TriStar 3000

Operator's Manual

V6.08

(For serial number 1001 and higher)

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TABLE OF CONTENTS

1. GENERAL DESCRIPTION

ganization of the Manual
Conventions
uipment Description
SmartPrep Degasser
Vacuum Pump
Analysis Program
Report System
Online Help
ecifications

2. INSTALLATION

Unpacking and Inspecting the Equipment	. 2-1
Equipment Damage or Loss During Shipment	
Equipment Return	
Installing the Analyzer	
Connecting the Computer	
Connecting the Vacuum Pump	
Connecting Peripheral Equipment to the Computer	
Connecting the Gas Supply	
Installing the SmartPrep Degasser	
Installing the Software	
Initial Installation	
Using the Setup Program for Other Functions	
Installing Subsequent Software Versions	
Adding an Analyzer	
Moving an Analyzer from one PC to another PC	
Removing an Analyzer	
Changing an Analyzer Setup	
Reinstalling the Calibration Files	
Uninstalling the Analysis Program	
Specifying Gas Ports	
	. 2-23

3. USER INTERFACE

Turning the Analyzer On and Off	3-1
Turning the Analyzer On	3-1
Turning the Analyzer Off	3-1
Using the Software	3-2
Shortcut Menus	3-2
Shortcut Keys	3-2

Dialog Boxes and Subdialog Bo	oxes	 	. 3-4
Selecting Files		 	3-6
File Name Conventions		 	.3-8
Menu Structure		 	.3-9
Main Menu Bar		 	.3-9
Windows Menu		 	.3-10
Help Menu		 	.3-11

4. OPERATIONAL PROCEDURES

Specifying Sample Defaults
Basic Format
Advanced Format
Defining Parameter Files
Sample Tube
Degas Conditions
Analysis Conditions
Adsorptive Properties
Report Options
Creating a Sample File
Basic and Restricted Formats
Advanced Format
Cleaning and Labeling Sample Tubes
Calibrating Sample Tubes
Preparing the Sample for Analysis
Weighing the Sample
Degassing the Sample
Installing the Sample Tube
Installing the Analysis Dewar
Performing an Analysis
Standard Analysis
QuickStart Analysis
Printing File Contents
Generating a List
Exporting Isotherm Data
Generating Graph Overlays
Multiple Graph Overlays
Multiple Sample Overlays

5. FILE MENU

Description		 																		.5	-1
Open	•	 																 		5	-3
Sample Information																				.5	-4
Basic		 •																		5	-5
Advanced	•		•	•		•		•	•	•	•	•		•		•	•		•	.5	-8

6. UNIT MENU

Description
Sample Analysis
QuickStart
Enable Manual Control
Show Instrument Schematic
Show Status
Show Instrument Log
Unit Configuration
Calibration
Temperature
Pressure Scale
Zero Pressure

Reset Pressure Calibration
Servo Valve
Volume
Save to File
Load from File

7. REPORTS MENU

Description
Start Report
Close Reports
DFT Plus
Printed Reports
Header
Onscreen Reports
Tool Bar
Shortcut Menus
Tabular Reports
Graphs
Zoom Feature
Axis Cross Hair
Report Examples
Analysis Log
Isotherm Plot
BET Surface Area Report
BET Surface Area Plot
Langmuir Surface Area Plot
t-Plot Report
t-Plot Graph
Alpha-S Method
f-Ratio Method
BJH Adsorption: Pore Distribution Report
BJH Adsorption: Cumulative Pore Volume Plot
BJH Adsorption: dV/dD Pore Volume
BJH Adsorption: dV/dlog(D) Pore Volume
BJH Adsorption: Cumulative Pore Area Plot
BJH Adsorption: dA/dD Pore Area Plot
Summary Report
Sample Log Report

8. OPTIONS MENU

Description																						.8-1
Option Presentation																						.8-3
Advanced																						.8-4
Basic																						8-5
Restricted	•		•		•		•				•	•	•		•		•	•	•	•	•	.8-6

Data Presentaton
Sample Defaults
Basic
Advanced
System Configuration
Parameter Files Directory
Service Test Mode

9. TROUBLESHOOTING AND MAINTENANCE

Cleaning the Analyzer
Replacing Port Frit and O-ring
Calibrating the System
Zero pressure
Pressure scale
Servo valve
Temperature
Volume

10. ORDERING INFORMATION

APPENDICES

Appendix A: Forms
Appendix B: Error Messages
Appendix C: Calculations
Saturation Pressure (Po)
Relative Pressure Calculations
Volume Adsorbed Calculations
Free Space
Measured, Calculated, and Entered
Quasi Gemini
Equilibration
BET Surface Area Calculations2
Langmuir Surface Area Calculations
t-Plot Calculations4
BJH Pore Volume and Area Distribution Calculations
Explanation of Terms
Calculations
Compendium of Variables
Dubinin-Radushkevich Calculations
Dubinin-Astakhov Calculations
MP-Method Calculations
Summary Page
References

Appendix D: Free-space Correction	D-1
Measure	D-2
Quasi-Gemini	D-2
Calculate	D-3
Enter	D-3
Appendix E: Maintaining High Purity Gases	E-1
Using Metal Gas Lines	E-1
Removing Trapped Air	E-1
Purge Method	E-2
Evacuation Method	E-4
Purging the System After Non-use	E-6

INDEX

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CHAPTER 1

GENERAL DESCRIPTION

- Organization of the Manual
- Equipment Description
- Specifications

GENERAL DESCRIPTION

This manual describes how to install and operate the TriStar 3000.

Organization of the Manual

This operator's manual is organized so that all operating procedures are located in Chapter 4. Chapters 5, 6, 7, and 8 are reference chapters should you need additional information while performing these procedures.

This manual is divided as follows:

Chapter 1	General Description. Provides a general description of the TriStar 3000 and its features and specifications.
Chapter 2	Installation. Describes how to unpack, inspect, install, and verify operation of the TriStar 3000.
Chapter 3	User Interface. Provides instrument and software interface.
Chapter 4	Operating Procedures . Provides step-by-step procedures for the operations performed with the TriStar 3000.
Chapter 5	File Menu. Provides a description of the choices available on the File menu.
Chapter 6	Unit [n] Menu . Provides a description of the choices available on the Unit menu.
Chapter 7	Reports . Provides a description of the choices available on the Reports menu.
Chapter 8	Options Menu . Provides a description of the choices available on the Options menu.
Chapter 9	Troubleshooting and Maintenance . Provides user maintenance and service information.
Chapter 10	Ordering Information . Provides ordering information for TriStar system components.
Appendix A	Forms . Contains forms you may use to record information prior to entering it into the system.

Appendix B	Error Messages. Lists the error messages that may be displayed by the software and includes a cause and action for each.
Appendix C	Calculations . Provides the calculations used by the TriStar pro- gram.
Appendix D	Free-space Correction . Provides a discussion of the free-space measurements available with TriStar.

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.

Equipment Description

The TriStar 3000 is an automated gas adsorption analyzer which contains three ports, allowing you to analyze up to three samples simultaneously. Or you can use port 2 for measuring the free space using the Quasi-Gemini method and/or port 3 to measure the saturation pressure (Po) on a continuous basis. You can operate up to four TriStar analyzers with one control module (computer). The TriStar 3000 system consists of the TriStar analyzer, a SmartPrep degasser (optional) for preparing samples, a vacuum pump, and a control module for entering analysis and report options.

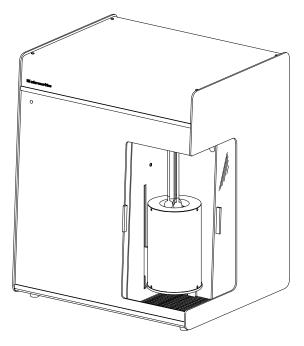


Figure 1-1. TriStar Analyzer

SmartPrep Degasser

The SmartPrep is a flowing-gas, degassing unit which removes adsorbed contaminants from the surface and pores of your sample in preparation for analysis. It contains six sample ports, each one independently controlled for greater flexibility. It contains two serial ports, one for connecting to the control module and the other available for connecting an additional SmartPrep. In fact, you can connect up to 4 SmartPreps, one to the other, allowing the capability of up to 24 preparation ports at one time! The SmartPrep is the recommended degassing unit for the TriStar 3000; however, you may furnish your own degassing method if desired. Chapter 10 provides ordering information for the SmartPrep.

Vacuum Pump

A vacuum pump is required for sample analysis with the TriStar analyzer. A recessed cavity is provided at the rear of the instrument for its placement.

Vacuum pumps used with the TriStar must meet the following criteria. They must:

- be able to achieve vacuums better than 20 μ mHg at the instrument inlet
- contain an anti-suckback valve to prevent vacuum pump oil from backing up into the analyzer in the event of a vacuum pump failure
- contain an NW16 inlet port for connection to the TriStar

An appropriate vacuum pump is available from Micromeritics. (Refer to Chapter 10 for ordering information.)

Analysis Program

The TriStar 3000 analysis program is designed to operate in the Windows[®] environment. This makes operation of the TriStar 3000 easier and allows you to run other applications while an automatic operation is in progress. If you are new to Windows, you should learn some basic Windows skills, such as using the mouse and choosing commands. Brief operating instructions are provided in **Using the Software** in Chapter 3. For more specific instructions, refer to the Windows operating manual or to the online tutorial.

Report System

The TriStar software 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. Refer to Chapter 7 for the options available for reports.

Online Help

An extensive online help system is provided for quick-and-easy access to a subject matter. The TriStar help system functions in the same manner as most other help systems. You can choose a subject from the **Contents** window where topics are organized in "books," locate your topic using the **Index** window where topics are listed alphabetically by key words, or search for information on a specific topic using the **Find** window.

Specifications

The TriStar 3000 has	s been designe	ed and tested to	o meet the	specifications provided
below.				

Characteristic	characteristic Specification				
	—— PRESSURE MEASUREMENT ———				
Range:	0 to 999 mmHg				
Resolution:	Within 0.05 mmHg				
Accuracy:	Within 0.5% of full scale				
Linearity:	Within 0.25% of full scale (transducer manufacturer's specification)				
	—— MANIFOLD TEMPERATURE ———				
Accuracy:	$\pm 0.25^{\circ}C$				
Resolution:	Within 0.1°C				
	VACUUM SYSTEM				
Vacuum Pump:	One required; 20 µmHg (maximum) with anti- suckback valve (May be purchased from Micromeritics or supplied by user.)				
	ENVIRONMENT				
Temperature:	10 to 35°C, operating; 0 to 50°C, nonoperating				
Humidity:	20 to 80% relative, noncondensing				
_	———— ELECTRICAL ————				
Voltage:	85 to 265 VAC				
Frequency:	50/60 Hz				
Power:	300 VA, maximum (110 VA for TriStar plus 190 VA for vacuum pump)				
———— PHYSICAL ————					
Height:	76 cm (30 in.)				
Width:	64 cm (25 in.)				
Depth:	53 cm (21 in.)				
Weight:	45 kg (100 lbs)				

CHAPTER 2

INSTALLATION

- Unpacking and Inspecting the Equipment
- Installing the Analyzer
- Installing the Software
- Specifying Gas Ports

INSTALLATION

This chapter describes how to:

- Unpack and inspect the equipment
- Install the TriStar system
- Verify operation of the system

Initially, your TriStar 3000 will be installed and verified for operation by an authorized Micromeritics service representative or a representative of a Micromeritics distributor. Installation instructions are provided in this manual in case the TriStar has to be relocated or reinstalled after a factory repair.

Unpacking and Inspecting the Equipment

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



If you need to declare equipment as damaged or lost, save the shipping cartons. The claims investigator must examine the cartons in order to complete the inspection report.

Equipment Damage or Loss During Shipment

If equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The freight carrier, not Micromeritics, is responsible for all damage or loss occurring during shipment. If you discover damage or loss of equipment during shipment, report the condition to the carrier immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, a customer may receive equipment which is not in working order. When equipment has been damaged (either during shipment or in use) and you wish to return the equipment to Micromeritics for repair or replacement, follow the steps below:

1. Pack the instrument in its original shipping carton if possible. If the original carton is unavailable, for a nominal fee, Micromeritics can provide another carton for your use.



Failure to package your instrument properly may result in shipping damage.

- 2. Tag or otherwise identify the defective equipment, noting the defect and circumstances, if any, the circumstances under which the defect occurred.
- 3. Reference the sales order or purchase order for the equipment, and provide the date the equipment was received.
- 4. Notify the Micromeritics Service Department of the defect and request shipping instructions. The Service Department will assign a Returned Materials Authorization (RMA) number. Write the RMA number on the outside of the carton.

Installing the Analyzer

Place the TriStar on an appropriately sized work table. Be sure to place the TriStar close enough to the control module so that cables may be connected adequately.

Connecting the Computer

Perform the following steps to connect the computer to the analyzer:

- 1. Plug the female end of the RS-232 cable into the computer.
- 2. Plug the other end into the connector labeled **RS232** on the back panel of the analyzer.

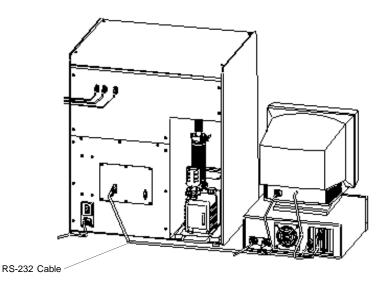


Figure 2-1. RS232 Connections

3. Tighten the retaining screws.



If you plan to install more than two instruments per computer, you must have additional serial I/O hardware. This hardware is available from Micromeritics; refer to Chapter 10 for ordering information. Instructions for installing the hardware are given in Appendix E; they also are included with the hardware.

4. Connect the female end of the power cord into the power connector on the back panel of the analyzer. Insert the other end into an appropriate power source.

Connecting the Vacuum Pump

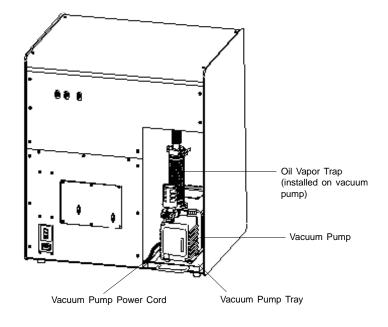
The TriStar requires a vacuum pump for operation. The instructions provided here are for installation of the vacuum pump available from Micromeritics (refer to Chapter 10 for ordering information). These instructions may vary slightly if you are providing your own vacuum pump. Perform the following steps to install the vacuum pump:

- 1. Prepare the vacuum pump (check oil, install exhaust filter, etc.) following the instructions provided in the vacuum pump manual. DO NOT connect the vacuum pump hose to the analyzer at this time.
- 2. Prepare the oil vapor trap included in the TriStar accessories kit following the instructions provided in its manual.
- 3. Install the oil vapor trap onto the vacuum pump following the instructions provided in the oil vapor trap manual.
- 4. Verify that the pump voltage (located on the pump motor) matches the voltage of the power outlet to which the pump will be connected.



Do not connect the pump power cord to a power source until the proper voltage has been verified. Doing so could result in electrical shock and/or damage to the pump.

- 5. Slide the vacuum drip tray provided in the accessories kit partially into the cavity at the rear of the analyzer.
- 6. Place the vacuum pump (with installed oil vapor trap) onto the tray.
- 7. Plug the power cord provided for the vacuum pump (to the left just inside the cavity) into the power outlet on the rear of the vacuum pump.
- 8. Place the vacuum pump ON/OFF switch in the ON position.



9. Slide the tray completely into the analyzer.

Figure 2-2. Vacuum Pump Connection

10. Connect the vacuum hose from the analyzer to the vapor trap.

Connecting Peripheral Equipment to the Computer

Follow the instructions provided below to connect the monitor, keyboard, printer, and mouse. The following illustration shows typical connections.



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

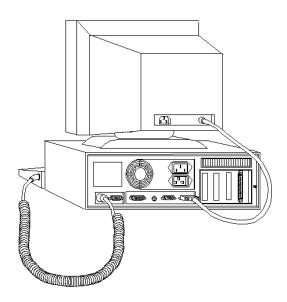


Figure 2-3. Typical Computer Connections

- 1. Plug the keyboard cable into the keyboard connector on the rear panel of the computer.
- 2. Plug the monitor cable into the monitor connector on the rear panel of the computer.
- 3. Plug the mouse cable into the mouse connector on the rear panel of the computer.
- 4. Plug one end of the printer cable into the connector on the rear panel of the printer. Plug the other end of the printer cable into the connector labeled **LPT1** (may be labeled **PARALLEL**) on the rear panel of the computer.
- 5. Plug all power cords into the appropriate power source.

Connecting the Gas Supply

Compressed gases with a purity of 99.99% are required for analyses performed using the TriStar 3000. Gas bottles or an outlet from a central source should be located near the analyzer. Refer to Appendix F for tips on maintaining gas purity.

Appropriate regulators which have been leak-checked and specially cleaned are required. Gas regulators are available from Micromeritics (refer to Chapter 10 for ordering information).

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

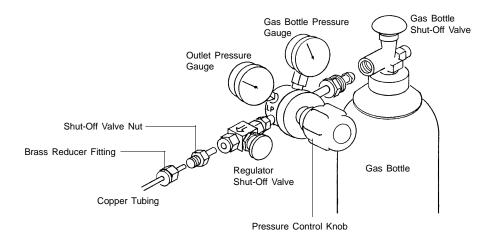


Figure 2-4. Connecting Two-Stage Regulator to Gas Supply

- 2. If the regulator has a 1/8-in. outlet, proceed to the next step. If the regulator has a 1/4-in. outlet, attach the reducer fitting to the outlet of the regulator shut-off valve.
- 3. Tighten the regulator shut-off valve nut.
- 4. Attach the copper tubing to the regulator or reducer fitting.



Do not over-tighten the fittings. Doing so could collapse the copper tubing and cause a leak.

- 5. Purge the regulator as follows:
 - a. Close the regulator shut-off valve by turning it fully clockwise.
 - b. Turn the pressure regulator control knob fully counterclockwise.

- c. Open the gas bottle valve by turning it counterclockwise, then close the gas bottle valve.
- d. Observe the outlet 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.
- e. Turn each pressure regulator control knob clockwise until the outlet pressure gauge indicates 15 psig (1.0 bar). Open each regulator shut-off valve by turning it counterclockwise briefly. Then close each valve.
- f. Make sure the gas bottle valve is completely closed.
- 6. Attach the other end of the copper tubing to the appropriate fitting on the back of the analyzer.

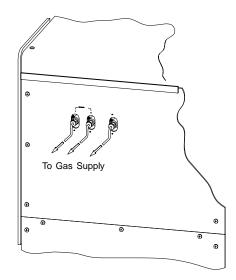


Figure 2-5. Gas Connections

- 7. Repeat steps 1 through 6 for an additional analysis gas (if desired) and for helium.
- 8. After installing the software, access the Unit Configuration dialog from the Unit menu to specify ports for gas attachment(s). (Refer to **Specifying Gas Ports** later in this chapter.)

Installing the SmartPrep Degasser

Refer to the SmartPrep operator's manual for installation instructions. When you install the SmartPrep, a menu item entitled "SmartPrep" is added to the main menu bar of the TriStar, allowing you complete control of the SmartPrep through the TriStar program.

Installing the Software

Your system must meet or exceed the following requirements before you can install the TriStar 3000 analysis program:

- Pentium 333MHz computer (or equivalent)
- CD-ROM drive
- 128 megabytes of main memory
- 1-gigabyte hard drive
- SVGA monitor (800 x 600 minimum resolution)
- Windows 2000 or Windows XP Professional

The TriStar analysis program is also available as a standalone option so that you can install it on a computer other than the one controlling the analyzer. This allows you to:

- create or edit sample and parameter files
- generate reports on completed sample files

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

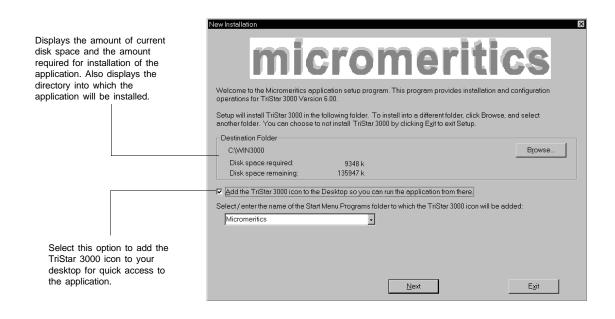
Initial Installation

The TriStar analysis program is supplied on a CD. Perform the following steps to install the program:

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

e:setup

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





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

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

- 6. If you want to run the application from the desktop, select the checkbox just below the Destination Folder group box to add an icon to the desktop.
- 7. The TriStar 3000 icon is added to the Micromeritics folder by default. If you prefer a different folder, enter or select one from the drop-down list.

mi	cromeritics
Step 1	Step 2
Select the number of analyzers that will be attached to this PC.	For each unit, enter the necessary information below. The analyzer serial number is on the analyzer identification label, typically located near the power switch.
Select 0 in the case that you are moving an analyzer from another PC to this PC or are doing off-line data reduction on this PC.	Unit Analyzer # Serial# 1 [102]
Analyzers: C 0	
C <u>4</u>	

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

9. In the Step 1 group box, click the radio button for the number of analyzers to be attached to this computer. If you are attaching multiple analyzers, make sure your computer has sufficient serial ports. Additional hardware may be required (refer to Chapter 10 for ordering information).



Choose 0 (zero) if you are installing this program for data reduction on a computer other than the one controlling the analyzer.

- 10. In the Step 2 group box, enter the serial number(s) for the analyzer(s) you are attaching to this computer.
- 11. Click Next ; the Calibration File Installation dialog is displayed. Read the information in the dialog(s) and proceed accordingly.



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

- 12. After the calibration files are installed, the Installation Complete dialog box containing the Readme file is displayed.
- 13. Use the scroll bar if you wish to read the contents of the file, then clickFinish to close the dialog.
- 14. Remove the Setup CD and store in a safe place. The original Setup CD contains the calibration files specific to your instrument. Upgrade CDs do not contain calibration files. Therefore, it is important that you maintain your original Setup CD in a secure location in the event calibration files need to be reinstalled.

Using the Setup Program for Other Functions

After initial installation of the TriStar analysis program, the application setup program is used to:

- Upgrade software
- Add an analyzer
- Move an analyzer from one computer to another computer
- Remove an analyzer from the computer
- Change the analyzer setup
- Reinstall calibration files
- Uninstall the analysis program

To start the application setup program:

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

Alternatively, you can click **Browse**, navigate to your CD-ROM drive, and select **setup.exe**.

6. Click oκ ; the setup Welcome screen showing the options available is displayed.

Welcome	<u>m</u> i micromeriti	× ics®
	licromeritics application setup program. This program provides installation Star 3000 Version 6.00.	and configuration
	Select which operation you wish to do	
	C <u>A</u> dd an analyzer	
	© Move an analyzer from one PC to another PC	
	C Rem <u>o</u> ve an analyzer	
	C Change analyzer setup	
	C Be-install calibration tiles for an analyzer	
	⊂ Uninstall	
[Start file installation	Exit

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

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

Ī	Welcome 🛛
	mi micromeritics ®
	Welcome to the Micromeritics application setup program. This program provides installation and configuration operations for DEMO TriStar 3000 Version 6.00. Select which operation you wish to do Re-install software version 6.00 Add an analyzer Move an analyzer from one PC to another PC Remgve an analyzer Change analyzer setup Re-install colibration files for an analyzer
Indicates status of ——— last operation.	C Uninstall Last operation status Re-install software was successful.
	Start file installation Exit

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

Installing Subsequent Software Versions

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

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

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

- 1. Start the Setup program. Choose the software option; remember, only the applicable option will display; it will be one of the following:
 - Upgrade software to version (number) from version (number)
 - Reinstall software version (number)
 - Downgrade software to version (number) from version (number)
- 2. Click **Start file installation**; the application installs the software and redisplays the setup Welcome dialog.
- 3. If no other operations are desired using this dialog, click **Exit** to close the dialog.

Adding an Analyzer

Add an analyzer to the existing application as follows:

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

Set up analyzer		mi	cro	omeri	tics®	×
				sary information below. Th located near the power swi		
Ent	er the communc	ations port r	iumber only, for	example, 1 for COM1.		
		Unit #	Analyzer Serial #	Communications Port#		
		2	112	1		
	< <u>B</u> ack]		Next >	<u>C</u> ancel]

- 2. Enter the serial number of the analyzer being added, then the communications port to which it is to be connected.
- 3. Click Next ; the Calibration Installation dialog is displayed. Read the information in the dialog and proceed accordingly.
- 4. After the calibration files are installed, the setup Welcome dialog is redisplayed.
- 5. If no other operations are desired using this dialog, click **Exit** to close the dialog.

Moving an Analyzer from one PC to another PC

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



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

1. Install the analysis program on the destination computer. Refer to **Initial In-stallation** earlier in this chapter. Be sure to select 0 as the number of instruments; all related instrument information will be transferred in the Move operation.

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

- 2. Start the application setup program on the source computer. Refer to Using the Setup Program for Other Functions earlier in this chapter.
- 3. In the Setup dialog, select **Move an analyzer from one PC to another PC**, then click Next ; the Move analyzer operation dialog is displayed.

Move analyzer operation
mi micromeritics ®
The Move analyzer operation is done following these steps.
 Install the analyzer software on the Destination PC if it is not already installed there. If the Destination PC already has the maximum number of analyzers a move cannot be done.
Proceed with the Move operation on the Source PC - this will gather the necessary information and files to be moved to the Destination PC.
3. Run the setup program on the Destination PC and select the Move operation.
 If you want to copy or move sample or parameter files you will have to do that using a file management program like Explorer or a backup / restore utility.
Is this the Source PC or the Destination PC?
© Source PC © Destination PC
<back <u="">Next> <u>C</u>ancel</back>

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

			eriti	CS [®]
Step 1 Select which analyzer	s to be moved from this	PC: Unit 1: S	5/N 201	
To select a different dr	yzer-being-moved files ve / folder, click Browse		ection .	
a:\ If possible, select a floppy	drive or a network drive	accessible by both t	he Source and Dest	Browse
If the files to-be-moved car and Destination PCs, you r there you must use a folde restore program can do thi Move operation.	nust choose a local folde transfer utility to copy th	er (which should have is folder from the Sou	e nothing in it). After urce PC to the Destir	the files are placed nation PC (a backup /
< <u>B</u> ack		<u>F</u> inish		Cancel

- 5. In the Step 1 group box, select the analyzer that is to be moved.
- 6. In the Step 2 group box, choose a location in which the moved files will be stored. If possible, choose a floppy drive or a shared network drive. If this is not possible, select a local folder. After the files are placed there, use a folder transfer utility to copy this folder from the Source PC to the Destination PC.

Calibration files are not included in the moved files. These files must be copied and moved separately to the destination computer. The calibration files are located in a subdirectory (folder) of the Hardware directory. The subdirectory is named 300-(serial number).

- 7. Click **Finish**; the files are moved and the setup Welcome screen is displayed.
- 8. Start the application setup program on the destination computer.
- 9. In the Setup dialog, select **Move an analyzer from one PC to another PC**; the Move analyzer operation dialog is displayed (shown on previous page).
- 10. Select **Destination PC**, then click **Next**; the following dialog is displayed.

Image: Second system Communications below. Step 1 Analyzer serial number is on the analyzer identification label. typically near the power switch. Enter the communications port number only. for example, 1 for COM1. Unit Analyzer 2 201 2 201 Secief Secief where the analyzer-being-moved files are located. 1 Browse	ove analyzer information to	this PC			
For the analyzer being moved to this PC, enter the necessary information below. Step 1 The analyzer serial number is on the analyzer identification label, typically near the power switch. Enter the communcations port number only, for example, 1 for COM1. Unit Analyzer Step 2 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .		: .			•
Step 1 The analyzer serial number is on the analyzer identification label, typically near the power switch. Enter the communications port number only, for example, 1 for COM1. Unit Analyzer 2 201 2 201 2 201 2 201 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .	I	l mi	icra	merit	JICS ®
Step 1 The analyzer serial number is on the analyzer identification label, typically near the power switch. Enter the communications port number only, for example, 1 for COM1. Unit Analyzer 2 201 2 201 2 201 2 201 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .			.		_
Enter the communications port number only, for example, 1 for COM1. Unit Analyzer Communications # Seriel # Port # 2 201 2 Step 2 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .		noved to this PU), enter the necess	ary information below.	
Unit Analyzer Communications # Serial # Port # 2 201 2 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .	The analyzer serial n				power switch.
# Serial # Port # 2 201 2 Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection.		ions portnumbe	a oniy, ior exampi		
Step 2 Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .					
Specify where the analyzer-being-moved files are located. To select a different drive / folder, click Browse, and make your selection .		2	201	2	
To select a different drive / folder, click Browse, and make your selection .					
a\Browse					
	a:\				B <u>r</u> owse
< Back Einish Cancel	< <u>B</u> ao	sk		Einish	Cancel

- 11. In the Step 1 group box, enter the serial number of the analyzer being moved and the communications port to which it will be attached.
- 12. In the Step 2 group box, click **Browse** and choose the location of the moved files.
- 13. Click Finish; the files are moved and the setup Welcome dialog is displayed.

Removing an Analyzer

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

- 1. Start the Setup program. Refer to Using the Setup Program for Other Functions earlier in this chapter.
- 2. From the Setup dialog, select **Remove an analyzer**, then click **Next**; the Remove an analyzer dialog is displayed.

Remove an analyzer	cs®
If your objective is to move an analyzer from one PC to another you should use the Move of	
Otherwise, to remove an analyzer from this PC select which analyzer to remove. Notes: The calibration and status files associated with this analyzer will be removed. The Unit number assignments will be re-arrranged if the analyzer being removed c not have the highest Unit number.	<u>M</u> ove
For example, if Unit #2 is removed: Before After Unit 1 = Serial # 155 Unit 1 = Serial # 155 Unit 2 = Serial # 210 Unit 2 = Serial # 341 Unit 3 = Serial # 341	
< Back Bernove	Cancel

- 3. From the drop-down list, choose the serial number of the analyzer you want to remove.
- 4. Click **Remove**; the analyzer is removed and the Welcome screen is again displayed.
- 5. Click **Exit** to close the dialog.

Changing an Analyzer Setup

Change the analyzer setup as follows:

- 1. Start the Setup program. Refer to Using the Setup Program for Other Functions earlier in this chapter.
- 2. From the Setup dialog, select **Change analyzer setup**, then click **Next**; the Change analyzer setup dialog is displayed.

Change analy	Zer setup Ter setup
	Change any of these item(s) for: Unit 1: S/N 201 Enter the communcations port number only, for example, 1 for COM1. Communications Fort #
	Current 1 New: 2
	< Back Einish Cancel

- 3. From the drop-down list, choose the analyzer you want to change.
- 4. Enter the new port number in the space provided.
- 5. Click **Finish**; the change is completed and the Welcome dialog is again displayed.
- 6. Click **Exit** to close the dialog.

Reinstalling the Calibration Files

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

- 1. Start the Setup program. Refer to Using the Setup Program for Other Functions earlier in this chapter.
- 2. From the Setup dialog, select **Re-install calibration files for an analyzer**, then click Next; the Re-install calibration files dialog is displayed.

Recinstal calibration files
Step 1 To re-install calibration files for an analyzer select the analyzer: Unit 1: S/N 201
Step 2 Please insert the calibration data medium (or specify the location) for this analyzer.
Existing calibration files will be backed up. Calibration file source location EAcalib Browse
<back einish="" qancel<="" td=""></back>

- 3. Select the analyzer whose calibration files you wish to reinstall from the dropdown list. If you have only one analyzer installed, a drop-down list is not included.
- 4. Insert the CD containing the calibration files; ensure that the CD drive is displayed as the calibration file source location. If not, click **Browse** and choose the CD drive.
- 5. Click **Finish**; the calibration files are reinstalled and the Welcome dialog is again displayed.
- 6. Click **Exit** to close the dialog.

Uninstalling the Analysis Program

When you uninstall the ASAP 2020 analysis program, the application removes the analysis program, status files, analyzer setup files, and resulting empty directories. It does not remove data files. Perform the following steps to uninstall the program:

- 1. Start the Setup program. Refer to Using the Setup Program for Other Functions earlier in this chapter.
- 2. From the Setup dialog, select **Uninstall**, then click **Next**; the Uninstall dialog is displayed.

	cs®
You have chosen the uninstall operation. This operation will remove the TriStar 3000 files as well as the analyzer status and calibration files. It will not remove any sample files that you have created. If any folders become empty as a result of the file remove be removed. If your intent is to move an analyzer from this PC to another PC you should perform the (press the Move button in this case).	data or parameter al the folder will also
	Move
< Back	Cancel

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



- 4. Choose one of the following:
 - Automatic: click Next ; the system uninstalls the analysis program automatically and the setup Welcome dialog redisplays.
 - **Custom**: click Next ; a series of dialogs is displayed, allowing you to choose the files you wish to uninstall. After all files are selected and uninstalled, the setup Welcome dialog redisplays.
- 5. Click **Exit** to close the Welcome dialog.

Specifying Gas Ports

After you have connected your analysis gas(es) to the analyzer, you must specify which gas is connected to each port.

- Valve 7 carries the free-space gas (He) and cannot be changed
- Valves 8 and 9 are designated for the analysis gas(es)

Specify gas connections as follows:

1. From the **Unit** menu, select **Unit configuration**; the Unit Configuration dialog box is displayed.

🔚 Unit configuratio	on (Unit 1 - S/N: 1083)		x		
Configuration		Volume Calibrati	on		
Comm Port:	COM4	Manifold Volu	me 18.2674 cm ³		
Serial #:	1083	Date	12/18/2002		
	Board ID				
Software Versions		Pressure Slope	Pressure Slope Calibration		
MIC BIOS:	Boot Block V1.04 May 15	Manifold	1/17/2003 97% of nominal		
Controller:	3000 MASS V5.00 Jan 9	Port 1	1/17/2003 98% of nominal		
Application:	TriStar 3000 V6.00	Port 2	1/17/2003 97% of nominal		
rippiloution.	110101 0000 10.00	Port 3	1/17/2003 98% of nominal		
Gas Selections		Pressure Offset	Pressure Offset Calibration		
Valve 9 gas:	N2	Manifold	1/17/2003		
Valve 8 gas:		Port 1	1/17/2003		
vaive o gas.		Port 2	1/17/2003		
Valve 7 gas:	Не	Port 3	1/17/2003		
Servo Valve		Temperature Calibration			
Calibrated	12/18/2002	Temperature	12/18/2002		
	0 <u>K</u>	<u>C</u> ancel			

2. In the Gas Selections group box, enter the mnemonic name for the gas installed for valve(s) 8 and 9; then click OK to close the dialog box.

CHAPTER 3

USER INTERFACE

- Turning the Analyzer On and Off
- · Using the Software
- Menu Structure
- Windows Menu
- Help Menu

USER INTERFACE

Turning the Analyzer On and Off

Turning the Analyzer On

- 1. Place the computer, monitor, and printer ON/OFF switches in the ON position.
- 2. Place the TriStar analyzer ON/OFF switch in the ON (|) position.
- 3. Place the vacuum pump ON/OFF switch in the ON position. This step is not necessary if your vacuum pump is connected to the TriStar's internal vacuum pump cable.

Turning the Analyzer Off



Always exit the analysis program and 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.

If an analysis is in progress, the following message is displayed:

Instrument is busy. Continue program exit?

Yes

No

Even if you select <u>Yes</u> and the analysis program closes, the analysis continues and data continue to be collected. 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 analyzer, the data collected *after exiting* the analysis program are lost.

- 2. Place the computer, monitor, and printer ON/OFF switches in the OFF position.
- 3. Place the TriStar analyzer ON/OFF switch in the OFF position.
- 4. Place the vacuum pump ON/OFF switch in the OFF position. This step is not necessary if your vacuum pump is connected to the TriStar's internal vacuum pump cable.

Using the Software

The TriStar 3000 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.

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 want 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.

<u>R</u> esize column R <u>e</u> name column
Move column → Align column →
Hide column
Column <u>f</u> ont Header f <u>o</u> nt
Edit <u>t</u> itle
<u>C</u> opy table as text

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 3-1 provides a list of the keys available in the TriStar 3000 analysis program.

Key(s)	Function
<u>F1</u>	Access online help
F2	Open a sample information file
F3	Open an analysis conditions file
F4	Open an adsorptive properties file
F5	Open a report options file
F6	Tile windows
F 7	Cascade windows
F8	Start report
F9	Close all open reports
F11	Start the DFT Plus program (if installed)
Alt F4	Exit the analysis 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

Table 3-1. Shortcut Keys

Dialog Boxes and Subdialog Boxes

Dialog boxes are displayed when an item followed by an ellipsis (...) is selected. Subdialog boxes are displayed when a push button on a dialog box is selected. Both types of boxes may contain one or more of the following:

- **Data entry field** These fields may be either numeric (numbers only) or alphanumeric (numbers, letters, or printable characters). If you make an invalid entry in either of these fields, an error message occurs.
- **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.

	Analysis Preparation
	<pre>Fast evacuation Evacuation rate: 5.0 mmHg/s Unrestricted evac. from: 5.0 mmHg Yacuum setpoint: 10 µmHg Evacuation time: 0.10 hours Leak test Leak test duration: 120 s</pre>
Displays range for - selected field.	Enter a value between 1.0 and 50.0.

A list contains selections from which you may choose one or more items. To select an item on the list, position the mouse pointer on the desired selection, then doubleclick. An item is selected if it is preceded with a check mark.

	Select Reports 🛛 🗵
Indicates item is selected.	 ✓ Isotherm ✓ Isotherm ✓ Langmuir Surface Area t-Plot Alpha-S Method I-Ratio Method BJH Adsorption BJH Desorption Dubinin MP-Method Options ✓ Summary Sample Log
	<u>OK</u> <u>C</u> ancel

List

Push ButtonA push button is used to display subdialog boxes in
which to enter additional information about the subject
matter. For example, this push button displays a dialog
for specifying a range of dates (explained later in this
chapter).

Push buttons also may be used to invoke an action. For example, **Cancel** closes the open dialog box, discarding any information you may have entered.

Radio ButtonRadio buttons are contained within a group box. These
buttons are used to select options; you may choose only
one item.

Thickness Curve		
C H <u>a</u> lsey		
Harkins-Jura		
C Carbon Black STSA		
C Broekhoff-de Boer		
C User-Defined		
<u>E</u> dit		

Check Box Check boxes also are used to select options. You may make as many selections as you wish.

<u>P</u> hysical volume below mark:	1.0000	cm³		
<u>□ U</u> se isothermal jacket				
🗌 Use filler r <u>o</u> d				

Drop-down List A drop-down list is indicated by a down arrow to the right of the field. Drop-down lists contain a list of options from which you may choose one.

All 🔹	- Down Arrow
All	
Analyzing	
Complete	
Entered	
No Analysis	
Prepared	
Preparing	

Some dialog boxes contain one or both of the following push buttons:

Browse... Displays a dialog box which allows you to select a file for the subject matter.

Replace...Allows you to copy file values from an existing file into
the one you are creating. You then can edit the values in
the new file without changing the ones in the original
file.

Selecting Files

Sample information is stored in files and saved under file names. Certain dialog boxes contain a **Files** list box, which displays a list of files available for that particular operation. For example, the Open Sample Information dialog box:

Specify a new Status category to limit the files displayed in	Open Sample Information File	
the Files: list box.	File name: 0176.SMP Complete	
	Selection Criteria	
	Status: All	
Click to specify a	- Dates	
range of dates.		Directories:
	Files:	g:\\tristar\data
	0121.smp ALUMINA REFERENCE 004/16816/00 C1	F []
	0136.smp 13 X REF 004-16826-00 MAL 2818 12M 0140.smp 13 X REF 004-16826-00 MAL 2818 13L	[-a-] Choose the
	0141.smp 13 X REF 004-16826-00 MAL 2818 14H	drive/directory.
Choose the file	0176.smp 13X REFERENCE 004/16826/00 12L	[[-d-]
you wish to open.	0177.smp 13X REFERENCE 004/16826/00 13L	[-e-]
,,	0178.smp 13X REFERENCE 004/16826/00 14H	
	0196.smp 13X REFERENCE 004/16826/00 12M 0200.smp 13X REFERENCE 004/16826/00 13L	[[-g-]
	0200.smp 13X REFERENCE 004/16026/00 13E	[-h-] [-q-]
	0309.smp 13 X Molecular Sieve Powder	- [-w-]
	0310 smn 13 X Molecular Sieve Powder	[-z-]
	* *	*
	OK	

From the list of files in the **Files** list box, move the mouse pointer to the file you wish to open and double-click. If you do not see the desired file, it may be in another directory. Go to the **Directories** list box and choose the correct drive and/or directory.

