: Confirm your login name and password.

2346- This user account is disabled. Please contact an administrator.

Cause: You attempted to log into the application using a password and user name that have been disabled.

Action: Contact your Administrator.

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 a Micromeritics 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: Several ASAP 2420 files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the ASAP 2420 program.

Action: Select **Yes** to update the directory information with data from each new file. This operation may take a minute.

Select **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 you wish to access.

2434- File (file name) — Subset # (number) wrote (number) bytes, expected (number) bytes.

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 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 nn).

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.

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 {number}) 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 {number}) 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 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, you may wish to 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 file containing initialization information and system options information used during program startup does not exist.

Action: Run the ASAP 2420 Setup program, select Change analyzer setup, and create the control file used by the ASAP 2420.

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 box until you correct the errors.

Action: Check the entries, correct the errors, and close the dialog box

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, press **Accept**.

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 2420 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 2420 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.

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.

2535- The requested directory is not a repository location. Resetting to the default location.

Cause: The directory last accessed is no longer available; the application defaults to the default location.

Action: Choose an appropriate directory to select a file.

2536- The maximum number of versions has already been created for this file.

Cause: The current file contains the maximum number of 676 versions.

Action: If additional versions are required, select **File**, **Save As** to create a new file from the current version.

2537- The directory (name) has already been added to the repository.

Cause: An internal conflict with the repository structure has occurred.

Action: Contact your Micromeritics service representative.

2538- The archive location name (name) has already been used.

Cause: An internal conflict with the repository structure has occurred.

Action: Contact your Micromeritics service representative.

2539- The repository location (name) cannot be created.

Cause: You attempted to create a new directory (category) or subdirectory (sub-category) but the directory-creating function of the Windows operating system has prevented you from doing so.

Action: Contact your Administrator to review security permissions and local disk space.

2540- Error getting log file name.

Cause: The Confirm application was unable to access (or create) the system log.

Action: Contact your Administrator to review user rights and local disk space.

2541- Windows and application user's rights conflict.

Cause: You attempted to log into the application, but the windows user rights and Confirm application rights do not agree.

Action: Contact your Administrator to review user rights.

2542- The file (name) could not be written. Check that the directory permissions are correct.

Cause: The system has prevented you from saving your file.

Action: Contact your Administrator to review user rights and local disk space.

2543- Cannot read from the repository. Check that the directory permissions are correct.

Cause: The system has prevented you from accessing a file in the repository.

Action: Contact your Administrator to review user rights for the application and the Windows operating system.

2544- Cannot write to the repository. Check that the directory permissions are correct

Cause: The system has prevented you from saving your file to the repository.

Action: Contact your Administrator to review user rights for the application and the Windows operating system.

2545- The repository appears to be empty. Check that the directory permissions are correct.

Cause: The application was unable to determine the repository structure.

Action: Contact your Administrator to review user rights and local disk space.

2546- The file list for the current repository location is missing. Contact an administrator to update the list.

Cause: You attempted to access a file but the repository does not contain any files.

Action: Contact your Administrator to review user rights and local disk space.

2547- The instrument is busy. Exiting the application will result in the termination of the analysis. Continue with program Exit?

Cause: You tried to exit the ASAP 2420 program with an analysis in progress.

Action: Click **Yes** to terminate the analysis and exit the ASAP 2420 program. Click **No** to continue the analysis.

2549- Error accessing online manual file (code #).

Cause: The operator's manual file could not be located.

Action A: Reinstall the application.

Action B: Copy of the contents of the \manual folder on the setup CD to the application directory.

2550- Attempts to acquire the instrument's status timed out.

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.

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.

4002- Thermal Transpiration correction had no effect.

Cause: The Thermal transpiration correction option was selected on the Report Options dialog box. However, the correction did not change any pressure by more than one percent.

Action: De-select this option to disable this message. This correction is only meaningful for very low pressures.

4003- Error Converting Pressures.
4004- Error Computing Quantity 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.

4005- Pressures were not smoothed. Not enough pressures below 0.10 P/Po.

Cause: The **Smooth pressures below 0.10 P/Po** option was selected on the Report Options dialog. There must be at least 10 pressures within this range for smoothing to occur.

Action: Deselect this option to disable this 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 desired reports in the sample's report options and save the sample file.

4010- Summary Report was not included in the Selected Reports.

Cause: You did not select a plot or a table for any of the Selected Reports in the Report Options dialog; you selected the reports only to generate an entry in the summary. However, the Summary was not included in the Selected Reports.

Action: Select a plot or table in one or more of the Selected Reports, and/or add the Summary to the Selected Reports.

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 box.

4012- Psat gas in sample file does not match Psat gas in unit.

Cause: You attempted to start an analysis using a sample information file in which the Psat gas does not match the Psat gas entered in the unit configuration.

Action: If necessary, attach the appropriate gas bottle, then enter the gas in the Unit Configuration dialog box.

4014- File (name) is not a valid file for conversion.

Cause: The file selected for conversion is not a MICMOS 2400 or 2405 file.

Action: Select only files that have been created by MICMOS 2400 or 2405 programs.

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 or CD 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.

4021- The entered Po value (Po and Temperature Options of the Analysis Conditions) is outside the range of the pressures listed in the Psat vs. Temperature Table (Adsorptive Properties).

Cause: The temperature that corresponds to the entered Po cannot be found because the entered Po is outside of the range of values in the Psat vs. T table.

Action A: Enter a Po that is within the range of the table.

Action B: Use a different Psat vs. T table.

4022- The entered bath temperature value (Po and Temperature Options of the Analysis Conditions) is outside the range of the temperatures listed in the Psat vs. Temperature Table (Adsorptive Properties).

Cause: The Po that corresponds to the entered temperature cannot be found because the entered temperature is outside of the range of values in the Psat vs. T table.

Action A: Enter a temperature that is within the range of the table.

Action B: Use a different Psat vs. T table.

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.

4026- Cannot calculate Dubinin-Astkahov: bad least squares data.

Cause: Less than two selected data points are within the fitted pressure range.

Action: Edit the calculation assignments on the Collected Data dialog, or change the fitted pressure range on the Dubinin report options dialog.

4027- Fewer than two sample files have data suitable for heat of adsorption reports.

Cause: Less than two of the sample files you selected for heat of adsorption reports contain appropriate data.

Action: Edit the Quantity Adsorbed table, or select other sample files.

4028- Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

Cause: Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.

Action: Access the Dubinin Report Adsorptive options in the sample file and enter an appropriate value for the analysis gas.

4029- At least two fitted data points are needed for Alpha-S calculations.

Cause: Fewer than two data points fall within the selected Alpha-s range.

Action: Edit the calculation assignments or the fitted Alpha-s range, or use a different reference curve.

4030- Preparations failed in primary data.

Cause: Appropriate data were not available to generate the report.

Action: This message was preceded by a different error message; refer to the cause/action of the preceding message.

4031- Not enough points with a relative pressure in the range (n,n)

Cause: Fewer than two data points selected for the Dubinin report falls within the selected relative pressure range.

Action: Edit the calculation assignments or the fitted relative pressure range.

4033- Not enough points to generate Dubinin Tabular Report.

Cause: There are fewer than two valid data points available for the Dubinin tabular reports.

Action: Examine the calculation assignments in the Collected Data dialog of the sample file. You must have at least two micropore pressures selected for inclusion in the Dubinin report.

4034- Fewer than 2 points available for Dubinin calculations.

Cause: There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.

Action: Examine the calculation assignments in the Collected Data dialog of the sample file. You must have at least two micropore pressures selected for inclusion in the Dubinin report.

4035- Cannot calculate optimized Astakhov exponent: Not enough points with a relative pressure in the range [(pressure), (pressure)].

Cause: There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.

Action: Examine the calculation assignments in the Collected Data dialog for the sample file. You must have at least two pressures in the given range selected for inclusion in the Dubinin report.

4036- Fewer than 2 points available for Horvath-Kawazoe calculations.

Cause: You must select at least two data points for inclusion in the Horvath-Kawazoe analysis on the Collected Data dialog. No report will be produced.

Action: From the Collected Data dialog, select the data points to be analyzed.

4037- Computations failed while processing the primary data set. No reports will be produced.

Cause: The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded with another one containing additional information.

Action: Refer to the number of the error message which preceded this one for an explanation.

4038- Fewer than 2 points available for the Langmuir Qm computation. Cheng/Yang correction will not be applied.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.

Action: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to prevent this message from appearing on future reports.

4039- The isotherm does not meet the constraints of the Cheng/Yang assumption. Cheng/Yang correction will not be applied.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.

Action A: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to prevent this message from appearing on future reports.

Action B: Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them and reproduce the Horvath-Kawazoe reports.

4040- The value of Qm computed from the Langmuir equation is too low. The pore size will not be computed for all data points.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.

Action: The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of Vm. The pore size will not be calculated for data points with an invalid volume adsorbed. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options dialog to clear this message.

4041- Cheng/Yang correction is inappropriate for some P/Po.

Cause: The Cheng/Yang correction is usually inappropriate for any P/Po above the isotherm knee. In some instances, the computed pore sizes may decrease above the knee. While it is possible to include these relative pressures (usually above 0.1 P/Po) in the analysis, the computed pore sizes for these pressures are usually meaningless.

Action: Change the data points selected for the Horvath-Kawazoe report to include only relative pressures at or below the knee of the isotherm, or change the Horvath-Kawazoe report options so that the Cheng/Yang correction is not applied.

4042- 0.0 cannot be a starting or ending pressure for a geometric progression from low pressure.

Cause: You selected to generate a pressure table from a geometrically progressing range.

Action: Change the 0.0 entered value.

4043- 1.0 cannot be a starting or ending pressure for a geometric progression toward saturation.

Cause: You selected to generate a pressure table from a geometrically progressing range.

Action: Change the 1.0 entered value.

4044- Points in the Langmuir report pressure table lie outside the collected data.

Cause: Calculation assignments are not being used and more than one of the report pressure table points is above the range of the collected data, and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4045- Points in the report pressure table lie outside the collected data.

Cause: Calculation assignments are not being used and more than one of the report pressure table points is above the range of the collected data, and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4046- (file name) could not be opened for reading.

Cause: A thickness curve file could not be opened.

Action: If the problem persists, restart your computer and optionally perform a media integrity check (using ScanDisk).

4047- Warning: An error occurred while reading (file name).

Cause: An error happened during a read operation of a thickness curve file.

Action: If the problem persists, restart your computer and optionally perform a media integrity check (using ScanDisk).

4051- The sample (file name) does not have any data in the range of the requested quantities adsorbed.

Cause: The sample's data cannot be interpolated to any of the quantities adsorbed.

Action: Edit the quantities adsorbed so that they are within the range of the collected data.

4052- Fewer than two points are selected for this report.

Cause: At least two points are required for the BET calculations.

Action: Edit the calculation assignments for the BET report.

4053- At least two data points must be selected for t-Plot calculations.

Cause: At least two points are required for the t-Plot calculations.

Action: Edit the calculation assignments for the t-Plot report.

4054- Fewer than two data points are inside the fitted thickness range.

Cause: At least two points must be within the fitted thickness range for the t-Plot calculations.

Action A: Edit the calculation assignments for the t-Plot report.

Action B: Edit the fitted thickness range in the t-Plot report editor.

4055- A positive BET surface area could not be calculated.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the t-Plot report editor.

4056- A positive Langmuir surface area could not be calculated.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the t-Plot report editor.

4057- At least two data points are needed for Freundlich calculations.

Cause: Less than two data points have been selected for the Freundlich report; at least two are required.

Action: Select Freundlich points on the Collected Data dialog. If calculation assignments are not being used, edit the Freundlich Report options, **Absolute pressure range** in the sample file.

4058- At least two data points are needed for Temkin calculations.

Cause: Less than two data points have been selected for the Temkin report; at least two are required.

Action: Select Temkin points on the Collected Data dialog. If calculation assignments are not being used, edit the Temkin Report options, **Absolute pressure range** in the sample file.

4059- Fewer than 2 points available for MP-Method calculations.

Cause: At least two points are required for the MP-Method calculations.

Action: Edit the calculation assignments for the MP-Method report.

4060- Sample (file name) contains no data points.

Action: An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.

Cause: Repeat the **save as t-curve** or **save as alpha-S** operation after opening a sample that has collected data.

4061- The t-curve must contain at least 2 points.

Cause: At least two points are required in a thickness curve definition.

Action: Edit the thickness curve.

4062- Error during report preparation.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

4063- The data requested on this report are not available.

Cause: There is no information in the sample log to report.

Action: You selected a sample file for which no instrument operations have been used. Select a sample file with a status of **Prepared**, **Preparing**, **Analyzing**, or **Complete** to obtain a valid sample log report.

4067- No data points are within the range of pressures in the reference isotherm.

Cause: There are no collected data points within the range of pressures in the reference isotherm.

Action: Select data points that are in the range of the reference isotherm, or select a more appropriate reference isotherm.

4068- No points were selected for the f-Ratio report.

Cause: The collected data column for the f-Ratio report does not have any points selected.

Action: Edit the collected data dialog and select points for the f-Ratio report.

4069- Dosing method choice is invalid. The Analysis Conditions choice of Absolute pressure dosing requires that the Adsorptive Properties (Dosing Method) is set to Normal.

Cause: There is an incompatibility between the analysis conditions choice of **Absolute pressure dosing** and the adsorptive properties **dosing** method.

Action: Edit one of the choices.

4070- Unable to load deconvolution model (name).

Cause: For some reason, the list of available models was corrupted, therefore, the model selected could not be loaded for the deconvolution.

Action: Exit the program and reinstall the software, then try again.

4071- The selected pressures points do not form a valid set for deconvolution.

Cause: The data points selected for analysis do not contain enough information to allow a DFT data reduction.

Action A: Edit data points in the table of the Collected Data dialog, or select another sample file. At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction.

Action B: Edit the pressure range on the DFT report options dialog.

4072- The range of pressures selected is too small to deconvolute using this model.

Cause: A null result was found using the selected model.

Action: Edit data points in the table of the Collected Data dialog, or select another sample file. At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT data reduction.

4073- The analysis gas (name) does not match the model gas (name).

Cause: The model assumes a specific gas and the sample file uses a different one.

Action: Select a model that assumes the same gas.

4076- Invalid sample file - Adsorptive Properties "Non-condensing adsorptive" cannot be used unless Analysis Conditions "Absolute pressure dosing" is selected.

Cause: You selected a sample file that has **Non-condensing adsorptive** selected in the Adsorptive Properties but does not have **Absolute pressure dosing** selected in Analysis Conditions.

Action A: If you plan to use a non-condensing adsorptive, select **Absolute pressure dosing** on the Analysis Conditions dialog.

Action B: If you are not using a non-condensing adsorptive, deselect the **Non-condensing adsorptive** option on the Adsorptive Properties dialog, or choose an adsorptive that you are sure in not non-condensing.

4077- Cannot get surface area for: (file name)

Cause: The Isotherm report for the named overlay file has **Per gram** selected for the **Volume Adsorbed**, and the Isotherm report for the primary file has a surface area option selected for the **Volume Adsorbed**.

Action A: Edit the Isotherm report for the named overlay file and select a surface area option for **Volume Adsorbed**.

Action B: Click **Overlays** on the Report options dialog of the primary file and remove the named overlay file from the list.

4078- Slope and Y-Intercept cannot be determined from the selected points.

Cause: The Langmuir report cannot be generated from the selected points.

Action: Edit the calculation assignments in the Langmuir column on the Collected Data dialog.

4079- Points found with negative pressure values will not appear on the graph.

Cause: Collected data contains negative pressure values.

Action: Negative pressure points are automatically removed from isotherm graphs. If you wish to have negative pressure values appear on the graph: Open the sample file and click the **Collected Data** tab. Deselect **Use calculation assignments** and then select the points with negative pressures as Outliers.

6100 Series

6100- Unable to establish the TCP connection with the instrument.

Cause: A communications problem between the computer and the analyzer has occurred.

Action A: Check the cable connection between the computer and the analyzer.

Action B: Select **Unit [n] > Unit Configuration** from the main menu and verify that the TCP/IP configuration for the computer and the analyzer are correct.

Action C: Exit the 2420 application and turn off the analyzer. Then turn on the analyzer and restart the 2420 application. If the problem persists, contact your Micromeritics representative.

6101- Configured serial number does not match instrument.

Cause: The serial number of the instrument is different than the serial number the application expects.

Action: Run the setup program on the installation CD; choose the **Remove** operation to remove the incorrect serial number. Then choose the **Add** operation to add the correct serial number.

6102- The instrument (unit number) is not calibrated.

Cause: One or more calibration operations have not been performed.

Action: If using the setup CD, reinstall the calibration files. If this does not correct the problem, contact your Micromeritics representative.

6200 Series

6201- 'GetOpenFile()' reports error code (number). File selection cannot be made.

Cause: You specified a file name which does not exist.

Action: Confirm that the file name used is valid and that the file exists.

6203- No serial number available. Unit cannot initialize.

Cause: The ASAP 2420 control file does not contain a serial number for the unit.

Action: Run the ASAP 2420 Setup program, select **Change analyzer setup**, and define the serial number for the instrument.

6204- This sample has an invalid status and cannot be used for degassing.

Cause: You tried to degas a sample that has a status other than No Analysis.

Action: Select a sample with a status of No Analysis.

6206- Error dosing.

Cause: The degas system failed to dose the backfill gas to a pressure of 850 mmHg.

Action: The gas regulator may be set too low or turned off.

6209- Analysis startup failed. (Secondary message number and text.)

Cause: The ASAP 2420 Program was unable to start an analysis with the indicated unit.

Action: Refer to the specified error number and text for instructions.

6212- Calibrating. This action must be completed before the application exits.

Cause: The unit is calibrating.

Action: Do not exit the application until the calibration is complete.

6213- Error overheating on port (number). Current = (temperature), Target = (temperature), Limit = (temperature).

Cause: A heating mantle and/or its thermocouple are not plugged into the correct port.

Action: Verify heating mantle connections. If the problem persists, contact your service representative.

6216- Instrument could not initialize. Reason: (reason).

Cause: An internal processing and/or hardware error has occurred.

Action: The specific reason given may identify a possible corrective action. Contact your service representative if you continue to receive this error message.

