mi micromeritics®

ChemiSoft TPx

Operator's Manual

V1.03

Windows is a registered trademark of Microsoft Corporation

TABLE OF CONTENTS

4. GENERAL OPERATING PROCEDURES

5. FILE MENU

6. UNIT MENU

7. REPORT MENU

8. OPTIONS MENU

9. ORDERING INFORMATION

A. FORMS

B. ERROR MESSAGES

C. CALCULATIONS

D. FORMAT OF EXPORTED DATA

E. PEAK DETECTION/INTEGRATION OPTIONS

F. TUTORIAL

INDEX

1. GENERAL DESCRIPTION

This manual describes how to install and operate the ChemiSoft TPx program in conjunction with the ChemiSorb 2750 and 2720 analyzers.

Organization of the Manual

The ChemiSoft operator's manual is organized as follows:

Chapter 1 **General Description**	Provides a general description of the ChemiSoft program, its features, and specifications.
Chapter 2 **Installation**	Provides instructions for installing the program.
Chapter 3 **User Interface**	Provides basic software and instrument interface.
Chapter 4 **Operating Procedures**	Provides brief step-by-step procedures for the operations you can perform with the program.
Chapter 5 **File Menu**	Provides a description of the commands available on the File menu.
Chapter 6 **Unit Menu**	Provides a description of the commands available on the Unit menu.
Chapter 7 **Reports Menu**	Provides a description of the commands available on the Reports menu.
Chapter 8 **Options Menu**	Provides a description of the commands available on the Options menu
Appendix A **Forms**	Provides a form for you to copy and use for recording values to be entered in the controller.
Appendix B **Error Messages**	Provides a listing of error messages that may be displayed by the ChemiSoft program, as well as causes and actions for each.

Appendix C **Calculations**	Contains the calculations used by the software to produce reports.
Appendix D **Format of Exported Data**	Provides the ASCII and Grams/32 formats for exported data.
Index	Provides quick access to a subject matter.

Conventions

This manual uses the symbols shown below to identify notes of importance and warnings:

Notes contain important information pertinent to the subject matter.

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

Cautions contain information that help you prevent actions that could damage the controller..

Description

The ChemiSoft program has been developed to interface with the ChemiSorb 2750 and 2720 analyzers. This program allows you to:

- monitor and record analysis signal and sample temperature data
- monitor and record the furnace temperature
- create and store sample files
- create analysis conditions files for specialized analyses, such as TPR, TPO, etc.
- perform analyses using prompts on the software screens and manually interface with the analyzer
- integrate detector signal peaks, automatically and manually
- perform peak deconvolution using peak-fitting algorithms
- generate and customize reports
- display and edit peak graphs and reports
- establish calibration curves for calculation of unknown sample concentrations
- export data in ASCII text for use in other applications

Online Help

An extensive online help system is provided for quick-and-easy access to a subject matter. The ChemiSoft 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.

Report System

The ChemiSoft program includes a report system which allows you to manipulate and customize reports. You can zoom in on portions of the graphs or shift the axes to examine fine details. Scalable graphs can be copied to the clipboard and pasted into other applications. Reports can be customized with your choice of fonts and a company logo added to the report header for an impressive presentation. Refer to Chapter 7 for the options available for reports.

Peak Editor

The ChemiSoft program includes an integrated Peak Editor. The Peak Editor provides an easy-to-use interface enabling you to view TCD signal and temperature data for sample files containing up to 16 experiments. the Peak Editor also allows you to specify and integrate peaks in the TCD signal so that the volume and gas consumed or evolved may be quantified. Peak fitting (deconvolution) is also included for TCD signals that feature overlapping peaks. Chapter 7 provides details for the Peak Editor.

Types of Analyses

Regardless of which type of experiment you perform, the basic concept is the same: the Thermal Conductivity Detector (TCD) detects changes in the gas mixture flowing through it. The sample, gas selection, and analysis conditions determine what changes occur. Some of the analyses you can perform using the ChemiSoft option are described below.

TPD Analysis

Temperature-Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows through the sample and adsorbs on the active sites. Programmed desorption begins by raising the temperature linearly with time while a steady stream of inert carrier gas flows through the sample.

At a certain temperature, the heat overcomes the activation energy; therefore, the bond between the adsorbate and adsorbent will break and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperatures. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measures the gas concentrations. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

TPR Analysis

Temperature-Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which the reduction occurs. An important aspect of TPR analyses is that the sample need not have any special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) through the sample, usually at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by the reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

TPO Analysis

Temperature-Programmed Oxidation (TPO) examines the extent to which a catalyst can be oxidized or, by inference, was previously reduced.

Usually the sample is pretreated and the metal oxides are reduced to the base metal, typically with a gas mixture of hydrogen with either nitrogen or argon. Then the reactant gas, typically 2-5% oxygen with helium, is applied to the sample as a steady stream.

The furnace heats the sample tube and sample according to the user-selected temperature program. The oxidation reaction occurs at a specific temperature. The uptake of oxygen. is determined.

When using any mixture of gases for either TPR or TPO analyses, make sure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity.

Pulse Chemisorption Analysis

A Pulse Chemisorption analysis determines active surface area, percent metal dispersion, and the average active particle size by applying measured doses of reactant gas to the sample, usually while the sample is held at a steady temperature.

The gas reacts with each active site until all sites have reacted. Once the active sites have completely reacted, the discretely injected gas volumes elute from the sample tube unchanged. The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that eluted from the sample. The quantity of each pulse of reactant gas is determined by the loop volume on an automatically controlled valve, or from syringe injections. Three loops of different volumes are provided with the ChemiSorb 2750 analyzer.

BET Surface Area Analysis

The BET Surface Area analysis lets you evaluate total surface area of the catalyst before and after chemisorption. Pore-plugging phenomena which might occur due to the irreversible adsorbed species during chemical reactions, as well as the occurrence of sintering, can be studied.

After outgassing the sample, mixtures of nitrogen and helium (typically 5 to 30% nitrogen) flow over the sample which is immersed in a liquid nitrogen (LN_2) bath. The adsorption and, upon removal of the LN_2, the desorption of the N_2 are recorded. The amount of nitrogen desorbed and the sample mass are used to calculate total specific surface area.

The entire BET analysis — or even repeat analyses — is performed in situ.