You may limit the list of files displayed in the **Files:** list box by choosing one of the following:

• Use wildcard characters in the path name you enter in the File name field.

Wildcard characters such as * and ? can be used to filter file names. For example, you can limit the list of files displayed to those beginning with **01** as by entering 01*.smp.

• Enter a range of dates. Select Dates...; the Select Dates dialog is displayed.

-	Select Dates
O Show	All Dates
Show	Date Range:
From	: 01/01/70 🛔
To:	01/01/70
0 <u>K</u>	Cancel

Select the **Show Date Range** radio button. This enables the **From** and **To** fields allowing you to enter a beginning and ending date. Alternatively, you may double-click in each field to display a calendar to set a date. The range of dates remains the default until you change the dates or select *Show All Dates*.

For convenience, the following function keys are available when the Select Dates dialog box is displayed:

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



You may change the date format by using the International Date Format function on the Windows Control Panel.

• Select a file status from the Status drop-down list. Table 3-2 describes each file status.

Status	Description
All	All sample information files in the specified directory and within the specified range of dates.
Analyzing	Sample information files that are currently being used for analysis or are in the degassing process.
Complete	Sample information files that were used in an analysis that has been completed.
Entered	Sample information files that contain manually entered data.
No analysis	Sample information files that have not been used to perform an analysis.
Prepared	Sample information files that have been used in an automatic degas operation, but not for an analysis.
Preparing	Sample information files currently being used in an automatic degas operation.

Table 3-2. File Status and Description

File Name Conventions

For sample information files, a default file name (the next available sequence number) and a default extension display. For Sample tube, Degas conditions, Analysis conditions, Adsorptive properties, and Report options, only a default extension displays.

The following table shows the file name extensions for the TriStar 3000 analysis program.

File Type	Extension	
Sample information	SMP	
Sample tube	STP	
Degas conditions	DEG	
Analysis conditions	ANC	
Adsorptive properties	ADP	
Report options	RPO	
Export to disk (ASCII)	EXP	
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	

Table 3-3. Default File Name Extensions

Menu Structure

SmartPrep drop-down	🛄 TriS	Star 3000					_ 🗆 ×
menu displays here if a	<u>F</u> ile	Unit <u>1</u>	<u>R</u> eports	Opt <u>i</u> ons	<u>W</u> indows	<u>H</u> elp	
SmartPrep unit is being used with the TriStar.							-

Main Menu Bar

All functions for TriStar 3000 are accessed from the main menu bar. Brief descriptions are provided below; refer to the chapter given in parentheses for a detailed description of the options contained on that menu.

File	Allows you to manage sample and parameter files. (Chapter 5)
Unit [n]	Enables you to perform analyses, calibrations, and other instrument operations. (Chapter 6)
SmartPrep	Displays only if you have a SmartPrep degassing unit in- stalled on the TriStar. The options contained on this menu are explained in the SmartPrep operator's manual.
Reports	Enables you to generate, close, and customize reports. (Chapter 7)
Options	Allows you to edit sample defaults, specify your system configuration, and select data presentation formats. (Chapter 8)
Windows	Enables you to arrange the windows and icons on your screen. It also displays the names of all open windows. (this chapter)
Help	Displays Help information. (this chapter)

Windows Menu

<u>W</u> indows	
<u>T</u> ile	F6
<u>C</u> ascade	F7
<u>A</u> rrange Icons	
<u>1</u> Unit1-Status	
✓ <u>2</u> C:\3000\DATA\SI0001.SMP	

The choices on this menu allow you to arrange your open windows and icons.

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.

The Windows menu also displays all open files; the active window is preceded with a check mark.

Help Menu

<u>H</u> elp	
<u>H</u> elp	F1
About TriStar 3000	

Help	Lists help topics for the TriStar 3000 analysis program.
About TriStar 3000	Displays information about the TriStar 3000 analysis pro- gram.

CHAPTER 4

OPERATIONAL PROCEDURES

- Specifying Sample Defaults
- Defining Parameter Files
- · Creating a Sample File
- Cleaning and Labeling Sample Tubes
- Calibrating Sample Tubes
- Preparing the Sample for Analysis
- Performing an Analysis
- Printing File Contents
- · Generating a List of File Statistics
- Exporting Isotherm Data
- Generating Graph Overlays

OPERATIONAL PROCEDURES

This chapter contains step-by-step procedures for operating the TriStar 3000. It does not provide detailed descriptions of the fields in the dialogs used to perform these procedures. Refer to Chapters 5 through 8 for field descriptions.

Specifying Sample Defaults

Sample information files include the information required by the TriStar to perform an analysis and collect data. The TriStar automatically generates sample information file names and assigns the default values which you specify.

The sample information presentation can be presented in a basic or advanced format. Accordingly, sample defaults also are specified in the basic and advanced formats. If desired, you may define parameter files before entering sample defaults (refer to **Defining Parameter Files** in this chapter).

Basic Format

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



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

1. From the **Options** menu, select **Sample Defaults**; the Sample DeFaults dialog is displayed.

Sample Information					
Se <u>q</u> uence: 000	Seguence: 000-000				
Sample:					
<u>M</u> ass:	0.00 g <u>D</u> ensity:	0.00 g/cm³			
Sample <u>t</u> ube:	Sample Tube	_			
Degas conditions:	Degas Conditions	-			
Analysis conditions:	Run Conditions	•			
Adsorptive properties:	Nitrogen	•			
Report options:	Report Options	•			
	Rep <u>l</u> ace All	Add Log E <u>n</u> try			
Save	Close	Advanced			

- 2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File**, **Open**, **Sample information**.
- 3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the **\$** symbol if you wish to have the sample file number included as part of the identification.



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

- 4. Enter a default value in the **Mass** field. You can edit this value at the time of analysis.
- 5. In the **Density** field, enter a value for the material to be analyzed. This information is required only for the Quasi-Gemini and computed free-space methods.
- 6. Select the down arrow to the right of the following fields to specify default parameter files:
 - Sample Tube
 - Degas Conditions
 - Analysis Conditions
 - Adsorptive Properties
 - Report Options
- 7. Click Save , then Close .

Advanced Format

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



Select *Option Presentation* on the *Options* menu and ensure that *Advanced* is selected as the format.

1. From the **Options** menu, select **Sample Defaults**; the Default Advanced Sample Information dialog is displayed.

🖱 Sample	Defaults	5				_ 🗆 ×
<u>≺</u> < Prev	Sample Information	Sample	Degas Ana Conditions Con	lysis Adsorpt ditions Properti		Next ≥>
s	eq <u>u</u> ence	000-002				
Sample:		\$				
Operator:						<u>□ 0</u> mit
Submitter	:					🗖 Omi <u>t</u>
	<u>M</u> ass:	1.0000			cally collected	
	<u>D</u> ensity:	1.000	g/cm³	C Manually	entered	
Comments	e					
					Add Log Er	ntry
					Rep <u>l</u> ace Al	I
Enter a valu	ie betwee	n 0.0010 and	500.0000.			
	<u>S</u> ave		<u>C</u> lose		B <u>a</u> sic	

- 2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File**, **Open**, **Sample information**.
- 3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the \$ symbol if you wish to have the sample file number included as part of the identification.



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

- 4. Edit the **Operator** and **Submitter** lines as desired. Or you may have either (or both) of these lines omitted entirely by selecting **Omit**.
- 5. Enter default values for the sample's **mass** and **density**. Both of these values may be edited at the time of analysis.

- 6. In the **Type of Data** group box, choose whether you wish to collect data automatically or enter it manually.
- 7. After you complete the Sample Information dialog, click on the parameter tabs to open associated dialogs; specify defaults as desired.

The defaults you specify for parameters serve as defaults for new parameter files. For example, the values you specify in the Analysis conditions portion of the sample file display as the default values for newly created stand-alone Analysis conditions files.

8. Click Save , then Close .

Defining Parameter Files

The following file types 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

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

Several predefined parameter files (located in the PARAMS directory) are included with the TriStar 3000 analysis program. Although these files may come close to the needs of your laboratory, you may wish to define additional ones. Or you can use a predefined file as a starting point. You can do this by creating a new file and then selecting **Replace**. A dialog is displayed so that you can select the existing parameter file. Then you can make any changes you need to make and the original file remains unchanged.

Make sure you save these files to the directory specified as the Parameter File directory (refer to Chapter 8 for additional information). If you do not save these files to the specified directory, they will not display in the drop-down lists on the Basic sample file editor.

Parameter files may be defined using either the Basic or Advanced format. The following sections provide instructions for using the Basic format; the dialogs for the Advanced format are the same as the ones on the Basic format.

Sample Tube

Sample tube files are used only for the *Quasi-Gemini* or *Calculated* free-space methods.

Perform the following steps to define a sample tube file:

- 1. From the **File** menu, select **Open**, **Sample tube**; the Open Sample Tube dialog is displayed.
- 2. Enter a name in the **File name** field, then click **OK**.

3. Click Yes to create the file; the Sample Tube dialog is displayed:

Sample Tube Sample Tube	Physical volume 1.0000 cm [*]
● <u>1/4 inch</u> ○ 3/ <u>8</u> inch ○ 1/ <u>2</u> inch	☐ <u>U</u> se isothermal jacket ☐ Use filler r <u>o</u> d
Save	

- 4. Enter a description in the **Sample Tube** field. Be sure to use an intuitive description so that you can recognize it easily.
- 5. Select the stem diameter of the sample tube you plan to use.
- 6. Enter the volume of the sample tube below the red line on the sample tube stem. This value is obtained by calibrating the sample tube. (Refer to **Calibrating Sample Tubes** in this chapter.)



This value is required only if you plan to use the quasi-Gemini or calculated method for free-space measurement.

- 7. Select whether you plan to use a filler rod and/or an isothermal jacket.
- 8. Click Save , then Close .

Degas Conditions

Degas Conditions files contain degassing information for sample preparation. These files are used only if you are using the SmartPrep Degasser.

Perform the following steps to define a degas conditions file:

- 1. From the **File** menu, select **Open**, **Degas conditions**; the Degas Conditions dialog is displayed.
- 2. Enter a name in the **File name** field, then click **OK**.
- 3. Click Yes to create the file; the Degas Conditions dialog is displayed:

Degas Conditions				
Description Degas Conditions Replace				
Soak Temperature Ramp Rate (°C) (°C/min) (°C/mi	Insert Delete Clear Ctrl/down-arrow to append			
	ose			

- 4. Enter a description in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily.
- 5. Enter up to five stages of degassing (soak temperature, ramp rate, and soak time).
- 6. Click Save , then Close .

Analysis Conditions

Analysis conditions specify the data used to guide an analysis.

Perform the following steps to define an analysis conditions file:

- 1. From the **File** menu, select **Open**, **Analysis conditions**; the Analysis Conditions dialog is displayed.
- 2. Enter a name in the **File name** field, then click <u>οκ</u>.
- 3. Click Yes to create the file; the Analysis Conditions dialog is displayed:

Analysis Conditions			
Description Run Conditions Replace			
Isotherm Collection © Target Pressures Pressures Options O Dose Increments Options			
Prep <u>a</u> ration <u>Free Space</u> <u>Po and T</u>	Equilibration		
SaveClose]		

- 4. Enter a description in the **Description** field. Be sure to use an intuitive description so that you can recognize it easily.
- 5. Choose whether you wish to have the isotherm collected by target pressures or dose increments. Then specify the options required for your choice.
- 6. Click Preparation... to specify evacuation and leak test values.
- 7. Click **Free space...** to specify how the free space is to be measured.
- 8. Click **Po and T**... to specify how the saturation pressure (Po) is to be measured and the analysis bath temperature.
- 9. Click Equilibration... to specify the equilibration interval and its delay time.
- 10. Click Save , then Close .

Adsorptive Properties

Adsorptive properties provide the adsorptive characteristics for the analysis.

Perform the following steps to create an adsorptive properties file:

- 1. From the **File** menu, select **Open**, **Adsorptive properties**; the Open Adsorptive Properties dialog is displayed.
- 2. Enter a name in the **File name** field, then click **OK**.
- 3. Click Yes to create the file; the Adsorptive Properties dialog is displayed:

Adsor Adsorptive: Nitrogen Mnemonic: N2	ptive Properties	Rep <u>l</u> ace Psat vs <u>T</u>
Maximum manifold pressure: Non-ideality factor: Density conversion factor: Molecular cross-sectional area:	1050.00 mmHg 0.0000620 0.0015468 0.162 nm ²	
		se

- 4. Verify that Nitrogen is displayed as the adsorptive. Nitrogen is the appropriate adsorptive in most analyses.
- 5. Enter information (or accept the defaults) in the following fields (refer to Adsorptive Properties in Chapter 5):

Maximum manifold pressure Non-ideality factor Density conversion factor Molecular cross-sectional area

6. Click Save , then Close

Report Options

Report options files specify the type of reports which can be generated from an analysis or from manually entered data. They also contain report details such as axis scale, axis range, and column headings. These report options files may be used to generate reports automatically at any time during or after the analysis. Of course, reports generated during analysis can only report on data collected up to the time of the report.

Report options files may contain tabular reports only, plots only, or both tabular reports and plots. They may also contain user-defined report tables.

Report options files also may be defined to include overlay options. The TriStar program enables you to overlay up to eight plots of different samples onto a ninth plot of the same type. Or you can overlay one plot type onto a different plot type from the same analysis. Refer to **Generating Overlays** in this chapter.

Perform the following steps to define a report options file:

- 1. From the **File** menu, select **Open**, **Report options**; the Open Report Options File dialog is displayed.
- 2. Enter a name in the **File name** field, then click **OK**.
- 3. Click Yes to create the file; the Report Options dialog is displayed:

Report Options			
Description: Report Options Replace			
Show report <u>t</u> itle	Micromeritics Instrument Corporation		
Show graphic	Micro.bmp	Browse	
O <u>v</u> erlays	Height: 0.250 in. Width: 2.000 in Selected Reports: Isotherm ✓ BET Surface Area Langmuir Surface Area V t-Plot Alpha-S Method 6-Ratio Method BJH Adsorption BJH Desorption Dubinin MP-Method Options Summary Sample Log	n. <u>E</u> dit	
Enter a value between 0.100 and 7.500.			
	<u>S</u> ave	ose	

4. 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**.

5. 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.

- 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.
- 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**, then **Close** to save the information and close the dialog.

Creating a Sample File

Whether you choose to create your sample file in advance or at the time of analysis, 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 may be created in any of the sample presentation formats:

- Advanced. This format provides complete access to all parts of the sample file, allowing you to edit parameters quickly and conveniently.
- **Basic.** This format presents a single window from which you select predefined parameter files. You can also switch to the Advanced format if editing is required.
- **Restricted.** This format presents a single window from which you select predefined parameter files. You cannot, however, switch to the Advanced format for editing.

Basic and Restricted Formats

The Basic and Restricted formats are the easiest and simplest way to create a sample file. You simply create your sample information file using *predefined parameter files* (explained in this chapter). If more detailed conditions or custom files are required, you must use the Advanced format (discussed next).

- If you are using the **Basic** format, you can switch to the Advanced format if editing is required.
- If you are using the **Restricted** format, you cannot edit parameter files.

Perform the following steps to create a sample information file using the basic or restricted format.

- 1. From the **File** menu, select **Open**, **Sample Information**; the Open Sample Information File dialog is displayed.
- 2. Accept the next sequenced file number or enter a new name in the **File name** field.

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

Sample Information Sample: 000-002 Mass: 1.0000 g Density: 1.000 g/cm ³			
Sample <u>t</u> ube: D <u>eg</u> as conditions: Analy <u>s</u> is conditions: Adsorptive properties:	.0000 g Density: 1.000 g/cm³ Sample Tube • Degas Conditions • Run Conditions • Nitrogen •		
Report <u>o</u> ptions:	Report Options Replace All Add Log Entry		
Save	<u>C</u> lose <u>A</u> dvanced		

- 4. Enter the sample's mass in the Mass field.
- 5. Enter the material's density in the **Density** field. This value is used for the Quasi-Gemini or Calculated free-space methods.
- 6. Click on the down arrow to the right of each of the following fields to choose parameter files:
 - Sample Tube
 - Degas Conditions (if using the SmartPrep degasser)
 - Analysis Conditions
 - Adsorptive Properties
 - Report Options



If you have an existing sample file containing the options you wish to use for this file, click Replace All... to choose the file and copy the options.

7. Click Save , then Close .

Advanced Format

The Advanced format presents all parts of the sample file in an index card manner. This format lets you customize your sample file, allowing you quick access to all parameters.

Perform the following steps to create a sample information file using the advanced format.

- 1. From the **File** menu, select **Open**, **Sample Information**; the Open Sample Information File dialog is displayed.
- 2. Accept the next sequenced file number or enter a new name in the File name field.
- 3. Click OK , then Yes to create the file; the Sample Information dialog is displayed.

These fields may not	≤< Prev Sample Sample Degas Analysis Adsorptive Report Next ≥>]
These fields may not display if omitted in	Sample: 000-001	
sample defaults.	Operator:	
	Submitter:	
	Mass: 1.0000 g Type of Data Density: 1.000 g/cm³ © Automatically collected	
	Comments:	
	Add Log Entry	
	Replace All	
	Enter a value between 0.0010 and 500.0000.	
	<u>S</u> ave <u>C</u> lose B <u>a</u> sic	

- 4. In the **Operator** field, enter the name of the person 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 sample's **mass** and **density**. The density is only relevant if you are measuring the free space using the Quasi-Gemini or Calculated method.

- 7. Choose whether you are going to use manually entered data or have it automatically collected.
- 8. After you complete the Sample Information dialog, click on the parameter tabs to open associated dialogs and edit as desired. Instructions for completing these dialogs are explained in Defining Parameter Files in this chapter.
- 9. After you have completed all parameters, click Save , then Close .

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 Table 4-1 for a list of materials needed to clean and weigh samples.

Table 4-1. Material	s Required to Cle	ean and Weigh S	Sample Tubes
---------------------	-------------------	-----------------	--------------

Supplied by Micromeritics	Supplied by User
Sample tube	Drying oven
Filler rod (if used)	Ultrasonic cleaning unit
Sample tube brush	Detergent
Stopper for sample tube	Rubber gloves or lint-free cloth
Sample tube rack	Acetone or isopropyl alcohol
Sample weighing support	Safety glasses
Sample data worksheet (copied	Waste container
from Appendix A of this manual)	Balance
	Pipe cleaners

- 1. Turn on the drying oven used for heating the sample tubes and filler rods and set the temperature to 110 $^{\rm o}$ C.
- 2. Check the reservoir of the ultrasonic cleaning unit to make sure it is clean.
- 3. Using 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water, fill the reservoir of the ultrasonic unit with enough water to cover the sample tubes 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.
- 4. Fill the sample tubes with warm water and place them in the reservoir of the ultrasonic cleaning unit. Place the filler rods in the bowl also. Turn on the ultrasonic cleaning unit for approximately fifteen minutes.

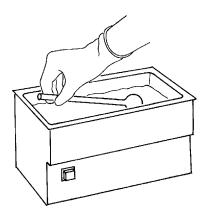


Figure 4-1. Cleaning Sample Tubes

- 5. Using rubber gloves, remove the sample tubes and filler rods from the reservoir.
- 6. Clean the interior of the sample tubes with the brush supplied with the TriStar System.
- 7. Rinse the sample tubes and filler rods thoroughly with hot water. Then rinse them with isopropyl alcohol or acetone using a waste container to collect used solvent.



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

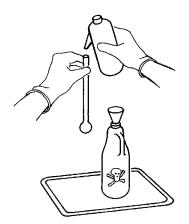


Figure 4-2. Rinsing Sample Tubes

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

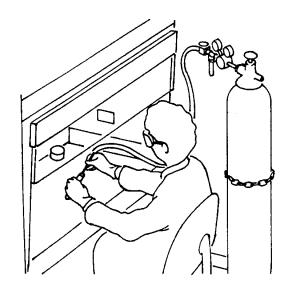


Figure 4-3. Drying Sample Tubes

- 9. Stand the sample tubes on the sample tube rack and place the filler rods in a basket or in the rack. Bake in a vacuum oven for two hours.
- 10. Remove the sample tubes and filler rods from the oven and allow them to cool.



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

- 11. Wipe a rubber stopper with a lint-free cloth.
- 12. Label the sample tube and stopper for identification.

Calibrating Sample Tubes

You **must** calibrate sample tubes if you wish to use them to perform Quasi-Gemini analyses or to calculate free space. The calibrated volume is entered in the sample tube parameter file.

Make a copy of the **Sample Tube Calibration Worksheet** provided in Appendix A, then proceed with the instructions provided below.

- 1. Write the Sample Tube Identification on the worksheet.
- 2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).
- 3. Place a clean, empty sample tube (without stopper) on the sample weighing support. Record the stabilized weight on the worksheet as *Empty sample tube weight*.

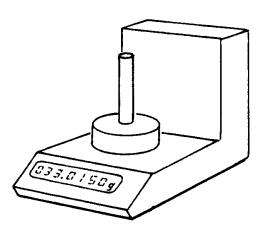


Figure 4-4. Weighing Sample Tube

4. Fill the sample tube with deionized water to the red line on the stem; the meniscus should be even with the line.

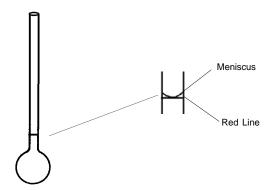


Figure 4-5. Level of Deionized Water

- 5. Place the sample tube containing water on the sample weighing support. Record this weight on the worksheet as *Sample tube plus water*.
- 6. Subtract the empty sample weight (step 3) from the weight of the sample tube containing water (step 5). Record this value on the worksheet as *Weight of the water*.
- 7. Insert a thermometer into the water to obtain its temperature, then refer to Table 4-2 to determine the water's density. Record the density on the worksheet.

Temperature (°C)	Density (g/cm ³)	Temperature (°C)	Density (g/cm ³)
15	0.9991016	23	0.9975412
16	0.9989450	24	0.9972994
17	0.9987769	25	0.9970480
18	0.9985976	26	0.9967870
19	0.9984073	27	0.9965166
20	0.9982063	28	0.9962371
21	0.9979948	29	0.9959486
22	0.9977730	30	0.9956511

Table 4-2. Water Density

- 8. Divide the weight of the water (step 6) by the density of water (step 7) to obtain the physical volume below the red line on the sample tube.
 - a. Record this value on the worksheet.
 - b. Enter this value in the **Physical volume below mark** field in the Sample Tube dialog box.

Preparing the Sample for Analysis

A sample must be properly weighed and degassed before installing it on the sample port for analysis.

Make a copy of the **Sample Data Worksheet** provided in Appendix A, then proceed with the instructions provided below.

Weighing the Sample

Analysis results are expressed in units of surface area per gram of sample; therefore, the true sample mass must be known. Carefully weigh each sample tube set (sample tube and stopper) and sample as described below.

- 1. Write the Sample Tube Identification on the Sample Data Worksheet.
- 2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).
- 3. Place the sample tube set on the sample weighing support. Record the stabilized weight on the Sample Data Worksheet as *Empty sample tube mass*. Remove the sample weighing support and sample tube set from the balance.

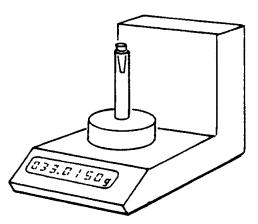


Figure 4-6. Weighing Sample Tube Set

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



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

- 5. Slowly add the desired amount of sample to the container.
- 6. Weigh the stabilized sample and record the value on the Sample Data Worksheet as *Sample mass (Before Degas)*.
- 7. Remove the rubber stopper from the sample tube.
- 8. Pour sample from the weighing container into the sample tube using the funnel provided.

If some sample clings to the inside of the sample tube above the red line, remove it using a pipe cleaner or lint-free wipe.

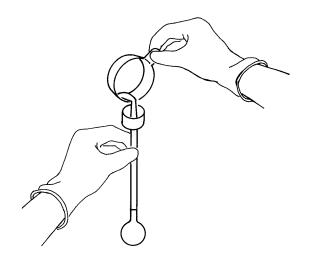


Figure 4-7. Placing Sample in Sample Tube

- 9. Replace the rubber stopper.
- 10. Weigh the sample tube set containing the sample and record the value on the Sample Data Worksheet as *Sample tube set plus sample mass (Before Degas)*.

Degassing the Sample

After the sample has been weighed, use a degassing unit to remove any contaminants which may have adsorbed to the surface or pores of your sample. Appropriate degassing units are available from Micromeritics. Refer to Chapter 10 for ordering information.

After degassing is complete, weigh the sample tube set containing the sample and record the weight on the Sample Data Worksheet as *Sample tube set plus sample mass (After Degas)*.

Installing the Sample Tube

You may use the three analysis ports as follows:

- Port 1 for a sample analysis
- Port 2 for a sample analysis or a Quasi-Gemini (blank sample tube) measurement
- Port 3 for a sample analysis or continuous Po measurement

To attach a sample tube to a port, follow the steps below.

- 1. Remove the sample tube stopper.
- 2. If using an isothermal jacket: slide the jacket down over the stem of the sample tube until it touches the bulb of the sample tube.
- 3. Install the dewar cover by inserting the sample tube through one of the holes in the cover. Make sure the tapered side of the cover is toward the bulb of the sample tube.

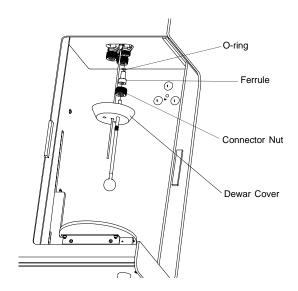


Figure 4-8. Attaching a Sample Tube to an Analysis Port

- 4. Place the connector nut, ferrule, and O-ring onto the sample tube stem.
- 5. Carefully insert a filler rod (if used) into the sample tube.
- 6. Attach the sample tube to the analysis port. Make sure it is fully in the port. Secure it in place by screwing the connector nut onto the analysis port. Hand-tighten the connector nut.
- 7. Slide the dewar cover upward until it stops.

Installing the Analysis Dewar

Prepare the analysis Dewar after you have installed the sample tube(s),



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

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

- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.
- When pouring liquefied gases from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the liquefied gas slowly to prevent splashing, and 3) vent the receiving container to the atmosphere.
- Use a plastic stirring rod when stirring substances in a Dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some type of protective coating.
- Do not remove the mesh covering from the Dewar flask. This covering is in place to minimize the risk of flying particles if the Dewar is accidentally knocked over or dropped and broken.
- Do not handle heavy objects above the Dewar. If unavoidable, place a protective cover over the Dewar's opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

Install the analysis Dewar as follows:

1. Fill the dewar with the analysis bath liquid (liquid nitrogen) to about 5 cm (2 in.) from the top.



Incorrect fluid levels can lead to measurement errors. You should check the level of the bath liquid before each analysis.

2. Insert the dipstick into the dewar and check the level of the analysis bath liquid.

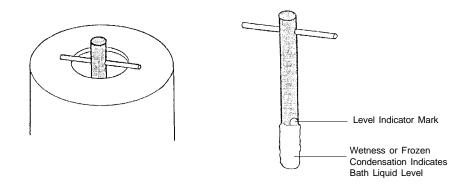


Figure 4-9. Analysis Bath Fluid Dipstick

3. Allow approximately 30 minutes (for best results) for the temperature of the dewar to stabilize with that of the bath liquid. Then recheck the level of the bath liquid; add additional liquid if necessary.



It is unnecessary to allow time for stabilization unless the dewar has not been in use for a while.

- 4. Place the analysis dewar onto the elevator.
- 5. Close the analysis compartment doors.

Performing an Analysis

Once a sample has been degassed and transferred to an analysis port, you may begin analysis. The TriStar provides two types of analyses from which you may choose:

- Standard allows you to perform up to three analyses using different analysis conditions.
- QuickStart allows you to perform multiple sample analyses using t analysis conditions.

Refer to chapter 6 for a detailed description of the dialog boxes shown in any seen ton.

Standard Analysis

1. From the **Unit** menu, select **Sample Analysis**; the following dialog box is displayed.

	🗖 Analysis (Unit 1 - S/N: Demo)	•
Indicates Freespace Meas- urement and shows only the Sample Tube File	View Operation Port 1: C;\WIN3000\000-003.SMP Browse Clear	
field and the Browse push button when this port is be-	Sample: 000-003 Clear Sample weight 1.000 g Sample density 1.000 g/cm ²	
ing used for the Quasi Gemini Method.	Port 2: C:\WIN3000\D00-019.SMP Browse Clear	
	Sample: 000-019 Sample weight 1.000 g Sample density 1.000 g/cm ²	
Indicates Po Measurement (no other fields are shown when this port is being used	Port 3:	
to measure the Po on a con- tinuous basis.	Sample weight 1.000 g Sample density 1.000 g/cm ^a Po: 760.000 mmHg Bath temperature: 77.30 K Report After analysis	
	Start Close Close	
	Port Sample Status 1 Idle 2 Idle 3 Idle	

- 2. Click Browse... to choose a sample information file to be used for each port.
- 3. The sample identification, sample weight, and sample density contained in the sample information file are displayed. Verify the sample weight and density. The density value is irrelevant unless you are using the Quasi-Gemini or Calculate method for the free-space determination.

- 4. The Po and bath temperature are displayed; these may be edited if desired.
- 5. Select **Report after analysis** to have a report generated automatically when the analysis completes.
- 6. Click **Start** to start the analysis; an analyzing view of the window is displayed so that you can view data as it is collected. A short delay is encountered before the port status changes from the *Idle* state.

QuickStart Analysis

It is not necessary to prepare sample files in advance when using the QuickStart method. The information in the sample files is inserted automatically from the parameter files you choose.

1. From the **Unit** menu, select **QuickStart**; the following dialog box is displayed.

		QuickStart (Unit 1 - S/N: test)	-
0	peration 🛓]	
	Sample Options Analysis Conditions: Adsorptive Properties: Report Options:	MICRO/BJH W/N2 @ 77K Nitrogen @ 77.35 K BJH Desorption Report Options	<u>.</u>
	Number of Re Destination	Report After Analysis sports 1 Printer 2 Stylsers(s&M)FRANKIEP(TRANSFER(T))	
	<u>N</u> ext >>		<u>Cancel</u> Close
Port Sample 1 2 3	Status Idle Idle Idle Idle		

- 2. Select the **Analysis Conditions**, **Adsorptive Properties**, and **Report Options** files to be used for the analyses.
- 3. Select **Report After Analysis** and enter the number of reports and report destination, if desired.

4. Click Next>> ; the following dialog is displayed.

-								
	-		QuickStart (Unit	1 - S/N: test)				- ▲
Indicates Freespace Meas- urement; shows only the	⊻iew Ope	eration 👤						
Sample Tube File field and the Browse push but-	Port 1:	C:\WIN3000\000-003.SMP			Browse	Clear]	
ton when this port is being	Sample:	000-003		Sample weight	1.000	g	-	
used for the Quasi Gemini Method.	Sample Tube		Ŧ	Sample density	1.000	g/cm³		
	Port 2:	C:\WIN3000\000-019.SMP			Browse	Clear]	
	Sample:	000-019		Sample weight	1.000	g		
	Sample Tube		Ŧ	Sample density	1.000	g/cm³		
	Port 3:				Browse	Clear]	
				Sample weight	1.000	g		
	Sample Tube		Ŧ	Sample density	1.000	g/cm°		
Indicates Po Measurement (no other fields are shown	Po: 76	0.000 mmHg	В	ath temperature:	77.30	к		
when this port is being used to measure the Po on a con-		<u>S</u> tart			<u>C</u> e	incel	C <u>l</u> ose	
tinuous basis)								

5. Assign a sample information file to each port:

a. Click Browse..., then OK .

b. Click Yes to create the file.

The analysis conditions, adsorptive properties, and report options selected on the previous screen are inserted automatically into the newly created files.

- 6. Select a sample tube file for the sample tube you plan to use.
- 7. Enter the sample's mass and density. The density is used only for the Quasi-Gemini or Calculated free-space method.
- 8. Ensure that the Po value and bath temperature are correct; edit if needed.
- 9. Click **Start** to start the analysis; the following actions occur:
 - A view of the data collection as it progresses is displayed. A short delay is encountered before the port status changes from the *Downloading* state. If desired, you can obtain a report on the data collected so far by selecting the appropriate **Report Port [n]** push button; the report is printed to the screen.
 - When the analysis finishes, the QuickStart dialog shown in Step 4 again is displayed.
- 10. Repeat steps 5 through 9 to perform additional analyses or click **Close** to close the QuickStart dialog.

Printing File Contents

You can print the contents of one or more of the following file types to the screen, a printer, or to a file on disk.

- Sample information
- Sample tube
- Degas conditions
- Analysis conditions
- · Adsorptive properties
- Report options
- 1. From the **File** menu, select **Print**. A drop-down list containing file types is displayed.
- 2. Choose the file type; a dialog similar to this one is displayed.

	_	Print S	ample Informatio	n File			
	File name: 0	I21.SMP	Complete				
	Selection Crite	eria	Settings				
	Status A	1	± Copies	1			
			Destination	Screen	Ŧ		
)ates	File name	G:\USERS	S&MFRANKIE		Choose the
							destination.
	Files:				Directories: q:\\tristar\data	a	
Choose the file you wish to print.	0121.smp 0136.smp 0140.smp 0141.smp 0176.smp 0176.smp 0178.smp 0186.smp 0200.smp 0201.smp 0309.smp 1310.smn *	13 X REF 004-16820 13 X REF 004-16820	04/16826/00 13L 04/16826/00 14H 04/16826/00 12M 04/16826/00 13L 04/16826/00 14H ve Powder	м _	[.] [-a-] [-b-] [-b-] [-b-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-d-] [-b-][-b-]	÷	
Click to complete		<u> </u>		Cancel			

- 3. From the **Files** list box, select the file you wish to print. If you plan to print multiple files, hold down **Ctrl** while clicking on the desired files.
- 4. From the **Settings** group box, choose a **Destination** for your file. If you choose *File* as the destination, enter a name in the **File name** field, or accept the default. If you have selected multiple files, individual files are exported as their file name with the extension .RPT.
- 5. Click **OK**; the file is printed to the specified destination.

Generating a List

You can generate a list of the following information on one or more files to the screen, a printer, or to a file on disk.

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- Status
- 1. From the **File** menu, select **List.** A drop-down list containing the following file types is displayed.
 - Sample information
 - Sample tube
 - Degas conditions
 - Analysis conditions
 - Adsorptive properties
 - Report options
- 2. Choose the file type; a dialog similar to this one is displayed.

	1		
		st Sample Information File	
	File name: 0121.SMP	Complete	
	Selection Criteria	Settings	
	Status All	Copies 1 A Destination Screen	L Choose the
	Dates	File name G:\USERS\S&M\FRANKI	lE destination.
	Files:	Directories:	
Choose the file(s) on which you wish	0121.smp ALUMINA REF 0136.smp 13 X REF 004-	g:\\tristar\d ERENCE 004/16816/00 C1	+
to generate a list.	0176.smp 13X REFEREN	-16826-00 MAL 2818 14H [] NCE 004/16826/00 12L [-d-]	
	0178.smp 13X REFEREN	NCE 004/16826/00 13L [-e-] NCE 004/16826/00 14H [-f-] NCE 004/16826/00 12M [-g-]	
	0201.smp 13X REFEREN 0309.smp 13 X Molecula	NCE 004/16826/00 13L [-h-] NCE 004/16826/00 14H [-q-] Ir Sieve Powder [-w-]	
	1310 smn 13 X Molecula *	r Sieve Powder	+
Click to complete the process.	<u> </u>	Cancel	

3. From the **Files** list box, select the file from which you wish to have a list generated. If you plan to list information for multiple files, hold down **Ctrl** while clicking on the desired files.

- 4. From the **Settings** group box, choose a **Destination** for your list. If you choose *File* as the destination, enter a name in the **File name** field, or accept the default. If you have selected multiple files, individual files are exported as their file name with the extension .LST.
- 5. Click $\bigcirc \kappa$; the list is printed to the specified destination.

Exporting Isotherm Data

Export allows you to copy the isotherm data in a sample information file and export it in a format it in ASCII text. If saved to a File, the data can be imported into applications such as spreadhseets. The output file consists of four columns containing the absolute pressure, relative pressure, specific volume adsorbed, and the elapsed time.

1. From the **File** menu, select **Export**; the Export Sample File dialog is displayed.

	Expo	ort Sample File	
	File name: 0121.SMP C Selection Criteria Status All 🛃 Dates	Complete Settings Export Type ASCI Copies 1 Destination Screen File name G:\USERS\S&M\FRANKI	Choose the type. Choose the destination.
Choose the file you wish to export	Files: 0121.smp ALUMINA REFERENC 0136.smp 13 × REF 004-16826-0 0140.smp 13 × REF 004-16826-0 0141.smp 13 × REF 004-16826-0 0176.smp 13 × REFERENCE 004, 0176.smp 13 × REFERENCE 004, 0178.smp 13 × REFERENCE 004, 0196.smp 13 × REFERENCE 004, 0200.smp 13 × REFERENCE 004, 0201.smp 13 × REFERENCE 004, 0201.smp 13 × REFERENCE 004, 0309.smp 13 × Molecular Sieve 0101.smp 13 × Molecular Sieve	0 MAL 2818 12M 1 ca-] 1 MAL 2818 13L 1 6b-] 1 6826/00 12L 1 6826/00 13L 1 6826/00 13L 1 6826/00 13H 1 6826/00 12M 1 6926/00 13L 1 6826/00 13L 1 6926/00 13L 1 6926/00 13L 1 6926/00 13H 1 -q-] 1 6826/00 14H 1 -q-] 1	
Click to complete	<u> </u>	Cancel	

- 2. From the **Files** list box, select the file you wish to export. If you plan to export multiple files, hold down **Ctrl** while clicking on the desired files.
- 3. From the **Settings** group box, choose a **Destination** for your exported file.

If you choose *File* as the destination, enter a name in the **File name** field, or accept the default. If you have selected multiple files, individual files are exported as their file name with the extension .ISO (isotherm).

4. Click **OK**; the file is exported to the specified destination.

Generating Graph Overlays

Use the graph overlay function when you wish to compare graphically multiple graph options. Overlays may be generated in two ways:

• Multiple Graph Overlays

Overlaying two different types of graphs from the same sample on top of each other. This type of overlay is available for BJH Adsorption/ Desorption reports only.

• Multiple Sample Overlays

Overlaying the same type of graph for up to eight samples on top of a previously selected sample.

Varying symbols are used to differentiate the graphical lines and reported in a legend at the top of the output. If color output is available, different colors are used instead.

Multiple Graph Overlays

To overlay two different types of graphs from the same sample file:

- 1. From the **File** menu, select **Open**, **Sample Information**. Double-click on the desired file. If your window is presented in the Basic format, click **Advanced**.
- 2. Select the **Report Options** tab.
- 3. Select the type of report on which you wish to have overlays generated. Remember, only BJH Adsorption/Desorption reports can be chosen for this type of overlay.

4. Click Edit...; the BJH Adsorption/Desorption dialog is displayed.

- BJH	Adsorption Report Options	l
Thickness Curve Type O Harkins and Jura Halsey Eguation Options Smooth differentials Adsorptive Range to use	Minimum BJH diameter: 1.700 nm Maximum BJH diameter: 300.000 nm Fraction of pores open at both ends: 0.00 Selected Reports Comutative Pore Volume dV/dD Pore Volume dV/dD Pore Volume dV/dlog(D) Pore Volume Cumulative Pore Area dA/dD Pore Area dA/dlog(D) Pore Area #	 Select the report variable Click to choose the second report variable.
		-

5. From the **Selected Reports** window, choose the report variable on which you wish to have another variable overlaid, then click **Edit...** . A dialog similar to this one is displayed.

BJH Adsorption Cumulative Pore Volume Options	
Plot Options Image: Plot option option Image: Plot curve Image: Plot option option	
X-Axis © Linear C Logarithmic □ Autoscale x-axis 1.0 To 1000.0 nm Y-Axis	Displays the first report variable.
Variable: Cumulative Pore Volume	
Autoscale y-axis 0.0000 To 9999.0000 cm²/g	Choose the second variable from this
	drop-down list.

6. From the **Overlay** drop-down list, choose the report variable you wish to overlay, then click Οκ.



You do not have to save these options before generating overlay reports. If you do save, the report options for your original sample file will be replaced with the new ones.