6217- Error thermocouple unplugged Current = (temperature), Target = (temperature).

Cause: The thermocouple is not plugged into the correct degas port.

Action: Check the heating mantle connections for the specified degas port.

6219- The file name (name) has already been used.

Cause: You entered a file name that is already in use.

Action: Enter a unique file name.

6220- There are currently no available ports to start a degas operation.

Cause: All degas ports are in use.

Action: Wait until the degas operation is complete on a port, then another degas operation may be started.

6221- An evacuation cannot be started because all degas ports are currently in use.

Cause: All degas ports are in use.

Action: Wait until the degas operation is complete on a port, then an evacuation can be started on the port.

6222- At least one port must be selected before an evacuation can be started.

Cause: You clicked **Start** on the Evacuate Degas dialog without first selecting a port.

Action: Select the port you wish to evacuate, then select **Start.**

6226- An analysis cannot be started because all ports are currently is use.

Cause: You tried to start an analysis while all ports are in use.

Action: Wait until the analysis completes on a port, then another analysis can be started.

6227- File (name) cannot be analyzed. It is currently being edited.

Cause: You tried to start an analysis using a sample information file that is currently being edited.

Action: Close the edit session on the sample information file, then the file can be used for analysis.

6228- Analysis gas in sample file does not match analysis gas in other active samples.

Cause: You tried to start an analysis using a sample file that contains an analysis gas other than the gas currently being used. All current analyses must use the same analysis gas.

Action A: Change the gas in the sample file to use the current analysis gas.

Action B: Wait until the current analyses are complete, then run the sample.

6229- This file already selected for the analysis.

Cause: You selected a sample file that is already being used for an analysis.

Action: Select another sample information file.

6230- Dosing method choice is invalid. The analysis requires that Adsorptive Properties\Dosing Method\is set to Normal.

Cause: You tried to perform a nitrogen or nitrogen-like analysis with **From Port 6** selected as the dosing method.

Action: Choose **Normal** for Dosing Method in the Adsorptive Properties dialog, then start the analysis.

6231- Dosing method choice is invalid. The Krypton analysis requires that Adsorptive Properties\Dosing Method\is set to From Port 6.

Cause: You tried to perform a Krypton analysis method with **Normal** selected as the dosing method.

Action: Choose **From Port 6** for Dosing Method in the Adsorptive Properties dialog, then start the analysis.

6232- Po gas in sample file does not match Po gas in other active samples.

Cause: You tried to start an analysis using a sample file that contains a Po gas other than the Po gas currently being used. All current analyses must use the same Po gas.

Action A: Change the gas in the sample file to use the current Po gas.

Action B: Wait until the current analyses are complete, then run the sample.

6233- (Gas name) gas in sample file does not match (gas name) gas in other active samples.

Cause: You tried to start an analysis using a sample file that contains an analysis gas other than the gas currently being used. All current analyses must use the same analysis gas.

Action A: Change the gas in the sample file to use the current analysis gas.

Action B: Wait until the current analyses are complete, then run the sample.

6234- Analysis Conditions Pressures or Dose Increments table has no entries.

Cause: You tried to start an analysis using a sample file that contains no entries in either the pressure table or dose increments table.

Action: In the Isotherm Collection group box on the Analysis Conditions dialog, select either **Target Pressures** or **Dose Increments**, then enter values in the associated table.

6236- An evacuation cannot be started because all analysis ports are currently in use.

Cause: You tried to evacuate one or more analysis ports while all ports are in use.

Action: Wait until the current operation completes to evacuate a port.

6240- Leak test failed.

Cause: With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.

Action: Check sample tube fitting and ensure that it is securely attached to the port. Then restart the analysis.

6241- (Unit [number]) Analysis canceled: Maximum time exceeded before the elevator reached the UP (or DOWN) position.

Cause A: The maximum time for the analysis was exceeded before the elevator reached the UP (or DOWN) position. Ice may be present in the bottom or the neck of the Dewar.

Action A: Check the Dewar and remove ice if necessary. Refer to **"Checking and Cleaning the Analysis Port Dewars" on page 8-8**. Then restart the analysis.

Cause B: The Psat tube is interfering with elevator movement.

Action B: Make sure the Psat tube is close to the sample tube and the Dewar cover is over both the sample and Psat tubes. Then restart the analysis.

6244- (Unit [number]) Analysis canceled: Time limit exceeded while storing analysis gas in Psat tube.

Cause A: The time limit (10 minutes) was exceeded while storing the analysis gas in the Psat tube. The Psat gas regulator may be set too low or turned off.

Action A: Set the Psat gas regulator to 15 psig (1 bar). Then restart the analysis.

Cause B: The Psat gas bottle is empty.

Action B: Connect a new Psat gas bottle. Then restart the analysis.

Cause C: The Psat tube fitting is loose.

Action C: Check the Psat tube fittings and ensure that the tube is attached securely to the port. Then restart the analysis.

6247- Warm Freespace failure: P1 = (pressure), P2 = (pressure).

Cause A: Helium gas line is not properly connected to the analyzer.

Action A: Check the helium gas connections.

Cause B: A valve, transducer, or elevator has failed.

Action B: Contact your Micromeritics representative.

6248- Cold Freespace failure: P3 = (pressure).

Cause A: Helium gas line is not properly connected to the analyzer.

Action A: Check the helium gas connections.

Cause B: A valve, transducer, or elevator has failed.

Action B: Contact your Micromeritics representative.

6249- Zeroing of a transducer failed. Analysis Canceled.

Cause: The transducer did not respond correctly.

Action: Contact your service representative.

6250- Matching of Sample port transducer failed. Analysis Canceled.

Cause: The transducer did not respond correctly.

Action: Contact your service representative.

6251- Matching of Psat port transducer failed. Analysis Canceled.

Cause: The transducer did not respond correctly.

Action: Contact your service representative.

6264- Krypton gas is not condensing in sample port 6 (pressure)(pressure units).

Cause A: The krypton gas may be contaminated.

Action A: Evacuate the krypton gas inlet line.

Action B: Manually verify the saturation pressure of the krypton gas:

1. Evacuate station 6 with an empty tube.
2. Backfill with krypton gas to 20 mmHg.
3. Raise the Dewar in Station 6.

These steps should condense your krypton to a pressure below 3 mmHg.

Cause B: The Dewar on sample port 6 does not contain enough cryogen.

Action B: Refill the Dewar.

6500 Series

6510- Main pressure transducer overrange detected.
6511- Sample transducer (number) overrange detected.
6512- Po transducer (number) overrange detected.
6513- Hardware pressure overrange detected.

Cause: A manifold pressure over 1000 mmHg was detected by the hardware.

Action: Observe caution when operating the analyzer manually. If the problem persists, contact your service representative.

6516- (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.

6517- (Unit [number]) Analysis canceled: Total volume dosed greater than (volume) cm3 is not allowed.

Cause A: More than (volume) cm^3 has been dosed onto the sample, possibly due to leaks in the system.

Action A: Perform leak checks on the system. Refer to **"TESTING THE ANALYZER FOR LEAKS" on page D-1**.

Cause B: The value for the Po may be too low, causing the instrument to mistake condensation for actual adsorption.

Action B: Change the analysis variables to directly measure the value of Po during the experiment.

Cause C: 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 C: 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.

6519- (Unit [number]) Analysis canceled: Psat gas is not condensing.

Cause A: The working Dewar does not contain enough bath liquid.

Action A: Fill the Dewar, then retry the operation.

Cause B: The Psat tubing from the regulator to the instrument is contaminated.

Action B: Pump out the tubing.

Cause C: The Psat gas is contaminated.

Action C: Replace the Psat gas supply.

6520- Power failure detected. The sample is in an unknown condition. A run termination will be performed for safety.

Cause: A total power failure occurred while an analysis was in progress.

Action: Allow the analysis to terminate.

6521- Time limit exceeded while dosing manifold to (target pressure) with (gas).

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.

6522- 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 D, **"TESTING THE ANALYZER FOR LEAKS" on page D-1**.

6523- Analysis suspended: Time limit exceeded while evacuating sample (unrestricted).

Cause: The maximum time for evacuating the sample through the unrestricted valve was exceeded.

Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Verify that the sample is properly degassed; then restart the analysis.

6524- Analysis suspended: Time limit exceeded while evacuating sample (servo).

Cause: The maximum time for evacuating the sample through the restricted valve was exceeded. Possible causes are a leak in the sample tube fitting, a crack in the sample tube, or a poorly degassed sample.

Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Verify that the sample is properly degassed; then resume the analysis.

6525- Time limit exceeded while backfilling manifold to (target pressure) 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 15 psig (1 bar). Then resume the analysis.

Cause B: The gas bottle is empty.

Action B: Connect a new gas bottle. Then resume the analysis.

C CALCULATIONS

Saturation Pressure (Po)	C-3
Relative Pressure	C-3
Free Space	C-4
Volume Adsorbed	C-5
Equilibration	C-6
Thermal Transpiration Correction	C-7
BET Surface Area	C-8
Langmuir Surface Area	C-9
t-Plot	C-10
BJH Pore Volume and Area Distribution	C-12
Horvath-Kawazoe	C-21
Dubinin-Astakhov	C-29
MP-Method	C-33
Freundlich Isotherm	C-35
Temkin Isotherm	C-35
DFT (Density Functional Theory)	C-36
Summary Report	C-38
SPC Report Variables	C-40
References	C-42

CALCULATIONS

Saturation Pressure (Po)

Po method is selected by the user on the Po and Temperature Options dialog box. It may be entered, measured, or calculated from temperature.

If entered, Po = user entered value

If measured, Po = equilibrated pressure reading after saturating Po tube

If calculated from temperature, interpolate Po from the Psat vs Temperature table on the Po and Temperature Options dialog box, given the analysis bath temperature

Relative Pressure

If Po was measured:

For points taken before the previous Po measurement,

$$Po_I = Po_1 + \frac{(Po_2 \angle Po_1) \times (T_I \angle To_1)}{(To_2 \angle To_1)}$$

For points taken after the previous Po, use the previous Po:

$$Po_I = Po_I$$

If Po was entered, use the entered Po:

$$Po_I = Po_E$$

If Po was calculated, use the calculated Po:

$$Po_I = Po_C$$

Calculate relative pressure for the I^{th} data point:

$$P_{rel_1} = \frac{P_1}{Po_I}$$

where

Po_I = saturation pressure for the I^{th} data point (mmHg)
Po_1 = most previous measured saturation pressure before I^{th} data point (mmHg)
Po_2 = first measured saturation pressure after the I^{th} data point (mmHg)
Po_C = calculated Po
Po_E = entered saturation pressure (mmHg)
T_I = time when the I^{th} data point was taken (minutes)
To_1 = time when Po_1 was measured (minutes)
To_2 = time when Po_2 was measured (minutes)
P_{rel_I} = relative pressure for the I^{th} data point (mmHg)
P_I = absolute pressure for the I^{th} data point, taken at equilibrium (mmHg)

Free Space

Free-space volumes are calculated using the following equations:

$$V_{FW} = \frac{V_{LOW}}{T_1} \times \left[\frac{P_1}{P_2} \angle 1\right] \times T_{STD}$$

$$V_{FC} = \frac{V_{LOW}}{T_2} \times \left[\frac{P_1}{P_3} \angle 1\right] \times T_{STD}$$

$$V_{ABT} = \frac{V_{FC} \angle V_{FW}}{1 \angle \frac{T_{ABT}}{T_{RM}}}$$

where:\

P_1 = system manifold pressure before dosing helium onto sample (mmHg)
P_2 = system manifold pressure after dosing helium onto sample (mmHg)
P_3 = sample pressure after raising Dewar and equilibrating with helium (mmHg)
T_{STD} = standard temperature (273.13 K)
T_{RM} = approximate room temperature (298 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_{FC} = volume of free space, cold (cm^3 at standard temperature)
V_{FW} = volume of free space, warm (cm^3 at standard temperature)
V_{ABT} = portion of cold free space at analysis bath temperature: used in non-ideality

correction (cm^3 at standard temperature)

T_{ABT} = analysis bath temperature (K), from the Po and Temperature Options dialog box

Volume Adsorbed

For the I^{th} dose:

$$Ngas_I = Ngas_{I\angle 1} + V_{LOW} \times \left[\frac{Psys1_I}{Tsys1_I} \angle \frac{Psys2_I}{Tsys2_I}\right] \times \frac{T_{STD}}{P_{STD}}$$

$$Nads_I = Ngas_I \angle Psam_I \div P_{STD} \times (V_{FC} + Psam_I \times V_{ABT} \times C)$$

$$V_I = \frac{Nads_I}{Wsam}$$

where:

C = non-ideality correction factor, user-entered on Adsorptive Properties dialog box

$Ngas_I$ = 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.

$$P_{AVG} = \frac{\angle 36(P_{11} + P_1) + 9(P_{10} + P_2) + 44(P_9 + P_3) + 69(P_8 + P_4) + 84(P_7 + P_5) + 89(P_6)}{429}$$

$$P_{CHG} = \frac{5(P_{11} \angle P_1) + 4(P_{10} \angle P_2) + 3(P_9 \angle P_3) + 2(P_8 \angle P_4) + (P_7 \angle P_5)}{110}$$

where the numerical constants are from the Savitzky-Golay 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.

Thermal Transpiration Correction

During data reduction, thermal transpiration correction is applied to the data if the user selected **Thermal transpiration correction** from the Report Options dialog box. Starting with the first collected pressure, the following calculations are performed until the pressure ratio (PC/P) is greater than or equal to 0.99.

$$Y = \frac{P \times SD \times MD^2}{2.33 \times T} \times 10^3$$

$$\mu = \frac{1 + G \times Y}{1 + H \times Y}$$

$$F = \frac{1}{(\alpha \times Y^2) + (\beta \times Y) + \mu}$$

$$PC = \left\{ 1 \angle \left[F \angle \left(F \left(\frac{T_{ABT}}{T_{RM}} \right)^{0.5} \right) \right] \right\} \times P$$

where:

P	=	equilibrated collected pressure measured by gauge at temp T_{RM} (mmHg)
SD	=	inside diameter of sample tube (mm), from the Report Options dialog box
MD	=	thermal transpiration hard sphere diameter of gas (Å), from the Adsorptive Properties dialog box
T	=	average temperature $[T_{ABT} + T_{RM}] / 2$ (K)
α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
G	=	Weber's coefficient, 2.5
H	=	Weber's coefficient, 2
T_{ABT}	=	analysis bath temperature (K), from the Po and Temperature Options dialog box
T_{RM}	=	room temperature (298 K)
PC	=	corrected equilibrated pressure at temperature T_{ABT} (mmHg)
F, Y, μ	=	intermediate values for subsequent calculations

BET Surface Area

For each point designated for surface area calculations, the BET[2] transformation is calculated as follows:

$$B_1 = \frac{P_{rel_1}}{(1.0 \angle P_{rel_1}) \times N_{ads_1}}$$

where B_I is in units of g/cm^3 STP.

A least-squares fit is performed on the (P_{relI} ,B_I) designated pairs where P_{relI} is the independent variable and B_I is the dependent variable. The following are calculated:

a. Slope (S g/cm^3 STP)

b. Y-intercept (Y_{INT} g/cm^3 STP)

c. Error of the slope (S_{ERR} g/cm^3 STP)

d. Error of the y-intercept (YI_{ERR} g/cm^3 STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

BET Surface Area (m^2/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times (S + Y_{INT})}$$

where

CSA = analysis gas molecular cross-sectional area (nm^2), user-entered on the Adsorptive Properties dialog box

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

Error of the BET Surface Area (m^2/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}{}^2 + YI_{ERR}{}^2)^{0.5}}{Y_{INT} + S}$$

Langmuir Surface Area

For each point designated for surface area calculations, the Langmuir[3] transformation is calculated as follows:

$$L_I = \frac{P_{rel_I}}{N_{ads_I}}$$

where L_I is in units of g/cm^3 STP

A least-squares fit is performed on the (P_{relI} ,L_I) designated pairs where P_{relI} is the independent variable and L_I is the dependent variable. The following are calculated:

a. Slope (S g/cm^3 STP)

b. Y-intercept (Y_{INT} g/cm^3 STP)

c. Error of the slope (S_{ERR} g/cm^3 STP)

d. Error of the y-intercept (YI_{ERR} g/cm^3 STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Langmuir Surface Area (m^2/g):

$$SA_{LAN} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times S}$$

where

CSA = analysis gas molecular cross-sectional area (nm^2), user-entered on the Adsorptive Properties dialog box

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{S}$$

Langmuir b Value:

$$b = (Y_{INT})(V_M)$$

Error of the Langmuir Surface Area (m^2/g):

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

t-Plot

For each point designated for t-Plot[4] calculations, the following calculations are done:

Thickness for the I^{th} point (Å):

$$t_I = HP1 \times \left[\frac{HP2}{\ln(P_{rel_I})}\right]^{HP3} \qquad \text{(Halsey[5])}$$

or

$$t_I = \left[\frac{HJP1}{HJP2 \angle \log(P_{rel_I})}\right]^{HJP3} \qquad \text{(Harkins and Jura[6])}$$

where:

t_I	=	thickness for I^{th} point
HP1	=	Halsey parameter #1
HP2	=	Halsey parameter #2
HP3	=	Halsey parameter #3
HJP1	=	Harkins and Jura parameter #1
HJP2	=	Harkins and Jura parameter #2
HJP3	=	Harkins and Jura parameter #3
P_{relI}	=	relative pressure for the Ith point (mmHg)

A least-squares analysis fit is performed on the (t_I, N_{adsI}) data pairs where t_I is the independent variable and $Nads_I$ is the dependent variable. Only the values of t_I between t_{MIN} and

t_{MAX}, the minimum and maximum thickness, are used. The following are calculated:

a. Slope (S cm^3/g-Å STP)

b. Y-intercept (Y_{INT} cm^3/g STP)

c. Error of the slope (S_{ERR} cm^3/g-Å STP)

d. Error of the Y-intercept (YI_{ERR} cm^3/g STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

External Surface Area (m^2/g):

$$SA_{EXT} = \frac{(S\ cm^3/g \angle A\ STP) \times (10^{10}\ A/m) \times (D\ cm^3\ liquid/cm^3\ STP)}{F \times (10^6\ cm^3/m^3)}$$

where

F = surface area correction factor, user-entered on the t-Plot Report Options dialog box

D = density conversion factor, user-entered on the Adsorptive Properties dialog box

Micropore Surface Area (m^2/g):

$$SA_{\mu P} = SA_{TOT} + SA_{EXT}$$

where SA_{TOT} is the BET surface area if the user enabled the BET report exclusively, or Langmuir surface area if the user enabled the Langmuir report exclusively. If neither report has been selected, SA_{TOT} is the BET surface area value calculated using a set of default parameters.