Langmuir Surface Area analysis

The Langmuir surface area analysis allows you to evaluate the total surface area of the catalyst and is especially useful for adsorbate/adsorbent systems that adsorb only a monolayer. These materials typically exhibit a Type 1 isotherm and are often microporous. The Langmuir surface area analysis may be best applied to zeolites and microporous carbons – typically the Langmuir surface area will exceed the BET surface area for these materials and provide a more accurate estimation of the total surface area.

The Langmuir surface area report may be applied to a wide range of gas concentrations and is not limited to the typical BET range (5-30%).

Total Pore Volume analysis

The Total Pore Volume is a single-point estimate of the pore capacity of a material. The total pore volume analysis is usually conducted near the saturation pressure of the adsorbate (0.995 P/Po). The total pore volume of a material can be determined on both fresh and used materials, the difference in pore volume may indicate pore plugging and directly relate to changes in the performance of catalysts and adsorbents.

For high surface area (> 100 m^2/g), a sample quantity less than 50 mg is recommended. The high sensitivity combined with the large quantity of adsorbed gas allows the use of smaller sample quantities while maintaining high precision. The reduced sample quantity will also reduce the likelihood of saturating the high sensitivity detector.

Specifications

Your system must meet or exceed the following requirements:

- Pentium 733 MHz computer (or equivalent)
- CD-ROM drive
- USB port
- 128 megabytes of main memory
- 1-gigabyte hard drive
- SVGA monitor (800 x 600 minimum; 1024 x 768 recommended)
- Windows 2000 or Windows XP Professional

2. INSTALLATION

This chapter describes how to install the ChemiSoft program.

The ChemiSoft option includes the following:

- Furnace controller and operating accessories
- One CD containing the software program
- Operator's manual

Notify Micromeritics immediately if you failed to receive all items (compare your packing list with items received).

Installing the Software

The ChemiSoft software is supplied on a CD. Perform the following steps to install the software:

1. Insert the program CD into the CD-ROM drive.

2. Select **Start** from the Status bar, then Run from the Start menu.

3. Enter the name of the drive designator, followed by setup. For example, if your CD-ROM drive is drive e, enter: **e:setup**

    Alternatively you may click **Browse**, navigate to the CD-ROM drive, and select **setup.**

4. Click **OK**; the New Installation dialog is displayed.
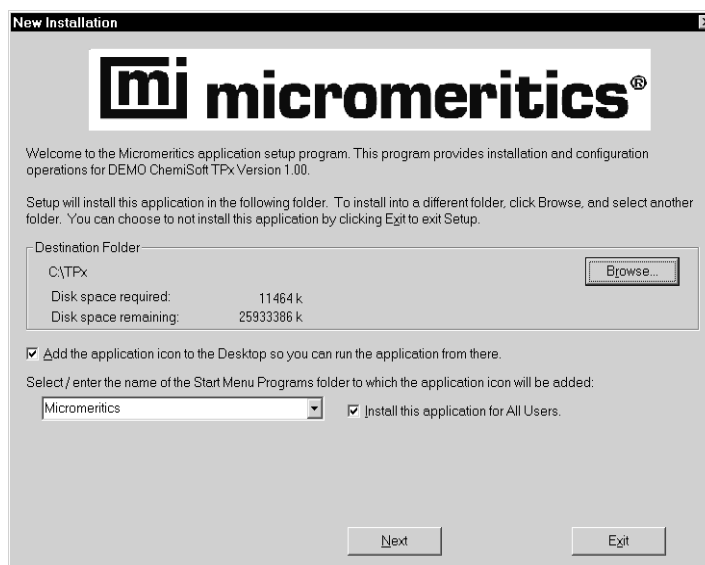
5. The **Destination** folder group box displays the amount of current disk space, the amount of disk space required for the application, and the directory into which the application will be installed. If you wish to install the application into a different directory, click **Browse** to choose the directory.

6. Click the **Add icon** option to run the ChemiSoft application from your desktop.

7. The ChemiSoft icon is added to the Micromeritics folder by default; if you prefer a different folder, click the down arrow and choose the desired folder.

8. Click **Next,** the Analyzer Configuration dialog is displayed.
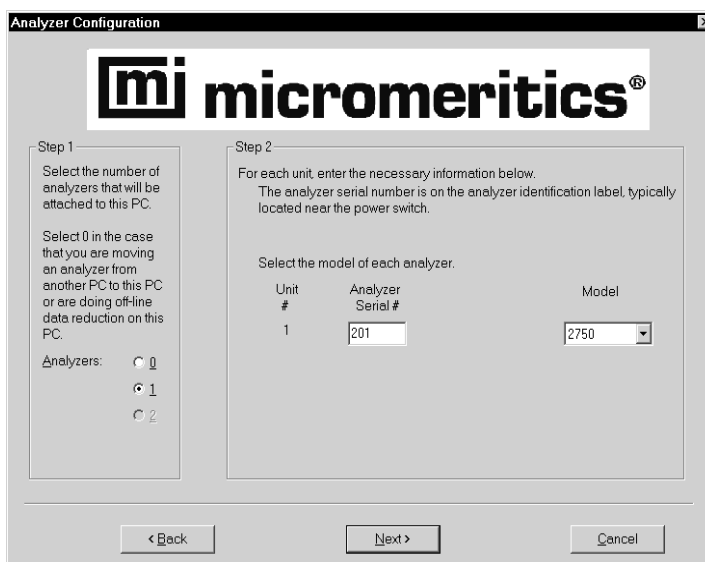
9. Ensure that 1 is selected in the Step 1 group box.

10. In the Step 2 group box, enter the serial number of the analyzer, then choose from the drop-down list the model number of the ChemiSorb that you have.

11. Click **Next** to complete installation; the Installation complete dialog box containing the Readme file is displayed.

12. Use the scroll bar if you wish to read the contents of the file, then click **Finish** to close the dialog box.

13. Do not remove the ChemiSoft program CD. The CD must remain in the drive when connecting the USB cable (refer to the next procedure).

Installing the Furnace Controller

Connecting the USB Cable

1. Connect one end of the USB cable to the **USB** port on the controller, and the other end to the USB port on the computer.

    • **Windows XP systems**: the Found New Hardware Wizard is displayed, prompting you through the process.

    • **Windows 2000 systems**: the driver is loaded automatically.