- 7. From the **Reports** menu, select **Start report**; the Start Report dialog is displayed. In most cases, your file name will be highlighted and displayed in the **File name:** field. If it is not, simply navigate to the appropriate directory and choose the file.
- 8. Click **OK**; the **Select Reports** dialog is displayed. Click **OK** again to close the dialog and generate the overlays.

Multiple Sample Overlays

To overlay the same type of graph on multiple samples:

- 1. From the **File** menu, select **Open**, **Sample Information**. Double-click on the desired file. If your window is presented in the Basic format, click **Advanced**.
- 2. Select the **Report Options** tab.
- 3. Choose the type of report from the **Selected Reports** window; then click **Edit...**.
- 4. Choose one:

If you are overlaying these types of reports	Then
Isotherm BET Surface Area Langmuir Surface Area t-Plot	Select the Overlay Samples checkbox. Click ок.
BJH Adsorption BJH Desorption	Choose the report variable from the Selected Reports window, then click Edit
	Click on the down arrow in the Overlay: field and choose Samples ; then click OK .
	Click ok again to close the BJH Adsorption/Desorption dialog and display the Report Options dialog.

5. From the Report Options dialog, click **Overlays...**; the Graph Overlays Samples dialog is displayed.

- 6. Click **Browse**... to the right of the Sample [n] field; the Open Sample Information dialog is displayed.
- 7. Choose the file containing the graph you wish to overlay, then click **OK**. You may choose up to eight files in this manner.



You do not have to save these options before generating overlay reports. If you do save, the report options for your original sample file will be replaced with the new ones.

- 8. From the **Reports** menu, select **Start report**; the Start Report dialog is displayed. Your primary file should be highlighted and in the **File name:** field. If it is not, simply navigate to the appropriate directory and choose the file.
- 9. Click **OK**; the Select Reports dialog is displayed. Click **OK** again to close the dialog and generate the overlays.

CHAPTER 5

FILE MENU

- Description
- Open
- Save
- . Save As
- · Save All
- Close
- · Close All
- Print
- List
- Export
- Exit

FILE MENU

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

Description

Open



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

Opens an existing file or creates a new one. When you select this item, a drop-down menu containing the following file types is displayed:

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

Save Saves the file in the active window.

Save AsSaves 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 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.
Export	Exports the isotherm data contained in a sample file.
Exit	Exits the analysis program.

Open

Open allows you to create or edit a sample or parameter file.

Regardless of which file type you select, a dialog box similar to the one shown here is displayed.

	Open Sample Information File	
File name:	0176.SMP Complete	
Selection (Criteria	
Status:	All	
Status.		
	Dates	
		Directories:
E .1		g:\\tristar\data
Files:	-	
0121.smp	ALUMINA REFERENCE 004/16816/00 C1 4	h [] 🔶
0136.smp	13 X REF 004-16826-00 MAL 2818 12M	[-a-]
0140.smp	13 X REF 004-16826-00 MAL 2818 13L	[-b-]
0141.smp	13 X REF 004-16826-00 MAL 2818 14H	[-c-]
0176.smp	13X REFERENCE 004/16826/00 12L	[-d-]
0177.smp	13X REFERENCE 004/16826/00 13L	[-e-]
0178.smp	13X REFERENCE 004/16826/00 14H	[-1-]
0196.smp	13X REFERENCE 004/16826/00 12M	[-g-]
0200.smp	13X REFERENCE 004/16826/00 13L	[-h-]
0201.smp	13X REFERENCE 004/16826/00 14H	[-q-]
0309.smp	13 X Molecular Sieve Powder	[- w -]
0310 smn	13 X Molecular Sieve Powder	
*	*	+
		_
	O <u>K</u> Cance	

File name

For *sample information* files, this field contains the next sequenced file name generated by the analysis program.

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

*.STB for sample tubes
*.DEG for degas conditions
*.ANC for analysis conditions
*.ADP for adsorptive properties
*.RPO for report options

If you are creating a new file, enter a name in the **File name** field. If you are editing an existing file, select a file from the list.

Refer to **Selecting Files** in Chapter 3 for a description of the other fields on this dialog box.

Open

Sample Information

Sample information files contain information used to control the analysis. Therefore, every analysis must be linked with a sample information file before the analysis can proceed. A sample information file is comprised of the following:

- sample identification
- sample tube parameters
- degas conditions (if using automatic degassing)
- analysis conditions
- · adsorptive properties
- · report options
- collected/entered data (when 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. Once it becomes part of the new sample file, you can edit it in any way you wish without changing the file from which it was copied.

Sample information files are presented in three formats: Advanced, Basic and Restricted.



Specify or change your format by selecting *Option presentation* from the Options menu.

Basic

Presents the entire sample information file in a single dialog. With this format, you can quickly create a sample information file using previously defined parameter files. This format also allows you to switch to the Advanced format to view or edit parameters.

Advanced

Presents all parts of the sample information file in a single, index card tabbed dialog. Each tab opens its associated dialog. Use this format to create customized files.

Restricted

Presents the sample information file in a single dialog similar to the Basic format. With the Restricted format, however, you cannot switch to the Advanced format to edit parameters. Some menu items also are unavilable.

Basic

When you open an existing sample information file or create a new one using the Basic format, all parts of that file are contained on a single dialog. With the Basic format, you can quickly create a sample information file using previously defined parameter files. This format also allows you to switch to the Advanced format to view or edit parameters. Chapter 4 provides step-by-step instructions for creating a Basic sample information file.

Sample Information	
Sample: 000-002	
<u>M</u> ass: 1	.0000 g <u>D</u> ensity: <u>1.000</u> g/cm ²
Sample <u>t</u> ube:	Sample Tube
Degas conditions:	Degas Conditions
Analysis conditions:	Run Conditions
Adsorptive properties:	Nitrogen 🗾
Report options:	Report Options
	Replace All Add Log Entry
Save	Close Advanced
<u> </u>	



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

Sample	Displays the next sequenced file description or the name of the file you are opening. You may enter a new descrip- tion or add to the existing one. <i>Range: 50 alphanumeric characters</i>
Mass	Enter the mass of the sample to be analyzed. Use <i>1.0000</i> when performing a blank analysis.
Density	Enter the density of the sample to be analyzed. This value is used only for the Quasi-Gemini or Calculated free-space method. Use 0.000 when performing a blank analysis.

Add Log Entry

Displays the Add Log Entry dialog.

Add the entry to the log report	
Degas by VacPrep at 350 deg for one hour.	
0 <u>K</u> ancel	

Enter information pertinent to the sample file. For example, if you are not using the SmartPrep for automatic degassing, you may wish to enter external degassing information. Any information you enter here is printed as part of the sample log report. You may make multiple entries by selecting this push button as many times as you wish.

Sample Tube Click the down arrow to display a list of previously defined sample tube files supplied with the analysis program, as well as any you may have created. Choose the sample tube file you wish to use.

Degas Conditions Click the down arrow to display a list of previously defined degas conditions files supplied with the analysis program, as well as any you may have created. Choose the degas conditions file you wish to use.

These files are pertinent only if using the SmartPrep for degassing samples.

Analysis Conditions Click the down arrow to display a list of previously defined analysis conditions files supplied with the analysis program, as well as any you may have created. Choose the Analysis Conditions file you wish to use.

Adsorptive Properties	Click the down arrow to display a list of previously de- fined adsorptive properties files supplied with the analy- sis program, as well as any you may have created. Choose the adsorptive properties file you wish to use.
Report Options	Click the down arrow to display a list of previously de- fined report options files supplied with the analysis pro- gram, as well as any you may have created. Choose the Report options file you wish to use.
Replace All	Allows you to copy <i>all</i> parameters of an existing sample file's values into the sample information file you are creating. An "open file" dialog is displayed so that you may select the desired file. Click OK ; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged.
Save	Saves the information you have specified for this sample file.
Close	Closes the dialog box. If a file containing unsaved changes is open, you are prompted to save before the dia- log box closes.
Advanced	Displays the sample information file in the advanced for- mat, allowing you to view or edit parameters.

Advanced

When you open an existing sample information file or create a new one using the Advanced format, all parts of the sample file are displayed in a tabbed dialog. The Advanced format allows you to customize sample files. Chapter 4 provides step-by-step instructions for creating an Advanced sample information file.

≤< Prev Sample Sample Degas Analys Information Tube Conditions Condit	
Sample: 000-001	
Operator:	
Submitter:	
<u>M</u> ass: <u>1.0000</u> g <u>D</u> ensity: <u>1.000</u> g/cm ²	Type of Data © A <u>u</u> tomatically collected © Manually <u>e</u> ntered
Co <u>m</u> ments:	
	Add Log Entry
	Rep <u>l</u> ace All
Enter a value between 0.0010 and 500.0000.	
<u>S</u> ave <u>C</u> lose	Basic

The prompts for the **Sample**, **Operator**, and **Submitter** fields may be customized by selecting **Options**, **Sample defaults**. Refer to Chapter 8 for instructions on customizing these prompts.

Sample	Displays the next sequenced file description or the name of the file you are opening. You may enter a new descrip- tion or add to the existing one.
	Range: 50 alphanumeric characters
Operator Submitter	Enter the name of the operator who will be performing the analysis and the name of the person (or department) submitting the sample for analysis. Either or both of these fields may be omitted from this dialog by choosing to do so when establishing defaults (Chapter 8).
	Range: 40 characters

Open

Mass	Enter the sample's mass.
	Use 1.000 when performing a blank analysis.
Density	This value is used only for the Quasi-Gemini or Calcu- lated free-space method.
	Use 0.000 when performing a blank analysis.
Type of Data	Choose whether data are to be collected automatically by the analysis program or manually entered.
	If you choose Manually entered , an "Entered" tab is added so that you may enter the desired data.
Add Log Entry	Displays a dialog box so that you may enter pertinent in- formation relating to the sample file. For example, if you are not using automatic degassing, you may wish to en- ter external degassing information. Any information you enter here is printed as part of the sample log report. You may make multiple entries by selecting this push but- ton as many times as you wish.
Replace All	Allows you to copy an existing sample file's values into the sample information file you are creating. An "open file" dialog is displayed so that you may select the de- sired file. Click OK ; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged. This replaces all parameters of the entire sam- ple information file, excluding any collected data from the other file.



Each of the individual tabbed dialogs contains a Replace... push button if you wish only to replace specific parameters and not the entire contents.

Comments	Enter comments about the sample or its analysis condi- tions. Comments entered here are printed in the header of the reports.
Save	Saves the information you have specified for this sample file.

Close	Closes the dialog box. If changes have been made in the dialog and have not been saved, you will be prompted to save before the dialog box closes.
Basic	Displays the sample information file in the basic format.

Sample Tube

This dialog allows you to enter the specifics for a sample tube. A sample tube file can be created as an independent parameter file or as part of the sample information file.



Be sure to save this file to the directory you specified as the Parameter Files directory if it is to be included in the drop-down list of the Basic Sample Information dialog (see Parameter Files Directory in Chapter 8).

	G:\\TRISTAR\000-001.SMP	•
CALL Complete Complet	e Degas Conditions Analysis Adsorptive Report Op Conditions Properties	tions Next >>
Sample Tube	Replace	
Stem Diameter • 1/4 inch	Physical volume 1.0000 cmª	
O 3/8 inch O 1/2 inch	Use isothermal jacket	
	Use <u>f</u> iller rod	
Save	<u>Close</u> Ba	sic

Sample Tube	Enter a description for the file.	
	Range: 40 alphanumeric characters (at least one character is required)	
Replace	Allows you to replace the values in the current sample tube file with those from an existing file. An "open file" dialog is displayed so that you may select the desired file. Click OK ; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged	
Stem Diameter	Select the diameter of the sample tube you are using for this analysis.	

Physical volume	Enter the volume below the red line on the sample tube.
below mark	Refer to Calibrating the Sample Tube in Chapter 2.
	This value is used only when using the Quasi-Gemini or Calculated method for measuring the free space.



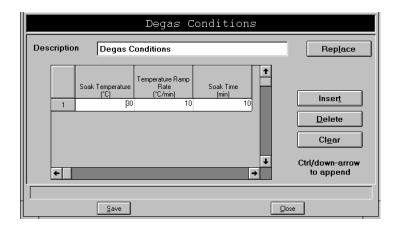
If you plan to use a calculated free space or the Quasi-Gemini method, it is a good idea to set up these files for your sample tubes.

Use isothermal jacket Use filler rod	Select whether you are using an isothermal jacket and/or a filler rod.				
	A <i>filler rod</i> is used to reduce the stem free-space volume, resulting in reduction of free-space error.				
	An <i>isothermal jacket</i> is used to maintain a constant temperature profile along the sample tube stem during an extended analysis (more than 1 or 2 hours).				

This dialog is used only if you are using the SmartPrep Degasser for degassing your sample.



Be sure to save this file to the directory you specified as the Parameter Files directory if it is to be included in the drop-down list of the Basic Sample Information dialog (see Parameter Files Directory in Chapter 8).



Description If you are opening an existing file, this field contains the description.

If this is a new file, enter a description.

Range: 40 alphanumeric characters (at least one character is required)

Replace...Allows you to copy the values of an existing degas conditions file into the file you have open. An "open file" dialog is displayed so that you may select the desired file.ClickOK; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged.

Conditions Table Allows you to enter up to five stages of degas conditions.

Soak Temperature	Enter the temperature at which the sample is to soak while flowing gas.
Ramp Rate	Enter the rate at which the temperature is to change when advancing to the soak temperature.
Soak Time	Enter the amount of time to soak the sample.
Insert	Inserts a row into the conditions table. A row is inserted above the selected line; the cursor moves to the new line.
Delete	Deletes the selected row from the conditions table.
Clear	Clears all entries contained in the conditions table. The rows do not have to be selected.

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.



Be sure to save this file to the directory you specified as the Parameter Files directory if it is to be included in the drop-down list of the Basic Sample Information dialog (see Parameter Files Directory in Chapter 8).

Analysis Conditions							
Descriptio	on Run Conditions <u>Replace</u>						
	Isotherm Collection Image: Target Pressures Options O Dose Increments Options						
Prep <u>a</u> ration	<u>Free Space</u> <u>Po and T</u> <u>Equilibration</u>						
	Save						

Description If you are opening an existing file, this field contains the description.

If you are creating a new file, enter a description.

Range: 40 alphanumeric characters (at least one character is required)

Replace...

Allows you to replace the values in the current analysis conditions file with those from an existing file. An "open file" dialog is displayed so that you may select the desired file. Click $\bigcirc K$; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged.

- **Isotherm Collection** The choices in this group box allow you to specify how you wish to have isotherms collected.
 - Target PressuresSelect this option to have isotherms collected by specify-
ing target pressures. When you select this option,Pressuresand Options(directly to the right of this
field) become enabled. Select each push button to verify
or edit data.

Pressures Displays the Entered Pressures dialog.

5	Ente	red Pressures	:							×
		Relative Pressure	BET Surf.	Lang. Surf.		BJH	вјн	Total Pore	-	X Use calculation assignments
	1	(P/Po) 0.000000001	Area	Area	t-Plot	Ads.	Des.	Vol.	[Insert <u>r</u> ange
										Insert predefined
										Insert
										Delete
										Cl <u>e</u> ar
									-	Ctrl/down-arrow to append
								,	·	
Í	Enter strictly increasing relative pressures up to a maximum of 1.0 followed by strictly decreasing values.									
			0 <u>K</u>							<u>C</u> ancel

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 0.995 P/Po. There must be one adsorption branch followed optionally by one desorption branch.

Several analysis conditions files containing complete pressure tables are included with the TriStar software; they are located in the PARAMS subdirectory. These files (and all other parameter files) can be changed. If you want to use any of these files, it is recommended that you use the **Save As** option on the **File** menu to save the file as a different name; then make your changes.

Pressures

(continued)

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
- BJH Adsorption
- BJH Desorption
- Total pore volume

Insert range...

Displays the Insert Pressure Range dialog.

— Insert Pressure R	ange	
Starting relative pressure	0.01000000	
Ending relative pressure	0.90000000	
Number of points	10	
Progression		
Linear		
O Geometric from low pressure		
O Geometric towards saturation		
	ancel	
Enter a value between 0.000000001 and 1.000000000.		

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.

Choose *Linear* to insert evenly spaced points into your table.

Choose Geometric from low pressure to insert geometrically spaced points from the low pressure range. For example, if you wish 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

Open

Pressures

(continued)

Choose *Geometric from saturation* to insert geometrically spaced points from the saturation pressure. For example, if you wish to insert 5 points with a 0.99 starting pressure and a 0.84 ending pressure, the following points are inserted into the table:

0	.99
0	.98
0	.96
0	.92
0	.84

Insert predefined

Displays the Insert Predefined Pressures dialog.

Insert Predefine	d Pressures 🛛 🗙	
S <u>u</u> rface area:	None	
t-Plot <u>m</u> icropore:	None	
<u>B</u> JH adsorption:	None	
B <u>J</u> H desorption:	None	
✓ Adsorption tota	al pore volume	
Description total pore volume		
□ <u>S</u> aturation		
Note: The predefined pressures make sense only if nitrogen is used as the adsorptive.		
0 <u>K</u> Cancel		

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

Click on the down arrow of 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 pressure table. A row is inserted above the selected line; the cursor moves to the new line.

Delete

Deletes the selected row.

Clear

Clears all entries from the table. The rows do not have to be selected.

Options

Allows you to specify target pressure options; the Target Pressure Options dialog box is displayed.

Target Pressure Options	×
Eirst Pressure Fixed Dose	cm3/g STP
🛛 Maximum volume increment: 25.000	cm3/g STP
Absolute pressure tolerance: 5.000	mmHg
Relative pressure tolerance: 5.0	%
Enter a value between 0.01 and 50.00.	
	<u>C</u> ancel

First Pressure Fixed Dose

Select this option to have the sample dosed 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. Once this first pressure point is reached, points are equilibrated and recorded in accordance with the specified pressure table.

You may wish to use this mode if you are performing a standard nitrogen analysis of microporous or highly mesoporous materials such as catalysts. If the first pressure table point is low, and you expect the gas uptake of the sample to be high, choosing this mode can shorten the time required to reach the first point on the pressure table.

Maximum volume increment

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

When using this option, reaching pressure points exactly is not important; therefore, the tolerances should be set relatively large (10 mmHg and 10% or so) for proper functioning of the algorithm. The pressure table should also have several points scattered over the region of interest.

Options

(continued)

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

Absolute pressure tolerance Relative pressure tolerance

These values 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. For example:

Experiment 1: you have an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg.

Experiment 2: you have an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg.

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

5-20

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.

Displays the Dose Increments dialog.

-		Dose I	ncrements	
	Ending Pressure (P/Po)	Dose Increment (cm3/g STP)	Percent of Previous (%)	
	0000000	1.000		Insert Edit Delete Clear
	<u> </u>		[Cancel

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

Options

Displays the Dose Increment Edit dialog.

- Dose Increment Edit
Ending pressure: 0.100000000 P/Po
Dose Increment 1.0000 cm3/g STP
O Percent of previous: 10.0000 %
O <u>K</u> <u>C</u> ancel
Enter a value between 0.000000001 and 1.000000000.

Ending pressure:

Enter the desired ending pressure of this segment.

Insert Edit (continued)	Dose increment Select this option to have the sample dosed with a speci- fied amount of gas for each isotherm point taken.
	Percent of previous Select this option to have the sample dosed with a per- centage of the volume adsorbed during the previous interval of the table. For example if in the previous seg- ment 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 <i>first</i> entry of the dose in- crement table.
Delete	Deletes the selected range.
Clear	Clears the table of all entries except one; one entry is re- quired.
Preparation	Displays the Analysis Preparation dialog.
	Analysis Preparation Fast evacuation Evacuation rate 50.0 mmHg/sec

Unrestricted evac from:

Evacuation time:

0<u>K</u>

Leak test
 Leak test duration:

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

5.0 mmHg

0.50 hours

120 secs

Cancel

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.

Preparation	In the Evacuation time field, enter the time you wish to
(continued)	allow for preliminary evacuation which takes place prior
	to free-space measurement.

Select **Leak test** to check for system leaks or sample outgassing before the analysis. The leak test seals the sample tube and monitors the pressure. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and you are notified with a message. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists.

If you select **Leak Test**, enter the amount of time you want the pressure monitored.

Displays the Free Space dialog.

- Free Space
 Measure Lower Dewar for evacuation Evacuation time: 0.5 hours Outgas test Outgas test duration: 180 secs
O Quasi-Gemini
O Calculate
O Enter
Warm free space: 16.0000 cm ³
Cold free space: 45.0000 cm ^a
OK Cancel

Specify the type of free-space measurement you wish to have performed. Refer to Appendix F for a discussion of free-space methods.

Measure Measures the free space automatically over the sample prior to data collection.

Lower Dewar for evacuation

Lowers the dewar during evacuation.

Evacuation time

Enter the time you wish to allow for evacuation after the free-space measurement.

Free Space...

Measure (continued)	Outgas test Checks the system for sample outgassing after the free- space measurement. The outgas test seals the sample tube and monitors the pressure. If the pressure rises more than 0.025 mmHg within the time specified for the outgas test duration , outgassing is present. If outgassing is found, the test repeats up to nine times with a 30-min- ute evacuation time between each test. If the 10th test fails, the analysis does not proceed and you are notified of its cancellation. Outgas testing slightly increases analy- sis time, but it prevents continuation of analysis and col- lection of erroneous data if outgassing is present. Enter the outgas test duration.
Quasi-Gemini	Select this option to have the free space of the sample tube continuously balanced against an identical empty sample tube. For each data point taken on the isotherm, a corresponding measurement is taken with the empty sample tube (must be connected on port 2) and that gas quantity subtracted from the gas quantity in the actual sample tube.
Calculate	Select this option to have the free-space measurement cal- culated using the sample and tube parameters.
Enter	 Enables the Warm free space and Cold free space fields so that you may enter the free space manually. <i>The warm free space</i> is the sample tube gas capacity measured at room temperature. <i>The cold free space</i> is the sample tube gas capacity measured with the Dewar raised.

Po and T	Displays the Po and Temperature Options dialog.
	Po and Temperature Options
	Choose one option.
	⊙ 1. Measure Po at intervals during analysis. Enter the Analysis Bath Temperature below.
	© 2. Enter Po below. Enter the Analysis Bath Temperature below.
	© 3. Measure Po over the sample. Enter the analysis bath temperature and an initial estimate for Po below.
	C 4. Measure Po continuously in port 3. Enter the analysis bath temperature below.
	Measurement inter <u>v</u> al: <u>120</u> min <u>I</u> emperature: 77.300 K
	Enter a value between 1 and 30000.
	0 <u>K</u>

This dialog provides four 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	a measurement interval (how often during analysis the Po is to be measured) and the analysis bath temperature
	This method may slow analysis time slightly since the sample port and the Po tube both require interaction with the manifold plumbing system.
2	a Po and analysis bath temperature
	This methoid uses the values you enter in the Po and temperature fields.
3	an analysis bath temperature and an estimate for the initial Po.
	This method measures the Po over the sample. After the Po is measured, the value is reapplied to all data points.

Option	Prompts for
4	an analysis bath temperature.
	This method measures the PO on a continuour basis; enabling you to measure every data point without slowing down the analysis. It does, however, require the dedicated use of one of the sample ports.

Equilibration...

Displays the Equilibration dialog box.

1	Equilibra	tion
	Equilibration interval: Minimum equilibration delay at P/Po >= 0.995:	20 sec 600 sec
En	ter a value between 1 and 10000.	
	<u>ok</u>	Cancel

Enter an equilibration interval.

The equilibration interval is the number of seconds between successive pressure readings during equilibration. At least 11 pressure readings for each equilibration are required to obtain an accurate reading of the pressure changes.

- Long equilibration intervals (15 100 seconds) tend to lengthen analyses but improve data integrity.
- Short equilibration intervals (1 14 seconds) produce faster analyses. For some samples, however, short equilibration intervals may result in less accurate data.

Enter a minimum time for equilibration in the **Minimum** equilibration delay at P/Po > = 0.995 field. This prevents premature equilibration which may be caused by saturation.

Adsorptive Properties

This dialog allows you to specify the characteristics of the gases used in the TriStar system. An adsorptive properties file can be created as an independent parameter file or as part of the sample information file.



If this is a new file, be sure to save it to the directory you specified as the Parameter Files directory to include it in the drop-down list of the Basic Sample Information dialog (see Parameter Files Directory in Chapter 8).

Adsor	ptive Properties	
Adsorptive: Nitrogen		Rep <u>l</u> ace
Mnemonic: N2		Psat vs <u>T</u>
Maximum manifold pressure: Non-ideality factor: Density conversion factor:	1050.00 mmHg 0.0000620 0.0015468	
Molecular cross-sectional area:	0.162 nm ²	
Save	<u>C</u> los	e .

Adsorptive	If this is a new file, this field contains either the name of the default adsorptive gas or blanks. Enter the name of the adsorptive gas.	
	Maximum number of characters: 40 (alphanumeric)	
Mnemonic	The mnemonic name for the adsorptive gas; for example, N2 for nitrogen, CO2 for carbon dioxide, etc.	
Maximum manifold pressure	The maximum gas pressure allowed in the manifold.	
Non-ideality factor	Enter a factor to compensate for the forces of attraction between molecules in a real gas.	
Density conversion factor	Enter the density conversion factor. The density conver- sion factor is determined by obtaining the ratio of the gas volume (STP) to the liquid volume.	

Molecular cross- sectional area	The molecular cross-sectional area.
Replace	Allows you to replace the values in the current adsorptive properties file with those from another file. An "open file" dialog box is displayed so that you may select the desired file. Click $\bigcirc K$; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged.
Psat vs t	Displays the Psat vs. Temperature dialog for the adsorp- tive gas.

	Saturation Pressure (mmHg)	Temperature (°K)	
1	600.193	75.400	
2	634.512	75.850	
3	674.383	76.350	Ins <u>e</u> rt
4	720.420	76.900	Delete
5	742.119	77.150	
6	759.833	77.350	
7	777.867	77.550	
8	805.525	77.850	
9	853.268	78.350	Ctrl/down-arrow
10	903.122	78.850	to append
9	853.268	78.350	Ctrl/down-arro to append

This table contains saturation pressures and their corresponding temperatures. The table may include as many as ten entries; at least two are required.

Edit the table by moving the cursor to the desired saturation pressure or temperature and entering a different value. Points always have to be in an ascending order. If you try to add a point which is less than the previous one, you will receive an error message.

Insert

Inserts a new row. A row is inserted above the selected line; the cursor moves to the new line.

Delete

Deletes the selected row.

Report Options

This dialog box allows you to specify report options for a new file or to edit an existing one. A report options file can be created as an independent parameter file or as part of the sample information file.



If this is a new file, be sure to save it to the directory you specified as the Parameter Files directory to include it in the drop-down list of the Basic Sample Information dialog (see Parameter Files Directory in Chapter 8).

	Report Options		
Descriptio <u>n</u> : Re	port Options	Rep <u>l</u> ace	
Show report <u>t</u> itle	Micromeritics Instrument Corporation		
☑ Show graphic	Micro.bmp Height: 0.250 in. Width: 2	<u>B</u> rowse	
0 <u>v</u> erlays	Selecte <u>d</u> Reports: Isotherm ✓ BET Surface Area Langmuir Surface Area ✓ t-Plot Alpha-S Method F-Ratio Method BJH Adsorption BJH Desorption Dubinin MP-Method Options Summary Sample Log	<u>E</u> dit	
Enter a value between 0.100 and 7.500.			
	ave	<u>C</u> lose	

DescriptionIf you are editing an existing file, this field displays a description of the report options file. You may change the name if you wish.If this is a new file, the default report options description is displayed. Enter a name for this file.Maximum number of characters: 40Show report titleThis 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. You can accept the default title or enter a new one.Maximum number of characters: 50If you deselect this option, a title will not display on the

report.

Overlays

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 enter the height and width in the appropriate fields. This image can also be edited from the report window.

Replace...Allows you to replace the values in the current report options file with those from an existing file. An "open file" dialog box is displayed so that you may select the desired file. Click OK ; the values are copied into the new file automatically. You may edit the values in the new file; the file from which they were copied remains unchanged.

Displays the Graph Overlay Samples dialog.

Graph Ove	erlay Samples	×
Sample 1	C:\WIN3000\DATA\000-180.SMP	Browse
Sample 2	C:\WIN3000\DATA\SI0102.SMP	Browse
Sample 3		Browse
Sample 4		Browse
Sample 5		Browse
Sample 6		Browse
Sample 7		Browse
Sample 8		Browse
overlaid by	You must select which graphs are selecting the Overlay samples on eacl he Report Options.	
	0 <u>K</u>	1

Choose the sample files you wish to overlay onto selected plots.

Click **Browse**... to the right of the sample [n] field to choose a file. You may select up to eight files. After choosing your file(s), be sure to select the "Overlay samples" option for each report type you plan to overlay. Refer to Chapter 4 for instructions on generating overlays.

Selected Reports Contains a list of available reports:

- Isotherm
- BET Surface Area
- Langmuir Surface Area
- t-Plot
- Alpha-S Method
- f-Ratio Method
- BJH Adsorption
- BJH Desorption
- Dubinin
- MP-Method
- Options Report
- Summary Report
- Sample Log Report

You can find examples of these reports in Chapter 7.

Using the mouse pointer or the arrow keys, move to the desired report and double-click the left mouse button, or press **Spacebar**. A report is selected when it is preceded with a check mark. You may deselect it in the same manner.

Edit

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

This button is disabled for some reports, such as the Options and Sample Log reports, since they do not contain parameters which may be edited.

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. Click **Edit** for Isotherm report options; the Isotherm Report Options dialog box is displayed.

	ls	otherm Report	Options	
Select Reports			Plot Option	ns
🗵 Tabular <u>R</u> eport			E Plot a	dsorption
Li <u>n</u> ear plot				•
Logarithmic plot			Plot d	esorption
Linear Plot Options				
Plot c <u>u</u> rve	•	🛛 Plot p <u>o</u> ints	□ 0 <u>v</u> e	erlay samples
		From	То	_
Autoscale <u>x</u> -axis	X:	0.000000000	1.000000000) P/Po
Autoscale <u>y</u> -axis	Y:	0.0000	9999.0000	cm [*] /g STP
Logarithmic Plot Option	ns—			
Plot curve	<u>1</u>	🛛 <u>P</u> lot points	🗆 0ve	erlay sa <u>m</u> ples
		From	То	_
🛛 🕅 Autoscale x-axis	X:	0.000000001	1.00000000) P/Po
🛛 Autoscale y-axis	Y:	0.0000	9999.0000	cm [®] /g STP
			<u>C</u> anc	el

Select Reports	Allows you to choose the type of report you wish to gen- erate. You may choose as many as you wish.
	Choices: Tabular Report, Linear plot, Logarithmic plot
	If calculation assignments are not used, any outlier points are included and marked in the analysis log re- port, but are not included in plots.
Plot Options	Choose which branch(es) of the isotherm you wish to plot.
	Choices: Plot adsorption, Plot desorption
Linear/Logarithmic Plot Options	Choose options for linear and logarithmic plots.
	Choices: Plot curve, Plot points, Overlay samples Autoscale x-axis, Autoscale y-axis
Overlay samples	Select this option to overlay this plot with data from other samples. The other samples are specified by selecting Overlays on the main Report Otions screen.

Autoscale x-axis Autoscale y-axis	Choose this option to have the x- and/or y-axes scaled automatically. The lowest and highest values collected during analysis are used as the beginning and ending points.
	Deselect the autoscale options to specify beginning and ending ranges; the corresponding From and To fields be- come enabled.
	The X-axis shows the relative pressure.
	The Y-axis shows the volume of gas adsorbed.
From/To fields	Specify beginning and ending values for the x- and y- axes ranges when you choose not to autoscale data. Data collected outside these ranges are not included in the plot.

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.

Click **Edit** for BET (or Langmuir) surface area report options; the BET (or Langmuir) Surface Area Report Options dialog box is displayed.

Displays as Langmuir	📰 BET Surface Area Re	epor	Options		×
Surface Area Report	✓ <u>I</u> abular report				
Options if the Langmuir report is being edited.	☑ <u>B</u> ET transform plot				
	<u>O</u> verlay samples		From	То	
	Autoscale <u>x</u> -axis	X:	0.000000000	1.000000000	P/Po
	🗹 Autoscale y-axis	Y:	0.000000	99.999999	g/cm ³ STP
	<u>■ B</u> ET Isotherm plot				
	Cverlay <u>s</u> amples		From	To	
	✓ <u>A</u> utoscale x-axis	X:	0.000000000	1.000000000	P/Po
	Autoscale y-axis	Y:	0.0000	9999.9999	cm³/g STP
	Pressures				
	Enter a value between 0.000	00000	00 and 1.00000000	0.	
	0 <u>K</u>			<u>C</u> ancel	



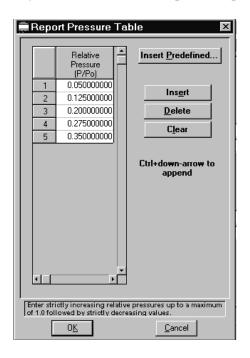
The Langmuir and BET Surface Area dialogs include the same fields; the operating instructions for both are the same.

Tabular report	Select this option to have a tabular report of the plotted data.
BET (or Langmuir) Transform plot	Generates a traditional BET (Langmuir) surface area plot that is used to determine monolayer volume and BET C constant.
BET (or Langmuir) Isotherm plot	Uses the BET (Langmuir) monolayer volume and con- stant 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.
	The X-Axis Range fields show the relative pressure.
	The Y-Axis Range fields show 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 begin- ning and ending ranges of the X- and/or Y-axis. Data col- lected outside these ranges are not included in the plot. The value entered in the To field must be greater than the value entered in the From field.

Pressures

Displays the Report Relative 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 dia- log, 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; the cursor moves to the new line.
Delete	Deletes the selected row.
Clear	Removes all but the one required entry from the table. The rows do not have to be selected. A warning message requesting confirmation is displayed before the table is cleared.

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 chacterizing complex mixed materials. Click **Edit** for t-Plot report options; the t-Plot Report Options dialog is displayed.

t-Plot Report Options		×	
Thickness Curve	Surface Area	Fitted thick <u>n</u> ess	
C H <u>a</u> lsey	⊙ <u>B</u> ET	range:	
Harkins-Jura	C Langmuir	0.35000 nm	
C Carbon Black STSA	C Entered	0.50000 nm	
C Broekhoff-de Boer	1.0000 m²/q		
C User-Defined		<u>S</u> urface area	
		correction factor:	
<u> </u>	Press <u>u</u> re Range	1.000	
✓ <u>T</u> abular report			
▼ t- <u>P</u> lot			
	From	To	
Autoscale <u>x</u> -axis X:			
		00.00000 nm	
Autoscale y-axis Y:	0.0000 100	000.0000 cm³/g STP	
Enter a value between 0.00000 and 99,90000.			
0 <u>K</u>		<u>C</u> ancel	

Thickness Curve

Presents the type of thickness curves available.

- Halsey
- Harkins-Jura
- Carbon Black STSA
- Broekhoff-de Boer
- User-Defined

You can also apply the Frenkel-Halsey-Hill thickness curve using the Halsey option, and 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.

Halsey

Displays the Halsey Thickness Equation dialog.

	Halsey Thickness Equation
t =	0 (-5.000) 0.333 In (P/Po)
Enter a value betw	en -1.000 and 99999.000.

You can edit the values for the multiplier, numerator, and exponent.

Refer to Appendix C, Calculations, for more information.

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

-	Harkins/Jura Thickness Equation
	t = (13.9900)
Er	ter a value between 0.0001 and 9999.0000.
	0 <u>K</u>

You can edit the values for the numerator, first element of the denominator, and exponent.

Refer to **Appendix C**, **Calculations**, for more information.

Carbon Black STSA	Displays the STSA Thickness Equation dialog.
	STSA Thickness Equation

-	STSA Thickness Equation		
	2.9800 + 6.4500 (P/Po) + 0.8800 (P/Po) ²		
	Enter a value between 0.0001 and 9999.9999.		
Ĭ.	0 <u>K</u>		

All coefficients can be edited.

🧱 Broekhoff-De Boer Thickn	ess Equation 🛛 🛛 🗙
log (P/Po) = 16.1100 t ² +	-0.1137 t 0.1682 e
Enter a value between -9999.9999	and -0.0001.
0 <u>K</u>	<u>C</u> ancel

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

You can edit the values for the multiplier, numerator, and exponent.

Refer to Appendix C, Calculations, for more information.

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

5	Ente	ered t-Curve		×
		Relative Pressure (P/Po)	Thickness (nm)	▲ Ins <u>e</u> rt
Ш	1	0.000000001	0.10000	Delete
Ш	2	0.00000002	0.20000	
Ш	3	0.00000003	0.30000	<u>Cl</u> ear
				Ctrl+down-arrow to append
				<u>O</u> pen Save <u>A</u> s
	Enter st	trictly increasing rel	ative pressure	s up to a maximum of 1.0
		0 <u>K</u>		<u>C</u> ancel

This table can also be created in another application if desired and imported into this dialog using <u>Open</u>. 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).

Open	Allows you to import the values from an existing thick- ness curve (THK) into this table. One predefined curve is shipped with the analysis program and is found in the Referenc directory. The Referenc directory is defaulted when you select this push button.
	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.
Clear	Clears the table of all but one entry; one entry is re- quired.
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 one of preference.
Pressure range	Displays the Report Relative Pressure Range dialog so that you may specify minimum and maximum relative pressures to use with this report.
	Report Relative Pressure Range
	Minimum Relative Pressure 0.010000000
	Maximum Relative Pressure 0.90000000

• 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..

Enter a value between 0.000000001 and 1.000000000.

• If **Use calculation assignments** is **selected**, collected data points which are assigned to this report type are used.

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



The values entered for the thicknesses can be expressed in angstroms or nanometers. Select *Data presentation* from the Options menu to specify desired units.

Surface area correction factor	This value corrects for surface areas that are not smooth and brings the values for BET surface area and micro- pore surface area into accordance. For most samples, the default value of 1.000 is adequate.
Tabular report	Select this option to have a tabular report generated.
t-Plot	Select this option to have a graphical representation of data.
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 begin- ning and ending ranges of the X- and/or Y-axis. Data col- lected outside these ranges are not included in the plot.
	The value entered in the To field must be greater than the value entered in the From field.
	The X-Axis Range fields show the relative pressureThe Y-Axis Range fields show the quantity of gas adsorbed.

Alpha-S Method	×
Relative Pressure (p/p [*]) Alpha-S 1 0.000000001 0.0001	Insert Fitted Alpha-S Range Delete 0.0000 Clear 1000.0000 Ctrl/down-arrow to append 1000.0000 Open Save As Belative pressure range when calculation assignments are not used 0.00000000 0.000000000 To 1.000000000 0.000000000 To 1.000000000
I abular report	
I I Alpha- <u>S</u> Plot	Το
□ □ <u>O</u> verlay samples	From
Autoscale <u>x</u> -axis X:	0.0000 1000.0000
✓ Autoscale y-axis Y:	0.0000 1000.0000 cm ³ /g STP
	0.0000 1000.0000 Gill 7g 311
0 <u>K</u>	Cancel

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).

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 and is located in the **Referenc** directory. The **Referenc** directory is defaulted when you click this push button. 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.
Clear	Clears the table of all entries.
Fitted alpha-S Range	Provides two fields for entering the minimum and maxi- mum relative pressures from which the fit will be deter- mined.
Relative pressure range	Provides two fields for entering a pressure range when calculation assignments are not used.
Tabular report	Choose this option to have a tabular report generated
Alpha-S 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 file(s). The other sample file(s1 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 begin- ning and ending ranges of the X- and/or Y-axis. Data col- lected outside these ranges are not included in the plot. The value entered in the To field must be greater than the value entered in the From field. • The X-Axis Range fields show the relative pressure.
	 The X-Axis Range fields show the relative pressure. The Y-Axis Range fields show the quantity of gas adsorbed.

f-Ratio Plot

f-Ratio Method		X
Reference isotherm:		
C:\\#IN2020\DATA\000-0	DO3.SMP	Browse
Relati <u>v</u> e pressure range v		ents are not used:
0.00000000 to 1.0	00000000 P/Po	
☑ <u>I</u> abular report		
☑ f- <u>P</u> lot		
C Overlay samples	From	То
🗖 Autoscale <u>x</u> -axis	X: 0.00000000 1.0	00000000 P/Po
🔽 Autoscale y-axis	Y: 0.0000	1000.0000
		Cancel

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 meas- ured 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 pres- sure, quantity adsorbed for the isotherm of interest, quan- tity adsorbed for the reference isotherm, and the ratio of the quantities adsorbed.
t-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 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 the axis, al- lowing you to enter a range.