Micropore Volume (cm^3 liquid/g):

$$V_{\mu P} = (Y_{INT}\ cm^3/g\ STP) \times (D\ cm^3\ liquid/cm^3\ STP)$$

BJH Pore Volume and Area Distribution

For adsorption data, the relative pressure and volume adsorbed data point pairs collected during an analysis must be arranged in reverse order from which the points were collected during analysis. All calculations are performed based on a desorption model, regardless of whether adsorption or desorption data are being used.

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (Pr), volume adsorbed (Va) pairs from (Pr_1, Va_1) to (Pr_N, Va_N) where (Pr_N = 0, Va_N = 0) is assumed as a final point. Each data pair represents an interval boundary (or desorption step boundary) for intervals i=1 to i=N-1 where N = total number of (Pr, Va) pairs.

Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin[7] equation. After the core has evaporated, a layer of adsorbate remains on the wall of the pore. The thickness of this layer is calculated for a particular relative pressure from the thickness equation. This layer becomes thinner with successive decreases in pressure, so that the measured quantity of gas desorbed in a step is composed of a quantity equivalent to the liquid cores evaporated in that step plus the quantity desorbed from the pore walls of pores whose cores have been evaporated in that and previous steps. Barrett, Joyner, and Halenda[8] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used on the ASAP 2420 is an implementation of the BJH method.

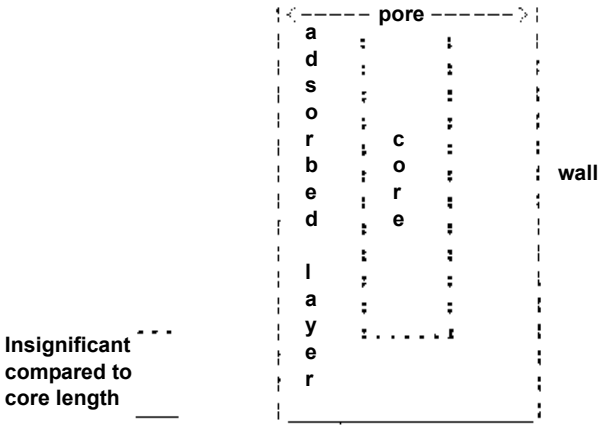
Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

a. The ***core*** - evaporates all at once when the critical pressure for that radius is reached; the relationship between the core radius and the critical pressure is defined by the Kelvin equation.

b. The ***adsorbed layer*** - composed of adsorbed gas that is stripped off a bit at a time with each pressure step; the relationship between the thickness of the layer and the relative pressure is defined by the thickness equation.

c. The ***walls of the cylindrical pore*** - the diameter of the empty pore is required to determine the pore volume and pore area. End area is neglected.



Calculations

The volumes adsorbed (Va) are converted to the liquid equivalent volumes (V1, cm^3/g):

$$VI_I = (Va_I)(D)$$

where D is the Density Conversion Factor from the Adsorptive Properties dialog box.

The relative pressure (Pr_1) is assumed to be close to unity so that substantially all the pores in the sample are filled.

The corresponding Kelvin core radius is calculated. Only pores smaller than this size will be included:

$$Rc_I = \frac{\angle A}{(1+F)[\ln(Pr_I)]}$$

where

A = adsorbate property factor (the BJH Adsorptive Options dialog box)
F = fraction of pores open at both ends (from the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box); assumed to be zero for desorption

This radius will be adjusted for the thickness of the adsorbed layer during subsequent calculation steps.

The thickness of the remaining adsorbed layer at this relative pressure is calculated:

$$Tw_1 = HP1\left[\frac{HP2}{\ln(Pr_1)}\right]^{HP3}$$

where

HP1, HP2, and HP3 are Halsey Parameters 1, 2, and 3 (respectively) from the Halsey Thickness Equation dialog box.

These calculations illustrate the use of the Halsey thickness equation. If the Harkins/Jura equation was selected, substitute the following wherever the thickness equation appears:

$$Tw_1 = \left[\frac{HJ1}{HJ2 \angle \log(Pr_1)}\right]^{HJ3}$$

where

HJ1, HJ2, and HJ3 are Harkins-Jura Parameters 1, 2, and 3 (respectively) from the Harkins-Jura Thickness Equation dialog box.

The following calculations (a-c) are made for each relative pressure interval based on the increment of volume desorbed during that interval. The variable I refers to the interval number, that is I=1 for the first interval from Pr_1 to Pr_2, and so on. J refers to each previous interval during which new pores were found. K refers to the total number of intervals in which new pores have been found. K is also the number of lines reported on the BJH table for collected data.

a. The thickness of the adsorbed layer at the end of the interval is calculated as follows:

$$Tw_{I+1} = HP1\left[\frac{HP2}{\ln(Pr_{I+1})}\right]^{HP3}$$

(For the last pressure interval from the lowest Pr_I to zero relative pressure, $Tw_{I+1} = 0$.)

For the first pressure interval, there are no previously opened pores so the volume desorbed from walls of previously opened pores is zero ($Vd_1 = 0$), and the remainder of Step a is skipped.

The change in thickness of the wall layer due to desorption from previously opened pores is calculated:

$$\Delta Tw = Tw_1 \angle Tw_{I+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all previously opened pores:

$$CSA_J = \pi[(Rc_j + \Delta Tw)^2 \angle Rc_j{}^2](10^{\angle 16}\ cm^2/A^2)$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_I = \sum(Lp_J)(CSAa_J) \text{ for all previously opened pores}$$

where LP_J = length of previously opened pores as calculated in Step b(2).

b. The physical processes occurring for this pressure interval are determined as follows:

(1) If Vd_I is greater than the current increment of volume desorbed (Vl_I - Vl_{I+1}), desorption from walls only is occurring. Total surface of walls exposed thus far (cm^2/g) is calculated as follows:

$$SA_W = \sum\pi(LP_J)(D_{avg_J})(10^{\angle 8}\ cm/A) \quad \text{for all previously opened pores}$$

where

D_{avgJ} = weighted average pore diameter calculated below in Step b(2).

A new layer thickness (ΔTw) that will not overcompensate for the actual volume desorbed in this interval is calculated:

$$\Delta Tw = \frac{(V1_I \angle V1_{I+1})(10^8\ A/cm)}{SAw_I}$$

Since no cores are evaporated in this pressure interval, no new pores are revealed. Thus no ending Kelvin radius and average pore diameter are calculated for this interval. Note that this means the report may have fewer tabulated intervals on the collected data report than experimental pressure intervals.

(2) If Vd_I is less than the volume increment desorbed during this interval, the remaining volume is due to new pores with core evaporation taking place in this interval. K, the number of intervals with new pores exposed, is increased by 1. (For the interval from the lowest to zero relative pressure, no new pore volume is calculated and the rest of Step b is skipped.)

The volume desorbed from newly opened pores in this interval is calculated as follows:

$$Vc_I = (VI_I \angle VI_{I+1}) \angle Vd_I$$

The Kelvin radius for the end of the interval is calculated as follows:

$$Rc_{K+1} = \frac{\angle A}{(1+F)[\ln(Pr_{I+1})]}$$

All new pores opened in this interval are represented by one pore having a length-weighted average pore diameter and a corresponding length sufficient to account for the required volume of adsorbate. The weighted average pore diameter is calculated as follows:

$$D_{avg_K} = \frac{(2)(Rc_K + Rc_{K+1})(Rc_K)(Rc_{K+1})}{Rc_K{}^2 + Rc_{K+1}{}^2}$$

D_{avgK} is the diameter of a pore which would have a surface area that is the average of the areas for pores with radius and, if its length was the mean of the lengths at those radii.

The relative pressure corresponding to is calculated as follows:

$$P_{avg_K} = \ln^{\angle 1}\left[\frac{\angle 2A}{(1+F)(D_{avg_K})}\right]$$

The thickness of the adsorbed layer at this pressure is calculated as follows:

$$Tw_{avg_K} = HP1\left[\frac{HP2}{\ln(P_{avg_K})}\right]^{HP3}$$

The decrease in thickness of the wall layer by desorption from the walls of new pores during the lower portion of the pressure interval is calculated as follows:

$$\Delta Td = Tw_{avg_K} \angle TW_{I+1}$$

The cross-sectional area of the newly opened pores is calculated as follows:

$$CSAc_K = \left[\frac{Davg_K}{2} + \Delta Td\right]^2 (10^{\angle 16}\ cm^2/A^2)$$

The length of the newly opened pores is calculated as follows:

$$LP_K = \frac{Vc_I}{CSAc_K}$$

c. Pore diameters and radii are adjusted for the change in thickness of the adsorbed wall layer during this interval. If new pores were opened during this interval, the average diameter is adjusted by the change in layer thickness during the second portion of the desorption interval as follows:

$$D_{avg_{Knew}} = D_{avg_{Kold}} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as follows:

$$D_{avg_{Jnew}} = D_{avg_{Jold}} + 2(\Delta Tw)$$

(not including D_{avgK})

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as follows:

$$Rc_{Jnew} = Rc_{Jold} + \Delta Tw$$

for all except Rc_{K+1}.

Steps a to c are repeated for each pressure interval.

After the above calculations have been performed, the diameters corresponding to the ends of the intervals are calculated as follows:

$$Dp_J = 2(rc_J)$$

for all Rc_J including Rc_{K+1}.

The remaining calculations are based on $DP_{(I)}$, $D_{avg(I)}$, and $LP_{(I)}$. These calculations are only done for values that fall between the Minimum BJH diameter and the Maximum BJH diameter specified by the operator on the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box.

1) Incremental Pore Volume (Vp_I, cm^3/g):

$$Vp_I = \pi(Lp_I)\left[\frac{D_{avg_I}}{2}\right]^2 10^{16}\ cm^2/A^2$$

2) Cumulative Pore Volume ($Vp_{CUM(1)}$, cm^3/g):

$$VP_{CUM_{(1)}} = \sum Vp_J\ for\ (J \le 1)$$

3) Incremental Surface Area (SA_I, m^2/g):

$$SA_I = \pi(LP_I)(10^{\angle 2}\ m/cm)(D_{avg_I})(10^{\angle 10}\ m/A)$$

4) Cumulative Surface Area ($SA_{CUM(I)}$, m^2/g):

$$SA_{CUM_{10}} = \sum SA_J\ for\ J \leq 1$$

5) dV/dD pore volume (dV/dD_I, cm^3/g-A):

$$dV/dD_I = \frac{VP_I}{Dp_I \angle Dp_{I+1}}$$

6) dV/dlog(D) pore volume ($dV/dlog(D)_I$, cm^3g):

$$dDv/d\log(D)_I = VP_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right)$$

7) dA/dD pore area (dA/dD_I, m^2/g-A):

$$dA/dD_I = \frac{SA_I}{Dp_I \angle Dp_{I+1}}$$

8) dA/dlog(D) pore area [$dA/dlog(D)_I$, m^2/g]:

$$dA/d\log(D)_I = SA_I/\log\left(\frac{Dp_I}{Dp_{I+1}}\right)$$

For fixed pore size tables (if selected), the following calculations are performed:

1) Average Fixed Pore Size (DF_{avgJ}, A):

$$DF_{avg_J} = \frac{Dp_{F_J} + Dp_{F_{J+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with between the Minimum BJH diameter and the Maximum BJH diameter.

2) Cumulative Pore volume (VpF_{CUMI}, cm^3/g):

$$VpF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = Dp_{J+1} and Y = VP_{CUMJ}, using an AKIMA semi-spline interpolation.

3) Incremental Pore Volume (VpF_I, cm^3/g):

$$VpF_I = VpF_{CUM_I} \angle VpF_{CUM_{I\angle 1}}$$

where $VpF_{CUM0} = 0$.

4) Cumulative Surface Area (SAF_{CUMI}, m^2/g):

$$SAF_{CUM_I} = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = Dp_{J+1} and Y = SA_{CUMJ}.

5) Incremental Surface Area (SAF_I, m^2/g):

$$SAF_I = SAF_{CUM_I} \angle SAF_{CUM_{I\angle 1}}$$

where $SAF_{CUM0} = 0$.

6) dV/dD pore volume ($dV/dDpF_I$, cm^3/g-A)):

$$dV/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = dV/dD_J.

7) dV/dlog(D) pore volume [$dV/dlog(DpF)_I$, cm^3/g]:

$$dV/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function X = D_{avgJ} and Y = $dV/dlog(D)_J$.

8) dA/dD pore area ($dA/dDpF_I$, m^2/g-A):

$$dA/dDpF_I = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = D_{avgJ}$ and $Y = dA/dD_J$.

9) dA/dlog(D) pore area [$dA/d\log(DpF_I)$, m^2/g]:

$$dA/d\log(DpF_I) = INTERP(DpF_{I+1})$$

where INTERP(x) is the value interpolated from the function $X = D_{avgJ}$ and $Y = dA/d\log(D)_J$.

Compendium of Variables

Va	=	quantity adsorbed expressed as a volume (cm^3/g STP)
Vl	=	liquid equivalent volume of volume adsorbed (cm^3/g)
D	=	density conversion factor from the Adsorptive Properties dialog box (cm^3/cm^3 STP)
Pr	=	relative pressure
Dp	=	pore (or core) diameter (A)
Rc	=	Kelvin radius (A) of core
A	=	adsorbate property factor; from the BJH Adsorptive Options dialog box
F	=	fraction of pores open at both ends; from the BJH Adsorption Report Options dialog box or the BJH Desorption Report Options dialog box
ΔTw	=	thickness of adsorbed layer desorbed during interval (A)
Tw	=	thickness of remaining adsorbed wall (A)

HP1, HP2, and HP3 are Halsey Parameters from the Halsey Thickness Equation dialog box.

HJ1, HJ2, and HJ3 are Harkins and Jura Parameters from the Harkins/Jura Thickness Equation dialog box.

Vd	=	volume of gas desorbed from walls of previously opened pores (cm^3/g)
Davg	=	average pore diameter (A)
CSAa	=	annular cross-sectional area of the desorbed layer (cm^2)
CSAc	=	cross-sectional area of opening of newly opened pores (cm^2)
SAw	=	total surface area of walls exposed (cm^2/g)
ΔTd	=	thickness of layer desorbed from walls of newly opened pores (A)
Vc	=	volume desorbed from cores of newly opened pores (cm^3/g)
Lp	=	length of pore (cm/g)

Horvath-Kawazoe

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/Po) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito/Foley)
- Sphere Pore Geometry (Cheng/Yang)

Slit Pore Geometry (original HK)

When you use the original Horvath-Kawazoe[9] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\frac{P}{Po} = \frac{K}{RT}\times\frac{IP\times 10^{32}\ JA^4/J\ cm^4}{\sigma^4\times(L\angle 2\times d_0)}\times\left[\frac{\sigma^4}{3\times(L\angle d_0)^3}\angle\frac{\sigma^{10}}{9\times(L\angle d_0)^9}\angle\frac{\sigma^4}{3\times d_0^3}+\frac{\sigma^{10}}{9\times d_0^9}\right]$$

where

K = Avogadro's number (6.023×10^{23})

R = gas constant (8.31441×10^7 ergs/mole K)

T = analysis bath temperature (K), from an entered or calculated value on the Po and Temperature Options dialog box

σ = gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

where:

Z_S = sample equilibrium diameter at zero interaction energy (Å) from the Horvath-Kawazoe Physical Properties dialog box

Z_A = zero interaction energy diameter from the Horvath-Kawazoe Physical Properties dialog box

d_0 = $\frac{D_A + D_S(A)}{2}$

where:

D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical Properties dialog box

D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical dialog box

L = pore width (nucleus to nucleus) (Å)
P = equilibrium pressure (mmHg)
Po = saturation pressure (mmHg)
IP = interaction parameter (10^{-43} ergs-cm^4) from the Horvath-Kawazoe Report Options dialog box

Cylinder Pore Geometry (Saito/Foley)

When you use the Saito-Foley[10] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{3}{4}\frac{\pi K}{RT} \times \frac{IP \times 10^{32}\ JA^4/J\ cm^4}{{d_0}^4} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1 \angle \frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} \angle \beta_k\left(\frac{d_0}{r_p}\right)^4\right\}\right]$$

where

K = Avogadro's number (6.023×10^{23})
R = gas constant (8.31441×10^7 ergs/mole K)
T = analysis bath temperature (K), from an entered or calculated value on the Po and Temperature Options dialog box
L = pore width (nucleus to nucleus) (Å)
P = equilibrium pressure (mmHg)
Po = saturation pressure (mmHg)
IP = interaction parameter (10^{-43} ergs-cm^4) from the Horvath-Kawazoe Report Options dialog box

$d_0 = \frac{D_A + D_S(A)}{2}$

where:
D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical Properties dialog box
D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical dialog box

$\alpha_k = \left(\frac{\angle 4.5 \angle k}{k}\right)^2$ $\alpha_{k \angle 1}, \alpha_0 = 1.0$

$\beta_k = \left(\frac{\angle 1.5 \angle k}{k}\right)^2$ $\beta_{k \angle 1}, \beta_0 = 1.0$

r_p = radius of the cylindrical pore, $\frac{L}{2}$

Sphere Pore Geometry (Cheng/Yang)

When you use the Cheng/Yang[11] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{Po}\right) = \frac{6(N_1\varepsilon^*_{12} + N_2\varepsilon^*_{22})L^3 \times 10^{32}\ J\ A^4/J\ cm^4}{RT(L \angle d_0)^3}$$

$$\left[\angle\left(\frac{d_0}{L}\right)^6\left(\frac{1}{12}T_1 + \frac{1}{8}T_2\right) + \left(\frac{d_0}{L}\right)^{12}\left(\frac{1}{90}T_3 + \frac{1}{80}T_4\right)\right]$$

where

R = gas constant (8.31441 x 10^7 ergs/mole K)

T = analysis bath temperature (K), from an entered or calculated value on the Po and Temperature Options dialog

$d_0 = \frac{D_A + D_S(A)}{2}$

where:

D_A = molecular diameter (Å) from the Horvath-Kawazoe Physical Properties dialog

D_S = diameter of sample atom (Å) from the Horvath-Kawazoe Physical dialog

L = pore width (nucleus to nucleus) (Å)

P = equilibrium pressure (mmHg)

Po = saturation pressure (mmHg)

$N_1 = 4\pi L^2 N_S$, where N_S = number of sample atoms/cm^2 at monolayer

$N_2 = 4\pi (L - d_0)^2 N_A$, where N_s = number of gas molecules/cm^2

$\varepsilon^*_{12} = \frac{A_S}{4{d_S}^6}$, where $A_S = \frac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$

$\varepsilon^*_{22} = \frac{A_A}{4{D_A}^6}$, where $A_A = \frac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$

$T_1 = \frac{1}{(1 \angle S)^3} \angle \frac{1}{(1 + S)^3}$

$T_2 = \frac{1}{(1 + S)^2} \angle \frac{1}{(1 \angle S)^2}$

$T_3 = \frac{1}{(1 \angle S)^9} \angle \frac{1}{(1 + S)^9}$

$$T_4 = \frac{1}{(1+S)^8} \angle \frac{1}{(1 \angle S)^8}$$

$$\text{where } S = \frac{L \angle d_0}{L}$$

Cheng/Yang Correction

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometrics:

$$\ln\left(\frac{P}{Po}\right) = G(L) \angle \left[1 \angle \frac{1}{\theta} \ln\left(\frac{1}{1 \angle \theta}\right)\right]$$

where

G(L) = one of the Horvath-Kawazoe equations given above

θ = degree of void filling; θ is estimated by first computing the monolayer capacity (V_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the volume adsorbed over V_m.