2. Remove the CD containing the ChemiSoft program and store in a secure location.

> **Do not disconnect the USB cable from the furnace controller while the ChemiSoft program is operating. Doing so will cause the program and any operation in progress to terminate.**

Selecting the Input Power

All units leave the factory set for 120 VAC and with the line fuse removed. The correct setting of the universal power entrance must be checked and the appropriate fuse installed before the furnace controller can be operated. The furnace controller operates with 100, 120, 200, or 240 VAC at 50 or 60 Hz. Voltage selection and fusing are made at the power connector, which is located on the rear panel of the unit.

> **The power cord should be disconnected from the controller before opening the cover on the input power connector. Failure to disconnect the power cord could result in electrical shock.**

1. Place the controller on the tabletop next to the analyzer with the rear panel facing outward.

2. Ensure that the power cord is disconnected from the controller.

3. Check the voltage setting on the cover of the power entrance, then open the cover and remove the fuse block.

> **If the voltage setting is incorrect, you will find instructions in the next step for selecting the correct voltage.**

a. Using a pointed object, pop the cover open.

b. The cover is hinged and cannot be removed; grasp the upper side with your fingers and swing it downward.

Fuse block

Swing the cover downward; the cover is hinged.
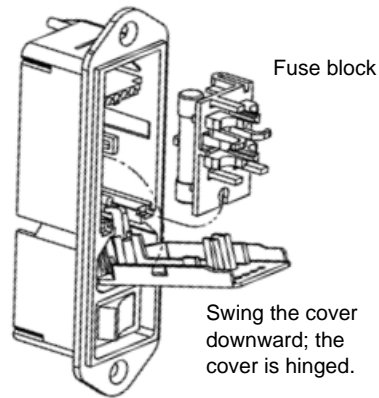
c. Remove the fuse block; you may have to use needle-nose pliers to grasp the fuse block.

4. Advance to Step 5 if the line voltage setting is correct. If the setting is incorrect, perform the following steps before proceeding:

a. Using needle-nose pliers, grasp the edge of the voltage selector card and pull it straight out of the power connector housing.

Grasp the card here and pull straight out of the housing.
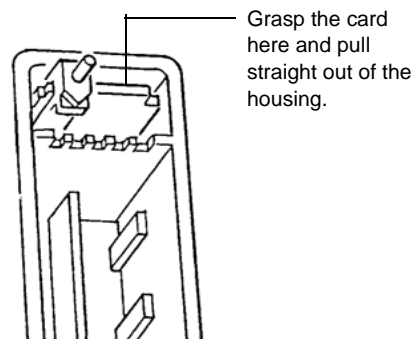
b. Orient the voltage selector card so that the desired voltage is indicated at the bottom. Orient the indicator pin so that it points upward.
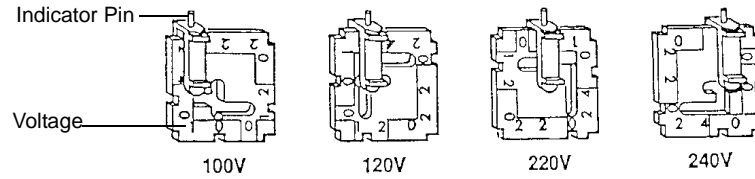
c. Insert the voltage selector card into the power connector housing with the edge containing the desired voltage first, and with the printed side toward the power connector.
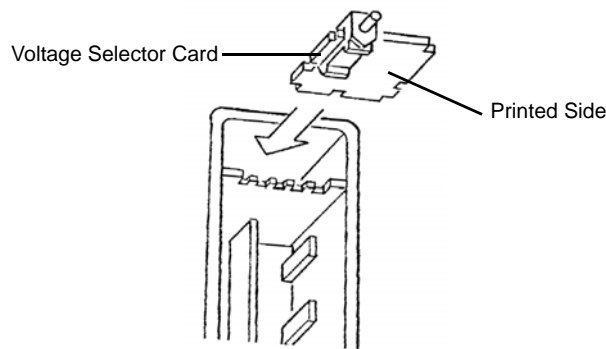
5. Fuse the input power line according to local safety practices. The input power connector can be used with either a single-fuse arrangement (100-120 VAC) or a double-fuse arrangement (200-240 VAC).

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

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

Power Source	Fuse
100-120 VAC	5.0 Amp (slow-blow) Type 3AG
200-240 VAC	2.0 Amp (slow-blow) 5 x 20 mm

6. Insert the fuse block into the input power connector. The arrangement shown here is for a single fuse.

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

7. Snap the fuse block into place.
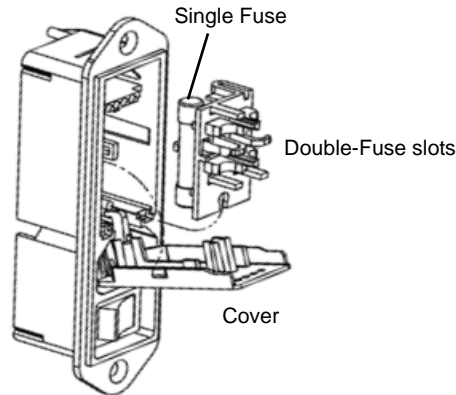
   • If the double-fuse arrangement is desired, the fuse block is positioned so that the side with the double-fuse slots is away from the cover.

When using the double-fuse arrangement, the fuse block will not snap into place. Simply position the fuse block properly. When you close the cover, the fuse block will seat properly.

8. Close the cover. Once the fuse block and cover are in place, the position of the indicator pin shows the input power selected.

9. Plug the controller power cord into an appropriate power source.

Programming the Controller

Program the Controller following the instructions provided in Chapter 5 of the Installation and Operation Handbook (included with your Controller).

Installing the Furnace

The furnace is installed on two metal supports (posts); one located on the underside of the top panel and one directly below on the shelf portion of the analyzer.

1. Position the furnace underneath the top support, aligning the support with the cavity in the furnace. Push the furnace upward until it stops.