Or BJH Desorption	BJH Adsorption Repo	ort Options
Report Options	Thickness Curve	Minimum BJH width:1.7000 nmMaximum BJH width:300.0000 nmFraction of pores open0.00at both ends:0.00
Not available on the Desorption dialog.	C User-Defined	Adsorptive Smooth differentials Selected Reports: Tabular Report Cumulative Pore Volume
	Pressure Range	dV/dw Pore Volume dV/dlog(w) Pore Volume Cumulative Pore Area dA/dw Pore Area dA/dlog(w) Pore Area
	0 <u>K</u>	Cancel

BJH Adsorption/Desorption Report Options



The BJH Adsorption and Desorption dialog boxes 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 both adsorption and desorption data.

Thickness CurveThe choices in this group box allow you to choose the
type of thickness curve you wish to use and change val-
ues of the equation, if desired.Choices:Halsey, Harkins-Jura, Carbon Black STSA,
Broekhoff de Boer, and User-DefinedYou must choose an equation for the type of thickness



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

curve to be used.

Edit	Displays the equation for the type of thickness curve se- lected. Refer to the previous section, t-Plot Report Op- tions, for an explanation of this push button.
Minimum BJH (diameter, radius, or width)	Enter the minimum diameter (or radius) of pores you wish to have included in the BJH reports. The value entered in this field must be <i>less</i> than the value entered in the Maximum BJH field.
Maximum BJH (diameter, radius, or width)	Enter the maximum diameter (or radius) of pores you wish to have included in the BJH reports. The value entered in this field must be <i>greater</i> than the value entered in the Minimum BJH field.
Fraction of pores open at both ends	During adsorption 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. In this field, you may enter the fraction of pores open at both ends to compensate for this error.
	• This field is not shown on the BJH Desorption Report Options dialog.

Displays the BJH Adsorptive Options dialog.

BJH Adsorptive Options			
	Adsorptive		Adsorbate Property Factor (nm)
1)	N2	Ŧ	0.95300
2)	AR	Ŧ	1.04400
3)		Ŧ	0.00000
4)		Ŧ	0.00000
5)		Ŧ	0.00000
6)		Ŧ	0.00000
7)		Ŧ	0.00000
8)		Ŧ	0.00000
 Enter a	value between 0.00000 and 10.00000.		
	OK		<u>C</u> ancel

The recommended adsorptives and their values are shown. You may specify up to six additional adsorptive/adsorbate property factor combinations.

Adsorptive

Adsorptive (continued)	The adsorbate property factor is the combination of con- stants in the numerator of the BJH equation which is spe- cific to the type of gas used. Adsorbate property factors may be specified in angstroms or nanometers. Select Options, Data presentation from the main menu to spec- ify units.
Smooth differentials	Smooths all differential calculations, eliminating vari- ations in the differential computation caused by noise in the input data.
Pressure range	<text><list-item></list-item></text>
Selected Reports	Contains a list of available BJH reports: • Tabular Report
	 Cumulative Pore volume dV/dW, dV/dD, or dV/dR Pore Volume dV/dlog(W), dV/dlog(D) ,or dV/dlog(R) Pore volume Cumulative Pore Area dA/dW, dA/dD,or dA/dR Pore Area dA/dlog(W), dA/dlog(D), or dA/dlog(R) Pore Area



You can measure pore width (w), pore radius (R), or pore diameter (D) for BJH reports. Select *Options*, *Data presentation* from the Main Menu to choose the desired measurement

Selected Reports (continued)	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. Reports are deselected in the same manner.
Edit	Allows you to edit the selected report. Editing options for available reports are shown in the following sections.

Tabular Report

Select **Edit** for the BJH Adsorption/Desorption tabular report to specify the method of data reduction; the BJH Adsorption (or Desorption) Tabular Report Options dialog box is displayed.

BJH Adsorption Tabular Report Options		
Tabular Data Defined by Fixed pore size table Collected points 	Co <u>l</u> umns T <u>a</u> ble	
OK	Cancel	

Fixed poreSpecify exact pore sizes for which volume or area datasize tableare reported. Only the pore sizes within the specifiedrange are reported. Select to enter or edit a fixed poresize table.

Collected points Includes all relative pressure points collected by the system.

Columns...

Displays the BJH Adsorption (or Desorption) Tabular Report Column Options dialog.

-	BJH Adsorption Tabular Report Column Options				
	Column 1:	Diameter Interval			
	Column 2:	Average Diameter			
	Column 3:	Incremental Pore Volume			
	Column 4:	Cumulative Pore Volume			
	Column 5:	Incremental Pore Area			
	Column 6:	Cumulative Pore Area			
$\ \Gamma$					
1					

Table...

Columns...The default column title appears next to the column num-
ber. Each column includes a pull-down list of the data
types available for inclusion in the report. You can meas-
ure pore width, pore radius, or pore diameter.

Enabled when you choose **Fixed pore size table**. Displays the BJH Adsorption (or Desorption) Fixed Pore Size Table dialog so that you may enter or edit a fixed pore size table.



The fixed pore size table must contain a minimum of two points and may include as many as 1000. The 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
 Clears all points from the pressure table; none have to be selected.

Click **Edit** for BJH Adsorption/Desorption plots to specify plotting methods and to customize plots. The following plots are available:

A dialog box similar to the following is displayed when you select a BJH plot.

	BJH Adsorption Cumulative Pore Volume Options 🛛
	Plot curve Plot points
	X-Axis
	C <u>L</u> inear © Logarithmic
	Autoscale 1.00 to 1000.00 nm
	Y-Axis
	Variable: Cumulative Pore Volume
	Overlay: None
	Auto <u>s</u> cale 0.0000 to 9999.0000 cm ³ /g
Malfal and an a disclose base	
Valid ranges display here.	Enter a value between 1.00 and 1000.00.
	0 <u>K</u>

Plot Options	Lists the data presentation styles available for BJH plots. You may choose to plot 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 loga- rithmic or linear scale.	
Autoscale	Select this option to have the x-axis scaled automatically. The lowest and highest values collected during analysis are used as the beginning and ending points.	
	When you deselect this option, the adjacent fields are en- abled so that you can specify beginning and ending values.	
	Data ranges are displayed in the information bar across the bottom of the dialog box when a cursor is in a nu- merical data field.	

Y-axis	Lists the options for the y-axis.
Variable	Choose the desired variable for the Y-axis.
Overlay	Displays a list of overlay choices. You may choose to overlay the current plot with data from other samples or with other plots from this sample.
Autoscale	Select this option if you wish to have the y-axis scaled automatically. The lowest and highest values collected during analysis are used as the beginning and ending points.
	When you deselect this option, the adjacent fields are en- abled so that you can specify beginning and ending values.
	Data ranges are displayed in the information bar across the bottom of the dialog box when the cursor is in a nu- merical data field

	🚍 Dubinin Report Options	x	
		Fitted relative pressure range: 0.000000 1.000000 Adsorpti <u>v</u> e	
		<u>C</u> ancel	
Report Type	erate. At least one type	the type of report you wish to gen- of report must be selected. generate the Radushkevich	
	report.	8	
	this choice, you may sel	erate the Astakhov report. With ect Optimize exponent . If you , the Exponent value field is en- tter a value.	
	<i>Refer to</i> Appendix C , C information.	alculations, for additional	
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 in the line fit.		
Selected Reports	double-clicking on the report name and press the	Lists the available Dubinin 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 (\checkmark).	
	The following reports ar	e available:	
	 Tabular Report Transformed Isotherm dV/dW Pore Volume (Astakhov only)	

Dubinin Report Options

Adsorptive

Pressure range

Allows you to specify up to ten adsorptive/affinity coefficient (beta) combinations. The Dubinin Adsorptive Options dialog is displayed.

🚅 Dubinin Adsorptive Options 🛛 🛛			
	Adsorptive	Affinity Coefficient (beta)	
1)	N2 🔹	0.33000	
2)	AR	0.26700	
3)	C02 •	0.46100	
4)	•	0.00000	
5)	·	0.00000	
6)		0.00000	
7)	•	0.00000	
8)	·	0.00000	
Enter a value between 0.00000 and 100.00000.			
0 <u>K</u>			

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.

Selecting **Tabular Report** from the Dubinin Report Options dialog box 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.

🚍 Dubinin Tabular Report Column Options 🛛 🛛 🛛			
C <u>o</u> lumn 1:	Absolute Pressure		
Co <u>l</u> umn 2:	Relative Pressure		
Col <u>u</u> mn 3:	Quantity Adsorbed		
Colu <u>m</u> n 4:	Log Quantity Adsorbed		
Column <u>5</u> :	Log (Po/P)^n		
Column <u>6</u> :	dV/dw Pore Volume		
	0 <u>K</u>		

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.

*The value for n (shown in Column 5) is the optimized exponent if **Optimized Astakhov exponent** is selected on the Dubinin Report Options dialog box. If not, then the value for n is the entered exponent value.

Transformed Isotherm Plot Options

Selecting Transformed Isotherm from the Dubinin Plot Option dialog enables you to restrict the line fit to a portion of the isotherm. The Dubinin Transformed Isotherm Plot Options dialog box is displayed.

Ę	Dubinin Transformed Isotherm Plot Options			
	<u> □</u> verlay samples			
	Autoscale <u>x</u> -axis 0.000000 to 1000.000000 Log (Po/P)			
I	Autoscale <u>v</u> -axis 0.00000 to 3.00000 Log (Q)			
Enter a value between 0.000000 and 99999.000000.				
	0 <u>K</u>			

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-axisSelect these options to have the X- and/or Y-axes auto-
matically scaled. Both anes begin at zero; the system
uses the highest values collected during analysis as the
ending points for anes ranges.

If you choose not to autoscale data for either (or both) axis, the corresponding fields are enabled so that you may enter a beginning and ending value.

- The X-axis shows the quantity of gas adsorbed at standard temperature and pressure.
- The Y-axis shows 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. When you select dV/dW Pore Volume from the Dubinin Report Options dialog box, the Dubinin dV/dw Pore Volume Options dialog is displayed.

🚍 Dubinin dV/dw Pore Volume Options 🛛 🛛 🛛				
✓ Plot curve	☑ Plot points	□ <u>O</u> verlay sample	s	
Autoscale <u>x</u> -axis	0.00 to	2.00 ni	n	
Autoscale y-axis	0.000 to	500.000 cr	n³/g∙nm	
Enter a value between 0.00 and 1000.00.				
0 <u>K</u>		<u>C</u> ancel		

Plot options	Choose to plot 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 auto- matically scaled. Both axes begin at zero; the system uses the highest values collected during analysis as the ending points.
	If you choose not to autoscale data for either (or both) axis, 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.
	Data ranges are displaye din the information bar across the bottom of the dialog when the cursor is in a numeri- cal data entry field.

MP-Method Report Options

MP-Method Report Options			
Thickness Curve Harkins-Jura Halsey Equation Pressure Range	<u>Selected Reports:</u> Tabular Report Cumulative Pore Volume dV/dw Pore Volume Cumulative Pore Area dA/dw Pore Area	<u>E</u> dit	
0 <u>K</u>	<u>C</u> ancel		



Pore size can be expressed in Angstroms or nanometers. Select *Data Presentation, Units* from the Options menu to secify desired unit.

Thickness CurveSelect the thickness curve type from this group box. You
must choose either the Halsey equation or the Harkins-
Jura equation. Use Equation to edit the values in the
equation.

EquationDisplays the equation for the type of thickness curve
selected.

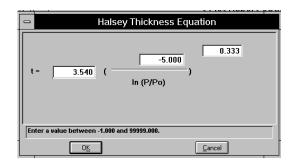
Harkins-Jura Displays the Harkins-Jura Thickness Equation dialog box.

Harkins/Jura Thickness Equation		
t = (13.9900 0.500 t = (0.034 <u>0</u> - log (P/Po))		
Enter a value between 0.0001 and 9999.0000.		
0 <u>K</u>		

You can edit the values for the numerator, first element of the denominator, and exponent.

Halsey

Displays the Halsey Thickness Equation dialog box.



You can edit the values for the multiplier, numerator, and exponent.

Refer to Appendix C, Calculations, for more information

Selected ReportsLists the available MP-Method reports. Choose a report
by double-clicking on the report name or highlight the re-
port name and press the spacebar. A report is selected
when it is preceded with a check mark (\checkmark).

The following reports are available:

- Tabular Report
- Cumulative Pore Volume
- dV/dW Pore Volume
- Cumulative Pore Area
- dA/dW Pore Area

The data for these reports are measured in pore width only.

Tabular Report Options

Selecting **Tabular Report** from the MP-Method Report Options dialog box 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.

🚍 MP-Method Tabular Report Column Options 🛛 🛛 🛛			
Column 1:	Pore Hydraulic Radius Interval		
Column 2:	ımn 2: Average Pore Hydraulic Radius		
Col <u>u</u> mn 3:	Incremental Pore Volume		
Colu <u>m</u> n 4:	Cumulative Pore Volume		
Column <u>5</u> :	dV/dw Pore Volume		
Column <u>6</u> :	2: Incremental Pore Area		
I			
	0 <u>K</u>		

The default column title appears next to the column number for columns three through six. Each of these columns includes a pull-down list of the data types to include in the report. The following data types are available:

Columns 1 and 2 are fixed and cannot be edited.



The MP-Method reports hydraulic radius only. If you select *Pore size in diameter* from the Data presentation menu, the MP-Method still reports pore size in radius.

Plot Options

Selecting an MP-Method plot option from the MP-Method Report Options dialog boxes enables you to specify the plotting method used for your report and to customize the plot. The following plots are available:

- Cumulative Pore Volume
- dV/dw Pore Volume
- Cumulative Pore Area
- dA/dw Pore Area

When you select any of these plots from the MP-Method Report Options dialog box, a dialog box similar to the following one is displayed. The fields on all dialog boxes are the same.

🚍 MP-Method Cumulative Pore Volume Options 🛛 🛛 🛛			
Plot curve Plot points			
X-Axis			
Y-Axis			
Yariable: Cumulative Pore Volume ▼			
Overlay: None			
✓ Auto <u>s</u> cale 0.0000 to 9999.0000 cm³/g			
Enter a value between 0.00 and 1000.00.			
0 <u>K</u>			

Plot options Choose to plot a curve, points, or both.

X-Axis Choose Autoscale x-axis to have the x-axis scaled automatically. The X-axis begins at zero and the system uses the highest values 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.

Data ranges are displaye din the information bar across the bottom of the dialog when the cursor is in a numerical data entry field.

Y-Axis		
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. Click Overlays on the Reports Options dialog box to choose the samples.	
Autoscale	Choose this option to have the y-axis scaled automat- ically. Y-axes begin at zero. The system uses the highest values collected during analysis as the ending points for axes ranges.	
	If you deselect this option, the corresponding fields are enabled so that you may enter a beginning and ending value. Data collected outside these ranges are not in- cluded in the plot.	
	Y-axis range fields show the volume of gas adsorbed.	
	Data ranges are displaye din the information bar across the bottom of the dialog when the cursor is in a numeri- cal data entry field.	

Summary Report

Summary Report Options			
Select All		Deselect All	
Surface Area ✓ Single-point <u>B</u> ET Multi-point BET Langmuir t-Plot micropore t-Plot external BJH cum. adsorption BJH cum. desorption	Pore Volume ✓ Adsorption total P/Po: 0.010000000 ✓ Desorption total P/Po: 0.900000000 □ t-Plot micropore □ BJH cum. adsorption □ BJH cum. desorption	Pore Size ✓ Average pore width ☐ BJH adsorption avg. pore diameter (4V/A) ☐ BJH desorption avg. pore diameter (4V/A)	
Micropore Reports			
	Microp	adushkevich ore surface ar <u>e</u> a iyer capacity	
Dubinin-Astakhov Micropore surface area Limiting micropore volum	ne Cumula	d Itive surface area Itive pore volume Je pore hydraulic radius	
0 <u>K</u>		<u>C</u> ancel	

The summary report provides a condensed listing of data results. Click **Edit** for Summary report options; the Summary Report Options dialog box is displayed.

The choices on this dialog box allow you to choose the types of data to include in the Summary report.

If you choose **Adsorption** or **Desorption total** for Pore Volume data the P/Po field is enabled so that you can enter the relative pressure at which to calculate the total pore volume. If **Use calculation assignments** (Collected Data screen) is not selected, the isotherm is interpolated to this value and that point used for the Total pore volume calculation. Otherwise, the point selected with calculation assignment is used.

Select all Selects all choices on the dialog box.

Deselect all Deselects all choices on the dialog box..

Options Report

The Options report is a subset of the contents report; it provides pertinent information for the following:

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

Collected/Entered Data

C]C:\V	VIN3000\C	ATA\CARB	ONG5.SMF	`					_ 🗆 ×
	<u><</u> < Pre	v Samp Inform	ile Sample nation Tube	Degas Conditions	Analys Condit		dsorptive operties	Report Options	Collected Data	Next ≥>
		Absolute Pressure (mmHg)	Relative Pressure (P/Po)	Quantity Adsorbed (cm³/g STP)	BET Surf. Area	Lang. Surf. Area	t-Plot	Alpha-S f-Ra	atio	
L	1	37.02406	0.049553765	1.9422	X					
L	2	54.39442	0.072802607	2.0887	X					
L	3	81.50283	0.109085058	2.2656	X					1
L	4	93.13522	0.124654091	2.3317	X					Insert
L	5	111.55423	0.149306466	2.4304	X					Delete
L	6	130.32841	0.174434220	2.5259	X				- -	Denete
L	7	149.14490	0.199618586	2.6191	X					Clear
L	8	167.99944	0.224853886	2.7116	X					
L	9	186.91739	0.250174062	2.8043	X					
L	10	205.88187	0.275556508	2.8980	X					
L	11	224.88440	0.300989887	2.9930	X					
1	•									
	₩ <u>U</u> se	e calculation	n assignments		E	o and 1	r	Fr <u>e</u> e Spac	:e	
Ì										
		<u>S</u> ave	•		<u>C</u> lo:	se			B <u>a</u> sic	

This dialog is displayed only in the Advanced mode of the sample file presentation, and only after an analysis has been completed.

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

Enabled only for entered data. Inserts a row into the pressure table.

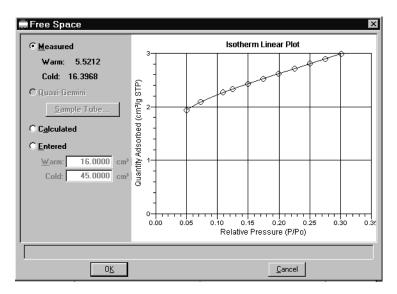
Insert

Delete	Enabled only for entered data. Deletes the selected row.
Clear	Enabled only for entered data. Clears the entire table; rows do not have to be selected.
Use calculation assignments	If selected, allows you to assign the points to be col- lected for each report type.
	If deselected, each report (with the exception of Lang- muir and BET) uses a range of pressures as selected in the report options. The Langmuir and BET reports inter- polate to entered relative pressures on the report options.
	The outlier points can be selected so that they will not be reported.
Po and T	Displays the Po and T dialog.
	Po and Temperature Options
	Choose one option.
	① 1. Measure Po at intervals during analysis. Enter the Analysis Bath Temperature below.
	C 2. Enter Po below. Enter the Analysis Bath Temperature below.
	O 3, Measure Po over the sample. Enter the analysis bath temperature and an initial estimate for Po below.
	C 4, Measure Po continuously in port 3. Enter the analysis bath temperature below.
	Temperature: 77.300 K
	Enter a value between 0.000 and 999.000.
	0 <u>K</u>

This dialog box allows you to edit the Po measurement and analysis bath temperature.

Free Space...

Displays the Free Space dialog, allowing you to edit free-space values.



This dialog also shows the isotherm for the analysis. The isotherm is redrawn each time values are edited.

Measure Quasi-Gemini	Either of these choices is enabled only if selected as the method used in the original analysis.	
	If Quasi-Gemini was selected, Sample Tube is enabled so that you may edit the parameters of the sample tube used as the balance in the Quasi-Gemini method.	
Calculated	Select this option to have the free-space measurement cal- culated using the sample conditions and sample tube pa- rameters.	
Enter	Enables the Warm free space and Cold free space fields so that you may enter the free space manually.	
	The warm free space is the sample tube gas capacity measured at room temperature.	
	Range: 1.0000 to 999.0000 cm ³ Default: 16.000 cm ³	
	The <i>cold free space</i> is the sample tube gas capacity measured with the Dewar raised.	
	Range: 1.0000 to 999.0000 cm ³ Default: 45.0000 cm ³	

Save

Save enables you to save any changes you have made to the file in the active window. The file is saved under its current name.

Save As

Save As enables you to:

- save a sample or parameter file in the active window under a different name. This option is useful for making a duplicate copy of a file that you can modify as desired without changing the original one. The original file remains open when you use this function, so be sure to open the new file before making any changes.
- 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.
- save as an ASCII file the relative pressures and corresponding thicknesses. 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 adsoptive.
- save as an ASCII file the relative pressures and resulting quantities adsorbed. These data are derived by taking the isotherm and dividing it by the quantity adsorbed at 0.4 relative pressure.

Save as Sample Information File	x
File <u>n</u> ame: ESMP	
Selection Criteria	
Status: Complete	
Dates	
Files:	Dir <u>e</u> ctories: c:\win3000\data
Carbond4.smp Carbon D-4 reference material carbong5.smp Carbon G-5 reference material sialref.smp Silica Alumina A-501-38 Adsorption Desorpti	[] [-a-] [-b-]
	[-c-] [-d-] [-e-]
	[-f-] [-g-] [-h-]
	[-q-] [-w-] [-z-]
0 <u>K</u> Cancel	

A dialog similar to the one shown below is displayed when you select the **Save** As command.

Enter a file name (up to eight characters) in the **File name** field; the appropriate extension is appended automatically when you click $\bigcirc \kappa$. The new file is saved as specified, but does not remain in the active window. Be sure to open the new file before making any changes to the file.

When saving Alpha-S files, the Save As t-Curve dialog box is shown first, allowing you to select the type of curve.

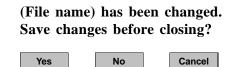
Save As t-Curve	. 🗙
Surface Area	
⊙ <u>B</u> ET	9.3343 m²/g
C <u>L</u> angmuir	1.0000 m²/g
C <u>E</u> ntered	1.0000 m²/g
0 <u>K</u>	<u>C</u> ancel

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.

Close

Close enables you to close the file in the active window. The following message is displayed if changes have been made to the file:

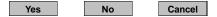


- Yes saves any changes and closes the active window.
- No discards any changes and closes the active window;.
- Cancel returns you to the open file.

Close All

Close All enables you to close *all* open files under their current names. The following message is displayed for each file in which changes have been made:

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



- Yes saves any changes and closes the active window.
- No discards any changes and closes the active window.
- Cancel returns you to the open file.

Print

Print enables you to print the entire contents of a file. When you select **File**, **Print** from the main menu, the following choices are presented:

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

Regardless of which file type you select, a dialog box similar to the one shown below is displayed.

	Print Sampl	e Information File
File name:	0121.SMP Cor	nplete
Selection C	Criteria	Settings
Status	All 🛓	Copies 1 Destination Screen File name G:\USERS\S&M\FRANKIE
Files: 0121.smp	ALUMINA REFERENCE	Directories: g:\\tristar\data
0136.smp 0136.smp 0140.smp 0141.smp 0176.smp 0177.smp 0196.smp 0200.smp 0200.smp 0309.smp n310.smp		MAL 2818 12M [-a-] MAL 2818 13L [-b-] MAL 2818 14H [-c-] i826/00 12L [-d-] i826/00 13L [-d-] i826/00 14H [-f-] i826/00 12M [-d-] i826/00 12M [-g-] i826/00 13L [-h-] i826/00 13L [-h-] i826/00 14H [-q-] wder [-w-] [-w-]
	0 <u>K</u>	Cancel

File name	The name of the file you select from the Files: list box is copied to this field.
Dates Files Directories	This push button and the contents of these list boxes are explained in Selecting Files in Chapter 3.
Destination:	Contains a drop-down list of the available print destina- tions.
	If you select <i>Screen</i> , <i>Printer</i> , or <i>Printer/Plotter</i> , the re- quested file is sent to that specified destination.

Destination: (<i>continued</i>)	If you select <i>File</i> , the requested file is converted to a text file which can be viewed with a text editor or other text file manipulation tools. You also must enter a name in the File name field of the Settings group box (or accept the default).
Settings, File name	Enabled only if you select <i>File</i> as the destination. A default name is displayed in the field, but you may enter a new one if you wish.

List

List enables you to generate a listing of the following information on a selected sample or parameter file.

- File name
- Date the file was created
- Time the file was created
- File identification
- File status

The List dialog is common to all file types. The title bar displays the type of file on which you have requested a list.

_	List Sample	e Information File
File name:	0121.SMP Co	mplete
Selection (Criteria	Settings
Status	All	Copies 1 Destination Screen
	Dates	File name G:\USERS\S&M\FRANKIE
Files:		Directories: g:\\tristar\data
0121.smp	ALUMINA REFERENCE	
0136.smp		MAL 2818 12M [-a-]
0140.smp	13 X REF 004-16826-00	
0141.smp	13 X REF 004-16826-00	
0176.smp 0177.smp	13X REFERENCE 004/10 13X REFERENCE 004/10	
0177.smp	13X REFERENCE 004/10	
0196.smp	13X REFERENCE 004/10	
0200.smp	13X REFERENCE 004/10	
0201.smp	13X REFERENCE 004/10	
0309.smp	13 X Molecular Sieve Po	
0310 smn	13 X Molecular Sieve Pr	
+		*
	0 <u>K</u>	Cancel

File name

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



You may request a list of multiple files by holding down <u>Ctrl</u> while selecting files. If no files are selected, a list is generated for *all* files.

Dates	
Files	

Files Directories This push button and the contents of these list boxes are explained in **Selecting Files** in Chapter 3.

Destination	Contains a drop-down list of the available print destina- tions.
	Choices: Screen, Printer, Printer/Plotter, File
	If you select <i>File</i> , enter a name in the File name field of the Settings group box.
Settings, File name	Enabled only if you select <i>File</i> as the destination. Enter a name in the File name field.

Export

Export allows you to copy the isotherm data in a sample information file and reformat it in an ASCII format acceptable to other programs, such as spreadsheets.

The output file consists of four columns containing the absolute pressure, relative pressure, specific quantity absorbed, and the elapsed time.



You may select multiple files by holding down Ctrl and clicking the left mouse button once on each file.

Select File, Export from the main menu; the Export Sample File dialog box is displayed.

Export Sample File 🛛 🛛 🛛			
File name: CARBOND4.SMP Cor	nplete		
Selection Criteria	Settings		
Status: Complete	Export Type Isotherm		
	C <u>o</u> pies 1 📉		
Dates	Destination Screen		
	File name C:\WIN3000\DATA\CA		
	Dir <u>e</u> ctories:		
Files:	c:\win3000\data		
carbond4.smp Carbon D-4 reference carbong5.smp Carbon G-5 reference			
sialref.smp Silica Alumina A-501-38	3 Adsorption Desorpti		
	[[-c-] [[-d-]		
	[-e-]		
	[[-t-] [-a-]		
	[-h-]		
	[-q-]		
x	[-q-] [-₩-]		
	[-q-] [- w -]		

File name	The name of the file you select from the Files list box is copied to this field.
Dates Files	This push button and the contents of these list boxes are explained in Selecting Files in Chapter 3.

Files Directories

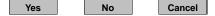
Settings Export Type	Fixed at Isotherm.				
Destination	Contains a drop-down list of the available print destina- tions.				
	Choices: Screen, Printer, Printer/Plotter, File				
	If you select File, the File name field is enabled.				
File name	Allows you to specify a name for your exported file, or you may accept the default.				

Exit

Exit enables you to exit the TriStar analysis program. Select **File**, **Exit** from the main menu. Even if an analysis is in progress, it will continue until completion; analysis data are collected and stored in an embedded controller.

• If a window containing a modified file is open, the following message is displayed:

Modifications have been made to (file name). do you want to save the modifications?



Yes saves any changes and exits the analysis program; **No** abandons any changes and exits the analysis program; and **Cance**l returns you to the open file.

• If an analysis is in progress, the following message is displayed:

CAUTION: Sample analysis is in progress. If you exit the program analysis will continue to completion, but the data are not saved to disk until the program is restarted. Do you want to exit the program?



Yes exits the analysis program; **No** leaves the analysis program active and allows the analysis to finish



Although data are stored in an embedded controller when you exit the program, data are not saved to disk in the control module until you reenter the program and save the data. If a power failure occurs and the analyzer does not have an Uninterruptible Power Supply (UPS), any unsaved data are lost.

• If a report is in progress, the following message is displayed:

CAUTION: Reports are being generated. If you exit the program these reports will be canceled. Do you want to cancel reports and exit the program?



Yes cancels the reports and exits the analysis program; **No** leaves the analysis program active and allows the reports to finish.

CHAPTER 6

UNIT MENU

- Description
- Sample Analysis
- QuickStart
- Enable Manual Control
- Show Instrument Schematic
- Show Status
- Show Instrument Log
- Unit configuration
- Calibration

UNIT MENU

The Unit menu contains the options for the operations which can be performed with the TriStar 3000. The main menu will contain a Unit menu for each attached analyzer. For example, if you have two attached analyzers, the main menu contains two Unit menus. For easy differentiation, the status displays associated with each unit are displayed in different colors. The unit number and the serial number also are displayed in the title bar of the operational windows. This is especially convenient if you have more than one analyzer attached to the same control module.



This menu does not appear on the menu bar if the analysis program is being used for offline data operations on a computer other than the one controlling the analyzer.

Description

Unit <u>1</u>
<u>S</u> ample analysis
QuickStart analysis
<u>E</u> nable manual control
Show instrument schematic
Sho <u>w</u> status
Show instrument <u>l</u> og
Unit configuration
<u>C</u> alibration
Service <u>T</u> est

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

Sample Analysis	Allows you to perform up to three analyses using differ- ent analysis conditions.
QuickStart	Allows you to perform multiple sample analyses using the same analysis conditions, allowing you to create sam- ple files at the time of analysis.
Enable Manual Control	Allows you to control the system manually.

Show Instrument Schematic	Displays the analyzer's schematic.
Show Status	Displays the status window.
Show Instrument Log	Displays a log of recent analyses, calibrations, and error messages.
Unit Configuration	Displays software version, hardware configuration, and calibration information. Also allows you to enter mne-monic names for port gases.
Calibrate	Allows you to calibrate the manifold temperature, the pressure scale, pressure offset, manifold volume, and servo valve. It also allows you to save calibration files as well as load previously saved files.
Service Test	Enables you to perform certain troubleshooting proce- dures. This option is enabled only with the direction of a Micromeritics service representative.

Sample Analysis

Use this mode of operation to schedule analyses which have different analysis conditions or report options. You may schedule up to three analyses at a time, depending on the number of ports you have available. All three ports are available for sample analyses if you are not using:

- port 2 for a Quasi-Gemini measurement
- port 3 for a continuous Po measurement

You can continue to schedule analyses by clicking Next>> (appears after the first series of analyses are finished) on the analyzing view of the Analysis dialog.

Select **Sample Analysis** from the **Unit** menu to display this view of the Analysis dialog box.

🗏 Analysis (U	nit 1 - S/N: 102)				
<u>⊻</u> iew: 01	peration				
Port 1	C:\WIN3000\000)-014.SMP			
Sample:	000-014			Browse	Clear
Mass:	1.0000 g	Density:	1.000 g/cm ³		
Port 2	C:\WIN3000\000)-019.SMP			
Sample:	000-019			Browse	Clear
Mass:	1.0000 g	Density:	1.000 g/cm ³		
Port 3					
Mass:	1.0000 g	Density:	1.000 g/cm³	Browse	Clear
Po: 760.00	0 mmHg	Bath temperature: 🗌	77.350 K	Report Aft	er analysis
	<u>S</u> tart			Cance	el <u>Cl</u> ose
Port Sample 1 2 3	Status Idle Idle Idle				
3	Tule				

View

Allows you to choose the view you wish to display in the window.

Choices: Operation, Instrument Schematic, Instrument Log

Operation displays the current mode of operation.

Instrument Schematic displays a schematic of the TriStar system. Refer to **Show Instrument Schematic** later in this chapter for additional information.

Instrument Log displays a list of recent analyses, calibrations, and errors or messages. Refer to **Show Instrument Log** later in this chapter for additional information.

Port [n]	This field is blank until you choose a file to use for the operation on this port.				
	• If port 2 is being used for a Quasi-Gemini free-space measurement, a message stating so is displayed.				
	• If port 3 is being used to measure the Po, a message stating so is displayed.				
Sample	Displays an identification of the sample file.				
Mass	Enter a value for the sample mass.				
	This field is not shown for ports 2 and 3 when:				
	Port 2 is being used for a Quasi-Gemini measurement.Port 3 is being used for a Po measurement.				
Density	Displays the density of the sample specified in the sample information file.				
	This field is not shown for ports 2 and 3 when:				
	Port 2 is being used for a Quasi-Gemini measurement.Port 3 is being used for a Po measurement.				
Browse	Click to:				
	• Select a sample information file when using the port for an analysis; the Open Sample Information dialog is displayed.				
	• Select a sample tube file when using the port for a Quasi-Gemini measurement; the Open Sample Tube dialog is displayed.				
	The file name you select is copied to the Port [n] field.				
Clear	Clears the information displayed for this port.				
Ро	Enabled if you have selected <i>Entered</i> for the Po measurement for at least one file. Enter the Po value, or edit the current one.				
Bath temperature	Enter the temperature for the analysis bath.				

Report after analysis ...

Allows you to have a report generated automatically after the analysis; displays the Report Settings dialog so that you may specify output options.

	Report Settings				
X Report After Analysis					
Number of Reports	1				
Destination	Printer 🛓				
File name C:\WIN30	J00\DATA*.RPT				
0 <u>K</u>	Cancel				
Enter a value between 1 and 4.					

Start

Starts the analysis; displays an analyzing view of the Analysis dialog box.

	🖻 Analysis (Unit 1 - S/N: 104)			
	<u>V</u> iew Operatio	n	S <u>k</u> ip	Suspend Resume
	Report Port 1	Report Port 2	Report Port 3	Close Valves
	A 37.0 000-014.5 000-014.5 55 35.0 000-016.5 33.0 33.0 33.0 92 32.0 33.0 92 32.0 30.0 000 30.0 -0.1	MP //	0.5 0.7 elative Pressure (P/Po)	0.9 1.1
Port Activity	Port 2: Equil port for	59 seconds. Int.=3 seconds, To 85 seconds. Int.=3 seconds, To 111 seconds. Int.=3 seconds, T	lerance=115.7 mmHg, Rate of	Change=0.039%.
Status Window	Port Sample 1 000-014.SMP 2 000-015.SMP 3 000-016.SMP	Next >> Last Point P(mmHg) 4 of 6 89.77 4 of 6 90.63 4 of 6 91.62	P/Po V(cm³/g) Last 0.12083 35.49 742. 0.12199 36.46 742. 0.12332 34.99 742.	98

This view also displays port activity as well as the status window.

Skip Skips an analysis; visible only when an analysis is in progress. The Skip dialog is displayed so that you may choose the port(s) containing the analysis you wish to skip. Skip (Unit 1 - S/N: 102) х Select the ports □ Port 1 E Port 2 Port 3 OK Cancel Suspend Suspends an analysis; visible only when an analysis is in progress. A dialog similar to the one shown above is displayed so that you may choose the port(s) containing the analysis you wish to suspend. Resume Resumes a suspended analysis; visible only when an analysis is suspended. A dialog similar to the one shown above is displayed so that you may choose the port(s) containing the analysis you wish to resume. Report Port [n] These push buttons allow you to generate reports on data collected at that time for the sample in the respective port. The reports are printed to the screen only. Close Valves Enabled when analysis is complete, allowing you to close all sample port valves immediately; otherwise the valves close in approximately five minutes. Cancel Cancels an analysis; enabled only when an analysis is in progress. A dialog similar to the one shown above is displayed so that you may choose the port(s) containing the analysis you wish to cancel. Start On this view of the Analysis dialog box, this push button changes to Next>> when analyses are finished. Next>> Returns you to the first view of the Analysis dialog box so that you may schedule additional analyses. Close Closes the Analysis dialog.

QuickStart

Use this mode of operation to analyze samples of the same type using the same analysis conditions, adsorptive properties, and report options. You may schedule up to three analyses at a time, depending on the number of ports you have available. All three ports are available if you are not using:

- port 2 for a Quasi-Gemini measurement
- port 3 for a continuous Po measurement

You can continue to schedule analyses by clicking Next>> (appears after the first series of analyses are finished) on the analyzing view of the Analysis dialog.

When you select this option from the Unit menu, the QuickStart dialog box is displayed.

⊐ ⊻iew	QuickStart (Unit 1 - S/N: test)
	Sample Options Analysis Conditions: MICRO/BJH W/N2 @ 77K & Adsorptive Properties: Nitrogen @ 77.35 K & Report Options: BJH Desorption Report Options &
	Report After Analysis Number of Reports 1 Destination Printer File name G.\USERS\S&MJFRANKIEP\TRANSFER\T
Port Sample 1 2 3	Next >> Close Close Close

View

Click the down arrow to choose the view you wish to have displayed in the window.

Choices: Operation, Instrument Schematic, Instrument Log

Operation displays the current mode of operation.

Instrument Schematic displays a schematic of the TriStar system. Refer to **Show Instrument Schematic** later in this chapter for additional information.

Instrument Log displays a list of recent analyses, calibrations, and errors or messages. Refer to **Show Instrument Log** later in this chapter for additional information.

Sample Options	This group box provides drop-down lists of previously de- fined files (including any you may have created) for analysis conditions, adsorptive properties, and report op- tions. Choose a file from each list to be used in the sam- ple files for this series of analyses.
Report Options	This group box allows you to have a report generated after each analysis. You may also specify the number of reports you want and a destination. If you choose <i>File</i> as a destination, the File name field is enabled. You can accept the default name or enter a new one.
Next>>	Advances you to the second view of the QuickStart dia- log.

The second view of the QuickStart dialog allows you to specify sample information for this set of samples.

Analysis (Unit 1 - S/N: 102)		D ×
View: Operation Close Valves		
Port 1		
File name: C:\WIN3000\000-012.SMP		
Sample: 000-012	Browse Clear	
Sample tube:		
Mass: 1.0000 g Density: 1.000 g/cm ³		
Port 2		
File name: C:\\WIN3000\000-014.SMP		
Sample: 000-014	Browse Clear	
Sample tube: Sample Tube		
Mass: 1.0000 g Density: 1.000 g/cm ³		
Port 3		
File name:		
	Browse Clear	
Sample tube:		
Mass: 1.0000 g Density: 1.000 g/cm ²		
1.000 g Density. 1.000 g/cm ²		
Po: 760.000 mmHg Bath temperature: 77.300 K		
Start	<u>C</u> ancel Close	

Port [n]

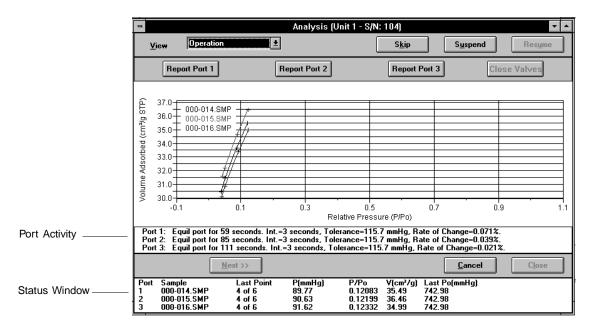
This field is blank until you choose a file to use for the operation on this port.

- If port 2 is being used for a Quasi-Gemini free-space measurement, a message stating so is displayed. No other fields are available for this port.
- If port 3 is being used to measure the Po, a message stating so is displayed. The **Sample Tube** field and **Browse...** are shown so that you can select a file.