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients -

$$A_S = \frac{6 \times MC^2 \times \alpha_S \times \alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$$

$$A_A = \frac{3 \times MC^2 \times \alpha_A \times \chi_A}{2}$$

where

MC^2 = kinetic energy of electron (0.8183 x 10^{-6} erg)

α_S = polarizability of sample atoms (cm^3)

α_A = polarizability of gas molecule (cm^3)

χ_S = diamagnetic susceptability of sample atom (cm^3)

χ_A = diamagnetic susceptability of gas molecule (cm^3)

$$IP = (N_A \times A_A) + (N_S \times A_S)$$

where:

N_A = number of gas molecules/cm^2 at monolayer from the Horvath-Kawazoe Physical Properties dialog

N_S = number of sample atoms/cm^2 from the Horvath-Kawazoe Physical Properties dialog

Refer to **Interaction Parameter Components** later in this Appendix for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):
(Shell to Shell)

$$AL_I = L_I \angle DS$$

Cumulative Pore Volume (cm^3/g):

$$V_{CUM_I} = V_I \times D$$

where

D = density conversion factor (cm^3 liquid/cm^3 STP) on the Adsorptive Properties dialog box

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_I} = \frac{V_{CUM_I} \angle V_{CUM_{I\angle 1}}}{AL_I \angle AL_{I\angle 1}}$$

Median Pore Width (Å):

$$V_{HALF} = \frac{V_{CUM_N}}{2}$$

$$D_{MED} = 10\left[\log(D_L) + [\log(V_{HALF}) \angle \log(V_L)] \times \frac{\log(D_G) \angle \log(D_L)}{\log(V_G) \angle \log(V_L)}\right]$$

where

V_{CUMN} = total cumulative pore volume (V_{CUMI}) for points designated for Horvath-Kawazoe calculations
V_{HALF} = 50% of total cumulative pore volume
V_L = cumulative pore volume (V_{CUMI}) for first point less than V_{HALF}
V_G = cumulative pore volume (V_{CUMI}) for first point greater than V_{HALF}
D_L = pore width (L_I) that corresponds to V_L
D_G = pore width (L_I) that corresponds to V_G

Table C-1. Interaction Parameters

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value*
Argon	87.3	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.61 5.89 3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.20 9.20 5.08
	273.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.34 9.35 5.22
	194.65	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.72 9.72 5.60
Nitrogen	77.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.84 6.53 3.49

*The interaction parameter is entered in the Horvath-Kawazoe Report Options dialog in the following field:

Interaction parameter: (calculated value) x 10^{-43} ergs-cm^4

The following values were used to calculate the values in Table C-1.

Carbon-Graphite

D_S = 3.40
N_S = 3.845 x 10^{15}
χ_S = 1.05 x 10^{-29} (Ross/Olivier)
13.5 x 10^{-29} (Horvath/Kawazoe, implicit)
α_S = 1.02 x 10^{-24}

Zeolite

D_S = 3.04
N_S = 3.75 x 10^{15}
χ_S = 1.94 x 10^{-29}
α_S = 0.85 x 10^{-24}

Nitrogen

D_A = 3.00
N_A = 6.71 x 10^{14}
χ_A = 3.6 x 10^{-29}
α_A = 1.76 x 10^{-24}

Argon

D_A = 2.95
N_A = 7.608 x 10^{14}
χ_A = 3.22 x 10^{-29}
α_A = 1.63 x 10^{-24}

Carbon Dioxide

D_A = 3.23
N_A = 4.567 x 10^{14} (25 ºC)
5.45 x 10^{14} (0 ºC)
7.697 x 10^{14} (-78 ºC)
χ_A = 5.0 x 10^{-29}
α_A = 2.7 x 10^{-24}

D_A values are from van der Waal's constant.
N_A values are from liquid densities.
χ and α values are derived from data found in Ross and Olivier[12].

The physical parameters referenced in Saito/Foley are as follows:

Aluminophosphate

D_S = 2.60
N_S = 1.48 x 10^{15}
χ_S = 1.3 x 10^{-29}
α_S = 2.5 x 10^{-24}

Aluminosilicate

D_S = 2.76
N_s = 1.31 x 10^{15}
χ_S = 1.3 x 10^{-29}
α_S = 2.5 x 10^{-24}

Dubinin-Radushkevich

The Dubinin-Radushkevich[9] equation is as follows:

$$\log(V) = \log(Vo)\angle\frac{B \times T^2}{\beta} \times \left[\log\frac{Po}{P}\right]^2$$

where:

V	=	volume adsorbed at equilibrium pressure (cm^3/g STP)
Vo	=	the micropore capacity (cm^3/g STP)
Po	=	saturation vapor pressure of gas at temperature T (mmHg)
P	=	equilibrium pressure (mmHg)
B	=	a constant
β	=	the affinity coefficient of analysis gas relative to Po gas (for this application β is taken to be 1)
T	=	analysis bath temperature (K), from the Po and Temperature Options dialog box

For each point designated for Dubinin-Radushkevich calculations, the following calculations are done:

$$LV = log\ (V)$$

$$LP = \log\left(\frac{Po}{P}\right)^2$$

The intercept, log(Vo) can be found by performing a least-squares fit on the (LP,LV) designated pairs where LP is the independent variable and LV is the dependent variable. Assuming the adsorption of gas is restricted to a monolayer, Vo is the monolayer capacity. Based on this assumption, the following are calculated:

a. Slope (S cm^3/g STP)

b. Y-intercept (YI cm^3/g STP)

c. Error of the slope (S_{ERR} cm^3/g STP)

d. Error of the y-intercept (YI_{ERR} cm^3/g STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Monolayer Capacity (cm^3/g STP):

$$Vo = 10^{YI}$$

Error of Monolayer Capacity (cm^3/g STP):

$$Vo_{ERR} = Vo\ x\ (10^{YI_{ERR}} - 1.0)$$

Micropore surface area (m^2/g):

$$SDP = \frac{\sigma \times Vo \times (6.023 \times 10^{23})}{22414\ cm^3 \times (10^{18}\ nm^2/m^2)}$$

where

σ = molecular cross sectional area of gas (nm^2) from the Adsorptive Properties dialog box

Dubinin-Astakhov

The Dubinin-Astakhov equation is a follows:

$$\log(V) = \log(Vo) \angle \left[\frac{RT}{\beta E_0}\right]^N \times \left[\log\frac{Po}{P}\right]^N$$

where

V = volume adsorbed at equilibrium pressure (cm^3/g STP)
Vo = the micropore capacity (cm^3/g STP)
Po = saturation vapor pressure of gas at temperature T (mmHg)
P = equilibrium pressure (mmHg)
T = analysis bath temperature (K)
R = the gas constant (0.0083144 kJ/mol)
E_0 = characteristic energy (kJ/mole)
N = Astakhov exponent, may be optimized or user entered from the Dubinin Report Options dialog box
β = the affinity coefficient of the analysis gas relative to the Po gas, from the Dubinin Adsorptive Options dialog box

For each point designated for Dubinin-Astakhov calculations, the following calculations are done:

$$LV = log(V)$$

$$LP = \left[\log\frac{Po}{P}\right]^{N}$$

A least-squares fit is performed on the (LP,LV) designated pairs where LP is the independent variable and LV is the dependent variable. If the user selected **yes** for the Optimize Astakhov Exponent prompt, a systematic search for the optimum value of N is conducted by recalculating the linear regression and selecting the value of N that gives the smallest standard error of the y-intercept. The exponent N is optimized to within 10^{-4}. If the optimum value for N is not found in this range, an exponent of 2 is used. The following are calculated:

a. Slope (S cm^3/g STP)

b. Y-intercept (YI cm^3/g STP)

c. Error of the slope (S_{ERR} cm^3/g STP)

d. Error of the y-intercept (YI_{ERR} cm^3/g STP)

e. Correlation coefficient (Cc)

f. Optimized Astakhov exponent (N)

Using the results of the above calculations, the following can be calculated:

Monolayer Capacity (cm^3/g STP):

$$V_0 = 10^{YI}$$

Micropore Volume (cm^3/g):

$$W_I = (V_I \, x \, D)$$

where

D = density conversion factor (cm^3 liquid/cm^3 STP) from the Adsorptive Properties dialog box

Limiting Micropore Volume (cm^3/g):

$$W_0 = (V_0 \, x \, D)$$

where

D = density conversion factor (cm^3 liquid/cm^3 STP) from the Adsorptive Properties dialog box

Error of Limiting Micropore Volume (cm^3/g):

$$W_{0ERR} = W_0 \, x \, (10YI_{ERR} - 1.0)$$

Characteristic Energy (KJ/mole):

$$E = \frac{2.303 \times R \times T}{\beta(2.303 \times S)^{1/N}}$$

Modal Equivalent Pore Width (nm):

$$w_{mode} = 2 \times \left\{ \left[\frac{3N}{3N+1} \right]^{1/N} \times \left[\frac{k}{E_0} \right] \right\}^{1/3}$$

where

k = the interaction constant for benzene, 8.969 kJ•nm^3/mol

Maximum Differential Pore Volume (cm^3/g-nm):

This value is also known as *frequency of the mode*[10]

$$\frac{dV}{dw_{mode}} Max = 0.5 \times (3N+1) \times W_o \times \left[\frac{3N+1}{3N} \right]^{1/3N} \times \left[\frac{E_0}{k} \right]^{1/3} \times \exp\left(\angle \left[\frac{3N+1}{3N} \right] \right)$$

Mean Equivalent Pore Width (nm):

$$w_{mean} = 2 \times \frac{\left[\frac{k}{E_0}\right]^{1/3}}{\Gamma\left(\frac{3N+1}{3N}\right)}$$

Micropore surface area (m^2/g):

$$SDA = 1000 \times 2.0 \times W_0 \times \left[\frac{E_0}{k}\right]^{1/3} \times \Gamma\left(\frac{3N+1}{3N}\right)$$

Γ is calculated by a polynomial approximation[11] over the domain $0 \le x \le 1$ as follows:

$$\Gamma(x+1) = 1 + b_1 x + b_2 x^2 + b_3 x^3 + b_4 x^4 + b_5 x^5 + b_6 x^6 + b_7 x^7 + b_8 x^8 + \varepsilon(x) \mid \varepsilon(x) \mid \le 3 \times 10^{-7}$$

where

$b_1 = -0.57719\ 1652$
$b_2 = 0.98820\ 5891$
$b_3 = -0.89705\ 6937$
$b_4 = 0.91820\ 6857$
$b_5 = -0.75670\ 4078$
$b_6 = 0.48219\ 9394$
$b_7 = -0.19352\ 7818$
$b_8 = 0.03586\ 8343$

and where

$$x + 1 = \left(\frac{3N+1}{3N}\right)$$

Equivalent Pore Width (nm):

$$w_i = 2 \times \left[\frac{\angle\left(\frac{k}{E_0}\right)^N}{\ln(W_i) \angle \ln(W_0)}\right]^{1/3N}$$

dV/dw Pore Volume (cm^3/g-nm):

$$\frac{dV}{dw_i} = 0.5 \times W_0 \times 3N \times \left[\frac{k}{E_0}\right]^N \times \left(\frac{w_i}{2}\right)^{\angle(3N+1)} \times \exp\left[\angle\left(\frac{k}{E_0}\right)^N \times \left(\frac{w_i}{2}\right)^{\angle 3N}\right]$$

MP-Method

For each point designated for MP-method[10] calculations, the following calculations are done:

Thickness for the I^{th} point (Å):

$$t_I = HP1 \times \left[\frac{HP2}{\ln(P_{rel_I})}\right]^{HP3} \qquad \text{(Halsey}^{6}\text{)}$$

or

$$t_I = \left[\frac{HJP1}{HJP2 \angle \log(P_{rel_I})}\right]^{HJP3} \qquad \text{(Harkins and Jura}^{7}\text{)}$$

where:

t_I	=	thickness for I^{th} point
HP1	=	Halsey parameter #1
HP2	=	Halsey parameter #2
HP3	=	Halsey parameter #3
HJP1	=	Harkins and Jura parameter #1
HJP2	=	Harkins and Jura parameter #2
HJP3	=	Harkins and Jura parameter #3
P_{relI}	=	relative pressure for the I^{th} point (mmHg)

With the (t_I,V_I) data pairs, the Akima semi-spline interpolation method is used to interpolate volume adsorbed values based on thickness values that are evenly spaced 0.2 Angstrom apart starting at the first outlier point. Outliers are defined as those points have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_I = \frac{t_I + t_{I\angle 1}}{2}$$

Remaining pore surface area for the Ith point (m^2/g):

$$S_I = \frac{(V_I \angle V_{I\angle 1}) \times (D \times 10^{\angle 6}\ m^3/cm^3)}{(t_I \angle t_{I\angle 1}) \times 10^{\angle 10} m/A}$$

where

D = density conversion factor (cm^3 liquid/cm^3 STP) on the Adsorptive Properties dialog box

Incremental pore surface area occluded for the Ith point (m^2/g):

$$S_{INC_I} = S_{I\angle 1} \angle S_I$$

Cumulative pore surface area occluded for the Ith point (m^2/g):

$$S_{CUM_I} = S_{INC_I} + S_{INC_{I\angle 1}} + \ldots + S_{INC_1}$$

dA/dR pore surface area for the Ith point (m^2/g-Å):

$$\frac{dA}{dR_I} = \frac{S_{INC_I}}{t_I \angle t_{I\angle 1}}$$

Incremental pore volume occluded for the Ith point (cm^3/g):

$$V_{INC_I} = (S_{INC_I} \times 10^4\ cm^2/m^2) \times (R_I \times 10^{\angle 8}\ cm/\text{Å})$$

Cumulative pore volume occluded for the Ith point (cm^3/g):

$$V_{CUM_I} = V_{INC_I} + V_{INC_{I\angle 1}} + \ldots + V_{INC_1}$$

dV/dR pore volume for the Ith point (cm^3/g-Å):

$$\frac{dV}{dR_I} = \frac{V_{INC_I}}{t_I \angle t_{I\angle 1}}$$

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) $RTQ_S/q_0\alpha$.

DFT (Density Functional Theory)

The adsorption isotherm is known to convey a great deal of information about the energetic heterogeneity and geometric topology of the sample under study. The data of physical adsorption have been used for many years as the basis for methods to characterize the surface area and porosity of adsorbents. Real solid surfaces rarely approach ideal uniformity of structure. It is accepted that in general, the surface of even a nonporous material presents areas of greater or lesser attraction for adsorbed molecules.

This energetic heterogeneity greatly affects the shape of the adsorption isotherm with the result that simple theories such as the Langmuir and BET formulas can, at best, give only approximate estimates of surface area. Porous solids virtually are never characterized by a single pore dimension, but instead exhibit a more or less wide distribution of sizes. The observed adsorption isotherm for a typical material is therefore the convolution of an adsorption process with the distribution of one or more properties which affect that process. This was first stated mathematically by Ross and Olivier[11] for the case of surface energy distribution and has become known as the integral equation of adsorption.

The Integral Equation of Adsorption

In a general form for a single component adsorptive, the integral equation of adsorption can be written as

$$Q(p) = \int da\ db\ dc \ldots q(p, a, b, c \ldots)\, f(a, b, c \ldots) \qquad (1)$$

where

Q(p)	=	the total quantity adsorbed per unit weight at pressure p,
a,b,c,...	=	a set of distributed properties,
f(a,b,c,...)	=	the distribution function of the properties, and
q(p,a,b,c,...)	=	the kernel function describing the adsorption isotherm on unit surface of material with fixed properties a,b,c,...

Equation (1), a Fredholm integral of the first kind, is a member of a class of problems known as ill-posed, in that there are an infinite number of functional combinations inside the integral that will provide solutions. Even when the kernel function is known, experimental error in the data can make solving for even a single distribution function a difficult task. Solving for multiple distribution functions requires more data than provided by a single adsorption isotherm.

Application to Surface Energy Distribution

Under certain conditions, an energetically heterogeneous surface may be characterized by a distribution of adsorptive energies. The conditions are that the sample is not microporous, i.e., that adsorption is taking place on essentially a free surface with no pore filling processes at least to about 0.2 relative pressure. Secondly, that each energetically distinct patch contributes independently to the total adsorption isotherm in proportion to the fraction of the total surface that it represents. This condition is satisfied if the patches are relatively large compared to an adsorptive molecule, or if the energy gradient along the surface is not steep. In mathematical terms, this concept is expressed by the integral equation of adsorption in the following form.

$$Q(p) = \int d\varepsilon \; q(p, \varepsilon) \, f(\varepsilon) \qquad (2)$$

where

Q(p)	=	the experimental quantity adsorbed per gram at pressure p,
q(p,e)	=	the quantity adsorbed per unit area at the same pressure, p, on an ideal free surface of energy e, and
f(e)	=	the total area of surface of energy e in the sample.