2. Then lower the furnace down onto the bottom support. Make sure the furnace is secure on both supports.

3. Connect the furnace power cord to the **Furnace Power** connector on the controller, then connect the other end to the furnace.
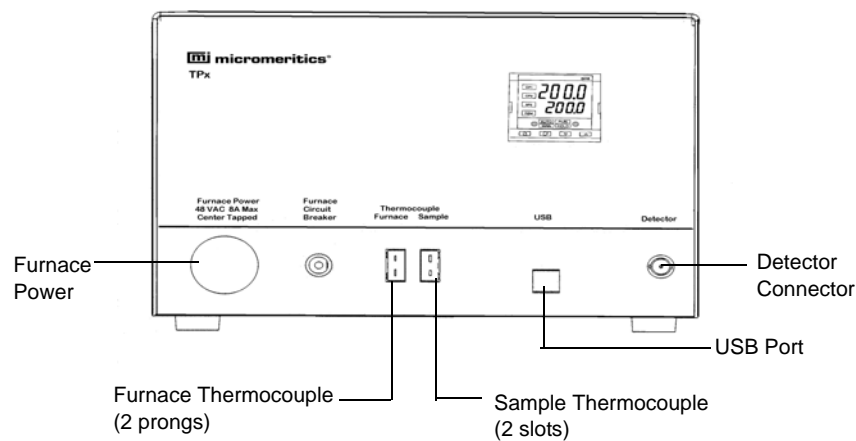
Connecting the Detector Cable

Connect one end of the detector cable to the **Detector** connector on the controller, and the other end to the connector on the analyzer labeled **Detector Signal Out**.

Attaching the Thermocouples

1. Attach one end of the Furnace thermocouple cable (with the two slots) to the **Furnace** thermocouple connector on the controller, and the other end to the connector on the side of the furnace.

Thermocouple
Connector

2. Attach the pronged end of the Sample thermocouple cable to the **Sample** thermocouple connector on the controller, and the slotted end to the sample thermocouple.

Using the Setup Program for Other Functions

After initial installation of the ChemiSoft program, the application setup program is also used to:

- • Upgrade software
- • Move an analyzer from one computer to another one
- • Remove an analyzer from the computer on which it is installed
- • Uninstall the ChemiSoft program

To start the application setup program.

1. Ensure that the ChemiSoft program is not operating.

2. Insert the CD into your CD-Rom drive.

3. Select Start from the Status bar, then Run from the Start menu.

4. Enter the drive designator of the CD-ROM drive, followed by setup. For example:**e:setup**

    Alternatively you may click **Browse**, navigate to the CD-ROM drive, and select **setup.**

5. Click **OK**; the Setup Welcome screen showing the options available is displayed.
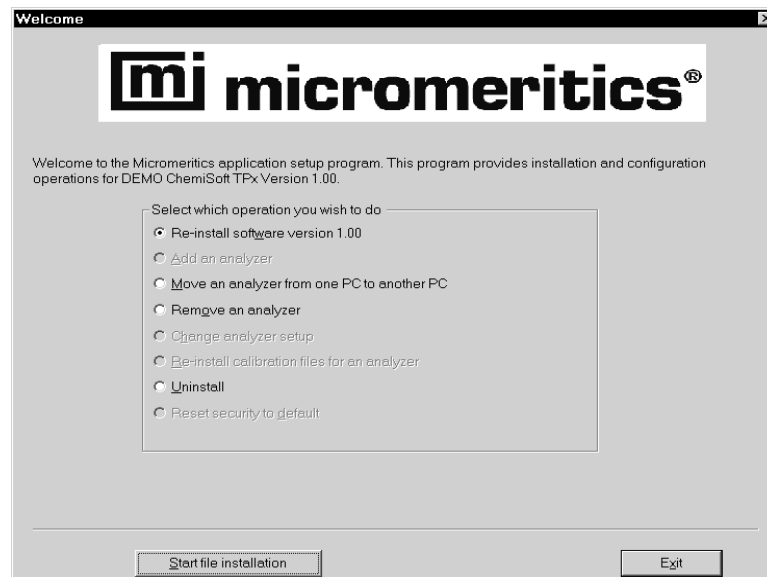
6. Choose the desired option.

Reinstalling the Software

When you install a software upgrade the system installs all of the application files that do not already exist on the computer. There are three types of subsequent installations:

- 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:

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

Moving an Analyzer from One Computer to Another Computer

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

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

1. Install the analysis program on the destination computer. Refer to **Installing the Software** 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.
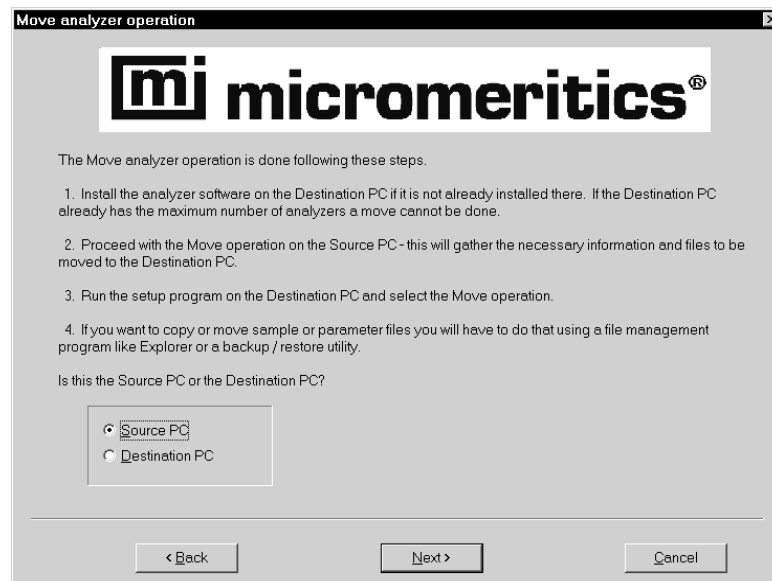
4. Select **Source PC**, then click Next; the following dialog is displayed.
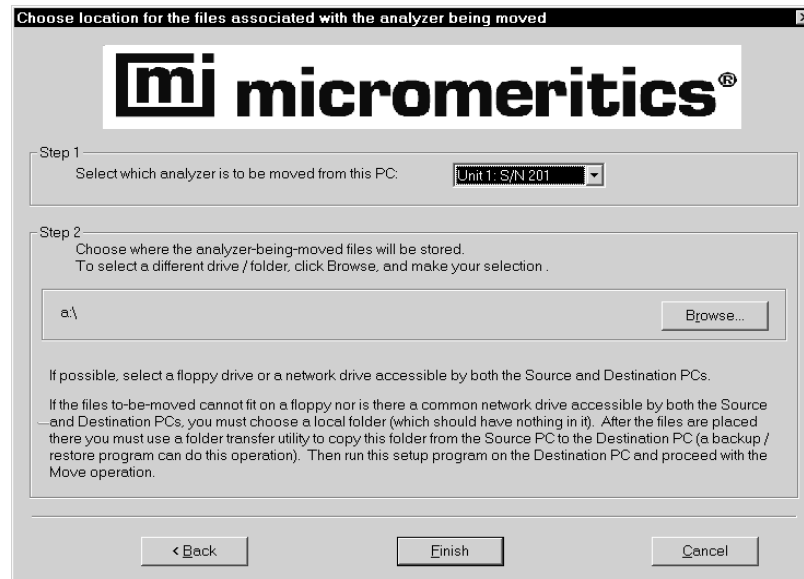
5. In the Step 1 group box, the current analyzer should display in the field of the drop-down list.