Sample	Displays the identification of the sample file.
Sample Tube	Select the sample tube file you wish to use with this analysis.
Mass	Enter a value for the sample mass.
	This field is not shown for ports 2 and 3 when:
	Port 2 is being used for a Quasi-Gemini measurement.Port 3 is being used for a Po measurement.
Density	Displays the density of the sample specified in the Analy- sis conditions file.
	This field is not shown for ports 2 and 3 when:
	Port 2 is being used for a Quasi-Gemini measurement.Port 3 is being used for a Po measurement.
Browse	Click to display:
	 The Open Sample Information File dialog (if using the port for an analysis), allowing you to create a file for the analysis on the current port. The analysis conditions, adsorptive properties, and report options selected on the previous screen are used.
	• The Open Sample Information File dialog (if using the port for an analysis), allowing you to create a file for the analysis on the current port. The analysis conditions, adsorptive properties, and report options selected
Clear	 The Open Sample Information File dialog (if using the port for an analysis), allowing you to create a file for the analysis on the current port. The analysis conditions, adsorptive properties, and report options selected on the previous screen are used. The Open Sample Tube dialog (if using the port for a Quasi-Gemini measurement), allowing you to select a
Clear	 The Open Sample Information File dialog (if using the port for an analysis), allowing you to create a file for the analysis on the current port. The analysis conditions, adsorptive properties, and report options selected on the previous screen are used. The Open Sample Tube dialog (if using the port for a Quasi-Gemini measurement), allowing you to select a sample tube file.

Start

Starts the analysis; displays an analyzing view of the Analysis dialog box.

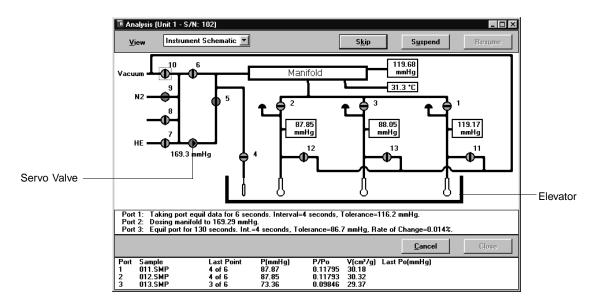


This view contains data collection for each analysis, as well as port information. Refer to the previous section for an explanation of the push buttons contained on this dialog.

When analyses are complete, the second view of the dialog again is displayed, allowing you to quickly start the next set of samples.

Enable Manual Control

Select this option to control certain components of your system manually. The instrument schematic must be visible to use this option. If it is not visible, select **Show Instrument Schematic.**



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 to move to the desired component and click the left mouse button once to select it.

Each component that can be actuated has a shortcut menu displaying the operations available for that particular component. These menus may be accessed by selecting the desired component, then clicking the right mouse button once, or pressing \uparrow Shift [F9].

The options available for the components on the instrument schematic are:

Valves 1 through 13 Open, Close, Pulse

Open and *Close* open and close the valves. You may also open and close the valves by double-clicking on the valve or by pressing **Spacebar** while the valve is selected.

Valves 1 through 13Pulse quickly turns the valve on and off, allowing the op-
eration to proceed in small increments. You may also
pulse the valve by pressing P while the valve is se-
lected.

Servo Valve Set, Close

Set displays the Servo Valve Settings dialog so that you may choose to dose or evacuate and specify a target pressure. You may also display this dialog by pressing **Spacebar** while the servo valve is selected.

Servo Valve Settings
Direction
• Dose
O Evacuate
Target Pressure 760.0 mmHg
OK Cancel

Close closes the servo valve. You may also close the servo valve by pressing **Spacebar** while the servo valve is selected.

Elevator

Raise, Lower, Stop

Raise and *Lower* raise and lower the elevator. You may also raise and lower the elevator by double-clicking in the elevator area or pressing **Spacebar** while the elevator area is selected.

Stop stops the elevator at its current position. You may also stop the elevator by double-clicking in the elevator area or pressing **Spacebar** while the elevator area is selected.

Show Instrument Schematic

Select this option to display a schematic of the analyzer. Refer to the previous section for an explanation of the components contained on the instrument schematic.

Even if manual control is not enabled, you still can determine the state of the valves by their color.

- Green = open
- White = closed

If you wish to change the state of a valve, you must enable manual control.

Show Status

Select this option to show only the status window of the analyzer.



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

🚏 Sta	atus (Unit 1 - S7	'N: 102)				_ 🗆 ×
Port 1 2 3	Sample 011.SMP 012.SMP 013.SMP	Last Point 2 of 6 2 of 6 2 of 6 2 of 6	P(mmHg) 60.19 60.32 60.44	P/Po 0.08079 0.08097 0.08112	V(cm³/g) 28.27 28.42 28.45	Last Po(mmHg)

The status window shows the current status for each port.

You may wish to use this option if:

- You have an automatic operation in progress and you wish to edit a sample file. This way, you can keep up with the status of your operation while performing another task.
- You have multiple units attached to your control module. You can select **Show Status** on each unit menu and have the status for all units displayed at one time.

Show Instrument Log

Displays a log of recent analyses, calibrations, and errors or messages. By default, this information is logged for a 7-day period for analyses and a 30-day period for messages and calibrations. You may change the time for which this information is retained in the Unit section of the WIN3000.INI file.

	la estre	ward less (light 1 - Cible tes	~	
	Instr	ument Log (Unit 1 - S/N: tes		▼ ↑
⊠ <u>A</u> nalysis		⊠ <u>C</u> alibration	⊠ <u>M</u> essage	
03/18/98 16:02:59 03/18/98 16:01:34 03/18/98 15:47:28 03/18/98 15:47:28 03/18/98 15:38:46 03/18/98 15:24:54 03/13/98 15:22:11 03/06/98 16:25:30 02/20/98 16:36:22 02/20/98 16:36:22 02/20/98 16:15:27 02/20/98 09:36:46	Analysis: Analysis: Analysis: Analysis: Analysis: Calibration: Calibration: Calibration: Calibration: Calibration: Calibration: Calibration:	Starting analysys of file Starting analysys of file Starting analysys of file Starting analysys of file Starting analysys of file Pressure offset calibrat Pressure offset calibrat Calibrate servo valve. Reset pressure scale cc Pressure offset calibrat Calibrate servo valve.	000-003. SMP on port 1. 000-003. SMP on port 2. 000-019. SMP on port 1. 000-019. SMP on port 1. 000-018. SMP on port 1. ion (Man, P1, P2, P3). ion (Man, P1, P2, P3). allbration to nominal.	
		<u>R</u> eport		
Port Sample 1 2 3	Status Idle Idle Idle			

You can choose the type of information to display in the window by selecting only the desired item(s). For example, select only the **Calibration** option to display calibration information:

Γ

😑 Instrument Log (Unit 1 - S/N: test) 🔽 🔺						
⊂ <u>A</u> nalysis		∝ <u>C</u> alibration	□ <u>M</u> essage			
03/13/98 15:27:11 03/06/98 16:25:30 02/20/98 16:36:22 02/20/98 16:15:27 02/20/98 15:50:46 02/20/98 09:36:46	Calibration: Calibration: Calibration: Calibration: Calibration: Calibration:	Pressure offset calibrat Pressure offset calibrat Calibrate servo valve. Reset pressure scale ca Pressure offset calibrat Calibrate servo valve.	tion (Man, P1, P2, P3). alibration to nominal.			
Port Sample	Status	<u>R</u> eport				
1 2 3	ldie Idie Idie					

Analysis Calibration Message

These options allow you to choose which entries are displayed in the window.

Report...

Displays the Log Report Settings dialog so that you can specify report output options.

	Log R	eport Settings
Start Date	01/01/97	Number of Reports 1 문 Destination File +
		File name C:\WIN3000*.RPT
	<u> </u>	Cancel

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

- Highlight the field (or press **F2** to clear the field) and type in the desired date
- Double-click in the field (or press **F4**) to display a calendar to choose a date
- Press **F2** to clear the field, then **F3** to insert the *cur*rent date

Select the number of reports desired in the **Number of Reports** 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 in the **Destination** field. If you choose *File* as the destination, the **File name** field is enabled, allowing you to enter a name for the printed file (or you may accept the default).

Unit Configuration

Select this option to display hardware/software configurations, calibrations, and gas selections of the connected analyzer. When you select this option from the Unit menu, the Unit Configuration dialog box is displayed.

Configuration		Volume Calibration	
Comm Port:	COM4	Manifold Volume 18.2674 cm	n ³
Serial #:	1083	Date 12/18/200	2
	Board ID		
Software Versio	ns	Pressure Slope Calibration	
MIC BIOS:	Boot Block V1.04 May 15	Manifold 1/17/2003 9	7% of nominal
Controller:	3000 MASS V5.00 Jan 9	101(1)	8% of nominal
Application:	TriStar 3000 V6.00	Port 2 1/17/2003 9	7% of nominal
rippiloution.	110101 0000 10.00	Port 3 1/17/2003 9	8% of nominal
Gas Selections		Pressure Offset Calibration	
Valve 9 gas:	N2	Manifold 1/17/2003	
Valve 8 gas:		Port 1 1/17/2003	
		Port 2 1/17/2003	
Valve 7 gas:	He	Port 3 1/17/2003	
Servo Valve		Temperature Calibration	
Calibrated	12/18/2002	Temperature 12/18/2002	
	0 <u>K</u>	Cancel	

Configuration	Displays the number of the port being used to control the analyzer and the serial number of the selected ana- lyzer.
Software Versions	Displays the software versions of the MICBIOS, control- ler, and TriStar 3000 analysis program.
Gas Selections	Allows you to specify the mnemonic names for the analy- sis gas(es). Valves 8 and 9 are designated for the analy- sis gas. Valve 7 displays the free-space gas; this field cannot be edited.
Servo Valve	Displays the date the servo valve was last calibrated.
Calibration group boxes	The remaining group boxes display calibration informa- tion for their respective component.

Calibration

This option allows you to calibrate the manifold temperature, pressure scale, zero pressure, servo valve, and system volume. You can also save the current calibrations to a file or load a different calibration file. Refer to Chapter 9 for step-by-step instructions for each calibration procedure.

Unit <u>1</u>	
<u>S</u> ample analysis	
<u>Q</u> uickStart analysis	
Enable manual control	
Show instrument schematic	
Sho <u>w</u> status	
Show instrument <u>l</u> og	
Unit configuration	
<u>C</u> alibration ►	<u>T</u> emperature
Service <u>T</u> est	<u>P</u> ressure scale
	<u>Z</u> ero pressure
	<u>Reset pressure calibration</u>
	<u>S</u> ervo valve
	<u>V</u> olume
	Save to <u>f</u> ile
	<u>L</u> oad from file

Temperature

Use this option to adjust the offset of the manifold temperature sensor to agree with a value obtained using a calibration device. The Calibrate Manifold Temperature dialog is displayed so that you may enter the measured value.

Calibrate Manifold Temperature (Unit 1 - S/N: test)				
Current manifold temperature	25.63 °C			
<u>ок</u>	Cancel			
Enter a value between 10.00 and 100.00.				

Pressure Scale

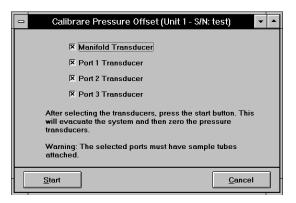
Use this option to adjust the scale of the pressure transducers. The Calibrate Pressure Scale dialog is displayed.

Calibrate Pressure Scale (Un	nit 1 - S/N: Demo)	х
Actual manifold pressure	760.00	mmHg	
🛛 Manifold <u>T</u> ransducer			
Port <u>1</u> Transducer			
Port <u>2</u> Transducer			
Port <u>3</u> Transducer			
<u> </u>	<u>C</u> ancel		
Enter a value between 500.00 and	1000.00.		

This dialog allows you to adjust the scale by entering a value obtained using a pressure gauge, as well as specifying which transducer(s) are at that pressure.

Zero Pressure

Use this option to evacuate the manifold and zero the selected transducers. The Calibrate Pressure Offset dialog is displayed.



Choose the transducer(s) you wish to have offset to zero. Click **Start** and a dialog indicating evacuation is in progress is displayed. When evacuation is complete, the dialog closes automatically.

Reset Pressure Calibration

Use this option to restore factory calibration settings for all transducers; a warning dialog is displayed.

DEMO 1	TriStar 3000 V4.00
	Reseting the pressure calibration will restore the factory calibration settings. The current calibration settings will be lost.
	OK Cancel

Click **OK** to restore the values; current values will be overwritten. Click **Cancel** to retain current values.

Servo Valve

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

0	Calibrare Servo Valve (Unit 1 - S/N: test)	•	
	This procedure will calibrate the servo valve to the		
	manifold pressure transducer.		
	14-1		
	Make sure that the pressure transducer was calibrated before starting.		
_			_
	Start Cancel		
J			-

The only action required here is to click **Start**. The servo valve is then used to fill and equilibrate the manifold, first to 100 mmHg and then to 760 mmHg. The pressure transducer readings (previously calibrated) at these points are used to calibrate the servo set point. Status messages are displayed during this procedure, then the dialog closes automatically.

Volume

Done

Next>>

Use this option to calibrate the system volume. The Calibrate Volume dialog is displayed so that you may specify how you want the volume calibrated.

	Calibrate Volume (Unit 1 - S/N: test)	•
Displays as Next>> if you choose Measured.	Previous 8.0000 cm ^e Entered 18.0000 cm ^e O Measured	
<u> </u>		

PreviousRetains the current system volume which is displayed to
the left of this choice.

Entered Enter a value to use for the system volume.

Measured Allows you to measure the system volume using an entered reference volume.

Closes the dialog; displays for the **Previous** and **Entered** selections.

Advances you to the next view of the Calibrate Volume dialog; displays for the **Measured** selection.

This view of the Calibrate Volume dialog allows you to enter the parameters required when using a reference volume to calibrate the system volume.

Calibrate Volume (Ur	nit 1 - S/N: Demo) 🔹 🔺
Reference Volume:	50.0000 cm ^a
Reference Volume Temp.	0.00 *C
Perform measurement:	3 times
Thermal equilibration time:	10 🌩 minutes
Reference volume must be 2.	e attached to ports 1 and
Start	<u>C</u> ancel

Reference Volume Enter the volume of the reference device.

Reference Volume Enter the temperature of the reference volume. **Temp.**

Perform Enter the number of measurements you wish to make. **measurement**

Thermal Enter the amount of time you wish to have the gas equilibration time equilibrate in the reference volume before taking the pressure measurement.

Start

Begins the calibration; a status view is displayed. After the calibration has finished, a confirmation view is displayed.

- Calibrate Volu	me (Unit 1 - S/N: test) 🗾 🔽
Previous	1 6.9000 cm³ 16.9000
O Entered	18.0000 cm ³
O Measured	17.2380 cm ³
O Average	17.1908 cm [*]
Last	9 🖉 measurements
Std.Dev.	0.0058 cm [*]
Done	Cancel

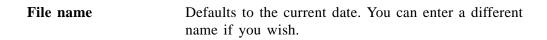
This dialog allows you to confirm the calculated volume. The **Measured** field contains the value of the last measurement; for example, the value for the third measurement is shown if you selected three measurements. The **Average** field contains an average of the number of selected measurements. This dialog also gives you the opportunity to return to the **Previous** or **Entered** value.

Click **Done** to close this dialog.

Save to File

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

	Select C	alibration F	ile
File name:	02-18-98.CAL		
			Directories:
Files:			g:\\hardware
*	<u>0K</u>	* * * <u>Car</u>	[] * [-a-] [-b-] [-c-] [-d-] [-d-] [-f-] [-f-] [-f] [-q-] [-w] [-w] [-w] [-z] #



Load from File

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

CHAPTER 7

REPORTS MENU

- Description
- Start Report
- Cancel Reports
- DFT Plus
- Printed Reports
- Sample Reports

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

<u>R</u> eports	
<u>S</u> tart report	F8
<u>C</u> lose reports	F9
<u>D</u> FTPlus	F11

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 window(s).
DFT Plus	Invokes the DFT Plus program, allowing you to generate DFT Plus reports. This option is functional only if the DFT Plus program
	is installed.

Start Report

	Sta	rt Report		
Selection Crit		Settings Copies Destination File name	1 Printer C:\WIN3000\DAT.	₹ A* BPT
Files: 000-013.smp 0136.smp 0140.smp 0140.smp 0176.smp 0176.smp 0176.smp 0176.smp 0186.smp 0200.smp 0201.smp n309.smn	000-013 ALUMINA REFERENCE (13 × REF 004-16826-00 13 × REF 004-16826-00 13 × REFENENCE 004/16 13 × REFERENCE 004/16 13 × Molecular Sieve Por	MAL 2818 12M MAL 2818 13L MAL 2818 14H 826/00 12L 826/00 13L 826/00 14H 826/00 14H 826/00 13L 826/00 14H	g:\\t	tories: ristar\data 7
	0 <u>K</u>	[Cancel	

Select this option to generate a report on a sample analysis. Select **Start report** from the **Reports** menu to display the Start Report dialog.

File name

The name of the sample file you select from the **Files** list window is copied to this field. If you select multiple files, the last one selected is displayed. If you have a sample file open, its name defaults to this field.

- If you select multiple files on which to have reports generated, the reports specified in each sample file are printed to the output destination.
- If you choose a single file, the Select Reports dialog containing the available reports is displayed. This dialog allows you to choose the reports you wish to generate. Refer to Chapter 5 for information on available reports.

Iect Reports Isotherm BET Surface Area Langmuir Surface Area LiPlot Alpha-S Method F-Ratio Method BJH Adsorption BJH Desorption Dubinin MP-Method Options Summary Sample Log
0 <u>K</u> ancel

File name (continued)	Select (or deselect) reports by double-clicking on the desired report (reports are selected when they are preceded with a check mark). Alternatively, you can hightlight the report and press the Spacebar .
Status	This drop-down list determines what type of sample files are displayed in the Files list window in the specified directory for all dates, or within the specified range of dates (using Dates push button).
	Choices: All, Analyzing, Complete, Entered
	All displays all types of samples files (Analyzing, Complete, and Entered).
	Analyzing displays sample files that are currently being used in an analysis.
	<i>Complete</i> displays sample files that have been used in an analysis.
	<i>Entered</i> displays sample files that contain maually entered data.
Dates	Displays the Select Dates dialog so that you may specify a range of dates. Refer to Selecting Files in Chapter 3 for a description of this push button.
Copies	Enabled when the <i>Printer</i> or <i>Printer/Plotter</i> destination is chosen. This option allows you to print up to four copies of the selected report(s).
Destination	Displays a drop-down list of output destinations.
	Choices: File, Printer, Printer/Plotter, Screen
	If you select <i>Printer</i> or <i>Printer/Plotter</i> , requested reports are printed to the selected printer and/or plotter. This option also enables the Copies field allowing you to print up to four copies of each report.
	If you select <i>Screen</i> , reports are displayed onscreen. Using this option allows flexibility for manipulating and customizing reports. Refer to Printed Reports later in this chapter.

Destination (continued)	If you select <i>File</i> , the tabular reports of the requested file are converted to a text file which can be viewed with a text editor or other text file manipulation tool.
File name	Enabled when you select <i>File</i> as the destination. Allows you to enter a name, or you may accept the default.
Files list box	Displays a list of the available sample files for the choice shown in the Status field and within the range of dates specified in the Dates dialog
Directories	Displays a list of available drives and directories. The drive and directory last accessed is displayed immediately above the Directories list box.

Close Reports

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

DFT Plus

DFT Plus[®] is an enhanced data reduction application. This program allows you to calculate pore volume, surface energy, and surface area distribution from the adsorption isotherm of materials which have been previously analyzed on a Micromeritics physisorption analyzer.

This option is nonfunctional if the DFT Plus program is not installed. See Chapter 10 for ordering information.

Printed Reports

Header

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

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

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

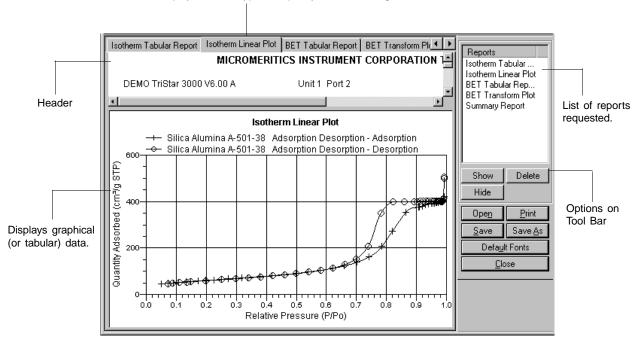
• Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.

Onscreen Reports

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

- a tool bar
- 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**.



Tabs display for each type of report you choose to generate.





Reports

Show

Contains a list of all requested reports.

Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible. DeleteDeletes 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.HideHides (removes) the selected report from the report window. The report's associated tab is also removed.OpenAllows you to open a previously saved report file.PrintDisplays 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.

Print			? ×
Printer —			Isotherm Tabular Report
<u>N</u> ame:	HP LaserJet 4/4M Post	✓ Properties	Isotherm Linear Plot BET Tabular Report
Type: H	Default printer; Ready HP LaserJet 4/4M PostScript; LP	YT6:	BET Transform Plot Summary Report
Where: M Comment:	AIC_SERVER	Print to file	
Print range		Copies	
© AI		Number of <u>c</u> opies: 1	
C Pages	from: to:	1 2 3 Collate	C <u>u</u> rrent <u>A</u> ll
C Selection	n		S <u>h</u> own Clear
		0 <u>K</u> Cancel	

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 nonhighlighted 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 for- mat using the same name as the sample file, only with 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:
	<i>Report system (*.rep)</i> : Saved in a format which allows you to reopen the file using the Open 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 Fonts	Displays the Default Fonts dialog so that you can edit report fonts.

Font Type	Close
Text	
Table title	Load
Table header	
Table column Graats Wa	Save
Graph title Graph axis title	<u></u> ave
Graph axis scale	F -0
Graph curve label	<u>E</u> dit
Graph legend	

The window on the left lists the items for which the font can be edited.

Click **Edit** to edit the font of a selected item; a font dialog is displayed so that you can specify the desired font and attributes.

Click **Save** to save the changes as the default. If you do not click **Save**, the changes will apply to the current report set only. The next reports will revert to the default font.

Default Fonts Click Load to load the default font of a selected item.

(continued)

Click **Close** to close the dialog.

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 to insert line feeds.
Move column	Allows you to move the location of the selected column to the left or to the right.
Align column	Enables you to right-align, left-align, or center the data in the selected column.
Hide column	Displays a list of all columns, enabling you to select the one 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 at- tributes for the tabular data in the current report.
Header font	Displays a Font dialog, allowing you to change font at- tributes 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. Colums are tab-delimited, allowing easy alignment.

Graphs

<u>A</u> utoscale	
<u>S</u> how curve <u>H</u> ide curve	+ +
Edit <u>c</u> urve Edit a <u>x</u> is Edit <u>l</u> egend))
Edit <u>t</u> itle	
Copy as meta <u>f</u> i C <u>o</u> py as text	le

Autoscale	Autoscales all axes of the graph. This function is useful for returning to a full view after having zoomed in.
Show curve	Shows any curve(s) that has been hidden. This option is disabled (greyed) if no curves have been hidden.
Hide curve	Hides (removes from view) any unwanted curve(s).

Edit curve

Displays the Curve Properties dialog, allowing you to edit curve properties.

I

Curve Propertie	es		×
Tjtle:	Desorption	_	
<u>S</u> tyle:	Curve and Points	•	
Curve Interpol <u>a</u> tion:	Akima Spline		
Point style:	Circle	▼ C <u>o</u> lor	
Pe <u>n</u> Style	Solid	•	
Histogram Fill Style	Back Diagonal	▼ Cojor	
La <u>b</u> el:	Center	<u></u>	
	0 <u>K</u>	<u>C</u> ancel	

Title	Displays the title of the curve you are editing.
Style	Drop-down list containing 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	Allows you to specify the type of fill as well as the color if Histogram is chosen as the style for collected data.

Edit axis Displays the Axis Properties dialog, allowing you to edit axis properties.

Axis Properties		×
Title Volume Adsorbed	cm²/g STP	Title <u>f</u> ont
Scale		
Einear	☐ In <u>v</u> ert Scale	Scale f <u>o</u> nt
C Logarithmic		
✓ Autoscale minimum	0	
Z Autoscale ma <u>x</u> imum	400	
<u>G</u> ridlines: Dotted	•	
0 <u>K</u>	<u>C</u> ancel	

Edit legend Displays the Legend Properties dialog, allowing you to edit the placement of the legend.

Legend Properties C Do not show Wertical above C Horizontal above C Left C Right C Bottom	Eont
<u> </u>	<u>C</u> ancel

Edit title Displays the Title Properties dialog, allowing you to edit the current graph's title and font.

Title Prop	erties		×
Tjtle:	Isotherm Plot		<u>E</u> ont
	0 <u>K</u>	<u>C</u> ancel	

Copy as metafile Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

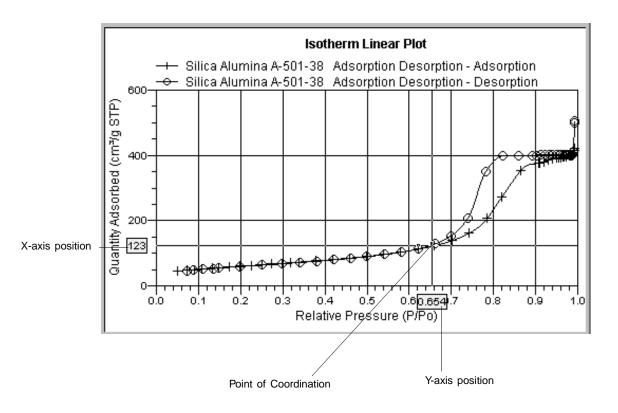
Copy as text Copies the data used to generate the graph as a series of tab-delimited columns of text.

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. Rightclick 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.



Right-click in the graph area and choose **Autoscale** form the shortcut menu to remove cross-hair lines and return to the normal view. Alternatively, you can click outside of the graph area.

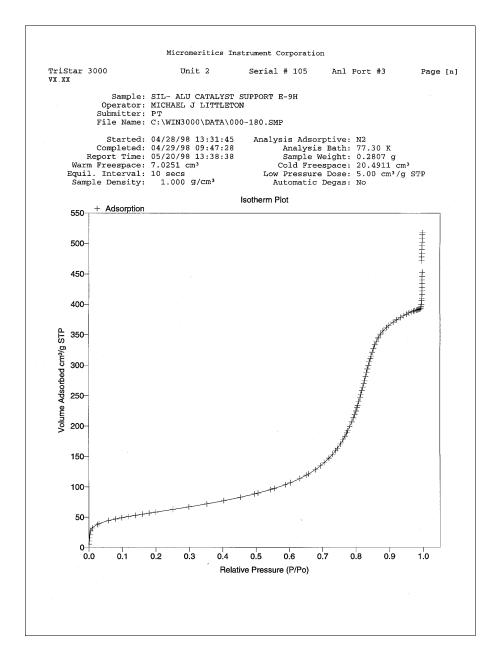
Report Examples

The remainder of this section contains samples of some of the reports which may be generated by the TriStar 3000 Analysis Program. Most of the reports can be varied through entries on the report options screens.

Analysis Log

	Minungalai	- Trackersen bold		
T ui (han 2000		cs Instrument Corpo		#2 D
TriStar 3000 VX.XX	Unit 2	Serial #	105 Anl Port	#3 Page [n]
Operator: Submitter:	MICHAEL J LITT		Н	
Completed:	10 secs	7:28 An 3:38 Sa Col Low Pr	Adsorptive: N2 alysis Bath: 77. mple Weight: 0.2 d Freespace: 20.4 essure Dose: 5.00 matic Degas: No	807 g 4911 cm³
		Analysis Log		
Relative Pressure	Pressure (mmHg)	Vol Adsorbed (cm³/g STP)	Elapsed Time (HR:MN)	Saturation Press.(mmHg)
0.000206635 0.000815369 0.001234223 0.002122194 0.004456607 0.009639227 0.024885514 0.02980067 0.058142523 0.079783313 0.097983914 0.117938122 0.138227411 0.158639562 0.179079633 0.199553211 0.250737177 0.298620570 0.352334403 0.403166524 0.453216784 0.495800293	0.15489 0.61118 0.92514 1.59074 3.34054 7.22529 18.65347 22.33727 43.58196 59.80328 73.44592 88.40302 103.61129 118.91165 134.23294 149.57935 187.94537 223.83739 264.09973 302.20203 339.71826 371.63763	5.2954 10.6694 15.8147 21.7052 27.2224 32.0209 38.0317 39.2723 44.3500 47.13711 49.1568 51.1696 53.0812 54.9166 56.7035 58.4680 62.8663 67.1035 57.21453 77.3413 83.0573 88.5249	00:35 00:44 00:53 00:59 01:04 01:17 01:25 01:31 01:38 01:45 01:51 01:57 02:03 02:09 02:15 02:22 02:30 02:37 02:47 02:58 03:10 03:21	
0.997040056 0.996375532 0.995582435 0.995783478 0.99584040 0.996113012 0.996353140 0.996626815 0.996626815 0.996626811 0.997107070 0.997430986	747.35254 746.85443 746.25995 746.41064 746.46602 746.65765 746.83765 747.04279 747.08881 747.40277 747.64557	$\begin{array}{c} 459.1459\\ 466.0022\\ 472.0712\\ 478.0933\\ 484.1599\\ 490.2154\\ 496.2256\\ 502.2635\\ 508.2940\\ 514.2736\\ 517.5811 \end{array}$	$16:25 \\ 16:29 \\ 16:33 \\ 16:40 \\ 16:40 \\ 16:43 \\ 16:46 \\ 16:49 \\ 16:52 \\ 16:59 \\ 17:03$	

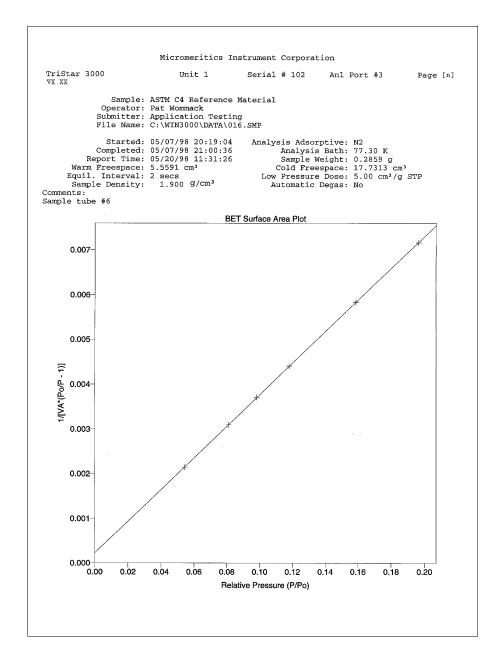
Isotherm Plot



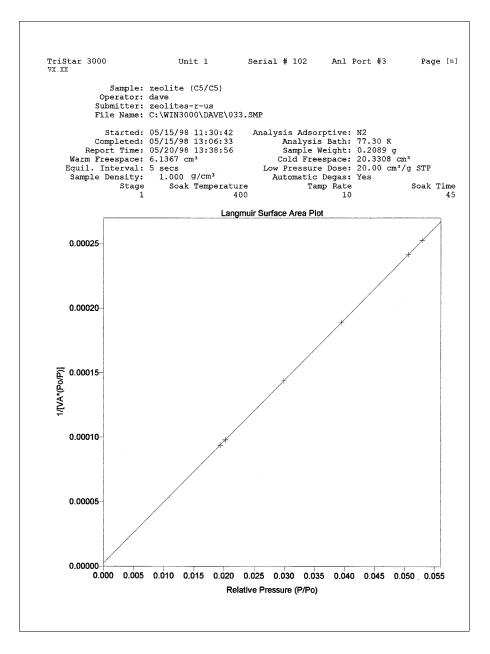
BET Surface Area Report

```
Micromeritics Instrument Corporation
 TriStar 3000 Unit 1
VX.XX
                                                                    Serial # 102 Anl Port #3
                                                                                                                            Page [n]
                  Sample: ASTM C4 Reference Material
Operator: Pat Wommack
Submitter: Application Testing
File Name: C:\WIN3000\DATA\016.SMP
Comments:
Sample tube *C
                                                                    Analysis Adsorptive: N2
Analysis Bath: 77.30 K
Sample Weight: 0.2859 g
Cold Freespace: 17.7313 cm<sup>3</sup>
Low Pressure Dose: 5.00 cm<sup>3</sup>/g STP
Automatic Degas: No
 Sample tube #6
                                                  BET Surface Area Report
                                                     122.3865 ± 0.2405 m<sup>2</sup>/g
0.035340 ± 0.000069
0.000229 ± 0.000009
                      BET Surface Area:
                      Slope:
                      Y-Intercept:
C:
VM:
                                                               155.088376
                      VM: 28.114154 cm<sup>3</sup>/g STP
Correlation Coefficient: 9.999923e-01
                      Molecular Cross-section:
                                                                 0.1620
                                                                                    nm²
                 Relative
Pressure
                                                                                                     1/
[VA*(Po/P - 1)]
                                                                 Vol
                                                              Adsorbed
                                                           (cm<sup>3</sup>/g STP)
               0.054499044
                                                                 26.8852
                                                                                                              0.002144
                                                                 20.8852
28.4721
29.3896
30.3727
32.3108
               0.081066623 0.098413885
                                                                                                             0.003098
               0.118128220
0.158576996
0.196421988
                                                                                                             0.004410
                                                                 34.1120
                                                                                                              0.007166
```