The exact form of the energy-dependent term depends on the form of the model isotherms expressed in the kernel function and is provided in the model description (refer to **"DFT MODELS " on page E-1** for model descriptions).

Application to Pore Size Distribution

Similarly, a sample of porous material may be characterized by its distribution of pore sizes. It is assumed in this case that each pore acts independently. Each pore size present then contributes to the total adsorption isotherm in proportion to the fraction of the total area of the sample that it represents. Mathematically, this relation is expressed by

$$Q(p) = \int dH \; q(p, H) \, f(H) \qquad (3)$$

where

Q(p)	=	the experimental quantity adsorbed at pressure p,
q(p,H)	=	the quantity adsorbed per unit area at the same pressure, p, in an ideal pore of size H, and
f(H)	=	the total area of pores of size H in the sample.

Numerical values for the kernel functions in the form of model isotherms can be derived from modern statistical mechanics such as density functional theory or molecular simulations, or can be calculated from one of various classical theories based on the Kelvin equation. Several types are found in the models library.

Performing the Deconvolution

The integrations in equations (2) and (3) are carried out over all surface energies or pore sizes in the model. The functions q(p,e) and q(p,H), which we call the kernel functions, are contained in numeric form as model isotherms. Because, in general, there is no analytic solution for equation (1), the problem is best solved in a discrete form; the integral equation for any distributed property Z becomes a summation:

$$Q(p) = \sum_i q(p, Z_i)\, f(Z_i) \qquad (4)$$

Given a set of model isotherms, q(p,Z), from a model chosen from the models library and an experimental isotherm, Q(p), contained in a sample information file, the software determines the set of positive values f(Z) that most nearly, in a least squares sense, solves equation (4). The distributed property, surface energy or pore size, is then displayed on the Report Options dialog box as a selection of tables or graphs.

Regularization

DFT allows a selectable regularization (also referred to as smoothing) constraint to be applied during the deconvolution process to avoid over-fitting in the case of noisy data or ill-fitting models. The method used is based on co-minimization of the second derivative of the distribution. The relative weight given to this term is determined by the value of the regularization parameter, which is set on the DFT Pore Size or Surface Energy dialog box and also is shown in the header of reports. The value of the regularization parameter varies from zero (for no second derivative constraint) to ten (indicating a weight equal to minimizing the residuals), or even larger. When the distribution and residuals obtained change little with the value of the regularization parameter, it indicates that the chosen model provides a good representation of the data. Conversely, a large sensitivity to the regularization parameter might indicate inadequate data or a poor choice of model to represent the data.

Summary Report

The following calculations and the results of previous calculations (as noted) are used to generate the summary report:

a. BET Surface Area

   See BET Surface Area Calculations

b. Langmuir Surface Area

   See Langmuir Surface Area Calculations

c. Single Point Surface Area (m^2/g)

$$S_{1PT} = \frac{[Va \times (1 \angle Pr)] \times CSA \times (6.023 \times 10^{23})}{22414\ cm^3\ STP \times 10^{18}\ nm^2/m^2}$$

where

Pr = pressure closest to 0.3 of the relative pressure points designated for surface area calculations.

Va = volume corresponding to Pr

d. Micropore Area

See t-Plot calculations.

e. Micropore Volume

See t-Plot calculations.

f MP-Method Cumulative Surface Area of Pores

$MPS_{TOT} = S_{CUMI}$, (see MP-method Calculations) for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative surface area was computed.

g. MP-Method Cumulative Pore Volume of Pores

$MPV_{TOT} = V_{CUMI}$, (see MP-method Calculations) for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative pore volume was computed.

h. Average Pore Hydraulic Radius (A)

$$MP_{DAVER} = \frac{MPV_{TOT}}{MPS_{TOT}} \times 10^{\angle 6} m^3/cm \times 10^{10}\ Å/m$$

SPC Report Variables

Regression Chart Variables

The line of best fit for the Regression Chart is calculated by the usual Least Squares methd. (Refer to *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.) If there is only a single point or all N points have the same x-value, there can be no line of best fit in the standard form.

$$XAve = \frac{\Sigma\, x_i}{N}$$

$$XAve = \frac{\Sigma\, y_i}{N}$$

$$Slope = \frac{\Sigma\, (x_i \angle XAve)(y_i \angle XAve)}{\Sigma\, (x_i \angle XAve)^2}$$

The coefficient of Correlation for this line is also calculated in the usual way. (Refer to *Mathematical Handbook for Scientists and Engineers*, by Granino A. Korn and Theresa M. Korn, Copyright 1961, 1968 McGraw Hill, Sec. 18.4.)

$$\sigma_x = \sqrt{\frac{\Sigma\, (x_i \angle XAve)^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\Sigma\, (y_i \angle XAve)^2}{N}}$$

$$Cov(x, y) = \frac{\Sigma\, (x_i \angle XAve)(y_i \angle XAve)}{N}$$

$$Correlation\ Coefficient = \frac{Cov(x, y)}{\sigma_x \sigma_y}$$

Control Chart Variables

$$Mean = \frac{\Sigma\, y_i}{N}$$

$$StdDev = \sqrt{\frac{\Sigma\, (y \angle Mean)^2}{N \angle 1}}$$

$$CoefVar = \frac{StdDev}{Mean}$$

$$PlusNSig = Mean + n \cdot StdDev$$

$$MinusNSig = Mean \angle n \cdot StdDev$$

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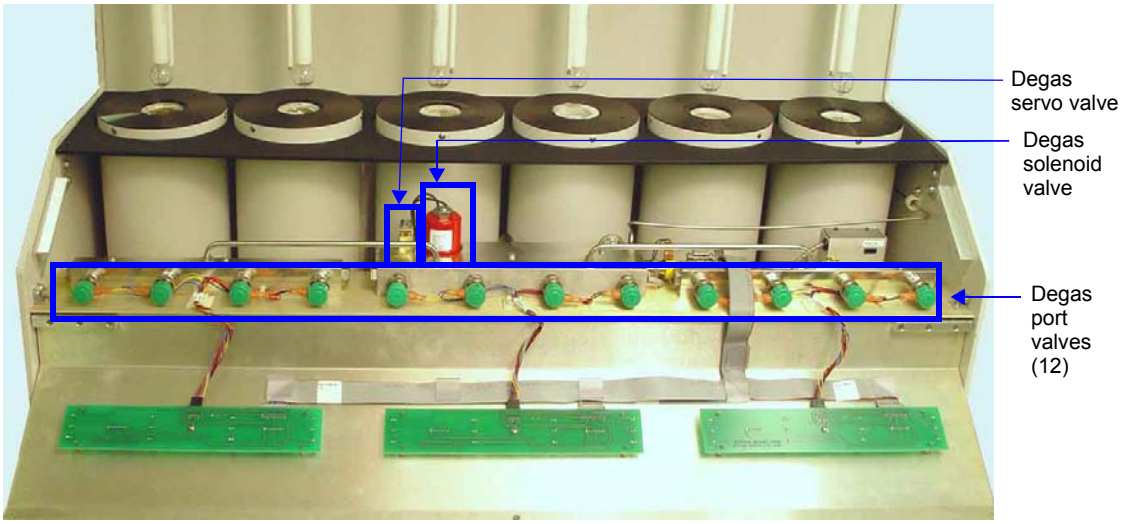
D TESTING THE ANALYZER FOR LEAKS

Valve Locations	D-3
Degas Manifold Valves	D-7
Analysis Manifold Valves	D-9
Gas Inlet Valves	D-11
Po Valves	D-13
What To Do If You Detect a Leaking Valve	D-15
Valve Test Data Sheet	D-18

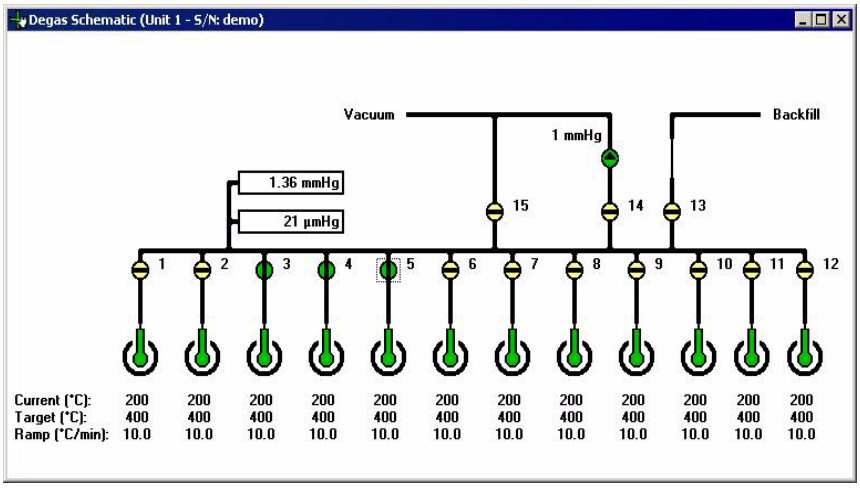
Valve Locations

Valves are located in the degas and analysis system manifolds. The valves are labeled with numbers that correspond to the numbers on the degas and analysis schematics.

Degas Manifold



Instrument Front View with Panel Lowered

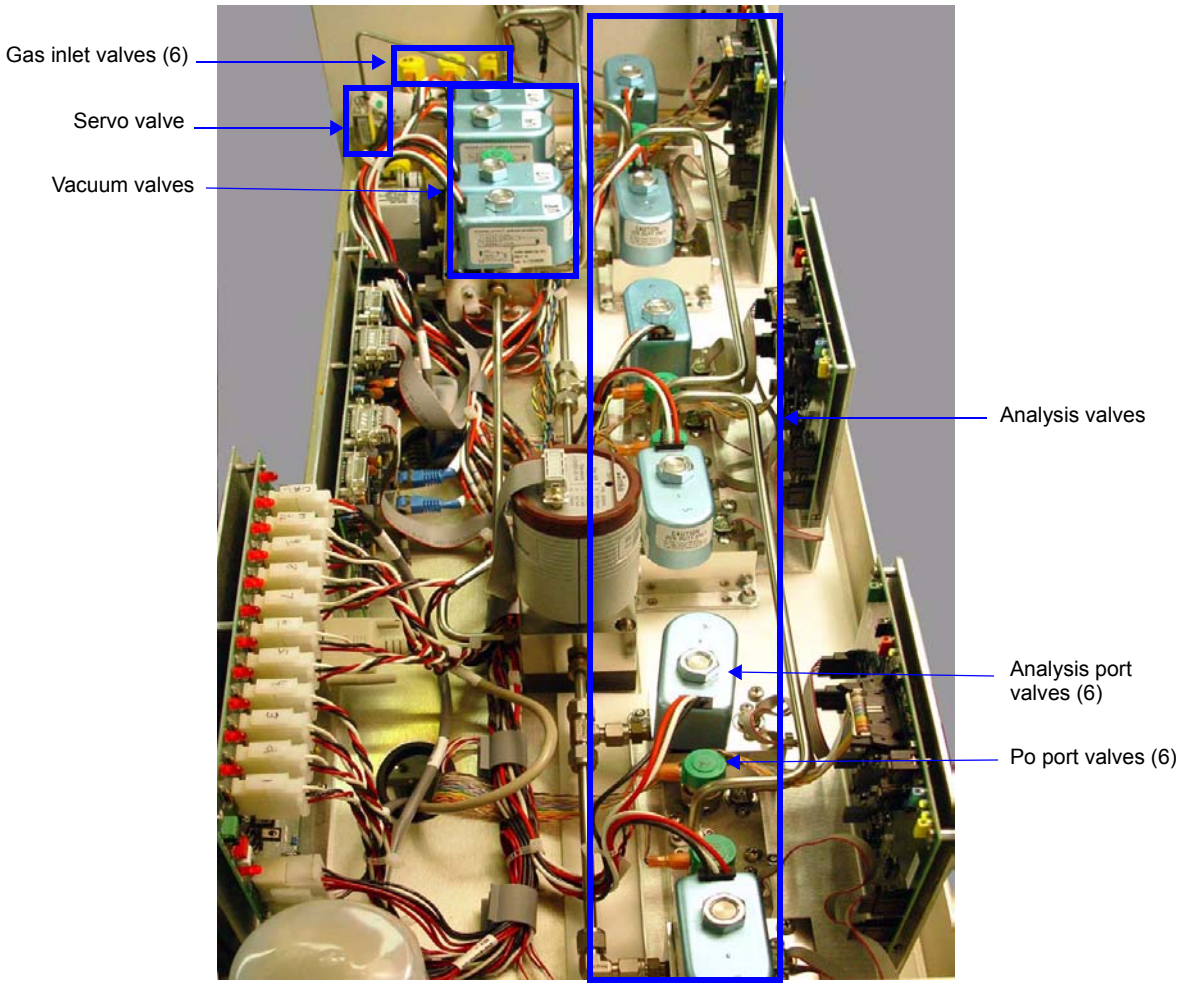


Degas Schematic

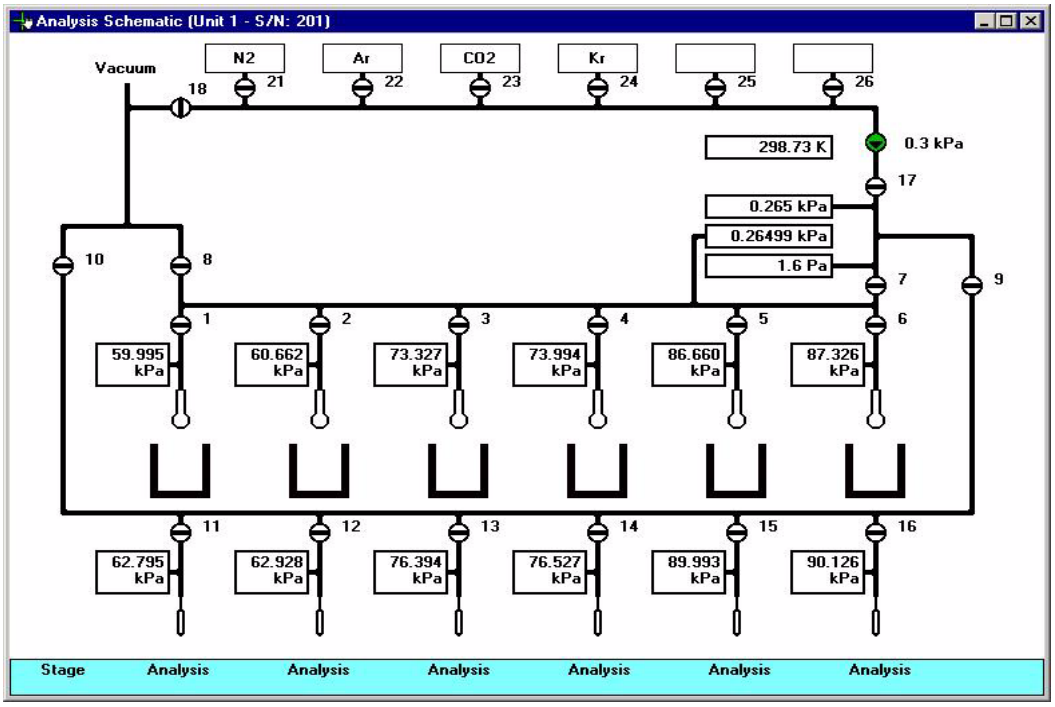
Valve	Description
1 through 12	Sample ports
13	Backfill
14	Servo vacuum
15	Unrestricted vacuum

Degas Valves

Analysis Manifold



Instrument Top View with Panel Removed



Analysis Schematic

Valve	Description
1 through 6	Sample ports
7	Lower manifold isolation
8	Sample ports unrestricted vacuum
9	Po ports access
10	Po ports unrestricted vacuum
11 through 16	Po ports
17	Upper manifold isolation
18	Gas inlets unrestricted vacuum
21 through 26	Gas inlets
Unmarked	Servo

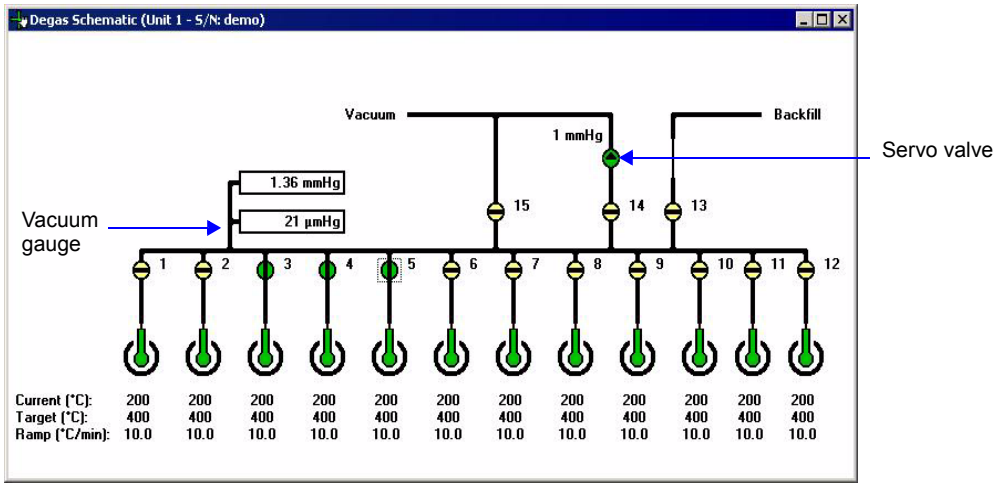
Analysis Valves

Degas Manifold Valves

The degas system must be in *idle* mode (no degas operations running) in order to perform the leak test procedures.

1. Place empty sample tubes on all 12 degas ports.
2. If the degas schematic is not displayed, select **Unit > Degas Schematic.**

   If the degas schematic is not in manual mode, select **Unit > Enable Manual Control** to display the degas schematic in manual control mode.