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 here, use a folder transfer utility to copy this folder from the Source PC to the Destination PC.

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

8. Remove the CD from the Source computer and insert it into the appropriate drive on the Destination computer.

9. Start the application setup program on the Destination computer.

10. 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).

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

12. In the Step 1 group box, enter the serial number of the analyzer being moved.

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

14. Click **Finish**; the files are moved to the Destination computer and the setup Welcome dialog is displayed.

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

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 application 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.
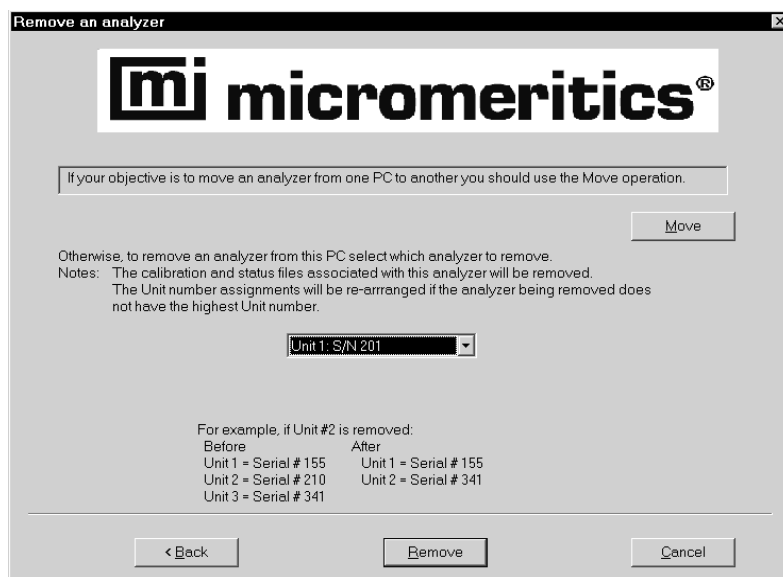
3. The serial number of the analyzer should display in the drop-down list.

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

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

Uninstalling the Software

When you uninstall the ChemiSoft program, the application removes the ChemiSoft program; it does not remove data files. Perform the following steps to uninstall the program:

1. Start the application setup program. Refer to **Using the Setup Program for Other functions** earlier in this chapter.

2. From the Setup dialog, select **Uninstall**, then click **Nex**t; the Uninstall ChemiSoft TPx dialog is displayed.
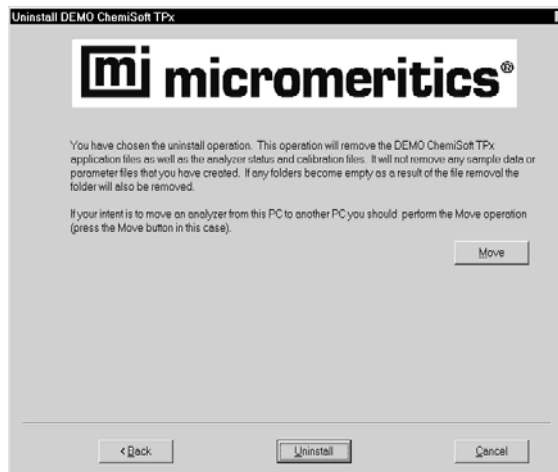
3. Click **Uninstall**; the Select Uninstall Method dialog is displayed.
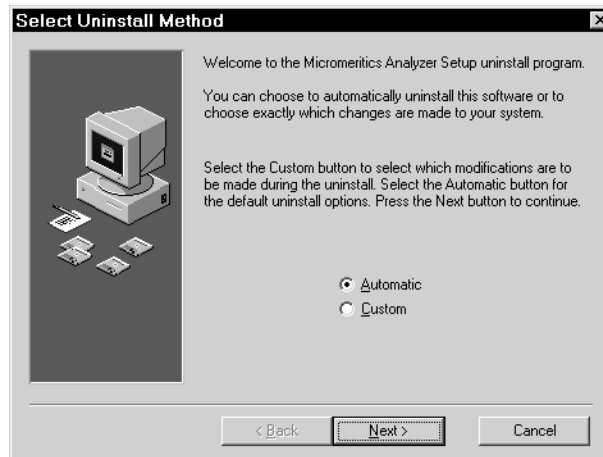
4. Choose one of the following:

   • **Automatic**: the system uninstalls the ChemiSoft program automatically
   • **Custom**: displays a dialog allowing you to choose the items you wish to uninstall

5. Click **Next**; the Perform Uninstall dialog is displayed. Click **Finish** to complete the uninstall operation.

3. USER INTERFACE

This chapter provides instructions for using the software, as well as the menu structure of the ChemiSoft program. It also contains a brief description of the front panel display on the Controller. Refer to Chapter 2 of the Controller Installation and Operation Handbook for a more detailed description.

ChemiSoft Software

The ChemiSoft program operates in the Windows environment and requires familiarity with standard Windows operations such as using the mouse, menus, and dialog boxes. This manual provides brief instructions for standard operations; however, you may need to refer to your Windows documentation or the Windows help system to clarify functions which are specific to Windows. Since most Windows operators use a mouse, this manual is written for mouse-driven operations.

Shortcut keys

Shortcut keys may be used instead of, or in addition to, a mouse. With the main menu displayed, simply press the shortcut key(s) for the function desired. For example, if you wish to open a Sample Information file, press **F2** and the Open Sample Information dialog box is displayed. You may also access any function that contains an underlined letter by pressing **Alt** plus the underlined letter. For example, to access the **File** menu, simply press **Alt**, then **F**.

Table 3-1 lists the shortcut key(s) available for operating the ChemiSoft program.