BET Surface Area Plot



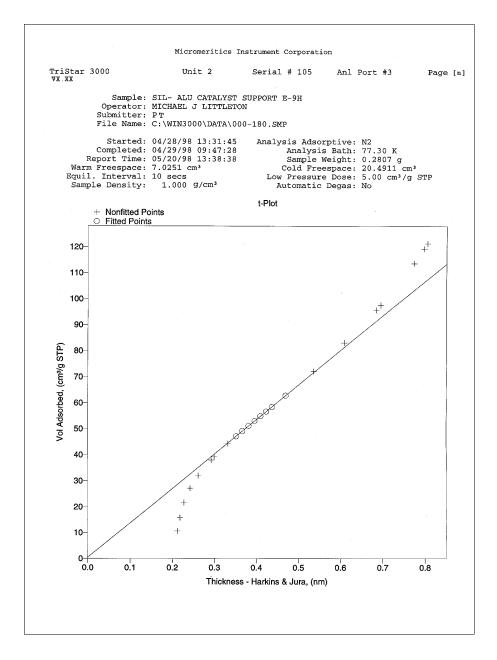
Langmuir Surface Area Plot



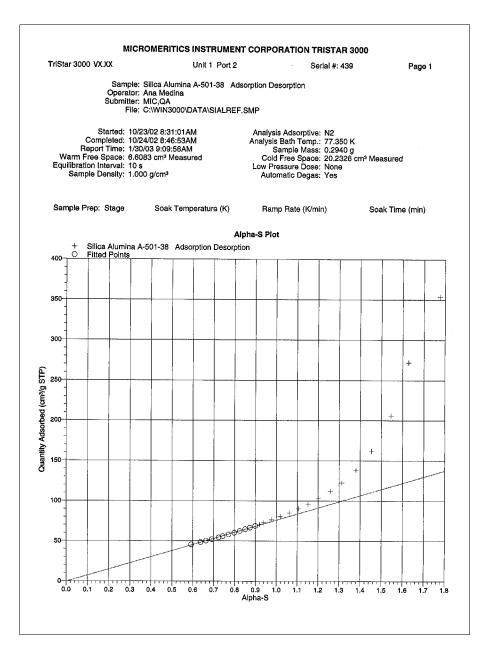
t-Plot Report

<text></text>							
VI.XX Sample: SIL- ALU CATALYST SUPPORT E-9H Operator: MICHAEL J LITTLETON Submitter: PT File Name: C:WIN3000\DATA\000-180.SMP Started: 04/28/98 13:31:45 Analysis Adsorptive: N2 Completed: 04/29/98 03:43:36 Sample Weight: 0.2807 g Warm Freespace: 7.0251 cm ³ Cold Freespace: 20.04911 cm ³ g Equil. Interval: 10 secs Low Pressure Dose: 5.00 cm ³ /g STP Automatic Degas: No t-Plot Report Micropore Volume: 0.000675 cm ³ /g Micropore Area: 205.6841 m ² /g Slope: 0.436509 ± 0.027469 Y-Intercept: 0.3500 to 0.5000 nm t = [13.9900 / (0.0340 - log(P/P0)]] 0.5000 Surface Area Correction Factor: 1.00 Density Conversion Factor: 1.00 Surface Area Correction Factor: 1.00 Micropore Thickness, (nm) (cm ³ /g) 0.000815569 0.2117 10.6684 0.00123223 0.2167 15.8147 0.00153569 0.2117 10.6684 0.00123223 0.2160 15.8147 0.00124223 0.2161 15.8147 0.00124223 0.2161 15.8147 0.0036500 0.2612 32.22 0.04485141 0.2922 38.017 0.028900667 0.2422 32.2723 0.02848514 0.2925 39.2723 0.02890067 0.2422 32.2723 0.02848514 0.2925 39.2723 0.05814253 0.3210 44.500 0.079783313 0.3515 47.1371 0.05814253 0.3216 47.1371 0.058639522 0.4212 56.017 0.028900667 0.3422 56.017 0.028900667 0.3422 56.0317 0.028900667 0.3422 56.0317 0.05814253 0.3210 44.500 0.079783313 0.3515 47.1371 0.05814253 0.3210 44.500 0.079783313 0.3515 47.1371 0.05814253 0.3220 44.500 0.079783313 0.3515 47.1371 0.05814253 0.3320 44.500 0.079783313 0.3515 47.1371 0.05814253 0.3220 44.500 0.079783313 0.3515 47.1371 0.35839562 0.4097 55.663 0.11793975 53.042 0.11793975 53.042 0.13829552 0.13829552 0.13829552 0.433234403 0.5366 57.755 0.3326468 57.772 0.332648552 0.6634 97.5772 0.5312688550		Micromeritics Instr	ument Corporation				
Operator: MICHAEL J LITTLETON Submitter: PT File Name: C:WUNN000/DATA\000-180.SMP Started: 04/28/98 03:47:28 Analysis Bath: 77.30 K Completed: 04/29/98 09:47:28 Analysis Bath: 77.30 K Report Time: 05/20/98 13:38:38 Sample Weight: 0.2807 g Warm Freespace: 7.0251 cm³ Cold Freespace: 20.4911 cm³ Equil. Interval: 10 secs Low Pressure Dose: 5.00 cm³/g STP Sample Density: 1.000 g/cm³ Automatic Degas: No t-Plot Report Lice Portersure Dose: 0.07469 Micropore Volume: 0.000675 cm³/g Micropore Area: 205.6841 m²/g Slope: 13.297392 ± 0.111480 Correlation Coefficient: 9.99987e-01 Thickness Range: 0.3500 to 0.5000 nm t = [13.9900 / (0.0340 - log(P/Po)]) 0.5000 Surface Area Correction Factor: 1.00 Density Conversion Factor: 1.00 Density Conversion Factor: 1.00 Pressure Thickness, (nm) (cm³/g) 0.0013569 0.2117 10.6694 0.002122194 0.2273 21.7052 0.0045607 0.22822 38.0317 0.0023227		Unit 2	Serial # 105	Anl Port #3	Page [n]		
$ \begin{array}{c} \mbox{Completed: 04/29/98 09:47:28} & Analysis math: 77.30 K \\ \mbox{Report Time: 05/20/98 13:83:8} & Sample Weight: 0.2807 g \\ \mbox{Cold Preespace: 20.4911 cm^3} \\ \mbox{Equil. Interval: 10 secs} & Low Pressure Doce: 5.00 cm^3/g \\ \mbox{Mutmatic Degas: No} \\ \mbox{t-Plot Report} \\ \mbox{Micropore Volume: 0.000675 cm^3/g \\ \mbox{Micropore Area: 6.6672 m^3/g \\ \mbox{Micropore Area: 6.6672 m^3/g \\ \mbox{Micropore Area: 0.3500 to 0.27469 } \\ \mbox{Y-Intercept: 0.436509 \pm 0.111480 \\ \mbox{Correlation Coefficient: 9.99987e-01 \\ \mbox{Thickness Range: 0.3000 to 0.5000 nm \\ \mbox{t = [13.9900 / (0.0340 - log(P/Po)]] 0.5000 \\ \mbox{Surface Area Correction Factor: 1.00 \\ \mbox{Density Conversion Factor: 0.001547 \\ \mbox{Total Surface Area (by BET): 212.3512 \\ \mbox{Relative Statistical Vol Adsorbed \\ \mbox{Pressure Thickness, (nm) (cm^3/g) \\ \mbox{0.002132223 0.2117 10.6694 \\ \mbox{0.002132232 0.2217 21.7052 \\ \mbox{0.004456607 0.2422 277 221 \\ \mbox{0.004456607 0.2422 277 222 \\ \mbox{0.003639227 0.2612 32.0209 \\ \mbox{0.00345514 0.2925 39.9723 \\ \mbox{0.009783313 0.3515 47.1371 \\ \mbox{0.09783313 0.3515 47.1371 \\ \mbox{0.09783314 0.3663 49.1568 \\ \mbox{0.1793632 0.4432 56.18663 \\ \mbox{0.13827411 0.3957 53.0812 \\ \mbox{0.13827411 0.3957 53.0812 \\ \mbox{0.13827411 0.3957 53.0812 \\ \mbox{0.13833 45.16696 \\ \mbox{0.13737177 0.4695 66.28663 \\ \mbox{0.138334403 0.5360 72.1453 \\ \mbox{0.158468570 0.6948 97.5772 \\ \mbox{0.631268853 0.7736 113.7026 \\ \mbox{0.651269065 0.7797 119.3289 \\ \mbox{0.53289} \end{tabular}} } \end{tabular}$	Operator: Submitter:	MICHAEL J LITTLETON PT					
Micropore Volume: 0.000675 cm³/g Micropore Area: 5.6672 m²/g External Surface Area: 205.6841 m²/g Slope: 13.297392 ± 0.027469 Y-Intercept: 0.435509 ± 0.111480 Correlation Coefficient: 9.9987e-01 Thickness Range: 0.3500 to 0.5000 nm t = [13.9900 / (0.0340 - log(P/Po))] 0.5000 Surface Area Correction Factor: 1.00 Density Conversion Factor: 0.001547 Total Surface Area (by BET): 212.3512 Relative Statistical Vol Adsorbed Pressure Thickness, (nm) (cm³/g) 0.000815369 0.2117 10.6694 0.01234223 0.2180 15.8147 0.02122194 0.2273 21.7052 0.004456607 0.2422 27.2224 0.00465607 0.2422 38.0317 0.023800067 0.2995 39.2723 0.658142523 0.3310 44.3500 0.079783313 0.3515 47.1371 0.079783313 0.3515 47.1371 0.079783313 0.3515 <td< td=""><td>Completed: Report Time: Warm Freespace: Equil. Interval:</td><td>04/29/98 09:47:28 05/20/98 13:38:38 7.0251 cm³ 10 secs</td><td>Analysis Sample We Cold Frees Low Pressure</td><td>Bath: 77.30 K ight: 0.2807 g pace: 20.4911 cm³ Dose: 5.00 cm³/g S</td><td>STP</td></td<>	Completed: Report Time: Warm Freespace: Equil. Interval:	04/29/98 09:47:28 05/20/98 13:38:38 7.0251 cm ³ 10 secs	Analysis Sample We Cold Frees Low Pressure	Bath: 77.30 K ight: 0.2807 g pace: 20.4911 cm ³ Dose: 5.00 cm ³ /g S	STP		
Micropore Area: $6.672 \text{ m}^2/\text{g}$ External Surface Area: $205.6841 \text{ m}^2/\text{g}$ Slope: 13.297392 ± 0.027469 $\sqrt{-1ntercept:}$ 0.436509 ± 0.111480 Correlation Coefficient: $9.9987e-01$ Thickness Range: $0.3500 \text{ to } 0.5000 \text{ nm}$ t = [13.9900 / (0.0340 - log(P/Po))] 0.5000Surface Area Correction Factor: 1.00 Density Conversion Factor:Density Conversion Factor: 0.001547 Total Surface Area (by BET):Thickness, (nm)(cm ³ /g)0.0008153690.2117 10.6694 0.0012342230.2212270.261232.02090.024856070.22120.0248855140.229539.27230.0581425230.332044.35000.077833130.366349.116960.11710.66940.22190.22190.2211710.66940.22122.7.22240.0096392270.261232.02090.024885140.23200.0581425230.332044.35000.77780.138227411 <td <="" colspan="2" td=""><td></td><td>t-Plot</td><td>Report</td><td></td><td></td></td>	<td></td> <td>t-Plot</td> <td>Report</td> <td></td> <td></td>			t-Plot	Report		
Surface Area Correction Factor: 1.00 Density Conversion Factor: 0.001547 Total Surface Area (by BET): 212.3512 Relative Statistical Vol Adsorbed Pressure Thickness, (nm) (cm³/g) 0.000815369 0.2117 10.6694 0.001234223 0.2180 15.8147 0.002122194 0.2273 21.7052 0.004456607 0.2422 27.2224 0.009639227 0.2612 32.0209 0.024885514 0.2922 38.0317 0.029800067 0.2995 39.2723 0.058142523 0.3320 44.3500 0.079783313 0.3515 47.1371 0.097983914 0.3663 49.1568 0.1138227411 0.3957 53.0812 0.138237411 0.3957 53.0812 0.138237411 0.4366 58.4680 0.25037177 0.43655 62.8663 0.352334403 0.5360 72.1453 0.453216784 0.6086 3.0573	Mid Ext Sla Y- Co	cropore Area: ternal Surface Area: ope: Intercept: rrelation Coefficien	6.6672 205.6841 13.2973 0.4365 t: 9.9998	m ² /g m ² /g 92 ± 0.027469 09 ± 0.111480 7e-01			
Density Conversion Factor: 0.001547 Total Surface Area (by BET): 212.3512 Relative Pressure Statistical Thickness, (nm) Vol Adsorbed (cm³/g) 0.000815369 0.2117 10.6694 0.001234223 0.2180 15.8147 0.00456607 0.2422 27.2224 0.00456507 0.2422 27.2224 0.00456507 0.2995 39.2723 0.024885514 0.2995 39.2723 0.02980067 0.2995 39.2723 0.058142523 0.3320 44.3500 0.079783313 0.3515 47.1371 0.097983914 0.3663 49.1568 0.117938122 0.3813 51.1696 0.138227411 0.3957 53.0812 0.158639562 0.4097 54.9166 0.179079633 0.4232 56.7035 0.199553211 0.4366 58.4680 0.352334403 0.5360 72.1453 0.453216784 0.6086 3.0573 0.54368365 0.6844 95.6638 0.	t	= [13.9900 / (0.034	0 - log(P/Po))] (0.5000			
Pressure Thickness, (nm) (cm³/g) 0.000815369 0.2117 10.6694 0.001234223 0.2180 15.8147 0.002122194 0.2273 21.7052 0.004456607 0.2422 27.2224 0.00485514 0.2922 38.0317 0.028805514 0.29922 39.2723 0.029800067 0.2995 39.2723 0.058142523 0.3320 44.3500 0.079783313 0.3515 47.1371 0.097983914 0.3663 49.1568 0.117938122 0.3813 51.1696 0.138227411 0.3957 53.0812 0.158639562 0.4097 54.9166 0.179079633 0.4232 56.7035 0.199553211 0.4366 58.4680 0.250737177 0.4695 62.8663 0.352334403 0.5360 72.1453 0.453216784 0.6086 83.0573 0.54368365 0.6844 95.6638 0.554669870 0.6948 97.5772	Der	nsity Conversion Fac	tor: 0.0015				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$					1		
	$\begin{array}{c} 0.001234223\\ 0.002123423\\ 0.002456607\\ 0.009639227\\ 0.024885514\\ 0.029800067\\ 0.058142523\\ 0.079783313\\ 0.079783313\\ 0.079783314\\ 0.117938122\\ 0.138227411\\ 0.158639562\\ 0.179079633\\ 0.199552211\\ 0.250737177\\ 0.352334403\\ 0.453216784\\ 0.543683665\\ 0.554869870\\ 0.631268853\\ 0.651290065\end{array}$		1.2180 .2273 .2422 .2422 .2422 .2995 .3320 .3515 .3663 .3813 .3957 .4097 .4326 .4097 .4326 .4695 .5360 .6086 .6086 .6086 .6844 .6948 .7736 .7970	$\begin{array}{c} 15.8147\\ 21.7052\\ 27.224\\ 32.0209\\ 38.0317\\ 39.2723\\ 44.3500\\ 47.1371\\ 49.1568\\ 51.1696\\ 53.0812\\ 54.9166\\ 56.7035\\ 58.4680\\ 62.8663\\ 72.1453\\ 83.0573\\ 95.6638\\ 97.5772\\ 113.7026\\ 119.3289\end{array}$			

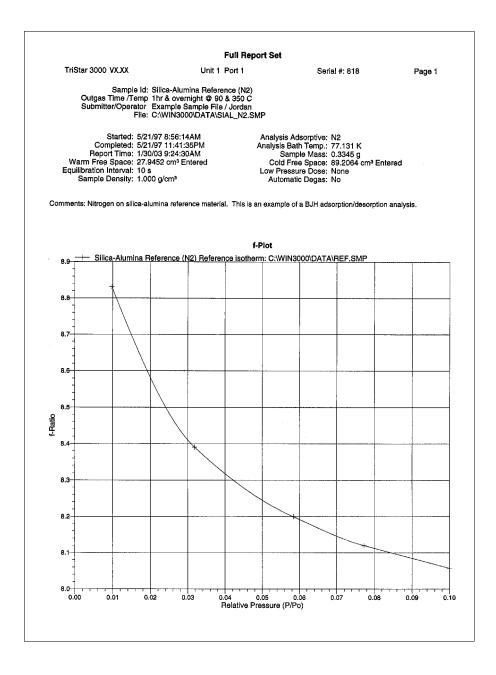
t-Plot Graph



Alpha-S Method



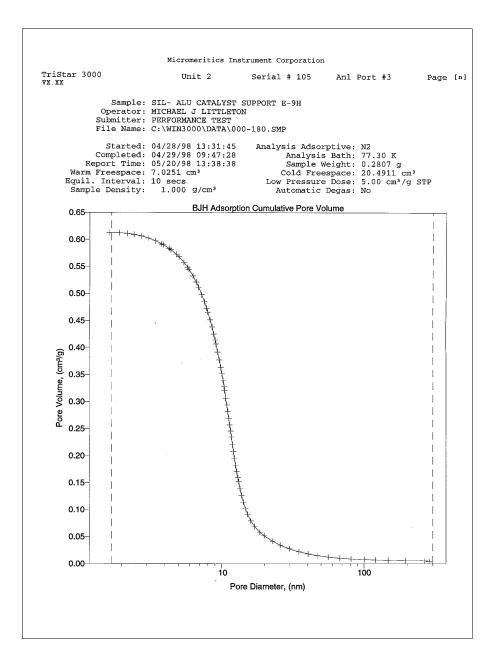
f-Ratio Method



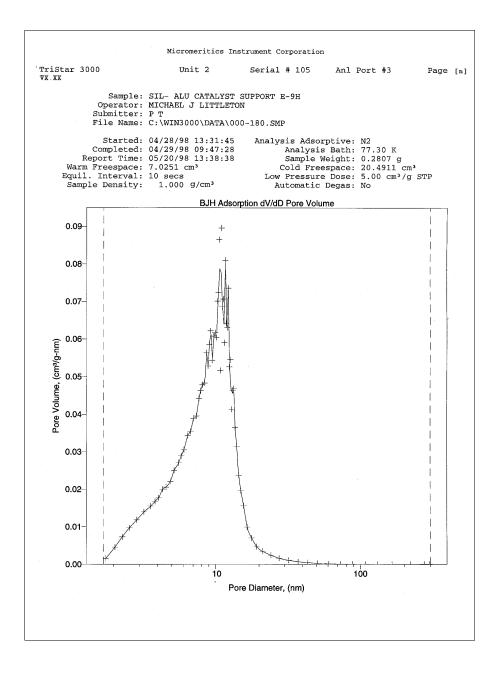
BJH Adsorption: Pore Distribution Report

TriStar 3000		meritics Instrum Unit 2	Serial # 105	Anl Port #3	B Pag
¥X . XX			Server " 100	THE FOLD IN	- Lug
		ALU CATALYST SU	JPPORT E-9H		
Subr	itter: PT	EL J LITTLETON			
File	Name: C:\WI	N3000\DATA\000-	180.SMP		
		/98 13:31:45 /98 09:47:28	Analysis Adso		
		/98 09:4/:28 /98 13:38:38		s Bath: 77.30 Weight: 0.280	
	space: 7.025			espace: 20.491	
Equil. Int Sample De	erval: 10 se	cs 00 g/cm³	Low Pressur	e Dose: 5.00 d	
Sample De	-			Degas: No	
		Adsorption Pore		-	
	t = 3	3.5400 x [-5.00	00 / ln(P/Po)](.3330	
		Diameter Range: Adsorbate Prope			
		Density Convers Fraction of Por	ion Factor: 0.	001547	
Pore	Average	Incremental	Cumulative	Incremental	Cumulati
Diameter	Diameter	Pore Volume	Pore Volume	Pore Area	Pore Are
Range (nm)	(nm)	(cm³/g)	(cm ³ /g)	(m²/g)	(m²/g)
314.8- 283.3	297.3	0.003298	0.003298	0.044	0.04
283.3- 264.3	273.1	0.001810	0.005107	0.027	0.07
264.3- 194.8 194.8- 147.4	219.1 164.6	0.000567	0.005674	0.010	0.08
147.4- 120.7	131.4	0.000463 0.000513	0.006138	0.011 0.016	0.09
120.7~ 100.2	108.5	0.000634	0.007285	0.023	0.13
100.2- 81.0	88.6	0.001176	0.008460	0.053	0.13
81.0- 67.6	73.1	0.001465	0.009925	0.080	0.26
67.6- 55.9	60.6	0.002071	0.011996	0.137	0.40
55.9- 47.2	50.8	0.002677	0.014673	0.211	0.61
47.2- 40.5	43.3	0.003278	0.017952	0.303	0.91
40.5- 34.9	37.3	0.004030	0.021981	0.432	1.34
34.9- 29.9	32.0	0.005496	0.027477	0.686	2.03
29.9- 25.8	27.5	0.006855	0.034331	0.995	3.02
25.8- 22.9 22.9- 19.9	24.1 21.2	0.007247 0.010134	0.041579 0.051713	1.201 1.913	4.22 6.14
19.9- 18.6	19.2	0.006386	0.058099	1.329	7.47
18.6- 17.1	17.8	0.010401	0.068500	2.337	9.80
17.1- 16.1	16.6	0.010599	0.079099	2.559	12.36
16.1- 15.3	15.7	0.012356	0.091454	3.157	15.52
•					
•					
•					
•					
3.4- 3.1	3.2	0.005168	0.602422	6.413	249.76
3.1- 2.7	2.9	0.003889	0.606310	5.404	255.16
2.7- 2.4	2.6	0.002991	0.609301	4.671	259.83
2.4- 2.2	2.3	0.001820	0.611121	3.180	263.01
	2.0	0.001143	0.612264 0.612721	2.243 1.038	265.26 266.29
2.2- 1.9 1.9- 1.6	1.8	0.000456			

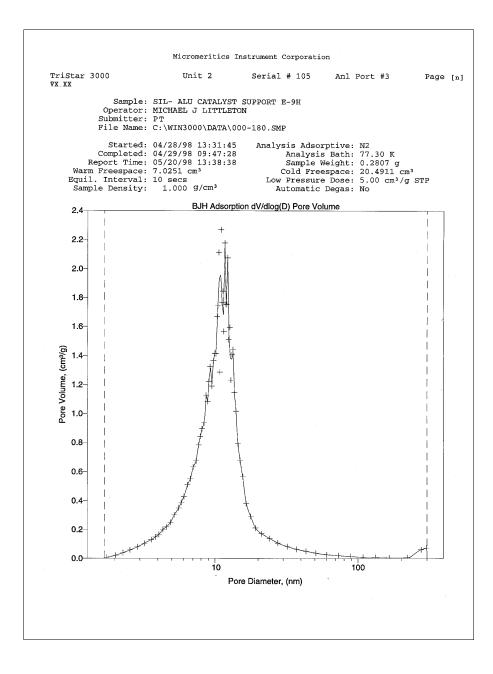
BJH Adsorption: Cumulative Pore Volume Plot



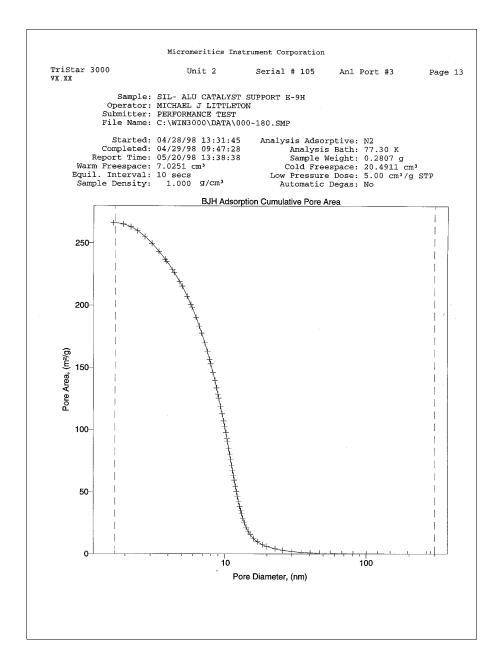
BJH Adsorption: dV/dD Pore Volume



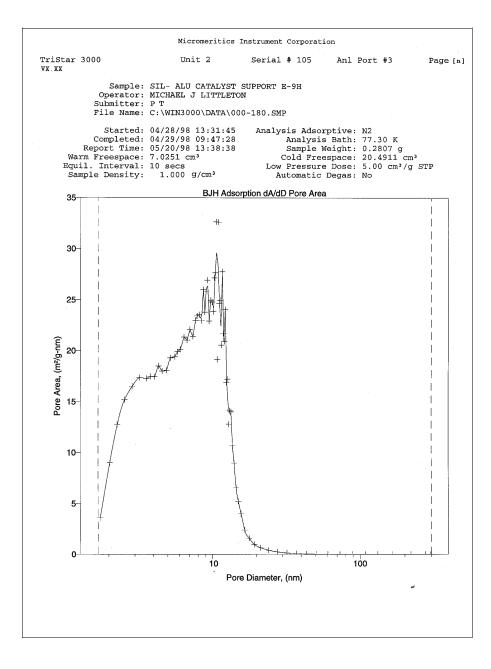
BJH Adsorption: dV/dlog(D) Pore Volume



BJH Adsorption: Cumulative Pore Area Plot



BJH Adsorption: dA/dD Pore Area Plot



Summary Report

		Micromeritics Inst	rument Corporation		
	iStar 3000 .XX	Unit 2	Serial # 105	Anl Port #3	Page [n]
	Operator: Submitter:	SIL- ALU CATALYST S MICHAEL J LITTLETON PT C:\WIN3000\DATA\000	ſ		
	Completed: Report Time:	04/28/98 13:31:45 04/29/98 09:47:28 05/20/98 13:38:38 7.0251 cm ³ 10 secs 1.000 g/cm ³	Analysis Sample W	rptive: N2 5 Bath: 77.30 K 4eight: 0.2807 g 2space: 20.4911 cm ³ 2 Dose: 5.00 cm ³ /g ST Degas: No	P
			y Report		
		А	rea		
Sir	gle Point Surface	Area at P/Po 0.1995	5321 :	203.7319	m²/g
BET	Surface Area:			212.3512	m²/g
Lar	gmuir Surface Area	1:		292.9783	m²/g
Mic	ropore Area:			6.6672	m²/g
Ext	ernal Surface Area	1:		205.6841	m²/g
BJH		ative Surface Area o and 300.000000 nm D		266.2989	m²/g
		Vo	lume		
Sir	gle Point Adsorpti 315.0478 nm I	on Total Pore Volum Diameter at P/Po 0.9	e of pores less 9384007:	than 0.612889	cm³/g
Mic	ropore Volume:			0.000675	5 cm³/g
BJH		tive Pore Volume of and 300.000000 nm D		0.612721	. cm³/g
		Pore	e Size		
Ads	orption Average Po	pre Diameter (4V/A b	y SINGLE POINT):	12.0332	nm
		e Pore Diameter (4V		9,2035	rım
				,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
-					

Sample Log Report

TriStar 3000 VX.XX	Unit 1	Serial	# 102	Anl	Port	#3	Pag	e [n]
Operator: « Submitter: s	silica gel (10/10) dave silica world C:\WIN3000\DAVE\025	. SMP						
Completed: Report Time: Warm Freespace: Equil. Interval:		C Low 1 Au	is Adsorpt Analysis F Sample Wei old Freesp Pressure I tomatic De Tamp	ath: .ght: bace: bose: egas:	77. 0.2 19. 10. Yes	092 g 7033 cm³ 00 cm³/g		Time 45
	Sample I	og Repor	t					
Date Time			Message					
05/12/98 03:47:46	Start degas stage	1 of 1:	ramp to 4	100°c	at	10°C/min	and	soak
05/12/98 03:47:46		1 of 1:	ramp to 4	00°c	at	10°C/min	and	soak
05/12/98 03:47:46	for 45min. Start degas stage for 45min.	1 of 1:	ramp to 4	00°c	at	10°C/min	and	soak
05/12/98 21:31:20 05/12/98 21:41:19	Start degas coold Measure freespace Starting Po in Po Measured Po over :	, Warm: tube mea	asurement.			19.7033	cm ³	

CHAPTER 8

OPTIONS MENU

- Description
- Option Presentation
- Data Presentation
- Sample Defaults
- System Configuration
- Parameter Files Directory
- Service Test Mode

OPTIONS MENU

The Options menu contains options which allow you to configure the system to your laboratory's requirements. With the options on this menu, you can:

- · Choose between basic, advanced, or restricted mode
- Edit program defaults
- Specify data presentation modes
- Specify printer/plotter options
- · Specify a directory for predefined parameter files

Description

Options

Option presentation

Service Test Mode

<u>D</u>ata presentation

Sample defaults... Tube <u>Characteristics...</u>

System configuration...

Parameter files directory...

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

►

Option presentation	Allows you to display the sample file dialog in either Ba- sic, Advanced, or Restricted format.
Data presentaton	Allows you to choose the way in which data are pre- sented on the screen and in printed reports.
Sample defaults	Allows you to specify defaults for the parameters con- tained in the sample information and parameter files.
Tube characteristics	Enabled only when in Service Test Mode.
System configuration	Allows you to specify the printer and/or plotter to which you wish to have reports sent.

Parameter files	Allows you to specify a location for the predefined pa-
diretory	rameter files used by the Basic format.
Service Test Mode	Enables you to perform certain troubleshooting proce- dures. This option is available only under the direction of a Micromeritics service representative.

Option Presentation

The sample editing dialogs for the TriStar 3000 analysis program may be presented in three different formats: Advanced, Basic, or Restricted.

Opt <u>i</u> ons		_
Service <u>T</u> est Mode		
Option presentation	Þ	<u>R</u> estricted
Data presentation	►	✓ <u>B</u> asic Advanced
Sample defaults Tube <u>C</u> haracteristics		
System configuration Parameter files directory		

Each format displays sample information and menu options differently.

- Advanced: displays all parts of the sample information file in a tabbed dialog as in an index card file. Clicking on each tab opens an associated dialog. Use this format to edit parameter files or create customized sample files.
- **Basic**: displays the sample file and parameter files in a single dialog. Use this format to create sample files using previously created parameter files.



Basic and Advanced formats contain a toggle push button so that you may switch from one format to the other. The push button displays as <u>Advanced</u> in Basic format and as <u>Basic</u> in Advanced format.

• **Restricted**: displays in the same manner as the Basic format. This format also is used to create sample files using previously created parameter files. However, you cannot switch to the Advanced format to edit parameters. You must also use a password for entering and exiting this format.

Advanced

The *Advanced* format presents all parts of the sample information file in a tabbed dialog. For example, if you wish to open or create a sample file using the Advanced format, the following dialog is displayed.

≤< Prev	
Sample: 000-001	
Operator:	
Submitter:	
Mass: 1.0000 g Type of Data Mass: 1.0000 g © Automatically collected Density: 1.000 g/cm³ © Manually entered	If you are open- ing an existing file, this dialog also contains a tab for Collected
Add Log Entry Replace All	or Entered Data
Enter a value between 0.0010 and 500.0000.	
Save Close Basic	

The Advanced format is used to create customized sample files — easily and quickly. You can also switch to the Basic format, if desired, by clicking **Basic**. Chapter 5 provides a detailed description of this dialog.

Basic

The *Basic* format presents the sample information file and its parameter files in a single dialog. For example, if you wish to open or create a sample information file using the Basic format, the dialog box is displayed in this manner.

s	Sample Information	
Sample: 000-0	002	
<u>M</u> ass: 1	.0000 g <u>D</u> ensity: 1.000 g/cm ³	
Sample <u>t</u> ube:	Sample Tube	
Degas conditions:	Degas Conditions	
Analysis conditions:	Run Conditions	
Adsorptive properties:	Nitrogen	This push button does
Report options:	Report Options	not display
	Replace All Add Log Entry	when in the Restricted format.
<u>S</u> ave		

The Basic format is used to create sample information files using previously defined parameter files. You can easily switch to Advanced format, if desired, by clicking Advanced. Chapter 5 provides a detailed description of this dialog.

Restricted

The **Restricted** format is identical to the Basic format, except that certain menu options are disabled, and you cannot switch to the Advanced format. This format is password-protected and is typically used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.

When you select Restricted, a dialog requesting a password is displayed:

Option Presentation Password	×
Please set a new password.	ОК
	Cancel

Any password (up to 31 characters) can be used to enable the Restricted format. The same password, however, must be used to exit the Restricted format. For example, if you enter "password" to enable the Restricted format, then you must use "password" to exit. If you forget the password, open the system INI file and navigate to the **Private** section. The current password will be shown immediately following "OptionPresentationPassword." Make a note of the password, exit the INI file, and enter the password where requested. Do not attempt to delete the password in the INI file.

Data Presentaton

The options on the Data presentation drop-down menu allow you to specify how you wish tabular and graphical data to display on screen and in printed reports.

Opt <u>i</u> ons	-
Option presentation	L
<u>D</u> ata presentation ▶	<u>U</u> nits
<u>Sample defaults</u> Tube <u>C</u> haracteristics	 ✓ Solid graph grid lines Dotted graph grid lines
System configuration	Show graph grid lines 🔶
Parameter files directory	
Service <u>T</u> est Mode	

Units

Displays the Unit Selections dialog box allowing you to choose the way in which certain criteria are to display.

Unit Selections			×
<u>A</u> mount Adsorbed	○ mmol/g	€ cm³/g STP	
<u>L</u> ength Unit	🖲 nm	⊖ Ă	
<u>B</u> JH Pore Dimension	• width	C diameter	C radius
<u>P</u> ressure Unit	⊖ kPa	C mbar	⊙mmHg
Pressure <u>S</u> ymbol	⊖р, р*	@ P, Po	
<u>T</u> emperature Unit	⊙к	O*C	
<u>A</u> nalysis Temperature	⊙ К	O*C	
	0 <u>K</u>	<u>C</u> ancel	

Solid graph grid lines Dotted graph grid lines	Allows you to choose whether to use solid or dotted lines when you choose to show graph grid lines.
	When you choose dotted graph grid lines, the screen out- put actually displays as solid; however, dotted lines dis- play when printed to a printer.
Show graph grid lines	The options on this cascading menu allow you to choose what type of lines to display on graphs, or you can choose to have none displayed.
	Choices: None, Horizontal, Vertical, Both

Sample Defaults

This option allows you to specify default parameters for sample information files. This option is presented in two formats: Advanced and Basic. Select **Option presentation** from the **Options** menu to choose the format you wish to use. Alternatively, you can switch between the two formats using the **Advanced** / **Basic** toggle push button displayed on the lower right side of the screen.

Basic

When you select **Sample Defaults** while using the *Basic* format, the Default Basic Sample Information dialog box is displayed.

Sample Information				
Seguence: 00	0-000			
Sample:				
<u>M</u> ass:	0.00 g <u>D</u> ensity: 0.00 g/cm ³			
Sample <u>t</u> ube:	Sample Tube			
Degas conditions:	Degas Conditions			
Analysis conditions:	Run Conditions			
Adsorptive properties:	Nitrogen 🔽			
Report options:	Report Options			
	Replace All Add Log Entry			
Save	Close Advanced			

Sequence

Specify a default sequence for the sample file name. The number you specify is sequenced incrementally each time you create a sample file. It is the number that appears in the **File name:** field when you select **File**, **Open**, **Sample information**.

- Use numbers, letters, or other printable characters, such as dashes. At least three numbers *must* be included.
- Use up to eight characters.
- DOS prohibits using such characters as * or ?. Refer to your DOS manual for more information.

Sample	Allows you to enter an additional identification that pro- vides more information than the sample file name alone.			
	In the field on the left, edit the prompt for <i>Sample</i> if you wish. For example, you may prefer to use <i>Test</i> or <i>Material</i> . The maximum number of characters is 20.			
	In the field on the right, specify a format for the sample identification.			
	• Use numbers, letters, or other printable characters, such as dashes.			
	• Maximum number of characters is 42, plus the \$ symbol.			
	• Include the automatically generated file name as part of the identification by using the \$ symbol where you want the sequence number to appear.			
	For example, if the sequence number is 000-001, en- ter the sample identification as follows:			
	Lab #25 - \$			
	The resulting sample identification for the first sam- ple information file would be:			
	Lab #25 - 000-001			
	and the second:			
	Lab #25 - 000-002, and so on.			
Mass	Enter a default value for the sample mass. Regardless of what weight you enter as the default, the value can be ed- ited in the sample information file.			
Density	Enter the density of the material you are analyzing.			

Sample Tube Degas Conditions Analysis Conditions Adsorptive Properties Report Options	Each parameter contains a drop-down list containing files of that type which were included with the software, as well as any you may have created. Choose the file you wish to use as the default.
Replace all	Allows you to replace all parameters of the default sample file you are creating with parameters from an existing sample file. For example, you may have an existing sample file that contains most (or all) of the parameters that you wish to use. You can click this push button, choose the file, and then $\bigcirc K$. All parameters of the file you choose are copied into the sample defaults dialog. You can even change the values in these files (if needed) without affecting the file from which they were copied.
Save	Saves the specified defaults.
Close	Closes the dialog.

Advanced

Sequence

When y	ou selec	t Sampl	e Def	faults	while	in 1	the A	Advance	d :	format,	the	Sample
Defaults	dialog	box with	the	Sampl	e Info	orma	ation	dialog	is	display	ed.	

🛅 Sample Defa	ults	_ 🗆 ×
≤< Prev Samp	ole Sample Degas Analysis Adsorptive Report natioi Tube Conditions Conditions Properties Options	Next >>
Sequence		
Sample:	\$	
Operator:		<u>□ 0</u> mit
Submitter:		🗖 Omi <u>t</u>
<u>M</u> ass:	: 1.0000 g Type of Data © A <u>u</u> tomatically collected	
Dens	ity: 1.000 g/cm³ C Manually <u>e</u> ntered	
Comments:		
	Add Log En	try
	Replace All	
Enter a value betw	veen 0.0010 and 500.0000.	
<u>S</u> ave	<u>Close</u> Basic	

Allows you to specify a default sequence for the sample file name. The number you specify is sequenced incrementally each time you create a sample file. It is the number that appears in the **File name:** field when you select **File**, **Open**, **Sample information**.

- Use numbers, letters, or other printable characters, such as dashes. At least three numbers *must* be included.
- Use up to eight characters.
- DOS prohibits using such characters as * or ?. Refer to your DOS manual for more information.

SampleAllows you to enter an additional identification that provides more information than the sample file name alone.

In the field on the left, you can edit the prompt for *Sample* if you wish. For example, you may prefer to use *Test* or *Material*. The maximum number of characters is 20.

In the field on the right, specify a format for the sample identification.

Sample (continued)	• Use numbers, letters, or other printable characters, such as dashes.
	• Maximum number of characters is 42, plus the \$ symbol.
	• Include the automatically generated file name as part of the identification by using the \$ symbol where you want the sequence number to appear.
	For example, if the sequence number is 000-001, en- ter the sample identification as follows:
	Lab #25 - \$
	The resulting sample identification for the first sam- ple information file would be:
	Lab #25 - 000-001
	and the second:
	Lab #25 - 000-002, and so on.
Operator Submitter	Allow you to enter the name (or other identification) of the operator performing the analysis and the name of the person, department, customer, etc. that submitted the sam- ple for analysis.
	The fields on the left can be edited to display something other than Operator and Submitter if you like.
	The fields on the right allow you to specify default names or titles.
	• Include the automatically generated file name as part of the identification by using the symbol \$ where you want the sequence number to appear. Refer to the example in the previous section.
	• Maximum number of characters for the prompt is 20.
	• Maximum number of characters for the identification is 42, including the \$ symbol.
	• Omit this item entirely from the sample information file by selecting Omit .

Mass	Enter a default value for the sample's mass. Regardless of the value you enter in this field, the value can be edited in the sample file.
Density	Enter a default density. If there is a material you analyze quite often, you may wish to use its default in this field. This value can be edited in the sample information file.
Comments	Allows you to enter comments about the sample or its analysis conditions. Anything you ener in this window is printed in the report header.
Add Log Entry	Allows you to enter comments about the sample or its analysis conditions. Anything you enter using this option appears in the Instrument Log Report; it does not display in the report header.
Replace all	Allows you to replace the contents of all parameter files contained in the current default sample file with those from an existing sample file. For example, you may have an existing sample file that contains most (or all) of the parameters that you wish to use. You can select this push button, choose the file, and then $\bigcirc K$. All parameter files of the file you choose are copied into the sample defaults dialog. You can even change the values in these files (if needed) without affecting the file from which they were copied.

Click on each remaining tab of the Advanced Sample dialog to establish parameter defaults. Refer to Chapter 5 if you need assistance on the fields of these dialogs.

System Configuration

This option allows you to specify printer and plotter destinations for report output; the System Configuration dialog box is displayed.

System Configuration
Printer name: HP LaserJet 4/4M PostScript (LPT1: 🛃 🗵 Default
Plotter name: HP LaserJet 4/4M PostScript (LPT1: 🛃 🗵 Default
O <u>K</u> Cancel

Printer name Plotter name

The printer and plotter names are shown exactly as they appear in the Operating System Print Manager.

Select **Default** if you wish to use the default printer and plotter as designated in the Print Manager.

If you wish to change the default printer, deselect **Default**. Then choose another printer (or plotter) from the drop-down list.

Parameter Files Directory

This option allows you to select the location for the predefined parameter files displayed in the drop-down lists on the Basic Sample Information dialog box.

When you select this option, the Parameter File Directory Selection dialog is displayed.

- Paramete	Parameter File Directory Selection				
Specify the location of the parameter files shown in the 'Basic-Mode' Sample presentation. Parameter files include Analysis Conditions and Report Options.					
Parameter file directory:	∴) * [-a-] * [-b-] - [-c-] - [-d-] -				
<u>OK</u>	Cancel				

The directory specified here is the one you should use when *creating* parameter files to be included in the drop-down lists on the Basic sample information dialog. It is where the software goes to obtain the files for the drop-down lists. If parameter files are not saved to this directory, they are not included in the lists.

Service Test Mode

Certain service tests are included in the TriStar operating program. These tests, however, 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, therefore, is known only by your service representative. You will be unable to perform these tests without his guidance.

After Service Test Mode has been enabled, the tests are accessible from the Unit menu.

CHAPTER 9

TROUBLESHOOTING AND MAINTENANCE

- Cleaning the Analyzer
- Replacing Port Frit and O-ring
- · Calibrating the System

TROUBLESHOOTING AND MAINTENANCE

The TriStar 3000 system has been designed to provide efficient and continuous service. However, certain maintenance procedures should be followed to obtain the best results over the longest period of time. This chapter includes maintenance procedures and calibration procedures.

Cleaning the Analyzer

The exterior casing of the analyzer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution.



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

Replacing Port Frit and O-ring

A 40- μ m frit is located in the analysis port of the analyzer. If the frit is contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated frit on the analysis port may be indicated by a leak or by a freespace reading much lower than normal.

A contaminated frit should be replaced as follows:

1. Remove the sample tube fitting from the analyzer using a wrench. Pry out the frit and O-ring.

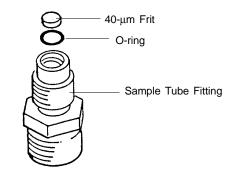


Figure 9-1. Analysis Port Frit and O-ring



To avoid degassing problems, the frit andO-ring should be clean and should not be touched with bare hands.

2. Replace the frit and O-ring. Carefully reassemble the sample tube fitting and tighten by hand. Then tighten with a wrench to prevent leaks.

Calibrating the System

A calibration file was created specifically for your analyzer and included with your accessories. Chapter 2 describes how to install this file. It is unnecessary to recalibrate your system unless you suspect that it may be out of calibration.

You may wish to recalibrate your system if:

- analysis on a known reference material or a blank tube gives unexpected results
- the transducers have been serviced

You can review calibration details of your analyzer by selecting Unit configuration on the Unit menu.

If it becomes necessary to recalibrate your system, the following order is recommended:

- Zero pressure
- Pressure scale
- Servo valve
- Temperature
- Volume

Zero pressure

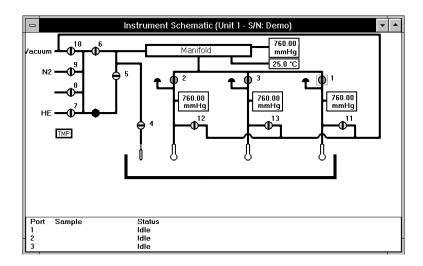
- 1. Install a blank sample tube on each port.
- 2. From the **Unit** menu, select **Calibration**, **Zero pressure**; the Calibrate Pressure Offset dialog is displayed.

Calibrare Pressure Offset (Unit 1 - S/N: test)	•
 Manifold Transducer Port 1 Transducer Port 2 Transducer Port 3 Transducer Port 3 Transducer After selecting the transducers, press the start button. This will evacuate the system and then zero the pressure transducers. Warning: The selected ports must have sample tubes attached.	
<u>Start</u>]

3. Ensure that all transducers are selected, then click **Start**. The dialog closes automatically when calibration is complete.

Pressure scale

- 1. Install a pressure reference on one port and sample tubes on the other two ports.
- 2. From the **Unit** menu, select **Enable manual control** (if the instrument schematic is not displayed, select **Show instrument schematic**).
- 3. Ensure that valves 1, 2, and 3 are open and the system is at a stable pressure near atmosphere.





In rare cases, calibrations may become corrupt so that manual control of the instrument is inoperative. If this occurs, select *Reset pressure calibration* from the Unit menu to restore factory defaults. Then proceed with the calibration.

4. From the **Unit** menu, select **Calibration**, **Pressure scale**; the Calibrate Pressure Scale dialog is displayed.

Calibrate Pressure Scale (Un	iit 1 - S/N: Dem	D) 🗵
Actual manifold pressure	760.00	mmHg
🛛 Manifold <u>T</u> ransducer		
Port <u>1</u> Transducer		
Port <u>2</u> Transducer		
Port <u>3</u> Transducer		
0 <u>K</u>	<u>C</u> ance	
Enter a value between 500.00 and	1000.00.	

5. Enter the pressure obtained from the reference gauge in the Actual manifold pressure field.

Servo valve



Make sure the pressure transducer has been calibrated before performing this procedure.

1. From the **Unit** menu, select **Calibration**, **Servo valve**; the Calibrate Servo Valve dialog is displayed.



2. Click **Start**; the dialog closes when calibration is complete.

Temperature

1. Remove the retaining screws and remove the top cover from the analyzer.

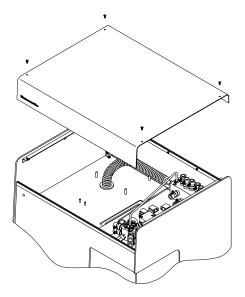


Figure 9-1. Removing the Top Cover

2. Locate the dosing manifold and insert the calibration device into the hole next to the RTD (resistance thermal device).

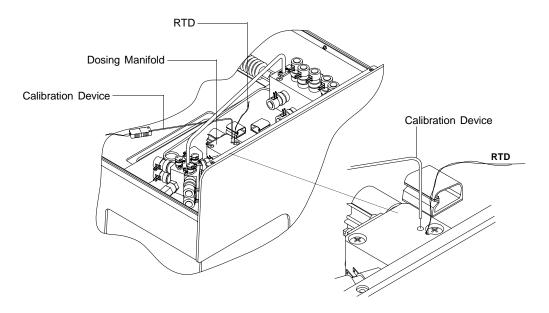


Figure 9-2. Inserting the Calibration Device

- 3. Wait until the temperature stabilizes (approximately 1 to 2 minutes).
- 4. From the **Unit** menu, select **Calibration**, **Temperature**; the Calibrate Manifold Temperature dialog is displayed.

Calibrate Manifold Temperature (Unit 1 - S/N: test)					
Current manifold temperature	25.63 °C				
<u>ок</u>	Cancel				
Enter a value between 10.00 and 100.00.					

5. Enter the temperature obtained using the calibration device, then click **OK**.

Volume



If you wish to use the previous volume or enter one manually, skip steps 1 and 2 and and begin at step 3.