3. Close all open degas valves by double-clicking the open valves.

   Green = open

   Yellow = closed

4. Open valve13 and backfill the degas manifold to approximately 800 mmHg with nitrogen. Close valve 13 when the pressure is set to the desired value.
5. Open degas valves 1 through 12.
6. Open valve 15 and evacuate the degas manifold to below 20 microns.
7. Allow the system to evacuate for at least an hour.
8. Close valves 1 through 12.
9. Allow the system to evacuate for another 30 minutes.
10. Close valve 15 and record the reading on the degas vacuum gauge.

Vacuum reading _____ microns

11. Wait 5 minutes, then immediately record the degas vacuum gauge reading.

    Vacuum reading _____ microns

12. Subtract the reading in step 11 from the reading in step 12. Divide the results by 5. This gives the system outgassing rate in microns/ min.

    Record the system outgassing rate _____ microns/min. All valves should be closed at this time and should remain closed.

13. While observing the degas vacuum gauge, remove the empty sample tube from station 1. Determine the outgassing rate as in the above steps 11 through 13. If the rate exceeds 1 micron/ min., the station one plunger is leaking. Clear the leak before proceeding.

14. Record the outgassing rate for station 1 in the space provided below step 17.

15. Clear all leaks and repeat steps 4 through 14 of this procedure for each station.

16. Record the outgassing rates below. If a rate exceeds 1 micron/ min., that station plunger is leaking. Clear the leak before proceeding.

    Station 1 outgassing rate _____ microns/min.

    Station 2 outgassing rate _____ microns/min.

    Station 3 outgassing rate _____ microns/min.

    Station 4 outgassing rate _____ microns/min.

    Station 5 outgassing rate _____ microns/min.

    Station 6 outgassing rate _____ microns/min.

    Station 7 outgassing rate _____ microns/min.

    Station 8 outgassing rate _____ microns/min.

    Station 9 outgassing rate _____ microns/min.

    Station 10 outgassing rate _____ microns/min.

    Station 11outgassing rate _____ microns/min.

    Station 12 outgassing rate _____ microns/min.

17. Open valve 15 and leave the system evacuating.

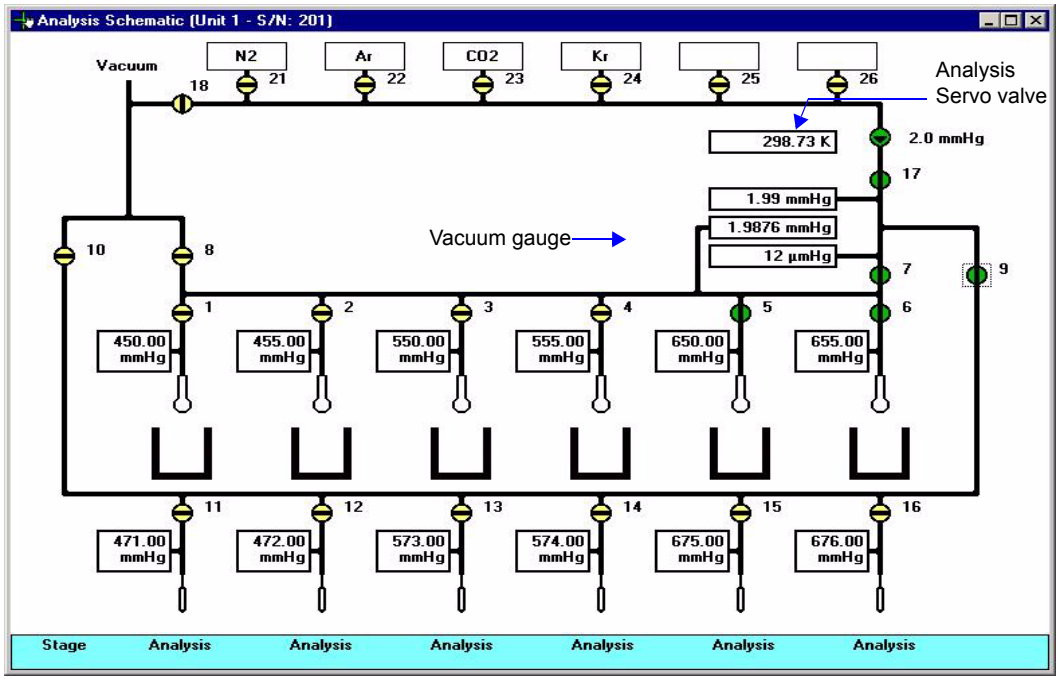
18. Remove the sample tubes and place plugs in all sample stations.

Analysis Manifold Valves

The analysis system must be in *idle* mode (no analyses running) in order to perform this procedure.

1. Place empty sample tubes on analysis stations 1 through 6.
2. Ensure the six Po tubes are mounted properly on stations 1 through 6.
3. Close all shut-off valves on the gas regulators.
4. Place plugs in any unused gas inlet ports.
5. If the analysis schematic is not displayed, select **Unit > Analysis Schematic**.

   If the schematic is not in manual mode, select **Unit > Enable Manual Control** to display the analysis schematic in manual control mode.



6. Close all valves except valves 7, 17 and 18. Leave these valves open.

   Green = open

   Yellow = closed

7. Set the analysis servo valve to evacuate to zero mmHg. This opens the servo completely.
8. Open valve 8.

9. Wait for 5 minutes.

10. Open valves 1, 2, 3, 4, 5 and 6.

11. Close valves 17 and 18 and the analysis servo valve.

12. Continue to evacuate for 2 hours or longer.

13. Close analysis valves 1, 2, 3, 4, 5 and 6.

14. Continue to evacuate for 2 hours.

15. Close valve 8 and record the analysis vacuum gauge reading.

    Vacuum reading _____ microns

16. Wait 5 minutes and record the analysis vacuum gauge reading.

    Vacuum reading _____ microns

17. Subtract the reading in step 15 from the reading in step 16. Divide the results by 5. This gives the system outgassing rate in umHg/min. The system outgassing rate should not exceed 0.7 umHg/min.

    Record the outgassing rate _____microns/min.

    All valves (except valve 7) should be closed at this time and should remain closed.

18. The sample port valves should remain closed during this procedure. While observing the analysis vacuum gauge, remove the empty sample tube from analysis station 1. Determine and record the outgassing rate as in steps 15 through 17 above.

19. If the outgassing rate exceeds 1 umHg/min., the station 1 plunger is leaking. Clear the leak before proceeding.

20. Record the outgassing rate for station 1 in the space provided in step 23.

21. Clear all leaks and repeat steps 15 through 17 of this procedure for that station before proceeding.

22. Repeat steps 15 through 20 of this procedure for each station.

23. Record the outgassing rate for stations 1 through 6 in the spaces provided below. If the outgassing rate exceeds 1 umHg/min., that station plunger is leaking. Clear the leak before proceeding.

    Station 1 outgassing rate _____ umHg/min.

    Station 2 outgassing rate _____ umHg /min.

    Station 3 outgassing rate _____ umHg /min.

    Station 4 outgassing rate _____ umHg /min.

Station 5 outgassing rate _____ umHg /min.

Station 6 outgassing rate _____ umHg /min.

24. Open valves 8 and 17, set the analysis servo valve to evacuate, and leave the system evacuating.

Gas Inlet Valves

The gas regulator shut-off valves must remain closed during this procedure.

The analysis system must be in *idle* mode (no analyses running) in order to perform this procedure.

1. Make sure the analysis schematic is displayed in manual control mode. (Refer to step 5 on page D-9.)
2. Open valves 7, 8 and 17, if closed. All other analysis valves should be closed.
3. Set the analysis servo valve to evacuate to zero mmHg.
4. Open valve 18.
5. When pressure is below 10 mmHg, open valve 21 and wait 5 minutes.
6. Open valve 22 and wait 5 minutes.
7. Open valves 23 through 26, waiting 5 minutes between each.
8. When valves 21 through 26 are open, allow the system to continue to evacuate for 2 hours.
9. All gas inlets should evacuate below 20 microns or there is a leak in one of the inlets. Find and clear the leak before proceeding.
10. Close valves 21 through 26 and allow the system to evacuate for 2 hours.
11. Close valves 8 and 18. Set the servo valve to dose to 1000 mmHg.
12. Record the analysis vacuum gauge reading.
13. Wait 5 minutes and record the analysis vacuum gauge reading.

    Vacuum reading _____ umHg

14. Subtract the reading in step 12 from the reading in step 13. Divide the results by 5. This gives the system outgassing rate in umHg/min.

Vacuum reading _____ umHg

15. Valves 21 through 26 should remain closed during this procedure. Open the gas regulator shut-off valve for the gas attached to gas inlet valve 21.

16. Determine and record the outgassing rate as in steps 11 through 14 above. If the outgassing rate exceeds 1 umHg/min., valve 21 is leaking.

17. Record the outgassing rate for valve 21 in the space provided in step 22.

18. Clear all leaks and repeat steps 11 through 15 of this procedure for valve 21 before proceeding.

19. While observing the analysis vacuum gauge, open the gas regulator shut-off valve for the gas attached to gas inlet valve 22.

20. Determine and record the outgassing rate as in steps 10 through 14. If the outgassing rate exceeds 1 umHg/min., valve 22 is leaking.

21. Record the outgassing rate for valves 22 through 26 in the spaces provided below.

22. Continue to test the remaining gas inlet valves in a like manner.

    Record outgassing rate _____ umHg/min. Valve 21

    Record outgassing rate _____ umHg/min. Valve 22

    Record outgassing rate _____ umHg/min. Valve 23

    Record outgassing rate _____ umHg/min. Valve 24

    Record outgassing rate _____ umHg/min. Valve 25

    Record outgassing rate _____ umHg/min. Valve 26

23. Open valves 8, 17 and 18, set servo valve to evacuate to zero mmHg, and leave the system evacuating.

Po Valves

The analysis system must be in *idle* mode (no analyses running) in order to perform this procedure.

1. Make sure the analysis schematic is displayed in manual control mode. (Refer to step 5 on page D-9.)
2. Close all open valves by double-clicking their icon on the analysis schematic.
3. Open valve 10.
4. Ensure all Po tubes are installed properly.
5. Open valves 11 through 16.
6. Wait 5 minutes.
7. Open valves 7, 8 and 9.
8. Allow the system to evacuate for 2 hours or longer.
9. Close valves 11 through 16.
10. Allow the system to evacuate for 2 hours or longer.
11. Close valves 8 and 10.
12. Record the reading on the analysis vacuum gauge.

    Vacuum reading _____ umHg

13. Wait 5 minutes and record the analysis vacuum gauge reading.

    Vacuum reading _____ umHg

14. Subtract the reading in step 12 from the reading in step 13. Divide the results by 5. This gives the system outgassing rate in umHg/min.

    Outgassing rate _____ umHg/min.

15. The Po valves must remain closed during this procedure. Remove the Po tube from station 1.
16. Determine and record the outgassing rate as in steps 12 through 14 above. If the outgassing rate exceeds 1 umHg, valve 11 is leaking.
17. Record the outgassing rate for valve 11 in the space provided in step 22.

18. Clear all leaks and repeat steps 10 through 14 of this procedure for valve 11 before proceeding.

19. While observing the analysis vacuum gauge, remove the Po tube from station 2.

20. Determine and record the outgassing rate as in steps 12 through 14 above. If the outgassing rate exceeds 1 umHg/min., valve 12 is leaking.

21. Record the outgassing rates for valves 12 through 16 in the spaces provided below.

22. Continue to test the remaining Po valves in a like manner.

    Outgassing rate _____ umHg/min.

    Outgassing rate _____ umHg/min.

    Outgassing rate _____ umHg/min.

    Outgassing rate _____ umHg/min.

    Outgassing rate _____ umHg/min.

    Outgassing rate _____ umHg/min.

23. Open valves 8, 10 and 17, 18, and set the servo valve to dose to 1000 mmHg. Leave system evacuating.

What To Do If You Detect a Leaking Valve

If you determine that a specific valve is leaking, remove differential pressure from the valve and continue testing the remaining valves for leaks.

As an alternative, you may repair or replace the leaking valve after removing the differential pressure on the valve. However, opening the analysis system to replace or repair a valve exposes the analyzer manifold to atmospheric gases and moisture. These gases and moisture greatly increase the time required to evacuate the analyzer and prepare it for further testing or operation.

Removing Differential Pressure from a Leaking Valve

1. Select either of the following actions:

   • Close the regulator outlet valve for the gas supply line to the valve.
   • Reinstall the plug that was removed to test the valve.

2. On the manual mode schematic screen, open the leaking valve.

3. Evacuate the analyzer until you obtain an outgas rate of less than 0.7 μmHg per minute. Refer to the previous sections for detailed instructions on determining the outgas rate for specific valves.

Repairing or Replacing a Leaking Valve

Whether you repair or replace a valve depends on the type of valve that is leaking.

- Analysis manifold valves should be repaired.
- Degas and inlet manifold valves should be replaced.
- Degas solenoid valve should be repaired.
- Servo valves should be replaced.

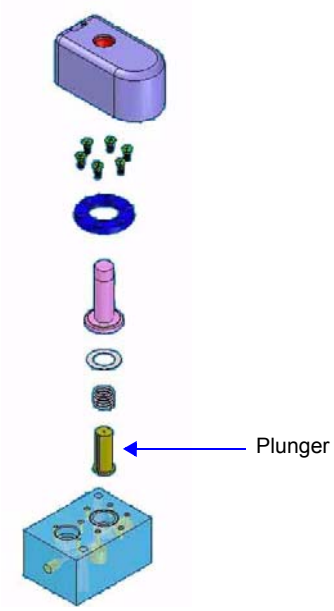
Repairing Valves on the Analysis Manifold

1. Backfill the manifold with nitrogen to approximately 800 mmHg.

   • Close off the helium gas supply if you are repairing the helium inlet valve.
   • Turn off the vacuum pump system if you are repairing a vacuum valve.

2. Remove and replace the plunger for the leaking valve.

3. Closely inspect the valve seat area for any debris which may cause another leak.



4. Reassemble the valve.

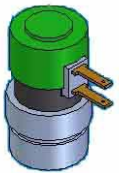
   • Reopen the helium supply if you are repairing the helium inlet valve.
   • Turn on the vacuum pump system if you are repairing a vacuum valve.

5. Using the Analysis Schematic in manual control mode, open and close the repaired valve approximately 20 to 30 times; this allows the valve to seat properly.

6. Evacuate the analyzer until the outgas rate is less than 0.7 µmHg per minute.

Replacing Valves on the Degas and Gas Inlet Manifold

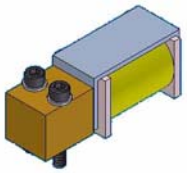
1. Backfill the manifold with nitrogen to approximately 800 mmHg.
2. Turn off the gas supply for the gas inlet valves.
3. Unscrew and remove the complete valve from the manifold.



4. Install the new valve, then reconnect the wiring.
5. Turn on the gas supply and vacuum pump system.
6. Evacuate the analyzer until the outgas rate is less than 0.7 µmHg per minute.

Replacing a Servo Valve

1. Unscrew and remove the servo valve.



2. Unplug the cable.

3. Install the new servo valve while ensuring the two sealing O-rings are in place on the manifold block.

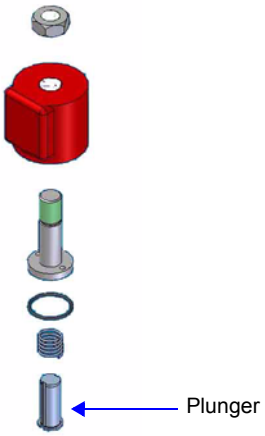
Repairing the Degas Solenoid Valve

1. Backfill the manifold with nitrogen to approximately 800 mmHg.

2. Ensure that the solenoid valve is closed (on the Degas Schematic in manual mode).

3. Unscrew and remove the valve from the manifold using a spanner wrench.

4. Remove and replace the plunger.



5. Closely inspect the valve seat area for any debris which may cause another leak.

6. Insert the new plunger and reassemble the valve.

7. Reassemble the valve.

8. Using the Degas Schematic in manual control mode, open and close the repaired valve approximately 20 to 30 times; this allows the valve to seat properly.

9. Evacuate the analyzer until the outgas rate is less than 0.7 µmHg per minute.

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			
1			
2			
3			
4			
5			
6			
7			
8			
Gas Inlet Valves			
17			
18			
21			
22			
23			
24			
25			
26			
Po Valves			
10			
11			
12			
13			
14			
15			
16			

VALVE(s)	P1 (1st pressure reading)	P2 (2nd pressure reading)	OUTGASSING RATE
Degas Valves			
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			

E DFT MODELS

Models Based on Statistical Thermodynamics	E-3
Models Based on Classical Theories	E-14
References	E-17

DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

Models Based on Statistical Thermodynamics

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

Theoretical Background

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (Vm), and the molar intensity of the gas-surface interaction, such as the Langmuir "K" constant or the BET "C" constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed.

A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.

Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal's forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.

If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:

$$U(s) = \left[\left(\frac{\sigma}{s}\right)^{12} \angle \left(\frac{\sigma}{s}\right)^{6}\right]$$

where

ε	=	a characteristic energy of the adsorptive,
σ	=	the diameter of the adsorptive molecule, and
s	=	the separation distance.

Molecular Simulation Methods

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

Molecular Dynamics Method

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

Monte Carlo Method

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is "equilibrated" through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerable less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

Density Functional Formulation

Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H. The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential* is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under **References** at the end of this appendix.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

*Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

The figure below shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.

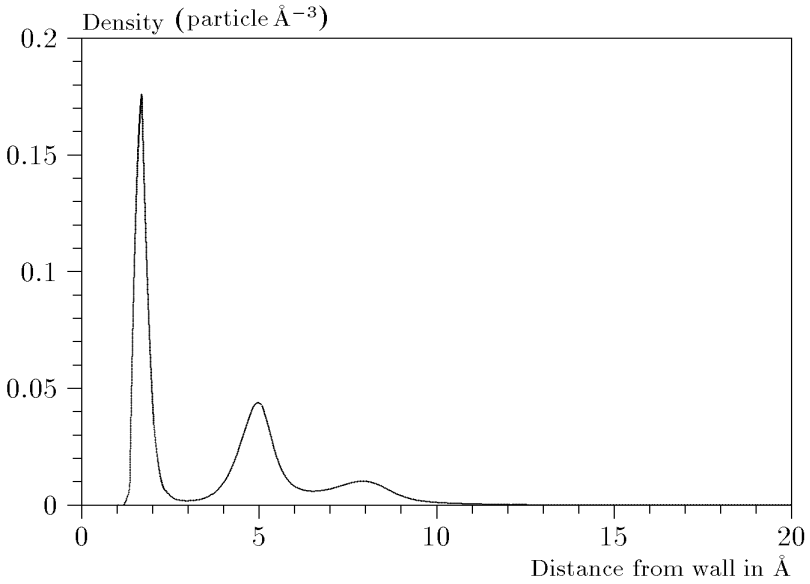


Figure E-1: Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

The figure below shows how the profiles change with pressure for a model pore with H = 40 Angstroms. The insets show the density profiles for the corresponding points of the isotherm.