Shortcut Menus

Shortcut menus (sometimes referred to as context-sensitive menus) are available for onscreen graphs, tabular reports, and for use in the Peak Editor. These menus are accessed by clicking in the graph or table area and clicking the right mouse button. For example, right-click in a column of an onscreen report and the following menu is displayed.
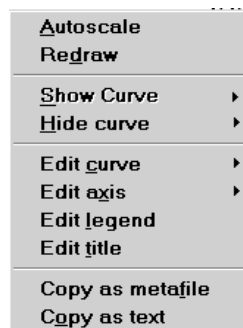
Table 3-1. Shortcut Keys

Key(s)	Function
F1	Access Help
F2	Open a sample information file
F3	Open an analysis conditions file
F4	Open a report options file
F6	Tile windows
F7	Cascade windows
F8	Start report
F9	Close all open reports
Alt + F4	Exit the ChemiSoft program
Shift + F2	List sample information files
Shift + F3	List analysis conditions files
Shift + F4	List report options files
Shift + F9	Displays the shortcut menu for onscreen reports

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.

Push Button   A push button is used to display subdialog boxes in which to edit, enter, or specify additional information about the subject matter. For example, click **Dates** on the Open Sample Information dialog to display a subdialog enabling you to specify dates in which to display sample files (explained later in this chapter).
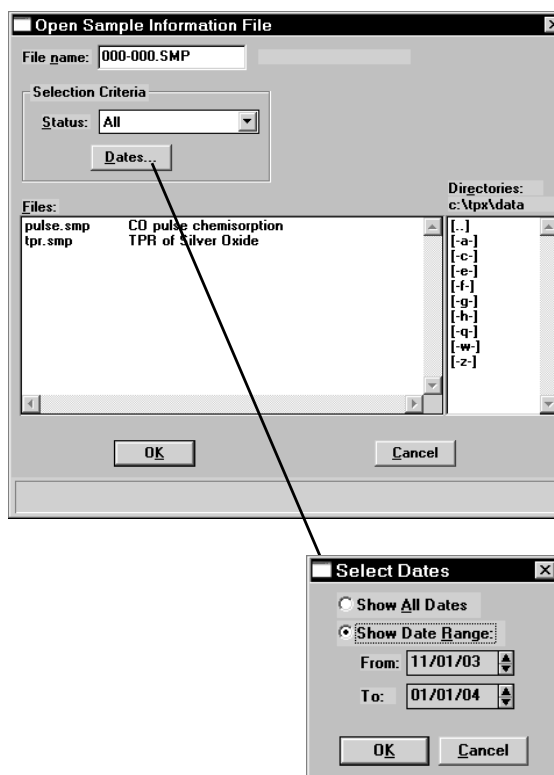
Push buttons are also used to invoke an action; for example, click **OK** on the Select Dates dialog to save the changes and close the dialog, or click **Cancel** to discard changes and close the dialog.

Radio Button   Radio buttons are contained within a group box. These
                             buttons are used to select options; you may choose only
                             one item.
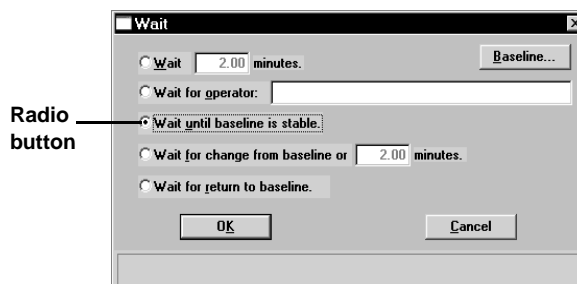
Check Boxes   Check boxes also are used to select options. You may
                             select as many items as you wish.
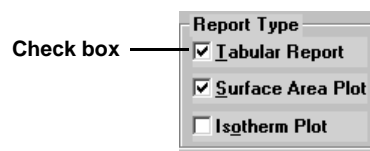
Drop-down lists   A drop-down list is indicated by a down arrow to the
                             right of the field. Drop-down lists contain a list of
                             options from which you may choose one.
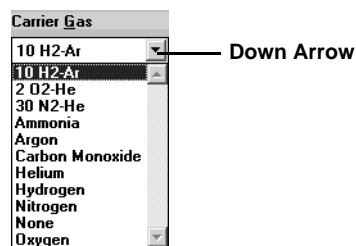
Information Bar   Some dialog boxes contain information pertinent to the
                             selected field in an information bar across the bottom of
                             the dialog. For example, a range is shown for fields in
                             which numeric entries are required.
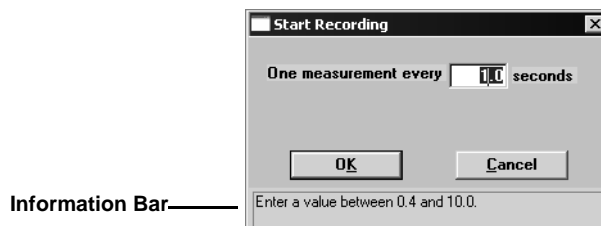
Selecting Files

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:
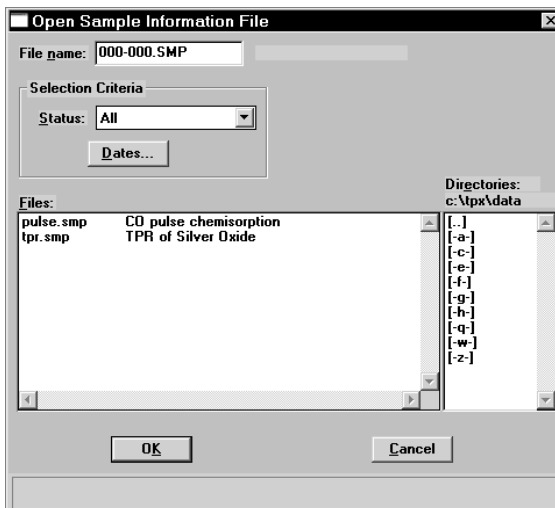
When opening sample information files, the next automatically sequenced file number appears in the **File name** field. For parameter files, a wildcard character with the default extension for the type of file you are opening appears in the **File name** field. Simply select the desired file, then click **OK** to open the file. Alternatively, you can double-click on the desired file.

You may limit the list of files displayed in the **Files** list box by choosing one (or more) 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 could filter a list to display files beginning with **P** by entering **P*.smp** (or other appropriate extension) in the **File name** field.

- Enter a range of dates. Click **Dates**; the Select Dates dialog is displayed.