1. Install the calibration volume onto ports 1 and 2.

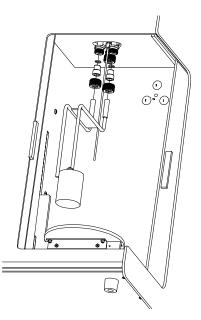


Figure 9-3. Installing the Calibration Volume

- 2. Insert the volume portion into an ice bath.
- 3. From the **Unit** menu, select **Calibration**, **Volume**; the Calibrate Volume dialog is displayed.

	Calibrate Volume (Unit 1 - S/N: test)	-
	Previous 8.0000 cm ³	
	C Entered 18.0000 cm ^s	
	O Measured	
Displays as Next>> if		
you choose Measured	<u>D</u> one	<u>C</u> ancel
as the method.		

- 4. Choose the method you wish to use for calibrating the volume.
 - **Previous**: simply click **Done** to close the dialog box; the previous volume is used.
 - Entered: enter the volume you wish to use, then click **Done** to close the dialog box.
 - Measured: click Next >> ; a second view of the Calibrate Volume dialog is displayed.

😑 Calibrate Volume (Ur	nit 1 - S/N: Demo) 🔹 🔺			
Reference Volume:	50.0000 cm ^a			
Reference Volume Temp.	0.00 *C			
Perform measurement:	3 🌲 times			
Thermal equilibration time:	10 🖨 minutes			
Reference volume must be attached to ports 1 and 2.				
Start	<u>C</u> ancel			

- 5. Enter the volume of the calibration volume device.
- 6. Ensure that the temperature is 0.00 ± 0.10 °C.
- 7. Select (or enter) the number of measurements you wish to make; generally, 3 is sufficient.
- 8. Select (or enter) an equilibration time; generally, 10 minutes are sufficient.
- 9. Click **Start** to begin calibration; an informational dialog displaying calibration status is displayed. After calibration is complete, a final view of the Calibration Volume dialog is displayed.

- Calibrate Volu	me (Unit 1 - S/N: test)	•
Previous	16.9000 ^{cm³} 16	.9000
O Entered	18.0000 cm ³	
O Measured	17.2380 cm ³	
O Average	17.1908 cm [*]	
Last	9 🖉 measurements	
Std.Dev.	0.0058 cm ³	
Done		ancel

10. Confirm that the value is acceptable and click **Done** to close the dialog.

CHAPTER 10

ORDERING INFORMATION

ORDERING INFORMATION

The TriStar 3000 Analysis System components and accessories can be ordered through our Customer Order Entry Department - (770) 662-3636. When ordering, please use the information provided in the following table.

Part Number	Item and Description
065-00000-00	SmartPrep 065, Windows interface allowing programmable ramp and soak rates; degasses up to six samples with flowing gas
060-00030-00	FlowPrep 060, degasses up to six samples at up to 400 °C with flowing gas
061-00030-00	VacPrep 061, degasses up to six samples at up to 400 °C; uses flowing gas or evacuation by vacuum (evacuation requires a vacuum pump and a regulator)
062-00000-11	Vacuum pump with built-in anti-suckback valve, 100/120 VAC, includes hose kit
062-00000-23	Vacuum pump with built-in anti-suckback valve, 220/240 VAC, includes hose kit
062-33002-00	Activated alumina oil vapor trap
004-16830-00	Activated alumina, 500 grams; for oil vapor trap
003-22617-00	Valve, solenoid 24 VDC 10-32 Mt, 0.025-in. orifice, for standard evacuation (Valves 1 through 10)
003-22626-00	Valve, solenoid 24 VDC 10-32 Mt, 0.060-in. orifice, for fast evacuation (Valves 11, 12, and 13)
004-62230-58	CGA 580 fitting, 30 psig (He, N ₂)
004-62230-32	CGA 320, 30 psig (CO ₂)
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure Relief Kit; prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
004-16833-00	Reference Material, Carbon black, SA~23.8 m ² /g
004-16821-00	Reference Material, Silica alumina, SA~215 m ² /g, PV~0.6 cc/g
003-20665-00	Cable, M/F 9-pin D 6 ft. straight thru

Table 10-1. TriStar 3000 System Components and Accessories

Part Number	Item and Description
004-25549-00	Reducer, 1/8-in. tube x 1/4-in. tube
230-02001-00	Gas inlet line
240-14855-00	Rack, sample tube
240-25853-00	Funnel, sample tube
240-25901-00	Dip stick
300-33601-00	Sample Tube Kit, 1/4-in; includes sample tubes, isothermal jackets, filler rods, ferrules, O-rings, caps, and dewar cover
300-33602-00	Sample Tube Kit, 3/8-in; includes sample tubes, brush (for cleaning sample tubes), isothermal jackets, filler rods, ferrules, O-rings, stoppers, and dewar cover
300-33603-00	Sample Tube Kit, 1/2-in; includes sample tubes, brush (for cleaning sample tubes), isothermal jackets, filler rods, ferrules, O-rings, stoppers, and dewar cover
300-61001-00	Sample tube, 1/4-in. stem OD; for analyzing powdered samples where free-space management (with a filler rod inserted) is critical
300-61002-00	Sample tube, 3/8-in. stem OD; for analyzing any type of sample
300-61003-00	Sample tube, 1/2-in. stem OD; for analyzing larger pieces of granular or pelleted samples
300-25901-00	Isothermal jacket, for sample tube
300-25902-00	Isothermal jacket, for 3/8-in. sample tube
300-25903-00	Isothermal jacket, for 1/2-in. sample tube
300-25904-00	Filler rod assembly, for 1/4-in. sample tube
300-25905-00	Filler rod assembly, for 3/8-in. sample tube
300-25906-00	Filler rod assembly, for 1/2-in. sample tube
004-25466-00	O-Ring, for sample port and 1/4-in. sample tube
004-25022-00	O-Ring, for 3/8-in. sample tube
004-25469-00	O-Ring, for 1/2-in. sample tube
300-31802-02	Dewar cover, for 1/4-in. and 3/8-in. sample tubes
300-31802-03	Dewar cover, for 1/2-in. sample tube
004-54104-00	Brush, for cleaning 3/8-in. sample tubes
004-54609-00	Brush, for cleaning 1/2-in. sample tubes

Part Number	Item and Description
300-25825-00	Ferrule, for 1/4-in. sample tube
300-25826-00	Ferrule, for 3/8-in. sample tube
260-25843-00	Ferrule, for 1/2-in. sample tube
004-27046-00	Frit, 40 µm, for sample port
240-32004-00	Stopper, rubber, for 3/8- and 1/2-in sample tubes
004-32604-08	Cap, plastic, for 1/4-in sample tube
300-32800-00	Support, sample weighing
300-34008-00	Vacuum pump tray
300-25861-20	Dewar
300-42832-00	Operator's manual, TriStar 3000
003-20690-00	USB Expander kit, for connecting multiple instruments to a single computer
202-33023-00	DFT Plus Program; allows you to use the total experimental isotherm to determine microporosity and mesoporosity as a continuous distribution of pore volume with respect to pore size
202-33024-00	DataMaster Program; allows you to generate classical or micropore data reductions

APPENDIX A

FORMS

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Sample Data Worksheet TriStar 3000

Use this worksheet to record the values necessary to calculate the sample mass (refer to **Weighing the Sample** in Chapter 4).

Sample Tube Identification:_____

Sample Mass

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

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Sample Tube Calibration Worksheet TriStar 3000

Use this worksheet to record the values necessary to calibrate a sample tube which is to be used in an analysis where the free space is to be determined by the quasi-Gemini or calculated method.

Sample Tube Identification:_____

Water Weight

Note: Record all weights in grams.

- A. Empty sample tube weight:
- B. Sample tube plus water:
- C. Weight of the water (B A):

Physical Volume

- D. Water temperature:
- E. Water density (refer to table):

Temperature (°C)	Density (g/cm ³)	Temperature (°C)	Density (g/cm ³)
15	0.9991016	23	0.9975412
16	0.9989450	24	0.9972994
17	0.9987769	25	0.9970480
18	0.9985976	26	0.9967870
19	0.9984073	27	0.9965166
20	0.9982063	28	0.9962371
21	0.9979948	29	0.9959486
22	0.9977730	30	0.9956511

Physical volume below the red line (C \div E):

APPENDIX B

ERROR MESSAGES

ERROR MESSAGES

Error messages for the 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, contact your service representative after making backup copies of any files involved in the operation.

- 2401- FATAL ERROR: (error message)
- 2402- MMDevice- Invalid state seen in T/O table.
- 2403- MMDevice- Invalid state seen in ERR table.
- 2404- MMDevice- Invalid state seen in DONE table.
- 2405- MMDevice- Invalid CmdType in DONE table.
- 2406- MMDevice- Invalid I/O State returned.
- 2407- MMDevice- CommandManager failed.
- 2408- MMDevice- XModem failed.
- 2409- MMDevice- SerialComm failed.
- 2410- MMDevice- Invalid UserID returned.
- 2411- MMDevice- Invalid state reached. cannot continue.
- 2412- MMDevice- Time-out. Canceling operation.
- 2413- MMDevice- Time-out during block receive. Retrying.
- 2414- MMDevice- Error. State was (number).
- 2415- CommandMgr- Invalid state reached. cannot continue.
- 2416- CommandMgr- Time-out. State was (number).
- 2417- CommandMgr- Parity or overrun error in receive.
- 2418- CommandMgr- Bad ACK received.
- 2419- CommandMgr- MICIC reports a command receive error: (number).
- 2420- CommandMgr- Unknown ACK (number number number) vs (number number number).
- 2421- CommandMgr- 'Last ACK' received from MICIC.
- 2422- XModem- Invalid state reached. cannot continue.
- 2423- XModemSender- Invalid state reached. cannot continue.
- 2424- XModemRcvr- Invalid state (number). cannot continue.
- 2425- XModemRcvr- Time-out. State was (number).
- 2426- XModemRcvr- Error. State was (number).
- 2427- XModemRcvr- Packet received out of sequence.
- 2428- XModemRcvr- Packet failed CRC validate.
- 2429- XModemRcvr- Duplicate packet received.
 - *Cause:* An internal processing and/or hardware error has occurred during communication with the analyzer.
 - *Action:* Contact your service representative if you continue to receive this message.

2430- Error accessing file (file name), error code = (number).

- Cause A: Diskette may be damaged.
- Action A: Clean the diskette drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.
- Cause B: Hard disk may be damaged.
- Action B: Contact your service representative.
- Cause C: A software error occurred when the file was accessed.
- Action C: Contact your service representative.
- Cause D: The name specified contains one or more invalid characters.
- Action D: Enter a valid DOS filename (refer to your operating systems manual).

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 diskette, 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 TriStar 3000 files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the TriStar 3000 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 wrong amount of data.

- *Cause:* An internal processing and/or hardware error has occurred.
- *Action:* Contact your service representative if you continue to receive this error message.

2435- SerialComm Rcv Time-out — Expected (number) | Rcv'd (number).

- *Cause:* An internal processing and/or hardware error has occurred during communication with the interface controller.
- *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.
- 2439- Could not register file.
- 2440- Subset not found.
- 2441- Seek within file failed.
- 2442- Bad header in subset file.
- 2443- Subset owner denied access.
- 2444- Not a valid file format.
- 2445- Subset wrote the wrong amount of data.
- 2446- Error reading data.
- 2447- Error writing data.
 - Cause: An unexpected error occurred when you tried to access a data file.
 - Action: Contact your service representative.

2448- File directory (path name) is invalid. Resetting to the installation directory.

- *Cause:* A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.
- Action: The installation directory will be substituted. The next time you open a file, use the **Directories:** list to move to the correct directory.

2449- This field does not contain a valid file specification.

- *Cause:* You entered an invalid file name.
- *Action:* See the description of file naming conventions in your DOS or Windows manual and re-enter the name.

2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.

- *Cause:* You are attempting to exit the application while the instrument is performing a critical operation. This operation must be completed before the application can be stopped.
- Action: Wait a short time and attempt to stop 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? (Yes, No)

- *Cause:* You are attempting to exit the application while an analysis is in progress. While this is possible, the data collected while the application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.
- *Action:* If you are not concerned with the potential for loss of data should a power failure occur, select **Yes** to continue; otherwise select **No**.

2460- Fatal Communications error on (Unit n - S/N: nnnn)

- *Cause:* There was a fatal error in the serial communications between the application and the software in the instrument. All displays for that instrument will be closed.
- *Action:* Ensure that the analyzer is connected to the computer on the communications port configured in the Setup program. Stop the TriStar 3000 software and then restart it. If this error persists, contact your service representative.

2461- No instruments are in operation. This application will unconditionally terminate.

- *Cause:* At least one instrument must be active for the application to operate. The initialization of all of the instruments 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 instrument's failure to initialize. See the instructions for that message.
- Action B: Ensure that the instrument is attached to the computer on the communications port configured with the Setup program. Verify that the instrument 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 instrument, contact your service representative.

2462- Non-Fatal timer error on Unit n - S/N: nnnn.

- *Cause:* The software has detected a non-fatal error in the timer.
- *Action:* One of the instrument's internal components has failed. Contact your service representative.

2463- Non-Fatal DMA error on Unit n - S/N: nnnn.

- *Cause:* The software has detected a non-fatal error in the analyzer's Direct Memory Access component.
- *Action:* One of the instrument's internal components has failed. Contact your service representative.

2464- Non-Fatal memory bank 3 error on Unit n - S/N: nnnn.

- *Cause:* The software has detected a non-fatal error in the analyzer's memory bank 3.
- *Action:* One of the instrument's internal components has failed. Contact your service representative.

2465- Non-Fatal memory bank 4 error on Unit n - S/N: nnnn.

- *Cause:* The software has detected a non-fatal error in the analyzer's memory bank 4.
- *Action:* One of the instrument's internal components has failed. Contact your service representative.

2466- Unit n - S/N: nnnn has no interface card and cannot be initialized.

- *Cause:* The software has detected that the analyzer's electronic components cannot support the software.
- Action: Contact your Mircromeritics Service Representative.

2467- Not enough memory error on Unit n - S/N: nnnn

- *Cause:* The software has detected insufficient memory in the instrument for the software to execute properly.
- *Action:* One of the instrument's internal components has failed. Contact your service representative.

2468- The instrument contains a different software version. Do you want to reset it?

- *Cause:* The application has discovered that a different version of software is already running on the instrument.
- Action: If there is no chance that some instrument other than the TriStar 3000 is connected to the instrument, select **Yes** to reset the instrument and download the proper software. Otherwise, select **No** to leave the instrument unchanged.

2469- The instrument software did not initialize properly on Unit n - S/N: nnnn.

- Cause: The software in the instrument failed to execute properly.
- Action: Reinstall the TriStar 3000 software, then restart it. If the problem persists, contact your service representative.

2470- Unable to read instrument software file for Unit n - S/N: nnnn.

- *Cause:* The application tried to read the instrument software file to download it to the instrument. It was unable to do so.
- Action: Reinstall the TriStar 3000 software, then restart it.
- 2471- Unit n S/N: nnnn has an invalid communications port specified. It cannot be initialized.
- 2472- Unit n S/N: nnnn communications port specified in the program control files is not a valid device. Unit cannot initialize.
- 2473- Unit n S/N: nnnn communications port specified in the program control files is not physically installed in the Control Module. Unit cannot initialize.
 - *Cause:* The communications port associated with this unit was not valid.
 - *Action:* Run the Setup program, specifying a valid port for this unit, when you are prompted to do so.

2474- Unit n - S/N: nnnn communications port specified in the program control files is already in use. Unit cannot initialize.

- *Cause A:* The communications port associated with this instrument is in use by some other program in the system.
- Action A: Close the other program to release the port. Restart the TriStar 3000 software.
- *Cause B:* The component associated with this unit is also being used by the SmartPrep.
- Action B: Run the Setup program and specify different communication ports for each TriStar and the SmartPrep.
- 2475- Unit n S/N: nnnn communications port specified in the program control files cannot be accessed. Unit cannot initialize.
- 2476- Unit n S/N: nnnn startup failed. Could not initialize the communications port.
 - Cause: The communications port associated with this unit was not valid.
 - *Action:* Run the Setup program, specifying a valid port for this unit, when you are prompted to do so.

2477- Unit n - S/N: nnnn did not properly initialize.

- *Cause:* The software was unable to initialize this instrument; this is usually caused by one of the conditions listed in the error messages above.
- Action: Correct the problem as described above, then restart the application.

2480- File <name> cannot be analyzed. It is currently being edited.

- *Cause:* You are trying to start an analysis using a file that is open for editing.
- Action: Finish editing the file, save and close it, then start the analysis.

2481- Error accessing the sample information file <name>.

- Cause: An unexplained error prevents you from accessing this file.
- Action: The hard disk drive may be corrupt. Run diagnositics.

2484- The edit session for <name> must be saved before the analysis. Save changes and continue with the analysis? (yes, no)

- *Cause:* You are trying to start an analysis using a file that contains unsaved changes and is open for editing.
- Action: Select Yes to save the changes and proceed with the analysis; select No to cancel the analysis and continue editing the Sample Information file.

2486- Could not construct (name) report type. Program will terminate.

- Cause A: You may not have full rights to the application's folders and files.
- Action A: Contact your system administrator to have full rights granted.
- 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.

2487- Could not start report generator. Error code (number). Program will terminate.

- Cause: An internal processing and/or hardware error has occurred.
- *Action:* 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 TriStar 3000 Setup program, select **Change analyzer** setup, and create the control file used by the TriStar 3000.

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.

- *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.

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 a value within the indicated range.

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- 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 **Sample defaults** from **Options** on the Main Menu and enter another 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.

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) cancelled.

- Cause: The requested print job was canceled at your request.
- Action: None required.

4000- Memory Allocation Error. 4001- Error Smoothing Data.

- *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:* Deselect this option to disable this message. This correction is only meaningful for very low pressures.

4003- Error Converting Pressures.

4004- Error Computing Volume Adsorbed.

- *Cause:* An internal processing and/or hardware error occurred during report generation.
- *Action:* Contact your service representative if you continue to receive this error message.

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 box. 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 collective data and restart the report.

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.

4015- Error creating export file for sample <sample file name>.

- Cause: A file error occurred during creation of an export output file.
- Action: The output file name may be invalid. Ensure that the target directory exists. Ensure that the target diskette is not full or write protected. The target disk drive may be damaged or inoperative. Verify that other files may be created on the same drive. Contact your service representative if you continue to receive this error message.

4016- Sample <sample file name> has no data for export to ISO/DFT.

- *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.

4023- The file <name> cannot be prepared for analysis. It is open for editing and contains errors.

- *Cause:* You are trying to start an analysis using a file that contains errors and is open for editing.
- Action: Finish editing this file, save and close it, then start the analysis.

4400- The communications port specified for the SmartPrep(s) is not physically installed in the Control Module. Cannot initialize.

- Cause: The communications port associated with this unit was not valid.
- Action: Run the setup program and set up the unit on a valid port.

4401- The communications port specified for the SmartPrep(s) is already in use. Cannot initialize.

- *Cause:* The communications port associated with this instrument is in use by some other program in the system.
- *Action:* Close the other program to release the port. Restart the TriStar 3000 application.

4402- The communications port specified for the SmartPrep(s) cannot be accessed. Cannot initialize.

- 4403- Cannot communicate with SmartPrep Unit %d S/N: %s.
 - *Cause:* The communications port associated with this unit was not valid.
 - Action: Run the setup program and set up the unit on a valid port.

4404- The application version of the SmartPrep Unit n - S/N: nn is invalid.

- *Cause:* The controller software running on the designated instrument is invalid.
- *Action:* Use the SmartPrep setup program to download the proper controller software to the instrument, or contact a service representative if unavailable.

4405- Fatal communications error with SmartPrep Unit n - S/N: nn.

- *Cause:* There was a fatal error in the serial communications between the application and the SmartPrep Instrument Controller. All displays for that SmartPrep will be closed.
- Action: Ensure that the SmartPrep is properly chained to the computer on the communications port configured in the Setup program. Stop and re-start the application. If this error persists, contact your service representative.

6000- An error occurred while loading the application control information. Data entry cannot be performed. (Code number)

- *Cause:* An error occurred accessing the control information disk file required by this application.
- *Action:* The disk drive may have failed or be corrupt. Run diagnostics on the disk drive.

6003- Unable to read the calibration file (number).

- *Cause:* Your attempt to load a previously saved calibration file was unsuccessful.
- *Action:* Make sure the file exists, and that you have entered the file name correctly, then try again.

6004- Unable to write the calibration file (number).

- *Cause:* Your attempt to save the calibration to a separate file was unsuccessful.
- Action: Make sure that the disk is neither full nor write-protected, then try again.

6005- The sample has an invalid status and cannot be used for this analysis.

- *Cause:* You selected a sample file with a status other than **No Analysis** or **Prepared**.
- Action: Select a sample file with a status of **No Analysis** or **Prepared**.

6006- Cannot do quasi-gemini freespace with sample selected on port 2.

- *Cause:* You attempted to select a file requesting Quasi-Gemini free space for analysis, but have already selected a sample for port 2. In Quasi-Gemini free space, port 2 is used for the balance tube and cannot have a sample selected.
- Action: Select a file that does not request Quasi-Gemini free space or clear the sample file on port 2 and reselect the file.

6007- Cannot do Po measurement in sample tube with sample selected on port 3.

- *Cause:* You attempted to select a file requesting Po in Port 3 measurement, but have already selected a sample for port 3.
- *Action:* Select a file that does not request Po in port 3 measurement or clear the sample file on port 3 and reselect the file.

6008- At least one sample must be selected to proceed.

- *Cause:* You attempted to start an analysis without selecting any sample files.
- Action: Select at least one file, then start the analysis.

6009- You must select a sample tube on port 2 to do a Quasi-Gemini analysis.

- *Cause:* You attempted to start an analysis requesting Quasi-Gemini freespace, but have not selected the sample tube file for the balance tube on port 2.
- Action: Select a sample tube file for port 2 describing the balance tube used.

6010- This sample requires a different adsorptive and cannot be analyzed at the same time as the other samples.

- *Cause:* You selected a sample file for analysis that requires a different adsorptive gas than the sample file already selected.
- *Action:* Select only sample files to be analyzed with the same adsorptive and perform the analysis. Then perform the analysis with the other adsorptive.

6011- The adsorptive required by this analysis is not available on this instrument.

- *Cause:* You attempted to start an analysis with an adsorptive that is not connected to the instrument.
- Action A: Make sure that the adsorptive is connected to the instrument, and then select the unit configuration item of this unit's menu to tell the application that the gas is connected.
- Action B: Select only sample files for which the analysis gas is available.

- 6012- Cannot read the analysis conditions parameter file.
- 6013- Cannot read the adsorptive properties parameter file.
- 6014- Cannot read the report options parameter file.
- 6015- Cannot read the sample tube properties parameter file.
 - *Cause:* The parameter set you selected on the QuickStart opening screen cannot be read.
 - Action: Close ands restart QuickStart analysis and reselect the parameters from the available set.

6016- Dosing manifold from valve (number) failed.

- *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.

6017- Leak test failed on port (number).

- *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.

6019- Elevator failed to reach upper/lower limit switch.

- *Cause A:* The maximum time for the analysis was exceeded before the elevator reached the UP position. Ice may be present in the bottom or the neck of the dewar.
- Action A: Check the dewar and remove ice if necessary. 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.

6020- Warning, servo valve performance is out of specification.

- *Cause:* The servo valve tried to dose to a pressure but was unable to reach it within specification. The analysis will continue.
- *Action:* At the next appropriate time, calibrate the servo valve to bring it back within specification (refer to Chapter 9).

6021- Servo calibration failed.

- *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.

6022- This file already selected for the analysis.

- *Cause:* You tried to choose a file for this analysis which has been selected for another analysis.
- Action: Choose a different file.

6023- The instrument (number) is not calibrated.

- *Cause:* The TriStar application is in the process of initializing the instrument and has noticed that no calibration information is available for that unit.
- Action: Click OK . When the instrument has initialized, open the calibration screen and calibrate the instrument.

6024- Evacuation failed.

- *Cause:* While attempting to zero the pressure transducers, the instrument was unable to evacuate to a pressure of less than 1 mmHg. This may be due to a leak or a bad calibration.
- Action A: Check the sample tube fitting and ensure that it is securely attached to the port.
- Action B: Calibrate the pressure offsset. If this calibration fails because the system cannot be evacuated, select Reset pressure calibration from the Calibration menu to restore the factory calibration settings. This will overwrite the current calibration settings.

6026- Psat gas is not condensing.

- Cause A: The working Dewar does not contain enough bath liquid.
- Action A: Refill the Dewar and try the operation again.
- Cause B: The Psat gas is contaminated.
- Action B: Replace the Psat gas supply.
- Cause C: The Psat tubing from the regulator to the instrument is contaminated.
- Action C: Pump out the tubing.

APPENDIX C

CALCULATIONS

CALCULATIONS

This appendix contains the calculations used in the TriStar program.

Saturation Pressure (Po)

Po method is selected by the user on the Po and Temperature Options dialog box. It may be entered or measured in the Po tube or on port 3.

- If entered, Po = user-entered value
- If measured, Po = equilibrated pressure reading after saturating Po tube or the sample tube

Relative Pressure Calculations

If Po was measured at intervals in the Po tube and several data points were collected in the interval:

• For points taken before the previous Po measurement,

$$Po_{I} = Po_{1} + \frac{(Po_{2} - Po_{1}) \times (T_{I} - To_{1})}{(To_{2} - To_{1})}$$

• For points taken after the previous Po, use the previous Po:

Po
$$_{I} = Po_{1}$$

If Po was entered, use the entered Po:

 $Po_I = Po_E$

Calculate relative pressure for the Ith data point:

$$P_{REL_{I}} = \frac{P_{I}}{Po_{I}}$$

Po _I	=	saturation pressure for the I th data point (mmHg)
Po ₁	=	most previous measured saturation pressure before Ith data
		point (mmHg)
Po ₂	=	first measured saturation pressure after the Ith data point (mmHg)
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}	=	relative pressure for the I th data point (mmHg)
P _I	=	absolute pressure for the I th data point, taken at equilibrium (mmHg)

Volume Adsorbed Calculations

Free Space

The free space can be measured, calculated, entered, or obtained using the Quasi-Gemini method. The quantities used for the data reduction are the warm free space (V_{FW}) , the cold free space (V_{FC}) , and the analysis bath temperature.

Measured, Calculated, and Entered

For **measured** free space, the cold and warm free space volumes are calculated as follows:

$$\begin{split} V_{FW} &= \frac{V_{MAN}}{T_1} \ x \left[\frac{P_1}{P_2} - 1 \right] x \ T_{STD} \\ V_{FC} &= \frac{V_{MAN}}{T_2} \ x \left[\frac{P_1}{P_3} - 1 \right] x \ T_{STD} \end{split}$$

For **calculated** free space, the cold and warm free space volumes are calculated in three steps.

First, the sample volume is determined:

$$V_{SAMP} = \frac{W_{SAMP}}{D_{SAMP}}$$

Second, the warm and cold portions of the free space are determined:

• With a filler rod:

$$V_{FWP} = (V_{PORT} + V_{STEMWARM}) x \frac{T_{STD}}{T_{MAN}}$$

$$V_{FCP} = (V_{BELOW} + V_{STEMCOLD} - V_{SAM}) \times \frac{T_{STD}}{T_{ANLS}}$$

• Without a filler rod:

$$V_{FWP} = (V_{PORT} + V_{STEMWARM} + V_{RODWARM}) \times \frac{T_{STD}}{T_{MAN}}$$

$$V_{FCP} = (V_{BELOW} + V_{STEMCOLD} - V_{SAMP} + V_{RODCOLD}) x \frac{T_{STD}}{T_{ANLS}}$$

Third, the warm and cold free space volumes are calculated:

$$V_{FC} = V_{FWP} + V_{FCP}$$
$$V_{FW} = V_{FWP} + V_{FCP} x \frac{T_{ANLS}}{T_{ROOM}}$$

The volume of the analysis bath temperature is calculated as follows:

$$V_{ABT} = \frac{V_{FC} - V_{FW}}{1 - \frac{T_{ABT}}{T_{RM}}}$$

D _{SAMP}	sample density (g/cm ³)
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_1	system manifold temperature before dosing helium onto sample (K)
T_2	system manifold temperature after raising Dewar and
	equilibrating with helium (K)
T_{ABT}	analysis bath temperature (K), from the Po and Temperature
	Options dialog box
T _{ANLS}	analysis temperature (K)
T _{RM}	approximate room temperature (298 K)
T _{STD}	standard temperature (273.13 K)
V_{ABT}	portion of cold free space at analysis bath temperature:
	used in non-ideality correction (cm ³ at standard temperature)
V _{BELOW}	volume of the sample tube below the red line
V _{FC}	volume of cold free space (cm ³ at standard temperature)
V _{FCP}	cold portion of free space (cm ³ at standard temperature)
V_{FW}	volume of warm free space (cm ³ at standard temperature)
V _{FWP}	warm portion of free space (cm ³ at standard temperature)
V _{MAN}	= manifold volume (cm ³)
VPORT	sample port volume (cm ³)

$$N_{GAS_{I}} = N_{GAS_{I-1}} + V_{MAN} x \left[\frac{P_{SYS1_{I}}}{T_{SYS1_{I}}} - \frac{P_{SYS2_{I}}}{T_{SYS2_{I}}} \right] x \frac{T_{STD}}{P_{STD}}$$

 $N_{\textbf{ADS}_{I}} \ = \ N_{\textbf{GAS}_{I}} \ - \ P_{\textbf{SAMP}_{I}} \ \div \ P_{\textbf{STD}} \ \textbf{x} \ (V_{FC} \ + \ P_{\textbf{SAMP}_{I}} \ \textbf{x} \ V_{ABT} \ \textbf{x} \ C)$

$$V_{I} = \frac{N_{ADS_{I}}}{W_{SAMP}}$$

С	=	nonideality correction factor, user-entered on Adsorptive
		Properties dialog box
N _{ADSI}	=	amount of gas adsorbed after equilibrating Ith dose (cm ³ STP)
NGASI	=	total amount of gas dosed into sample tube after Ith dose (cm ³ STP)
P _{STD}	=	standard pressure (760 mmHg)
P _{SYS1}	=	system manifold pressure before Ith dose of gas onto
		sample (mmHg)
P_{SYS2_I}	=	system manifold pressure after Ith dose of gas onto
1		sample (mmHg)
P _{SAMP1}	=	sample pressure after equilibrating I th dose of gas onto
1		sample (mmHg)
T _{STD}	=	standard temperature (273.13 K)
T_{SYS1_I}	=	system manifold temperature before Ith dose of gas onto
1		sample (K)
T_{SYS2_I}	=	system manifold temperature after Ith dose of gas onto
I		sample (K)
V _{ABT}	=	portion of cold free space at analysis bath temperature:
		used in non-ideality correction (cm ³ at standard temperature)
V _{FC}	=	volume of cold free space (cm ³ at standard temperature)
V _{MAN}		manifold volume (cm ³)
WSAMP		weight of sample (g)
VI	=	
1		\mathcal{C}

Quasi Gemini

The quantity adsorbed, $N_{\mbox{\rm ADS}_i},$ is calculated as follows:

$$N_{ADSI} = N_{DOSEDI}$$
 - ComputedColdSampleGas - BlankPressureCorrection x
(N_{BLANKI} - ComputedColdBlankGas)

ComputedColdSampleGas =

$$\begin{bmatrix}
\frac{P_{sam_{I}}}{P_{STD}} \times V_{FCP_{SAMP}} \times \left(\frac{R_{STD}}{T_{ANLS}}\right) \times (1 + P_{SAMP_{I}} \times C)$$

BlankPressureCorrection =
$$\frac{P_{SAMP_{I}} x (1 + P_{SAMP_{I}} x C)}{P_{BLANK_{I}} x (1 + P_{BLANK_{I}} x C)}$$

$$\begin{aligned} & ComputedColdBlankGas = \\ & \left[\frac{P_{BLANK_{I}}}{P_{STD}} x \; V_{FCP_{BLANK}} x \frac{T_{STD}}{T_{ANLS}} \right] x \; (1 + P_{BLANK_{I}} x \; C) \end{aligned}$$

N _{DOSED1}	=	total quantity dosed for the I th point
NBLANKI	=	volume dosed into the blank tube
PBLANK	=	pressure of the blank tube
P _{SAMP1}	=	pressure at which the equilibrated sample point was taken
P _{STD}	=	standard pressure (760 mmHg)
TANLS	=	analysis temperature (K)
T _{STD}	=	standard temperature (273.13 K)
V _{FCP_{BLANK}}	=	cold portion of free space for blank tube (see calculation for
DEAL		calculated free space)
V _{FCPSAMP}	=	Cold portion of free space for sample tube (see calculation for
57 1111		calculated free space)
VSAMP	=	sample volume

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-Golay1 ^convolution method for polynomial functions. The equations below are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

$$P_{AVG} = \frac{-36(P_1 + P_{11}) + 9(P_2 + P_{10}) + 44(P_3 + P_9) + 69(P_4 + P_8) + 84(P_5 + P_7) + 89(P_6)}{429}$$

$$P_{CHG} = \frac{5(P_1 - P_{11}) + 4(P_2 - P_{10}) + 3(P_3 - P_9) + 2(P_4 - P_8) + (P_5 - P_7)}{110}$$

$$P_{PCPI} = 100\% \frac{P_{CHG}}{P_{AVG}}$$

where the numerical constants are from the Savitzky-Golay convolution arrays, and

P _{AVG}	=	average pressure (mmHg)
P _{CHG}	=	change in pressure (mmHg)
P _{PCPI}	=	Percent change per interval
PI	=	Ith pressure reading taken at equilibrium intervals (mmHg)



If the value you enter for the maximum equilibration time is too small, 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.

BET Surface Area Calculations²

For each point designated for surface area calculations, the BET transformation is calculated as follows:

$$B_{I} = \frac{P_{rel_{I}}}{(1.0 - P_{rel_{I}}) \times N_{ads_{I}}}$$

where B_I is in units of g/cm³ STP.

A least-squares fit is performed on the (P_{rel_I}, B_I) designated pairs where P_{rel_I} is the independent variable and B_I is the dependent variable. The following are calculated:

- a. Slope (S g/cm³ STP)
- b. Y-intercept (Y_{INT} g/cm³ STP)
- c. Error of the slope (S_{ERR} g/cm³ STP)
- d. Error of the y-intercept (YI_{ERR} g/cm³ STP)
- e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

BET Surface Area (m²/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414 \text{ cm}^3 \text{ STP}) \times (10^{18} \text{ nm}^2/\text{m}^2) \times (S + Y_{INT})}$$

where

CSA = analysis gas molecular cross-sectional area (nm²), user-entered on the Adsorptive Properties dialog box

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

Volume of the Monolayer (cm³/g STP):

$$V_M = \frac{1}{C \ x \ Y_{INT}} = \frac{1}{S + Y_{INT}}$$

Error of the BET Surface Area (m²/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}^2 + YI_{ERR}^2)^{0.5}}{Y_{INT} + S}$$

Langmuir Surface Area Calculations

For each point designated for surface area calculations, the Langmuir³ transformation is calculated as follows:

$$L_{I} = \frac{P_{rel_{I}}}{N_{ads_{I}}}$$

where L_I is in units of g/cm³ STP

A least-squares fit is performed on the (P_{rel_I}, L_I) designated pairs where P_{rel_I} is the independent variable and L_I is the dependent variable. The following are calculated:

- a. Slope (S g/cm³ STP)
- b. Y-intercept (Y_{INT} g/cm³ STP)
- c. Error of the slope (S_{ERR} g/cm³ STP)
- d. Error of the y-intercept (YI_{ERR} g/cm³ STP)
- e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

Langmuir Surface Area (m²/g):

$$SA_{LAN} = \frac{CSA x (6.023 X 10^{23})}{(22414 \text{ cm}^3 \text{ STP}) x (10^{18} \text{ nm}^2/\text{m}^2) x S}$$

where

CSA = analysis gas molecular cross-sectional area (nm²), user-entered on the Adsorptive Properties dialog box Volume of the Monolayer (cm³/g STP):

$$V_M = \frac{1}{S}$$

Langmuir b Value:

$$b = (Y_{INT}) (V_M)$$

Error of the Langmuir Surface Area (m²/g):

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

t-Plot Calculations⁴

For each point designated for t-Plot calculations, the following calculations are done:

Thickness for the Ith point (Å):

$$t_{I} \ = \ HP1 \ x \left[\frac{HP2}{ln(P_{rel_{I}})} \right]^{HP3} \ (Halsey^{5} \)$$

or

$$t_{I} = \left[\frac{HJP1}{HJP2 - log(P_{rel_{I}})}\right]^{HJP3} (Harkins and Jura^{6})$$

or

$$t_I = STSA1(P_{rel_I})^2 + STSA2(P_{rel_I}) + STSA3$$

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
$P_{rel_{I}}$	= relative pressure for the Ith point (mmHg)
STSA1	= statistical thickness surface area parameter #1
STSA2	= statistical thickness surface area parameter #2
STSA3	= statistical thickness surface area parameter #3

A least-squares analysis fit is performed on the (t_I, N_{ads_I}) 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 cm³/g-Å STP)
- b. Y-intercept (Y_{INT} cm³/g STP)
- c. Error of the slope (S_{ERR} cm^3/g -Å STP)

- d. Error of the Y-intercept (YI_{ERR} cm³/g STP)
- e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

External Surface Area (m²/g):

$$SA_{EXT} = \frac{(S \text{ cm}^3/\text{g}-\text{\AA STP}) \text{ x } (10^{10} \text{ \AA/m}) \text{ x } (D \text{ cm}^3 \text{ liquid/cm}^3 \text{ STP})}{F \text{ x } (10^6 \text{ cm}^3/\text{m}^3)}$$

where

F	=	surface area correction factor, user-entered on the t-Polot Report
		Options dialog box
D	=	density conversion factor, user-entered on the Adsorptive
		Properties dialog box

Micropore Surface Area (m²/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 (cm³ liquid/g):

$$V_{\mu P} = (Y_{INT} \text{ cm}^3/\text{g STP}) \text{ x } (D \text{ cm}^3 \text{ liquid/cm}^3 \text{ STP})$$

BJH Pore Volume and Area Distribution Calculations

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 (Pr₁, Va₁) 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.

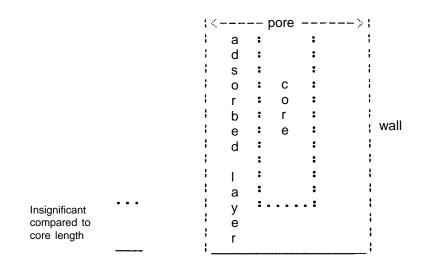
Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin⁷ 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 Halenda⁸ developed the method (known as the BJH method) which incorporates these ideas. The algorithm used on the TriStar 3000 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):

$$Vl_I = (Va_I)(D)$$

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:

$$Rc_1 = \frac{-A}{(1+F)[ln(Pr_1)]}$$

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:

$$\mathrm{Tw}_{1} = \left[\frac{\mathrm{HJ1}}{\mathrm{HJ2} - \mathrm{log}(\mathrm{Pr}_{1})}\right]^{\mathrm{HJ3}}$$

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:

$$\mathrm{Tw}_{1+1} = \mathrm{HP1}\left[\frac{\mathrm{HP2}}{\mathrm{ln}(\mathrm{Pr}_{1+1})}\right]^{\mathrm{HP3}}$$

(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 preivously opened pores is calculated:

$$\Delta Tw = Tw_{I} - Tw_{I+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all previously opened pores:

$$CSA_{J} = \pi \left[(Rc_{i} + \Delta Tw)^{2} - Rc_{i}^{2} \right] (10^{-16} \text{ cm}^{2}/\text{A}^{2})$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_I = \sum (Lp_J)(CSAa_J)$$
 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:

SAw =
$$\sum \pi (Lp_J)(D_{avg_J})(10^{-8} \text{ cm/A})$$
 for all previously opened pores

where

$$D_{avg_J}$$
 = weighted average pore diameter calculated below in Step b(2).

A new layer thickness (Δ Tw) that will not overcompensate for the actual volume desorbed in this interval is calculated:

$$\Delta Tw = \frac{(Vl_I - Vl_{I+1})(10^8 \text{ A/cm})}{\text{SAw}_{I}}$$

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 $(Vl_I - Vl_{I+1})$ 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 Pr_I 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:

$$Vc_{I} = (Vl_{I} - Vl_{I+1}) - Vd_{I}$$

The Kelvin radius for the end of the interval is calculated as follows:

$$Rc_{K+1} = \frac{-A}{(1+F)[ln(Pr_{I+1})]}$$

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_{avg_K} is the diameter of a pore which would have a surface area that is the average of the areas for pores with radius Rc_K and Rc_{K+1} , if its length was the mean of the lengths at those radii.