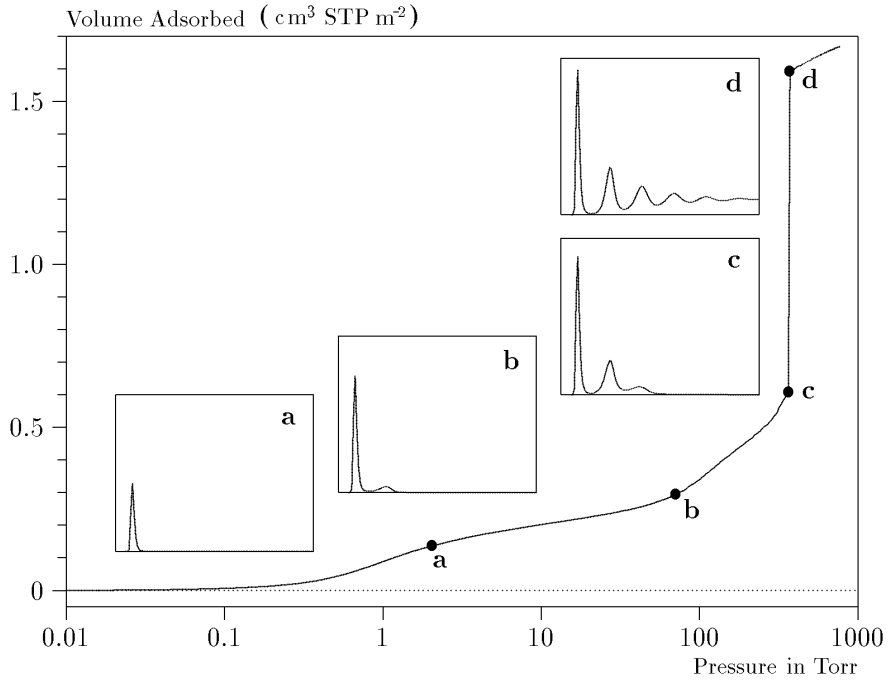


Figure E-2: Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

- Inset **a** shows the profile corresponding to point a on the isotherm where the surface is about half covered.
- At point **b**, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point **c**, a third layer is forming. Point c, for this size slit, is the critical pore-filling pressure. In inset c, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.
- Inset **d** shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

Note that the adsorption isotherms for pores larger than the one shown in Figure 4-2 is identical up to point c. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in the figure below, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.

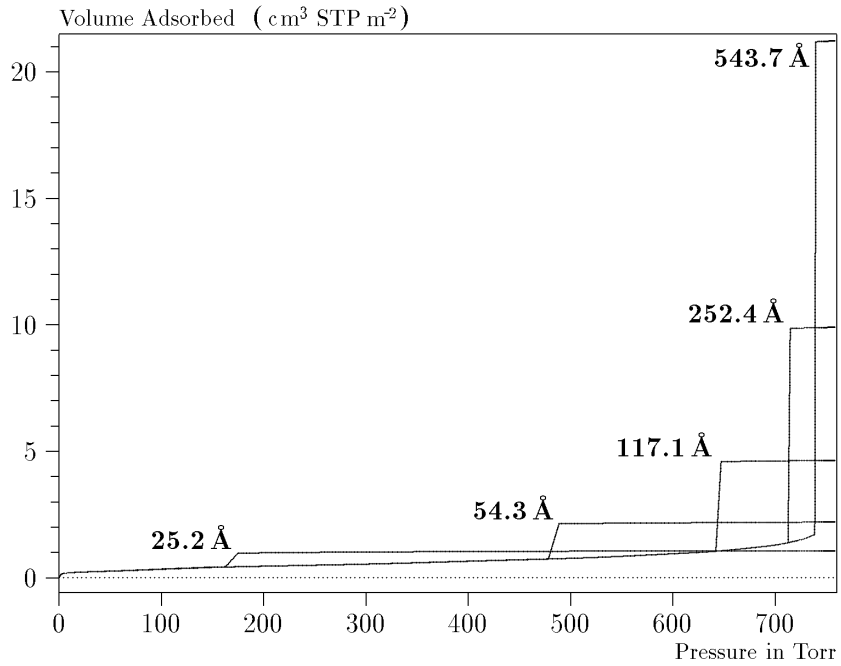


Figure E-3: Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

The figure below shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

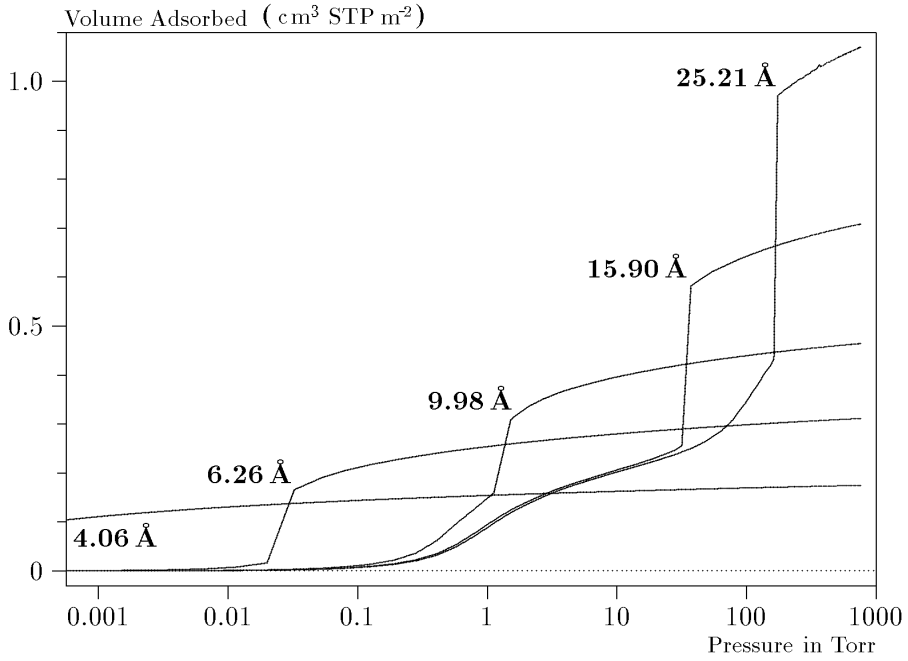


Figure E-4: Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density Independent Weights

N2 - DFT Model
AR - DFT Model

Geometry: Slit
Substrate: Carbon (graphite)
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near $1x10^{-6}$ to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Modified Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry: Free surface
Substrate: Surface energy
Method: Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (refer to references 3 and 4 on page E-17), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the Select Data Points dialog box) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, e, for the adsorptive/adsorbent pair divided by Boltzmann's constant. The units are therefore degrees Kelvin.

N2 - Cylindrical Pores - Oxide Surface

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. This model reports pore sizes ranging from 3.8 to 387 Angstroms.

Reference: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry: Cylinder
Substrate: Crystalline Silicate
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillionite.The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al,1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts.This model reports pore widths from 3.8 to 387 Angstroms.

References: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occell, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.

References: M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods", *Langmuir* 2002, 18, 9816-9823.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization Of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface area and microporosity of pillared rectorite catalysts from a hybrid density functional theory method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Carbon dioxide at 273 K

Model isotherms were calculated, using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional

Geometry: Free Surface
Substrate: Any
Category: Surface energy
Method: Argon at 87 K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is /k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 Angstroms.

Reference: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

Models Based on Classical Theories

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) on page C-36. Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

Surface Energy

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity, or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-deBoer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

Pore Size

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, it finally will predict a larger increment of adsorptive for a given pressure increment than is actually observed; since a negative pore volume is non-physical, the algorithm must stop. Conversely, if the thickness curve used underestimates film thinning, accumulated error results in the calculation of an overly large volume of (possibly nonexistent) small pores.

The use of equation (1) on page C-36 represents an improvement over the traditional algorithm. Kernel functions corresponding to various classical Kelvin-based methods have been calculated for differing geometries and included in the list of models.

Models Included

Kelvin Equation with Halsey Thickness Curve

N2 - Halsey Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{\angle 5.00}{\ln(P/\mathrm{Po})}\right)^{1/3}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Halsey Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948)

Kelvin Equation with Harkins and Jura Thickness Curve

N2 - Harkins and Jura Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left(\frac{13.99}{0.034 - \log(P/Po)}\right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Harkins and Jura Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

References: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944)
J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966)

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen at 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

Reference: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, "The Surface Area in Intermediate Pores," Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969)

References

The papers listed below provide additional information on DFT models:

1. "Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids," J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).

2. "Classification of Adsorption Behavior: Simple Fluids in Pores of Slit-shaped Geometry," Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).

3. "Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory," J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).

4. "The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory," J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

INDEX

A

Add archive location command, 7-4
Administrator, 2-3
Adsorption isotherm, E-3
Adsorptive Properties dialog, 4-34
Adsorptive properties file, 4-34
  creating, 3-14
  importing, 3-65
Alpha-S Method dialog, 4-57
Alpha-S report, 4-57
Alumina, replacing in oil vapor traps, 8-20
Analysis
  details, 5-34
  Dewar, checking, 8-8
  dialog, 5-5
  gas, 1-12, 3-51
  high throughput, 1-12, 3-51, 3-56
  Krypton, 1-12, 3-51, 3-54
  manifold, 1-15
  micropore, 1-12, 3-51, 3-58, 5-17
  performing, 3-51, 3-52
  reference, 8-36
  sample amount, 3-28
  skipping steps, 5-8, 5-11, 5-15, 5-19
  standard, 1-12, 3-51
  status, 5-33
  suspending, 5-8, 5-11, 5-15, 5-19
  viewing options, 5-6, 5-10, 5-14, 5-18
Analysis bath fluid, 3-39
  checking level, 3-40
Analysis Conditions dialog, 3-12, 4-19
Analysis conditions file, 4-19
  creating, 3-12
Analysis port, 2-4
  attaching sample tube, 3-43
  evacuating, 5-24
  replacing filter, 8-9
Analysis Preparation dialog, 4-27
Analysis program, 2-12
  commands, common, 2-16
  exiting, 2-13, 4-107
  functions, common, 2-18
  logging in, 4-106
  logging out, 4-106
Analysis system, 1-11
  manually controlling, 5-26
  schematic, 5-26
  specifications, 1-15
Analyst, 2-3
  creating a sample information file, 3-19
  opening a sample information file, 4-93
Analyzer
  cleaning, 8-26
  controls, indicators, and connectors, 2-4
  depth, 1-17
  description, 1-11
  front panel, 2-4
  height, 1-17
  leak testing, D-3
  maintaining, 8-7
  side panel, 2-8, 2-10
  specifications, 1-15
  troubleshooting, 8-3
  turning off, 2-11
  turning on, 2-11
  weight, 1-17
  width, 1-17
AR DFT model, E-10
AR Modified Density Functional, E-12
Archive
  adding location, 7-4
ASCII text, saving reports, 6-18
Automatic Degas dialog, 3-35, 3-36
Automatically collected data, 4-89
Autoscale, 6-22
  onscreen reports, 6-20
  X- and Y-axes, 6-20
Avogadro's number, C-21
Axis
  coordinates, viewing, 6-23
  properties, editing, 6-21
Axis Properties dialog, 6-21

B

Backfill
  gas, 1-17
  port, 2-8
  sample tube, 4-18
Balance
  weighing the sample, 3-28
Bath temperature, 5-7, 5-11, 5-15, 5-19
BET surface area report, 4-44
  calculations, C-8
BET Surface Area Report Options dialog, 4-44
BETsurface area report
  example, 6-28

BJH
Adsorption or Desorption plot dialog, 4-66
Adsorption/Desorption dialog, 4-61
Adsorption/Desorption plot report, 4-66
Adsorption/Desorption report, 4-61
Adsorption/Desorption tabular report, 4-64
Adsorptive Options dialog, 4-62
calculations, C-12
report example, 6-32
report examples, 6-32
Blank analysis, 4-14, 4-15
Boltzmann's constant, E-10
Bookmarks tab, 1-5
Breaker
valve circuit, 2-10
Broekhoff-de Boer equation, 4-54
Button
See Push buttons

C

Calculated free space, 4-30
Calculations, C-3
Calibrate Analysis command, 5-44
Calibrate Degas command, 5-44
Calibration Data command, 5-44
Calibration procedures, 5-44
Cancel command, 2-17
Cascade windows, 2-24
Category, 7-4
Cautions, 1-4
Check box, 2-15
Chemical potential, E-5
Cheng/Yang
calculations, C-24
Pore Geometry, C-23
Cheng/Yang correction, 4-69
Cleaning
analyzer, 8-26
Dewars, 8-8
filler rods, 3-24
sample tubes, 3-24
Close All command, 4-99
Close command, 2-17, 4-98
Close Reports command, 6-5
CO2 DFT Model, E-12
Collected Data dialog, 4-89
Columns, onscreen reports, editing, 6-19
Commands, common, 2-16
Cancel, 2-17
Close, 2-17
Replace All, 2-17
Save, 2-16
Comment dialog, 3-53
Computer, requirements, 1-17
Control Chart command, 6-11
Control Chart Graph Options dialog, 6-12
Control Chart Options dialog, 6-11
Control Chart report, 6-11
defining the Y-axis, 6-12
recalculating archived results, 6-12
Control Chart Sample Selection dialog, 6-13
Conventions, operator's manual, 1-4
Convert Sample Files dialog, 4-105
Copy as metafile command, 6-22
Copy as text command, 6-22
Create a Degas Conditions File dialog, 3-10
Create a Sample Information File dialog, 3-19, 3-22
Create a Sample Tube File dialog, 3-8
Create an Analysis Conditions File dialog, 3-12
Cross-hair, axis, 6-23
Cross-references, 1-8
Curve Properties dialog, 6-21

D

Data
automatically collected, 4-89
manually entered, 4-89
selecting display options, 7-6
Data entry field, 2-14
Dates, selecting range, 2-21
Degas
dialog, 5-21
manifold, 1-15
multiple samples, 3-36
sample, 3-33
single sample, 3-35
Degas Conditions dialog, 3-11, 4-16
Degas conditions file, 4-16
creating, 3-10
Degas port, 1-11, 2-8
attaching sample tube, 3-33, 3-34
connector, 2-6, 3-33
evacuating, 5-25
ferrule, 3-33
O-ring, 3-33
removing plug, 3-33
removing sample tube, 3-41
replacing filter, 8-11
Degas system, 1-11
accuracy, 1-17
indicator lights, 1-11, 2-5
manually controlling, 5-29
specifications, 1-17
Density conversion factor, 4-35

Density Functional Theory
See DFT
Density, sample, 4-7, 4-11
Detergent, for cleaning sample tubes, 3-25
Developer, 2-3
creating a sample information file, 3-22
opening sample information file, 4-93
Dewar, 1-12
checking, 8-8
cleaning, 8-8
filling, 3-39
insulator, 3-38
placing cover over sample tube, 3-44, 3-49
precautions, 3-38
raising and lowering, 5-27
safety shield, 1-12, 3-44, 3-50
DFT
calculations, C-36
models, E-3
pore size report, 4-75
Pore Size Report Options dialog, 4-75
surface energy report, 4-78
Surface Energy Report Options dialog, 4-78
theory, E-5
Diagnostics command, 5-40
Dialog box
closing, 2-17
description, 2-14
Dipstick, 3-40
Directories
adding, 7-4
editing labels for, 7-5
Dose Increment Edit dialog, 4-25
Dosing
absolute pressure, 4-19
amount, 4-25
in Krypton analyses, 3-54
increments, 4-25
percent of previous, 4-26
Dosing Options dialog, 4-23
Drop-down list, 2-15
Drying oven
setting temperature, 3-25
Dubinin
Adsorptive Options dialog, 4-80
calculations, C-28
dV/dw Pore Volume Options dialog, 4-82
pore volume report, 4-82
report, 4-79
Report Options dialog, 4-79
tabular report, 4-81
Tabular Report Column Options dialog, 4-81
Transformed Isotherm Plot Options dialog, 4-81
transformed isotherm report, 4-81

E

Edit curve command, 6-21
Edit Labels command, 7-5
Edit title command, 6-22
Elevator, 1-12, 5-27
lubricating screws, 8-25
raising and lowering, 5-26
Enable Manual Control command, 5-26
Entered free space, 4-29
Entered t-Curve dialog, 4-52
Equilibration
calculations, C-6
delay, 4-32
interval, 4-31
Equilibration delay, 4-25, 4-27
Equilibration dialog, 4-31
Ethernet connection, 2-9
Evacuate
analysis port, 5-24
degas port, 5-25
Evacuate analysis port dialog, 5-24
Evacuate Degas Port(s) dialog, 5-25
Evacuation, 1-17
entering conditions, 4-16
fast, 4-27, 5-24
rate, 4-17
time, 4-17
Exit command, 4-107
Exiting the analysis program, 2-13, 4-107
Expansion kit, 8-33
Export
directory, 3-66
isotherm data, 3-66
sample file, 4-104
Export command, 4-104
Export Data dialog, 3-66
Export text command, 2-22

F

Fast evacuation, 4-27, 5-24
Ferrule, 3-33
Field, data entry, 2-14
File
listing statistics, 2-13
opening, 2-13
selection, 2-19
status, 2-20, 4-92
See also Analysis conditions file
See also Degas conditions file
See also Report options file
See also Sample information file
See also Sample tube file