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

For convenience in choosing dates, the following function keys are available when the **From** or **To** field is selected:

F2 Clears the field
F3 Inserts the current date
F4 Displays a calendar from which to 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.
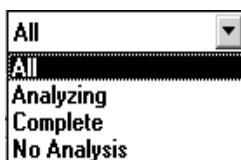
Table 3-2. File Status and Description

Status	Description
All	All sample information files in the specified directory and within the specified range of dates
Analyzing	Sample information files that are currently being used in an analysis.
Complete	Sample information files that were used in an analysis that has been completed.
No Analysis	Sample information files that have not been used in an analysis.

File Name Conventions

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

The following table shows the file name extensions for the ChemiSoft program.

Table 3-3. Default File Name Extensions

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

Menu Structure

All functions for the ChemiSoft program are located on menus which are accessed from the Menu bar. Each menu contains commands, and in some cases a submenu. A submenu is indicated when the command is followed by an arrow.
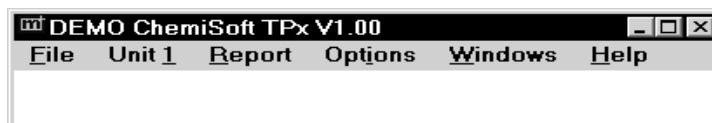
File	The options on the File menu enable you to create and manage sample and parameter files. (Chapter 5)
Reports	Enables you to generate and close reports, and use the Peak Editor. (Chapter 6)
Options	Allows you to choose sample editing mode, specify your system configuration, select data presentation formats, and specify a location for predefined parameter files. (Chapter 7)
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 information about the ChemiSoft program. (this chapter)

Windows Menu

```
Windows
  Tile                 F6
  Cascade              F7
  Arrange Icons

✓ 1 C:\TPX\DATA\000-005.SMP
  2 C:\TPX\DATA\000-010.SMP
```
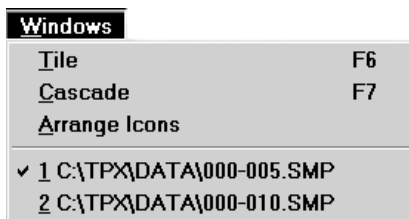
Tile   Resizes all open windows and arranges them side by side.

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 all minimized icons in an orderly manner.

Open Files   Displays all currently open files. The active file is preceded with a check mark.

Help Menu

```
Help
  Help              F1
  About ChemiSoft...
```

Help   Lists help topics for the ChemiSoft program.

About   Displays information about the ChemiSoft program

Controller Display

Certain functions on the controller are used to enter parameters for your analysis. This section provides a brief description of the controls on the front panel of the controller. Refer to the Installation and Operation Handbook for a more detailed description.
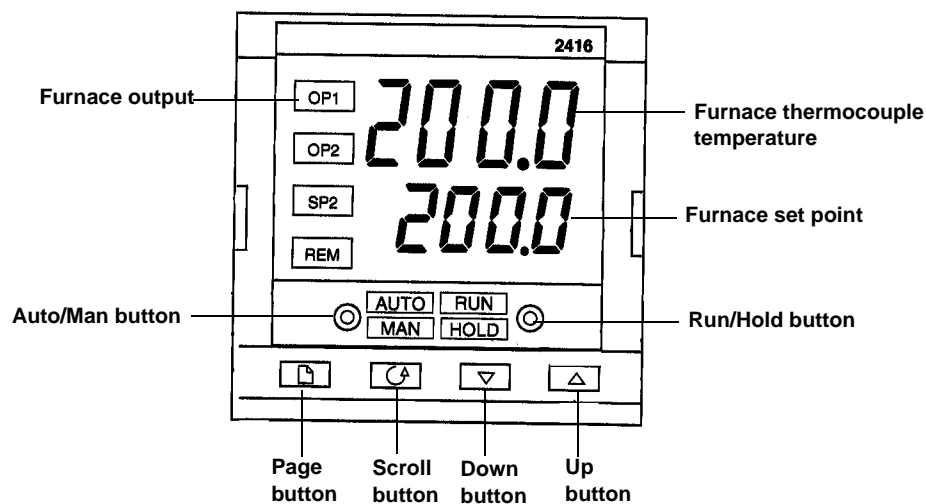
OP1	Furnace output; blinks while the furnace is heating during a dwell step
OP2 **SP2** **REM**	Not used with the ChemiSoft program
Auto/Man button	Enables you to toggle between Automatic and Manual mode. When in Automatic mode, **Auto** is illuminated and when in Manual mode, **Man** is illuminated.
Run/Hold button	Press the button once; **Run** is illuminated. You should use this mode when you start the analysis. If you press this button twice, the controller will be put on **Hold**. Press the button again to restart, or press and hold for two seconds to reset the controller.
Page button	Press to select a new list of parameters
Scroll button	Press to select a new parameter in a list
Down button	Press to decrease the set point value

Up button   Press to increase the set point value

Upper readout   Displays the temperature of the furnace thermocouple

Lower readout   Displays the furnace set point

4. GENERAL OPERATING PROCEDURES

This chapter provides brief step-by-step procedures for operating the ChemiSoft program with your ChemiSorb analyzer.

Specifying Sample Defaults

Sample information files include the information required to perform analyses and collect data. The ChemiSoft program automatically generates sample information file names and assigns default values. The program is shipped with default values, however, they may be unsuitable for your type of material. The Sample Defaults command on the Options menu enables you to specify values specific to your type of analysis. You can always edit the values when the sample file is created.

The sample information presentation can be presented in a basic, advanced, or restricted format. Sample defaults can be specified in the basic and advanced formats only; you cannot specify sample defaults using the restricted format. The sample defaults you specify for the basic format display for the restricted format.

If desired, you can define parameter files before entering sample defaults (refer to Defining Parameter Files in this chapter).

Advanced Format

1. Select **Sample defaults** from the Options menu; the Sample Defaults dialog is displayed.

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

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

> **You can also edit the word Sample that displays for the prompt in the sample file. For example, you may prefer to use *Test* or *Material* to display as the prompt.**

4. Edit the **Operator** and **Submitter** lines as desired. Or, you may have either (or both) of these limes omitted entirely by selecting **Omit**.

5. In the **Mass** field, enter a value to represent the amount of sample you typically use in your analysis. This value can be edited when you create your file, or after the analysis.

6. Click the **Analysis Conditions** tab and enter appropriate information for your most commonly analyzed sample material. Remember, you can always edit this information in the sample file when it is created.