The relative pressure corresponding to \boldsymbol{D}_{avg_K} is calculated as follows:

$$P_{avg_{K}} = \ln^{-1} \left[\frac{-2A}{(1+F)(D_{avg_{K}})} \right]$$

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:

$$\Delta Td = Tw_{avg_{K}} - Tw_{I+1}$$

The cross-sectional area of the newly opened pores is calculated as follows:

$$\mathrm{CSAc}_{\mathrm{K}} = \left[\frac{\mathrm{Davg}_{\mathrm{K}}}{2} + \Delta \mathrm{Td}\right]^{2} (10^{-16} \,\mathrm{cm}^{2}/\mathrm{A}^{2})$$

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:

$$D_{avg_{K_{new}}} = D_{avg_{K_{old}}} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as follows:

$$D_{avg_{J_{new}}} = D_{avg_{J_{old}}} + 2(\Delta Tw)$$

(not including $D_{avg_{K}}$)

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as follows:

$$Rc_{J_{new}} = Rc_{J_{old}} + \Delta Tw$$

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 $D_{avg_{(I)}}$ 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,cm³/g):

$$Vp_{I} = \pi (Lp_{I}) \left[\frac{D_{avg_{I}}}{2} \right]^{2} 10^{16} \text{ cm}^{2} / \text{A}^{2}$$

2) Cumulative Pore Volume $(Vp_{CUM_{(1)}}, cm^3/g)$:

 $Vp_{CUM_{(I)}} = \sum Vp_J \ {\rm for} \ J \leq I$

3) Incremental Surface Area (SA_I, m^2/g):

 $SA_{I} = \pi (Lp_{I})(10^{-2} \text{ m/cm})(D_{avg_{I}})(10^{-10} \text{ m/A})$

4) Cumulative Surface Area (SA_{CUM₀}, m^2/g):

$$SA_{CUM_{(I)}} = \sum SA_J \text{ for } J \leq I$$

5) dV/dD pore volume (dV/dD_I , $cm^3/g-A$):

$$dV/dD_{I} = \frac{VP_{I}}{Dp_{I} - Dp_{I+1}}$$

6) dV/dlog(D) pore volume $(dV/dlog(D)_I, cm^3/g)$:

$$dV/dlog(D)_{I} = Vp_{I}/log(Dp_{I}/Dp_{I+1})$$

7) dA/dD pore area (dA/dD_I, m^2/g -A):

$$dA/dD_{I} = \frac{SA_{I}}{Dp_{I} - Dp_{I+1}}$$

8) dA/dlog(D) pore area $[dA/dlog(D)_I, m^2/g]$:

 $dA/dlog(D)_{I} = SA_{I}/log(DP_{I}/Dp_{I+1})$

For fixed pore size tables (if selected), the following calculations are performed:

1) Average Fixed Pore Size (DF_{avg_1}, A) :

$$\mathrm{DF}_{\mathrm{avg}_{\mathrm{J}}} = \frac{\mathrm{Dp}_{\mathrm{F}_{\mathrm{J}}} + \mathrm{Dp}_{\mathrm{F}_{\mathrm{J}+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with DF_{avg_I} between the Minimum BJH diameter and the Maximum BJH diameter.

2) Cumulative Pore volume (VpF_{CUM₁}, 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 $Y = Vp_{CUM_J}$, using an AKIMA semi-spline interpolation.

3) Incremental Pore Volume (VpF_I, cm^3/g):

$$VpF_{I} = VpF_{CUM_{I}} - VpF_{CUM_{I-1}}$$

where $VpF_{CUM_0} = 0$.

4) Cumulative Surface Area (SAF_{CUM_I}, 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 $Y = SA_{CUM_J}$.

5) Incremental Surface Area (SAF_I, m^2/g):

$$SAF_{I} = SAF_{CUM_{I}} - SAF_{CUM_{I-1}}$$

where $SAF_{CUM_0} = 0$.

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 $X=D_{avg_J}$ and $Y=dV/dD_J.$

7) dV/dlog(D) pore volume $[dV/dlog(DpF)_I, cm^3/g]$:

 $dV/dlog(DpF_{I} = INTERP(DpF_{I+1}))$

where INTERP(x) is the value interpolated from the function $X = D_{avg_J}$ 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 $X=D_{avg_J}$ and $Y=dA/dD_J.$

9) dA/dlog(D) pore area [$dA/dlog(DpF_I)$, m²/g]:

 $dA/dlog(DpF_{I} = INTERP(DpF_{I+1}))$

where INTERP(x) is the value interpolated from the function $X = D_{avg_J}$ and $Y = dA/dlog(D)_J$.

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
	$(cm^3/cm^3 \text{ STP})$
Pr	= relative pressure
Dp	= pore (or core) diameter (A)
Rc	= Kelvin radius (A) of core
А	= 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.

STSA1, STSA2, and STSA3 are STSA parameters from the STSA 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)

Dubinin-Radushkevich Calculations⁹

The Dubinin-Radushkevich equation is as follows:

$$\log (V) = \log (V_0) - \frac{B \times T^2}{\beta} x \left[\log \frac{P_0}{P} \right]^2$$

where:

V	= volume adsorbed at equilibrium pressure (cm ³ /g STP)
Vo	= the micropore capacity (cm^3/g STP)
Ро	= saturation vapor pressure of gas at temperature T (mmHg)
Р	= equilibrium pressure (mmHg)
В	= a constant
β	= the affinity coefficient of analysis gas relative to Po gas
	(for this application β is taken to be 1)
Т	= 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 mono-layer capacity. Based on this assumption, the following are calculated:

- a. Slope (S cm³/g STP)
- b. Y-intercept (YI cm³/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 (cm³/g STP):

$$Vo = 10^{YI}$$

Error of Monolayer Capacity (cm³/g STP):

 $Vo_{ERR} = Vo x (10^{YI_{ERR}} - 1.0)$

Micropore surface area (m²/g):

SDP =
$$\frac{\sigma x \text{ Vo } x (6.023 x 10^{23})}{22414 \text{ cm}^3 x (10^{18} \text{ nm}^2/\text{m}^2)}$$

where:

σ

molecular cross sectional area of gas (nm²)
 from the Adsorptive Properties dialog box

Dubinin-Astakhov Calculations

The Dubinin-Astakhov equation is a follows:

$$\log (V) = \log (Vo) - \left[\frac{(R \times T)}{\beta E_o}\right]^N x \left[\log \frac{Po}{P}\right]^N$$

where:

V	=	volume adsorbed at equilibrium pressure (cm ³ /g STP)
Vo	=	the micropore capacity (cm ³ /g STP)
Ро	=	saturation vapor pressure of gas at temperature T (mmHg)
Р	=	equilibrium pressure (mmHg)
Т	=	analysis bath temperature (K)
R	=	the gas constant (0.0083144 KJ/K-mole)
E ₀	=	characteristic energy (KJ/mole)
Ν	=	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 cm³/g STP)
- b. Y-intercept (YI cm³/g STP)
- c. Error of the slope (S_{ERR} cm³/g STP)
- d. Error of the y-intercept (YI_{ERR} cm³/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 (cm³/g STP):

 $Vo = 10^{YI}$

Micropore Volume (cm³/g):

$$W_{I} = (V_{I} \times D)$$

where

D	=	density conversion factor (cm ³ liquid/cm ³ STP)
		from the Adsorptive Properties dialog box

Limiting Micropore Volume (cm³/g):

Wo =
$$(Vo \times D)$$

where

D = density conversion factor (cm³ liquid/cm³ STP) from the Adsorptive Properties dialog box

Error of Limiting Micropore Volume (cm³/g):

$$Wo_{ERR} = Wo \times (10YI_{ERR} - 1.0)$$

Characteristic Energy (KJ/mole):

$$E = \frac{2.303 \text{ x R x T}}{(2.303 \text{ x S})^{1/N}}$$

_

Modal Equivalent Pore Diameter (Å):

$$D_{\text{MODE}} = 2 x \left\{ \left[\frac{3N}{3N+1} \right]^{1/N} x \left[\frac{10^3 \text{ nm}^3/\text{\AA}^3}{\beta \cdot \text{E}_0} \right] \right\}^{1/3}$$

where

β

 affinity coefficient of the analysis gas relative to the Po gas from the Dubinin Adsorptive Options dialog box

Maximum Differential Pore Volume (cm³/g-Å):

This value is also known as frequency of the mode¹²

$$\frac{dV}{dDmode} Max = 0.5 \text{ x } (3N+1) \text{ x Wo } \text{ x} \left[\frac{3N+1}{3N}\right]^{V_{3N}} \text{ x} \left[\frac{\beta \cdot E_0}{(10^3 \text{ nm}^3/\text{\AA}^3)}\right]^{V_3} \text{ x } e^{-\left[\frac{3N+1}{3N}\right]}$$

Mean Equivalent Pore Diameter (Å)

$$D_{MEAN} = 2 x \frac{\left[\frac{10^3 \text{ nm}^3/\text{\AA}^3}{\beta \cdot \text{E}_0}\right]^{1/3}}{\Gamma\left(\frac{3N+1}{3N}\right)}$$

Micropore surface area (m²/g):

SDA = 10000 x 2.0 x Wo x
$$\left[\frac{\beta \cdot E}{10^3 \text{ nm}^3/\text{\AA}^3}\right]^{1/3} \text{x } \Gamma\left(\frac{3N+1}{3N}\right)$$

 Γ is calculated by a polynomial approximation 11 over the domain $0 \leq x \leq 1$ as follows:

$$\begin{aligned} \Gamma(x+1) &= 1 + b_1 x + b_2 x^2 + b_3 x^3 + b_4 x^4 + b_5 x^5 + b_6 x^6 + b_7 x^7 + b_8 x^8 + \epsilon(x) \\ &| \epsilon(x) | \leq 3 \ x \ 10^{-7} \end{aligned}$$

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

$$\mathbf{x} + \mathbf{1} = \left(\frac{3\mathbf{N} + \mathbf{1}}{3\mathbf{N}}\right)$$

Equivalent Pore diameter (Å):

$$D_{I} = 2 x \left[\frac{-\left(\frac{10^{3} \text{ nm}^{3} / \text{Å}^{3}}{\beta \cdot E_{0}}\right)^{N}}{\ln(W_{I}) - \ln(W_{o)}} \right]^{J_{3N}}$$

dV/dD Pore Volume (cm³/g-Å):

$$\frac{\mathrm{dV}}{\mathrm{dD}_{\mathrm{I}}} = 0.5 \ \mathrm{x} \left[\mathrm{Wo} \ \mathrm{x} \ 3\mathrm{N} \ \mathrm{x} \left[\frac{10^3 \ \mathrm{nm}^3/\mathrm{\AA}^3}{\beta \cdot \mathrm{E}_0} \right]^{\mathrm{N}} \ \mathrm{x} \left(\frac{\mathrm{D}_{\mathrm{I}}}{2} \right)^{-(3\mathrm{N}+1)} \right]^{\mathrm{N}}$$

$$x e \left[-\left(\frac{10^3 \text{ nm}^3/\text{\AA}^3}{\beta \cdot \text{E}_0}\right) x \left(\frac{\text{D}_{\text{I}}}{2}\right)^{-3\text{N}} \right]$$

MP-Method Calculations¹⁰

For each point designated for MP-method calculations, the following calculations are done:

Thickness for the Ith point (Å):

$$t_{I} = HP1 x \left[\frac{HP2}{ln(P_{rel_{I}})} \right]^{HP3} (Halsey^{5})$$

or

$$t_{I} = \frac{HJP1}{HJP2 - log(P_{rel_{1}})}^{HJP3} (Harkins and Jura^{6})$$

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_{rel}	=	relative pressure for the Ith 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):

$$R_{I} = \frac{t_{I} + t_{I-1}}{2}$$

Remaining pore surface area for the I^{th} point (m^2/g) :

$$S_{I} = \frac{(V_{I} - V_{I-1}) x (D x 10^{-6} m^{3} / cm^{3})}{(t_{I} - t_{I-1}) x 10^{-10} m / \text{\AA}}$$

where

Incremental pore surface area occluded for the I^{th} point (m^2/g) :

$$S_{INC_I} = S_{I-1} - S_I$$

Cumulative pore surface area occluded for the I^{th} point (m^2/g) :

$$S_{\text{CUM}_{\text{I}}} = S_{\text{INC}_{\text{I}}} + S_{\text{INC}_{\text{I}-1}} + \dots + S_{\text{INC}_{1}}$$

dA/dR pore surface area for the Ith point (m^2/g -Å):

$$\frac{\mathrm{dA}}{\mathrm{dR}_{\mathrm{I}}} = \frac{\mathrm{S}_{\mathrm{SINC}_{\mathrm{I}}}}{\mathrm{t}_{\mathrm{I}} - \mathrm{t}_{\mathrm{I}-1}}$$

Incremental pore volume occluded for the Ith point (cm³/g):

$$V_{INC_{I}} = (S_{INC_{I}} \times 10^{4} \text{ cm}^{2}/\text{m}^{2}) \times (R_{I} \times 10^{-8} \text{ cm}/\text{\AA})$$

Cumulative pore volume occluded for the Ith point (cm³/g):

$$V_{\text{CUM}_{\text{I}}} = V_{\text{INC}_{\text{I}}} + V_{\text{INC}_{1-1}} + \ldots + V_{\text{INC}_{1}}$$

dV/dR pore volume for the Ith point (cm³/g-Å):

$$\frac{dV}{dR_I} = \frac{V_{INC_I}}{t_I - t_{I-1}}$$

Summary Page

The following calculations and the results of previous calculations (as noted) are used to generate the summary page:

a. Single Point BET Surface Area (m^2/g)

$$S_{1PT} = \frac{[Va \ x \ (1-Pr)] \ x \ CSA \ x \ (6.023 \ x \ 10^{23})}{22414 \ cm^3 \ STP \ x \ 10^{18} \ nm^2/m^2}$$

where

Pr	=	pressure closest to 0.3 of the relative pressure points designated
		for surface area calculations.
Va	=	volume corresponding to Pr

b. Multipoint BET Surface Area

See BET Surface Area Calculations

c. Langmuir Surface Area

See Langmuir Surface Area Calculations

d. t-Plot Micropore Surface Area

See t-Plot calculations.

e. t-Plot External Surface Area

See t-Plot calculations.

- f. BJH Cumulative Adsorption
- g. BJH Cumulative Desorption
- h. Adsorption Total Pore volume
- i. Desorption Total Pore volume
- j. t-Plot Micropore Pore Volume

See t-Plot calculations.

- k. Average Pore Diameter
- 1. BJH Adsorption Average Pore Diameter
- m. BJH Desorption Average Pore Diameter

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APPENDIX D

FREE-SPACE CORRECTION

FREE-SPACE CORRECTION

Free space is that volume of the sample tube which is unoccupied by the sample. The quantity of gas dosed into the sample tube is calculated from the difference in pressures in the manifold before and after the dose is delivered. The quantity of gas adsorbed by the sample is calculated by subtracting the quantity of gas remaining in the free space of the sample tube after equilibrium is established from the quantity of gas originally dosed into the sample tube. Free space must be determined accurately to obtain a precise value for quantity adsorbed.

Static-volumetric systems such as the TriStar 3000 consist basically of a gas manifold joined to a sample tube by an isolation valve. The manifold section has connections for an absolute pressure transducer, a temperature gauge, and a vacuum system. It also has inlets for the adsorptive gas and helium. A Dewar flask containing a cryogenic liquid (usually LN₂ at approximately 77 K) is situated so that it can be raised to immerse most of the sample tube. Two temperature zones exist within the sample tube when immersed in the cryogenic bath: a warm zone (the volume above the liquid level and near ambient temperature) and a cold zone (the volume below the liquid level at cryogenic temperature). Not only must the total free-space volume be determined, but it also is necessary to determine the quantity of gas residing within the "cold" zone since a nonideality correction must be applied to only that quantity of gas.

The total quantity of gas in the partly immersed sample holder cannot simply be determined using n = PV/RT because temperature is not constant over the total volume, but instead is distributed as two temperature zones with a steep temperature gradient between them. A convenient method for resolving this problem is to derive two factors which, for the existing temperature profile, can be multiplied by the prevailing pressure to reveal the molar volume of gas contained in the cold zone and the total quantity residing in the free volume of the immersed sample holder (the cold free space).

The TriStar 3000 system provides four methods for free-space determination:

- Measure
- Quasi-Gemini
- Calculate
- Enter

Measure

Generally, this method, although requiring a little more time (approximately 10 minutes) is the most preferred one for determining free space. It is simple, automatic, requires very little information, and essentially is error-proof. With this method, the instrument first evacuates the manifold and sample tube (containing sample), then isolates the sample tube by closing the valve. Then the manifold is charged with helium, the pressure measured, and the valve opened allowing the helium to expand into the sample tube at ambient temperature. Again the pressure is measured.

The dewar is raised and the sample tube is cooled to cryogenic temperature. Again pressure drops; when pressure has equilibrated, the value is recorded. Warm and cold free spaces are calculated from (1) system volume, (2) system, ambient, and bath temperatures, and (3) measured pressures. From these, the value of the portion of cold free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable; free-space determination by the TriStar 3000 requires the use of helium
- Analysis speed is a major factor; a helium free-space measurement of 10 to 15 minutes is required
- Your sample tends to absorb and retain helium for a prolonged period of time or if it adsorbs helium

Quasi-Gemini

This method requires the dedicated use of port 2, allowing concurrent free-space correction. With this method, an empty sample tube (called the "balance" tube) is installed on port 2 to run concurrently with one or two analyses on the other port(s). You must enter the following information accurately for best results:

- Sample tube stem nominal diameter (found on the Sample Tube dialog)
- Indicate whether a filler rod is being used (found on the Sample Tube dialog)
- Analysis bath temperature (found on the Po and Temperature dialog)
- Sample tube volume below the index mark for the sample and balance tubes (refer to Calibrating the Sample Tube in Chapter 4)
- Sample weight and density (found on the Sample Information dialog)

A separate pressure table is not required for the balance tube. Its pressure table is a composite of the pressure table(s) for the analysis. Therefore, each data point collected for the analysis has a corresponding pressure point on the balance tube record from which a computed pressure/volume point may be interpolated to exactly the pressure obtained by the analysis.

Calculate

This method is the most rapid and efficient way of compensating for free space. You must enter the following information accurately:

- Sample tube stem diameter (found on the Sample Tube dialog)
- Indicate whether a filler rod is being used (found on the Sample Tube dialog)
- Sample tube volume below the index (red) mark
- Analysis bath temperature (found on the Po and Temperature dialog)
- Sample's weight and density (found on the Sample Information dialog)

The volume of the portion of the sample tube below the index mark is measured by filling the sample tube to the index mark, weighing, and calculating the volume using the weight and density of the fluid. This value is used in calculating the free space and should be engraved onto the sample tube for future use. Refer to **Calibrating the Sample Tube** in Chapter 4.

Enter

This method allows you to enter predetermined values for the warm and cold free spaces. The values to enter may be obtained in one of two ways:

- A pre-analysis free-space calibration of the sample tube containing sample
- The total free space of an empty sample tube is measured and the displacement of the sample calculated from its mass and density and subtracted from the total free space

In either procedure, ensure that the level (or, in cases where the Isothermal Jacket is used, the effective level) of the cryogen bath on the sample tube is the same when the analysis is performed as it was when gathering data for free-space calculations.

APPENDIX E

MAINTAINING HIGH PURITY GASES

Maintaining High Purity Gases

The TriStar 3000 was designed to measure accurately the surface area of all types of materials. It is important that the gases (nitrogen or helium) used for these measurements be of highest purity, especially when analyzing low surface area samples. Three ways to ensure high purity gases are to always maintain:

- thoroughly purged gas pressure regulators
- non-permeable gas lines
- leak-free connections

Impure gas is strongly indicated, for example, if a series of measurements on a low surface area material yields decreasing specific surface areas with decreasing quantities of sample. The TriStar uses very small amounts of helium; therefore any residual air in the regulator can distort results of subsequent analyses for quite some time.

Micromeritics offers the following suggestions to assist you in maintaining high purity gases (particularly helium) for the TriStar.

- Use metal gas lines only
- Remove trapped air from the regulator and gas lines
- Purge the regulator and instrument lines after periods of non-use (this step is unnecessary if you are using V2.00 or higher)

Using Metal Gas Lines

You should always use metal gas lines which have been carefully cleaned of any oils and greases used in the manufacturing process. *Do not use plastic or rubber gas lines*. When these types of permeable, nonmetallic gas lines are used with he-lium, contaminants accumulate at a much faster rate. This causes errors in analysis results and can also contaminate a clean sample.

Removing Trapped Air

When connecting the regulator to the gas bottle, air is unavoidably trapped on the high- and low-pressure sides of the regulator, as well as in the gas lines. You should remove as much of this air as is possible *before* opening the gas bottle valve. If this air is allowed to remain in the regulator, it will mix with the helium and cause inaccurate results in subsequent analyses. Or if the valve is open for any length of time, the air trapped on the high pressure side may diffuse back into the gas bottle and contaminate its entire contents.

There are two methods for removing trapped air from the regulator lines: the Purge Method and the Evacuation Method.

Purge Method

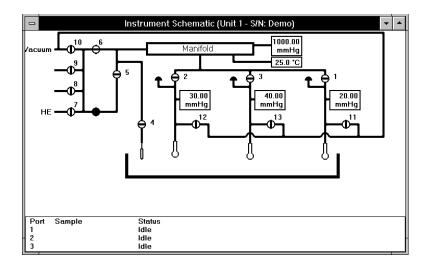
This is the preferred method for removing trapped air.

1. From the **Unit** menu, select **Enable manual control** (if the instrument schematic is not displayed, select **Show instrument schematic**).

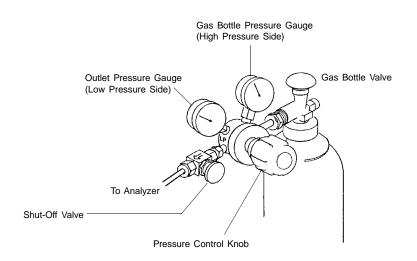


If you have multiple TriStars installed, make sure you choose the correct Unit menu.

2. Close all valves.



- 3. Open the regulator Shut-off valve.
- 4. Open the gas bottle valve *briefly* and allow the regulator to be charged with gas until the high pressure gauge reads just over half the tank pressure; then *quickly* close the valve.



- 5. Using the Pressure Control knob, set the output pressure (gas bottle pressure gauge) to 15 psig.
- 6. Loosen the fitting at the helium inlet (on the rear panel of the TriStar) until the low pressure side drops to approximatley 3 psig (0.02 MPa), then tighten the fitting.
- 7. Repeat steps 4, 5, and 6 three times.
- 8. Briefly open the gas bottle valve; then, using the Pressure Control knob, reset the regulator output pressure to 15 psig.
- 9. After the pressure has stabilized (indicating there are no leaks), open the gas bottle valve.

Evacuation Method



To use this method, your gas tank must be within 10 feet of the instrument.

1. Do one of the following:

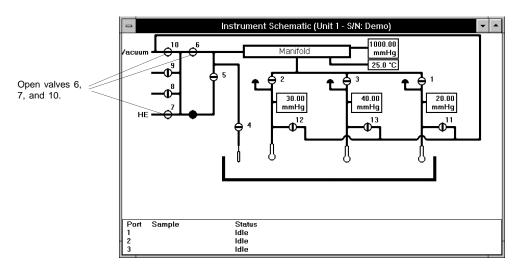
If	Then
The regulator has not	Close the gas bottle valve.
been filled with gas and the gas line is attached to the instrument:	Open the regulator Shut-off valve.
The regulator is filled	Close the gas bottle valve.
with gas:	Open the regulator Shut-off valve.
	Loosen the helium inlet fitting (or nut) on the rear panel of the instrument.
	Allow all of the gas in the regulator to expel from the line (pressure reading will be zero).
	Retighten the helium inlet fitting (or nut).

2. From the **Unit** menu, select **Enable manual control** (if the instrument schematic is not displayed, select **Show instrument schematic**).



If you have multiple TriStars installed, make sure you choose the correct Unit menu.

3. Close all valves; then open valves 6, 7, and 10.



4. Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas bottle. The manifold pressure transducer should fall close to zero.



Be sure to allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

5. Close valves 6, 7, and 10.

Purging the System After Non-use



These steps are unnecessary if your analysis program is V2.00 or higher. V2.00 (or higher) provides automatic purges before analysis if your system is idle for more than 24 hours.

You should always purge the regulator after periods of non-use. Even though the regulator and lines have been carefully cleaned, vapors sometimes permeate the O-rings and seals in the TriStar system. These permeated vapors gradually increase the level of contaminants in the helium line and regulator when the instrument is idle for more than hours. Use the following guidelines:

- Always purge the regulator and lines when the TriStar has been idle for 72 hours (3 days).
- Purge the regulator and lines when the TriStar has been idle for more than 24 hours if your sample has a total surface area of less than a few meters squared and you plan to *measure* the free space.

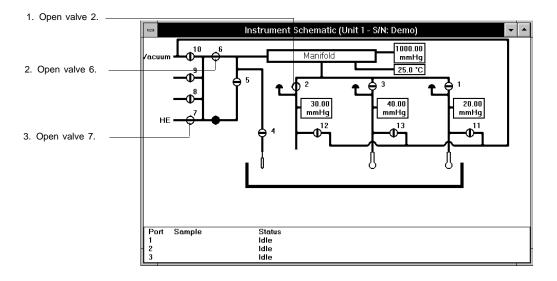
Using an open sample port, purge the regulator and lines as follows:

- 1. Remove the sample tube or plug from port 2 (in this example).
- 2. From the **Unit menu**, select **Enable manual control** (if the instrument schematic is not displayed, select **Show instrument schematic**).



If you have multiple TriStars installed, make sure you choose the correct Unit menu.

3. Open Valves 2, 6, and 7, allowing the helium to flow through the system to purge the lines.



E-6

- 4. After approximately 1 to 2 minutes, close valve 7.
- 5. Open the nitrogen valve (8 or 9); allow the nitrogen to flow through the system for one minute. This allows the helium to be flushed from the system.



It is important that you flush the helium from the manifold after purging. If helium is allowed to remain in the manifold for a sufficient amount of time, transducer offset problems may be encountered.

6. Close the nitrogen valve.

INDEX

INDEX

\$ symbol, 8-9

Α

Absolute pressure tolerance, 5-20 Add log entry, 8-13 Add Log Entry push button, 5-6, 5-9 Adding an analyzer, 2-16 Adsorbate property factor, 5-48 Adsorptive properties file, 5-27 defining, 4-9 printing contents, 5-71 Adsorptive push button BJH report, 5-47 Dubinin report, 5-54 Advanced format, 5-4, 5-8, 8-3 sample defaults, 8-11 sample information file, 4-14, 8-4 Alpha-S report example of, 7-23 options, 5-42 Analyses performing, 4-26, 6-3 QuickStart method, 4-27, 6-7 Analysis conditions file, 5-15 defining, 4-8 printing contents, 5-71 Analysis dewar See Dewar Analysis Log report example of, 7-16 Analysis Preparation dialog, 5-22 Analysis program exiting, 5-77 installing, 2-10 installing upgrade, 2-15 uninstalling, 2-23 using, 3-2 Analyzer adding another, 2-16 changing setup, 2-21 cleaning, 9-1 configurations, 6-17 installing, 2-3 moving to a different computer, 2-17 removing, 2-20 schematic, 6-11 turning on/off, 3-1

Astakhov report, 5-53 calculations, C-25 Autoscale, 5-33, 5-52, 5-61, 5-62 Axis coordinates, viewing, 7-14 properties, editing, 7-13 Axis Properties dialog, 7-13

В

Basic format, 5-4, 5-5, 8-3 sample file defaults, 8-8 sample information file, 4-12, 8-5 Bath temperature, 6-9 calculations, C-4 BET surface area report calculations, C-8, C-9 example of, 7-18 options, 5-34 BJH Adsorption/Desorption report calculations, C-13 examples of, 7-25 options, 5-46 BJH Adsorptive Property Factor dialog, 5-47 Broekhoff-de Boer thickness curve, 5-39 Browse push button, 3-6, 6-4, 6-9

С

Calibration information, 6-17 manifold temperature, 6-18, 9-5 Pressure scale, 6-19, 9-4 sample tube, 4-19 servo valve, 6-20, 9-5 system volume, 6-21, 9-7 zero pressure, 9-3 Calibration file loading, 6-23 reinstalling, 2-22 saving, 6-23 Cancel push button, 6-6 Carbon Black STSA thickness curve, 5-38 Catalysts, 5-19 Cautions, 1-2 Check box, 3-5 Close All command, 5-70 Close command, 5-70

Close Reports command, 7-5 Cold free space, 5-24 calculations, C-4 Collected data, 5-65 Columns, editing on onscreen reports, 7-10 Comments window, 8-13 Computer connecting analyzer, 2-3 connecting peripheral equipment, 2-6 Cross hair, axis, 7-14 Curve Properties dialog, 7-12

D

Data collected, 5-65 entered, 5-65 Data Presentation command, 8-7 Date format, changing, 3-7 Dates push button, 3-7 Default Basic Sample Information dialog box, 8-8 Degas conditions file, 5-13 defining, 4-7 printing contents, 5-71 Degassing unit, 1-3, 10-1 Density conversion factor, 5-27 Dewar installing, 4-24 precautions, 4-24 DFT Plus command, 7-5 Dialog boxes, using, 3-4 Dose increment, 5-21, 5-22 Dose Increments dialog, 5-21 Dubinin Adsorptive Options dialog box, 5-54 Dubinin report calculations, C-23 options, 5-53 types available, 5-53 Dubinin Report Options dialog box, 5-55 Dubinin Tabular Report Column Options dialog, 5-55 Dubinin-Radushkevich calculations, C-24

Ε

Elevator options, 6-12 Enable Manual Control command, 6-11 Entered data, 5-65 Equation Broekhoff-de Boer, 5-39 Halsey, 5-59 Harkins-Jura, 5-58 STSA Carbon Black, 5-38 Equation push button, 5-37, 5-47, 5-58 Equilibration calculations, C-7 push button, 5-26 Error messages, B-1 Evacuation options, 5-22 Exit command, 5-77 Exiting the analysis program, 5-77 Export command, 5-75 Exporting sample files, 4-32, 5-75

F

f-Ratio report example of, 7-24 options, 5-44 File printing contents, 4-29 printing to, 7-4 selecting, 3-6 status, 3-7 File menu Close All command, 5-70 Close command, 5-70 description, 5-1 Exit command, 5-77 Export command, 5-75 List command, 5-73 Open command, 5-3 Print command, 5-71 Save All command, 5-70 Save command, 5-68 File name default extensions, 3-8, 5-3 included as part of ID, 8-9 sequence, 8-8 Filler rod, 5-12 Fixed dose, 5-19 Forms, 1-1 Free Space, D-1 calculated method, 5-24, D-3 calculations, C-3 cold, 5-24 dialog, 5-23 entered method, 5-24, D-4 measured method, D-2 Quasi-Gemini method, 5-24, D-3 warm, 5-24 Function keys See Shortcut keys

G

Gas supply connecting regulators, 2-7 specifying, 2-25 Gases maintaining high purity, E-1 selecting, 6-17 Graph copying as metafile, 7-13 editing onscreen, 7-11 grid lines, 8-7 legend, editing, 7-13 Graph overlay, 4-33 for multiple graphs, 4-33 for multiple samples, 4-35 Graph Overlay Samples dialog, 5-30 Graphic, appearing in report title, 5-30 Grid lines. 8-7

Η

Halsey equation, 5-59 Halsey thickness curve, 5-38 Halsey Thickness Equation dialog box, 5-59 Harkins and Jura thickness curve, 5-38 Harkins/Jura equation, 5-58 Help menu, 3-11 Help, online, 1-5

I

Increment, maximum volume, 5-19 Insert Pressure Range dialog, 5-17 Installation analysis program, 2-10 sample tube, 4-23 Instrument log, 6-2, 6-15 Instrument Schematic dialog box, 6-11 Instrument schematic, showing, 6-13 Isotherm collecting, 5-16 data, exporting, 5-75 report options, 5-32 Isotherm collection dose increments, 5-21 target pressures, 5-16 Isotherm report example of, 7-17 options, 5-32 Isothermal jacket, 5-12

L

Langmuir surface area report calculations, C-9 example of, 7-20 options, 5-34 Leak test, 5-23 Legend Properties dialog, 7-13 List command, 5-73 List, generating, 4-30, 5-73 Log entry, 5-6 Log Report settings, 6-16 Low surface area sample, analyzing, E-1

Μ

Maintenance, system, 9-1 Manifold temperature, 6-18 Manual conventions, 1-2 organization, 1-1 Manual control, 6-11 accessing shortcut menu, 6-11 enabling, 6-11 Maximum BJH diameter (or radius), 5-47 Maximum volume increment, 5-19 Menu bar. 3-9 Menu structure, 3-9 Mesoporous materials, 5-19 Metafile, copying graphs as, 7-13 Minimum BJH diameter (or radius), 5-47 MP-Method Cumulative Pore Area Options dialog, 5-61MP-Method Cumulative Pore Volume Options dialog, 5-61 MP-Method dA/dR Pore Area Options dialog, 5-61 MP-Method dV/dR Pore Volume Options dialog, 5-61 MP-Method report, 5-58 calculation, C-29 options, 5-58 types available, 5-59 MP-Method Tabular Report Column Option dialog, 5-60

Ν

Next push button, 6-6 Non-ideality factor, 5-27 Notes, 1-2

0

O-ring, port replacing, 9-2 Open command, 5-3 Operator, specifying, 8-12 Option Presentation command, 8-3 Options menu Data Presentation command, 8-7 description, 8-1 Option presentation command, 8-3 Parameter Files Directory command, 8-15 Sample Defaults command, 8-8 Service Test Mode command, 8-16 System configuration command, 8-14 Options report, 5-64 Outgas test, 5-24 Overlay samples, 5-35, 5-41, 5-43, 5-44 Overlays push button, 5-30

Ρ

Parameter files, 5-4 defining, 4-5 specifying location, 8-15 Parameter Files Directory command, 8-15 Password, restricted format, 8-6 Physical volume below mark, 5-12 Po and T push button, 5-25 Po and Temperature Options dialog box, C-4, C-23 Po value, 6-4 Ports Gas connection, 3-10 Preparation push button, 5-22 Presentation options, 8-3 Pressure relative, calculations, C-2 saturation. C-1 table, 5-16 target, 5-16 tolerance, 5-20 Pressure offset, 6-19 Pressure scale calibration, 6-19 Pressures push button, 5-48 Print command, 5-71 Printer/plotter, specifying default, 8-14 Printing to a File description, 5-72 Psat vs t push button, 5-28 Push button, 3-5 Browse, 3-6 Dates, 3-7 Equilibration, 5-26

Free Space, 5-23 Overlays, 5-30 Po and T, 5-25 Preparation, 5-22 Psat vs t, 5-28 Replace, 3-6 Skip, 6-6 Suspend, 6-6

Q

Quasi-Gemini calculations, C-6 free-space method, D-3 QuickStart command, 6-7 QuickStart dialog box, 6-7

R

Radio button, 3-5 Radushkevich report, 5-53 calculations, C-23 Ramp rate, 5-14 Ranges, valid, 3-4 Relative pressure calculations, C-1 See also Pressure Relative pressure tolerance, 5-20 Removing an analyzer, 2-20 Replace All pushbutton, 5-7, 5-9 Replace push button, 3-6, 5-11, 5-13, 5-15, 5-28, 5-30 Report Alpha-S method, 5-42 Analysis Log, 7-16 BET Surface Area, 5-34 BJH Adsorption/Desorption, 5-46 closing, 7-5 displaying company logo in title, 5-30 Dubinin, 5-53 examples, 7-15 f-Ratio method, 5-44 generating, 7-2 graphs, editing onscreen, 7-11 header, 7-6 Isotherm, 5-32, 7-17 Langmuir surface area, 5-34 legend, editing, 7-13 onscreen, 7-6 onscreen, shortcut menus for, 7-10 Options, 5-64 Sample log, 5-64

saving in other formats, 7-9 Summary, 5-63 t-Plot, 5-37 Tabular. 5-49 Tabular, editing onscreen, 7-10 Tool bar, 7-7 types available, 5-31 zoom feature, 7-14 Report options file, 5-29 defining, 4-10 printing contents, 5-71 Reports header. 7-6 MP-Method, 5-58 Reports menu Close Reports command, 7-5 description, 7-1 DFT Plus command, 7-5 Start Report command, 7-2 Restricted format, 8-3, 8-6 sample information file, 4-12 Resume push button, 6-6 Returning equipment, 2-1

S

Sample degassing, 4-22 entering density, 8-9 low surface area, E-1 Sample Analysis command, 6-3 Sample defaults Advanced format, 4-3, 8-11 Basic format, 4-1 specifying, 8-8 Sample file name specifying default sequence, 8-11 Sample information file, 3-6, 5-4 Advanced format, 5-4, 5-8 Basic format, 5-4, 5-5 creating in Advanced format, 4-14 creating in Basic format, 4-12 exporting, 4-32, 5-75 generating a list, 5-73 printing contents, 5-71 Restricted format, 5-4 specifying defaults, 8-8 Sample Log report, 5-64 example of, 7-32 Sample tube calibrating, 4-19 cleaning and labeling, 4-16 file, 5-11

installing, 4-23 Sample Tube file, 4-5 Saturation pressure, C-1 Save All command, 5-70 Save As command, 5-68 Save command, 5-68 Schematic, 6-11 Select Dates dialog, 3-7 Selecting files, 3-6 Service Test mode, 8-16 Servo valve calibrating, 6-20 settings, 6-12 Setup program adding an analyzer, 2-16 changing an analyzer setup, 2-21 moving an analyzer to a different computer, 2-17 reinstalling analysis program, 2-15 reinstalling calibration files, 2-22 removing an analyzer from the computer, 2-20 uninstalling the software, 2-23 using for other functions, 2-13 Shortcut keys, 3-2 for Select Dates dialog, 3-7 list of. 3-3 Shortcut menu, 3-2 for graphs, 7-11 for instrument schematic, 6-11 for tabular reports, 7-10 Show Instrument Log command, 6-15 Show Instrument Schematic command, 6-13 Show Status command, 6-14 Skip push button, 6-6 SmartPrep Degasser, 1-3 Soak temperature, 5-14 time, 5-14 Software installing, 2-10 installing upgrade, 2-15 using, 3-2 Specifications, 1-6 Spreadsheet format exporting isotherm data, 5-75 saving reports, 7-9 Start push button, 6-6 Start Report command, 7-2 Start Report dialog box, 7-2 Status list, 7-3 Status window, 6-14 STSA thickness curve, 5-38 Submitter, specifying, 8-12

Summary Report calculations, C-32 example of, 7-31 options, 5-63 Surface area correction factor, 5-41 Suspend push button, 6-6 System configuration command, 8-14 System volume, calibrating, 6-21

Т

t-Plot report calculations, C-11 example of, 7-21 options, 5-37 Target pressure, 5-16 Temperature bath, 6-9 manifold, 6-18 soak, 5-14 Text, copy graph data as, 7-13 Thickness curve, 5-37, 5-46, 5-58 Broekhoff-de Boer, 5-39 Carbon Black STSA, 5-38 Halsey, 5-38, 5-59 Harkins and Jura, 5-38 Harkins-Jura, 5-58 User-defined, 5-39 Title Properties dialog, 7-13 Tool bar, reports window, 7-7 Transducer offset, 6-19 TriStar See also Analyzer description, 1-3 specifications, 1-6 unpacking/inspection, 2-1

U

Unit configuration, 6-17 Unit Menu description, 6-1 Enable Manual Control command, 6-11 QuickStart command, 6-7 Sample Analysis command, 6-3 Show Instrument Log command, 6-15 Show Instrument Schematic command, 6-13 Show Status command, 6-14 Unit Configuration command, 6-17 Units dialog box, 8-7 Use calculation assignments, 5-66 User-defined thickness curve, 5-39

V

Vacuum pump, 1-4 connecting to analyzer, 2-4 View, 6-7

W

Warm free space, 5-24 calculations, C-4 Warnings, 1-2 Weighing sample, 4-21 Window tile, 3-10 Windows menu, 3-10 Worksheet *See* Forms

Ζ

Zero pressure calibration, 6-19 Zoom feature, 7-14