File menu, 2-23, 4-3
Close All command, 4-99
Close command, 4-98
Exit command, 4-107
Export command, 4-104
Import command, 4-102
Log in command, 4-106
Log out command, 4-106
Print command, 4-99
Save All command, 4-98
Save as command, 4-96
Save As Template command, 4-98
Save command, 4-95
File name
default extensions, 2-15
Files list, 2-19
Filler rod, 3-24
cleaning, 3-24
inserting into sample tube, 3-27
Filter
port, 8-9
vacuum pump exhaust, 8-31
Find command, 1-9
Folder, directory, 3-4
f-Ratio Method dialog, 4-59
f-Ratio report, 4-59
Free space, 5-7, 5-10, 5-14, 5-18
calculated, 4-7, 4-11, 4-30
calculations, C-4
entered, 4-29
measured, 4-29
Free Space dialog, 4-28
Frequency, 1-15
Freundlich report, 4-47
calculations, C-35
Freundlich Report Options dialog, 4-47
Frit, seal, for degas port, 3-33
Fume hood, 8-31
Function keys
See Shortcut keys

G

Gas
amount added for each dose cycle, 4-23
analysis, 1-12, 3-51
backfill, 1-17, 3-27
changing, 8-33
connecting a regulator, 8-33
connecting to the analyzer, 8-35
guidelines for connecting to analyzer, 8-33
hazardous, 8-35
inlet ports, 1-12, 2-8
regulator pressure, 3-51
regulators, 1-13
requirements, 1-12
saturation pressure, 1-12, 3-51
Gas Configuration dialog, 5-38, 8-36
Graph
copying as metafile, 6-22
copying report data as text, 6-22
editing onscreen, 6-20
grid lines, 7-6
legend, 6-22
onscreen report, 6-20
overlays, 3-68
Graph Grid Lines command, 7-6
Graph Grid Lines dialog, 7-6
Graph Overlay Samples dialog, 3-70, 4-38
Graphic
showing in report title, 4-38
Grid lines, 7-6

H

Halsey equation, 4-53
Harkins and Jura equation, 4-54
Harkins and Jura Thickness Equation dialog, 4-84
Heater Settings dialog, 5-30
Heating mantle
cooling, 3-41
installing, 3-34
power connection, 2-6
power plug, 3-34, 3-47
side-lacing, 3-45
thermocouple connection, 2-6
Heating phase
entering conditions, 4-17
Help menu, 2-23, 2-24
Hide curve command, 6-20
High surface area samples, 3-28
High throughput analysis, 3-56
High Throughput Analysis dialog, 3-56
High Throughput Analysis screen, 3-57
Histogram, 6-21
Hold Pressure, 4-17
Horvath-Kawazoe calculations
Cylinder pore geometry (Saito/Foley), C-22
Slit pore geometry (original HK), C-21
Sphere pore geometry (Cheng/Yang), C-23
Horvath-Kawazoe Physical Properties dialog, 4-70
Horvath-Kawazoe Plot Options dialog, 4-73
Horvath-Kawazoe report, 4-69
calculations, C-21
plot, 4-73
tabular, 4-72

Horvath-Kawazoe Report Options dialog, 4-69
Humidity, 1-15

I

Icons, arrange, 2-24
Import command, 4-102
Import Sample Information dialog, 3-65
Importing files, 3-65, 4-102
Index, online, 1-8
Indicator lights, 1-11, 2-4, 2-5
Information bar, 2-14
Insert Predefined Pressures dialog, 4-22
Insert Pressure Range dialog, 4-21
Instrument log, 5-36
  add entry, 5-37
  report, 5-37
Instrument schematic
  See Schematic
Interaction parameter, C-24
IP address of analyzer, 5-39
Isotherm, E-3
Isotherm report, 4-42
  example, 6-25
Isotherm Report Options dialog, 4-42
Isothermal jacket, 1-12, 3-45, 4-14
  placing on sample tube, 3-42

K

Kernel function, E-14, E-15, E-16
Keys
  shortcut, 2-13, 2-21
Kruk-Jaroniec-Sayari Equation, 4-53
Krypton
  analysis, 1-12, 3-51, 3-54
  dosing port, 1-12, 3-51
  option, 1-12
Krypton Analysis dialog, 3-54
Krypton Analysis screen, 3-55

L

Labels, sample file, editing, 7-5
Langmuir surface area report, 4-44
  calculations, C-9
Langmuir Surface Area Report Options dialog, 4-44
Leak test
  data sheet for recording values, D-18
  performing, D-3
  removing differential pressure, D-15
Legend Properties dialog, 6-22
Legend, report, 6-22
Lennard-Jones interaction parameter, E-10
Lennard-Jones potential, E-4
Links, 1-7
List file statistics, 3-62
List Sample Information File dialog, 3-63
List Sample Tube dialog, 3-64
Log
  See Instrument Log
Log in, 2-12
Log in command, 4-106
Log out command, 4-106
Log Report Settings dialog, 5-37
Login screen, 3-53, 3-55, 3-57
Low pressure incremental dose mode, 4-24, 4-26

M

Main Menu bar, 2-23
Maintenance, routine, 8-33
Manifold
  analysis, 1-15, 1-16
  degas, 1-15
Manual control, 5-26
  enabling for analysis system, 5-26
  enabling for degas system, 5-29
Manually entered data, 4-89
Mass
  determining, 3-28
Maximum volume increment, 4-23
MCM-41 materials, E-11
Measured free space, 4-29
Menu
  File, 4-1
  Help, 2-24
  Options, 7-1
  Reports, 6-3
  shortcut, 2-12, 2-13
  structure, 2-23
  Unit, 5-1, 5-3
  Windows, 2-24
Mesoporous materials, 4-23
Metafile, copying graph as, 6-22
Micropore
  analysis, 1-12, 3-51, 3-58, 5-17
  low pressure incremental dose mode, 4-26
Micropore Analysis dialog, 3-58, 5-17
Models, E-3
  AR DFT model, E-10
  AR Modified Density Functional, E-12
  based on classical theories, E-14
  based on statistical thermodynamics, E-3
  CO2 DFT Model, E-12

N2 Broekhoff-de Boer Model, E-16, E-17
N2 Cylindrical Pores - Oxide Surface, E-11
N2 Cylindrical Pores Pillared Clay Surface, E-11
N2 DFT model, E-10
N2 Halsey Thickness Curve, E-15
N2 Harkins and Jura Thickness Curve, E-16
N2 Modified Density Functional, E-10
N2 Tarazona NLDFT, Esf = 30.0K, E-13
Molecular dynamics, E-4
Molecular simulation methods, E-4
Monte Carlo method, E-4
Montmorillionite, E-11
MP-Method
calculations, C-33
pore volume/pore area report, 4-86
report, 4-84
Report Options dialog, 4-84
tabular report, 4-86
Tabular Report Column Options dialog, 4-86

N

N2 Broekhoff-de Boer Model model, E-16
N2 Cylindrical Pores - Oxide Surface model, E-11
N2 Cylindrical Pores Pillared Clay Surface model, E-11
N2 DFT model, E-10
N2 Halsey Thickness Curve model, E-15
N2 Harkins and Jura Thickness Curve model, E-16
N2 Modified Density Functional model, E-10
N2 Tarazona NLDFT, Esf = 30.0K model, E-13
Non-ideality factor, 4-14
Notes, 1-4

O

Oil vapor traps
replacing alumina, 8-20
On/Off switch, 2-9, 2-11
Online manual
cross references, 1-8
index, 1-8
printing options, 1-10
using, 1-5
See also Operator's manual
Onscreen reports
autoscale, 6-22
axis coordinates, 6-23
graphs, 6-20
legend, editing, 6-22
shortcut menus, 6-19
title, editing, 6-22
zooming in and out, 6-22
Open Adsorptive Properties File dialog, 3-14
Open dialog, 2-18
Open Report command, 6-5
Open Report Options File dialog, 3-16
Operator's manual
conventions, 1-4
online, 1-5
organization, 1-3
Options menu, 2-23
Add Archive Location command, 7-4
description, 7-3
Edit Labels command, 7-5
Graph Grid Lines command, 7-6
Update File List command, 7-5
Options report, 4-88
Ordering information, 9-3
Outgassing rate
checking, 8-26
Oven, drying samples, 3-25
Overlays
for multiple graphs, 3-71
for multiple samples, 3-68
generating, 3-68

P

Parameter file, 4-6, 4-94
adsorptive properties, creating, 3-14, 4-34
creating, 3-8, 4-5
degas conditions, creating, 3-10
importing, 3-65, 4-102
listing, 3-62, 3-64, 4-101
printing, 3-61
report options, creating, 3-16, 4-37
sample tube, creating, 3-8
Parameter templates, 3-7
Parts, ordering, 9-3
Password, 2-12
changing, 4-106
logging in, 4-106
Po and Temperature Options dialog, 4-30
Pore filling pressure, E-6
Pore Geometry
cylinder (Saito-Foley), C-22
slit (original Horvath-Kawazoe), C-21
sphere (Cheng/Yang), C-23
Pore size, E-14
Port filter, replacing, 8-10, 8-11
Ports
gas connection, 2-8
gas inlet, 1-12, 2-8
Po, connector, 2-6
sample, connector, 2-6
specifying gas, 8-36

See also Analysis Port
See also Degas Port
Power
connector, 2-9
heating mantle, 2-6, 3-34, 3-47
panel, 2-9
specifications, 1-15
switch, 2-9
Pressure
absolute, 4-24
absolute, dosing, 4-19
absolute, tolerance, 4-24
measurement specifications, 1-15, 1-16
relative, 4-24
relative, calculations, C-3, C-4
relative, tolerance, 4-24
Pressure table
first point, 4-23
Fixed Dose mode, 4-23
Preventive maintenance schedule, 8-7
Print command, 4-99
Printing
files, 3-60, 4-99
Prompts, sample information, 7-5
Psat tube, 1-12
displayed on schematic, 5-28
Psat vs. Temperature Table dialog, 4-35
Pump
See Vacuum pump
Push buttons, 2-15

R

Radio button, 2-14
Ramp rate, 5-30
Redraw command, 6-20, 6-23
Reducer fitting, 8-34
Reference material, 8-36
Regression report, 6-7
recalculaing SPC values, 6-8
selecting X- and Y-Axes variables, 6-8
Regression Report command, 6-7
Regression Report Options dialog, 6-7
Regression Report Sample Selection dialog, 6-8
Regulators
attaching to gas supply, 8-33
expansion kit, 8-33
gas, 1-13
purging, 8-34
shut-off/isolation valve, 8-34
Replace All command, 2-17
Report
closing, 2-13, 6-5
Control Chart, 6-11
customizing, 1-13, 3-16
displaying company logo in title, 4-38
generated automatically, 5-7, 5-11, 5-15, 5-19
generating, 6-4
graphs, editing onscreen, 6-20
header, 6-14
number of copies, 2-22
onscreen, shortcut menu, 2-13
opening, 6-5
preview, 2-22
print, 2-22
Regression, 6-7
saving in other formats, 6-18
selecting, 6-5
starting, 2-13
Statistical Process Control (SPC), 6-6
system, 1-13
title, 4-37
zoom feature, 6-22
Report button, 6-10, 6-13
Report examples, 6-23
Report Options dialog, 3-17, 3-68, 3-71, 4-37
Report options file, 4-37
creating, 3-16
importing, 3-65
Report Relative Pressure Range dialog, 4-55
Report Settings dialog, 2-22
Reports menu, 2-23, 6-3
Close Reports command, 6-5
Control Chart command, 6-11
description, 6-3
Open Report command, 6-5
Regression Report command, 6-7
SPC Report Options command, 6-6
Start Report command, 6-4
Resolution, 1-16
Revert, 4-8, 4-12

S

Saito-Foley
calculations, C-22
Pore Geometry, C-22
Sample
degassing, 3-33
degassing multiple, 3-36
degassing single, 3-35
density, 4-11
determining mass, 3-28
dosing, 4-25, 4-26
high surface area, 3-28
inserting large granules or chunks in a tube, 3-28

inserting powder in a tube, 3-28
preparing, 3-24
weighing, 3-28
weight, recording, A-3
Sample Analysis screen, 3-53
Sample Backfill Options dialog, 4-32
Sample Data Worksheet, 3-28, 3-42, A-3
Sample Information dialog, 3-5, 3-22
Sample information file
Analyst, 4-6
converting, 4-105
creating, 3-3, 4-5
creating, Analyst, 3-19
creating, Developer, 3-22
Developer, 4-10
editing labels for, 7-5
exporting, 4-104
exporting isotherm data, 3-66
importing, 3-65, 4-102
list, 3-62
opening, 4-91
printing contents, 3-60, 4-99
revert to previous version, 4-8, 4-12
versions, 4-8, 4-12
Sample Log report, 4-88
Sample template
See Template
Sample tube
assembling, 3-43
attaching to analysis port, 3-43
attaching to degas port, 3-33
choosing, 3-24
cleaning, 3-24
displayed on schematic, 5-28, 5-30
filler rod, 3-24
labeling, 3-24
outside diameter, 3-24
rack, 3-26
removing from degas port, 3-41, 3-42
replacing O-ring, 8-12
rinsing, 3-26
set, 3-24
sizes, 1-11
stopper, 3-24, 3-27
thermal profile, 1-12
weighing, 3-42
Sample Tube dialog, 3-9
Sample tube file, 4-14
creating, 3-8
Sample, density, 4-7
Saturation pressure, calculations, C-3
Save All command, 4-98
Save as Adsorptive Properties File, 4-36
Save as Adsorptive Properties File dialog, 3-15
Save As Analysis Conditions File dialog, 3-13
Save as command, 4-96
Save As Degas Conditions File dialog, 3-11
Save as Report Options File, 3-18
Save As Sample Information File dialog, 3-23, 4-8
Save as Sample Tube File dialog, 3-9, 4-15
Save as Template command, 3-7, 4-98
Save as Template dialog, 3-7
Save command, 2-16, 4-95
Schematic
analysis system, 5-31
degas system, 5-32
shortcut menu, 2-13
Seal frit, sample tube, 3-24
Select Dates group box, 2-21
Selection Criteria group box, 2-18
Sequence number, 7-4
Service Test command, 5-44
Shield, safety, 3-44, 3-50
Shortcut keys, 2-13, 2-21
Shortcut menu, 2-12, 2-13
for graphs, 6-20
for tabular reports, 6-19
Show Analysis Schematic command, 5-31
Show Analysis Status command, 5-33
Show curve command, 6-20
Show Degas Schematic command, 5-32
Show Degas Status command, 5-34
Show Instrument Log command, 5-36
Soak Temperature, 4-17
Soak Time, 4-17
SPC Report Options command, 6-6
SPC Report Options dialog, 6-6
Specifications, 1-15
Spreadsheet format
saving reports in, 6-18
Start Analysis command, 3-52, 5-5
Start Micropore Analysis dialog, 3-58
Start Multiple Degas command, 5-22
Start Report command, 6-4
Start Report dialog box, 6-4
Start Single Degas command, 3-35, 3-36, 5-21
Status
selecting, 2-19
Stopper
for sample tube, 3-24
inserting in sample tube, 3-27
STSA Equation, 4-54
Subcategory, 7-4
Summary report, 4-41
calculations, C-38
example, 6-24
Surface Energy, E-14

T

Table of Contents, online, 1-7
Tarazona prescription, E-10
Target temperature, 4-17, 5-30
Temkin report, 4-49
  calculations, C-35
Temkin Report Options dialog, 4-49
Temperature, 1-11, 1-15
  control, 1-17
  displayed on schematic, 5-28
  range, 1-17
  setting, 5-30
  target, 5-30
  transducer specifications, 1-15
Temperature ramp rate, 4-16
Template, 3-4
  listing, 4-101
  opening, 4-94
  parameter, creating, 3-7
  sample information, creating, 3-5
  save as, 4-98
Tests, diagnostic, 5-40
Text, copy graph data as, 6-22
Thermal transpiration correction
  calculations, C-7
  choosing, 4-39
Thermocouple
  connection, 2-6
  plug, 3-34, 3-47
Thermodynamic law, E-4
Thumbnails tab, 1-10
Tile windows, 2-24
Title Properties dialog, 6-22
t-Plot report, 4-51
  calculations, C-10
  example, 6-30
t-Plot Report Options dialog, 4-51
Transducer, 1-16
  displayed on schematic, 5-28
TranSeal, 3-43, 4-28
Troubleshooting, 8-3

U

Ultrasonic cleaning unit, 3-25
Unit command
  Start Multiple Degas command, 5-22
Unit Configuration command, 5-38
Unit IP Setup dialog, 5-39
Unit menu, 2-23, 5-3
  Calibrate Analysis command, 5-44
  Calibrate Degas command, 5-44
  Calibration Data command, 5-44
  description, 5-3
  Diagnostics, 5-40
  Enable Manual Control command, 5-26
  Service Test command, 5-44
  Show Analysis Schematic command, 5-31
  Show Analysis Status command, 5-33
  Show Degas Schematic command, 5-32
  Show Degas Status command, 5-34
  Show Instrument Log command, 5-36
  Start Analysis command, 3-52, 5-5
  Start Single Degas command, 5-21
  Unit Configuration command, 5-38
Units Selections dialog, 7-6
Units, selecting, 7-6
Update file list command, 7-5
User levels, 2-3

V

Vacuum
  setpoint, 5-24, 5-25
  system specifications, 1-16
Vacuum pump
  exhaust filter, replacing, 8-31
  housing, 2-10
  power connectors, 2-10
  replacing alumina in oil vapor trap, 8-20
  specifications, 1-16
Vacuum setpoint, 4-17
Valve circuit breaker, 2-10
Valves
  analysis manifold, 5-26, D-5
  degas manifold, 5-29, D-3
  gas inlet, D-11
  location, D-3
  opening and closing, 5-26
  Po, D-13
  regulator, 8-34
  repairing or replacing, D-15
Van der Waal force, E-3
Versions of sample information file, 4-8, 4-12
Viewing options, 5-6, 5-10, 5-14, 5-18
Voltage
  selecting, 2-10
Volume
  calibration details, 5-39
Volume adsorbed
  calculations, C-5

W

Wall potential, E-13
Warnings, 1-4

Weighing sample, 3-28, 3-42
Windows
  arrange icons, 2-24
  cascade, 2-24
  tile, 2-24
Windows menu, 2-23, 2-24

Z

Zoom feature, 6-22