> **The default values you specify for Analysis conditions and Report options display as the defaults for newly created parameter files.**

7. Click the **Report Options** tab and choose the desired reports. Again, this selection can be edited in the sample file when it is created.

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

Basic Format

The defaults you specify for this format also display as the defaults for sample files created using the Restricted format.

1. Select **Sample defaults** from the Options menu; the Basic Sample Information Defaults dialog is displayed.
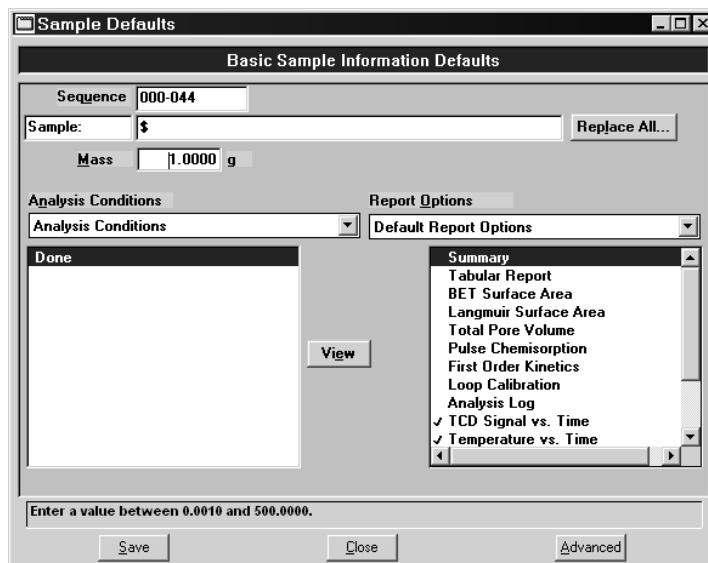
2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select File, Open, Sample information.

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

> **You can also edit the word Sample that displays for the prompt in the sample file. For example, you may prefer to use *Test* or *Material* to display as the prompt.**

4. In the **Mass** field, enter a value to represent the amount of sample you typically use in your analysis. This value can be edited when you create your file, or after the analysis.

5. Select the down arrow to the right of the **Analysis Conditions** field to choose a default parameter file. The steps included in this file will display in the window below the field. You may view the details of these steps by selecting **View** .

6. Select the down arrow to the right of the **Report Options** field to choose a default parameter file. The reports chosen (preceded with a check mark) for the selected file will display in the list of reports below the field. Edit selections if desired.

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

Defining Parameter Files

Analysis Conditions and Report Options files can exist as part of the sample information file or as an individual parameter file. Having these files exist independently allows you to use them over and over again. For example; unless you expect each analysis to use unique sets of analysis conditions and report options, you can create a group of standard parameter files containing the values you expect to use most frequently. The contents of these files can be copied and imported into multiple sample information files, providing speed and consistency. After these values have been copied and imported into the sample information file, they can be edited in any way that you wish. The original parameter file remains intact and ready for its next use.

This makes it possible for one person (such as a laboratory manager) to create standard parameter sets that can be used easily by others (such as laboratory technicians).

Several predefined parameter files (located in the PARAMS directory) are included with the ChemiSoft program. These files are examples only and the gases specified in these files may not match the gases connected to your analyzer. If you wish to use one of these files as a starting point, simply create a new file and select **Replace**. A dialog is displayed so that you can select an existing parameter file. After the values from this file are copied into the current file, you can edit them as desired.

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 parameter file directory, they will not display in the drop-down lists on the Basic and Restricted sample file editors.

Parameter files can also be created from the Advanced Sample information window using the **Save as** function on the **File** menu. For example, you have a sample information file open that contains exactly the analysis conditions that you use often in your analyses. Simply select **Save As**, **Analysis Conditions** from the File menu and enter a name for the file. After this file has been created, it can be copied into as many sample files as you wish using the **Replace** push button on the sample file editor.

Analysis Conditions

Analysis conditions specify data to guide an analysis.

1. Select **Open**, **Analysis conditions** from the File menu; the Open Analysis Conditions File dialog is displayed.

2. Enter a name for the file (up to eight characters) in the **File name** field.

3. Click **OK**, then **Yes** to create the file; the Analysis Conditions dialog is displayed.
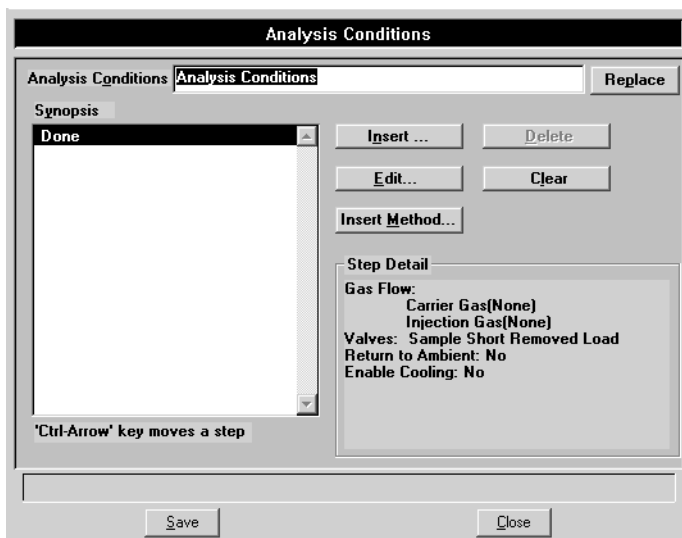
4. Enter a description in the **Analysis Conditions** field. Use a description that will help you identify the conditions specified. You can use up to 42 characters.

5. Click **Insert** to insert steps for the analysis. Each step you insert has an associated dialog, which contains the parameters for that step. You can insert up to 1000 steps in the Synopsis.

6. Choose **Experiment** (or Loop Calibration), then click **OK**; an associated dialog is displayed.

7. Define the experiment (or calibration) as desired, then click **OK**; the experiment (or calibration) will display in the **Synopsis** window; details of each step you specify will display in the **Step Detail** group box.

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

Report Options

1. Select Open, Report options from the File menu; the Open Report Options File dialog is displayed.

2. Enter a name for the file (up to eight characters) in the **File name** field.

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

```
┌─────────────────────────────────────────────────────────┐
│                    Report Options                        │
├─────────────────────────────────────────────────────────┤
│ Report Options │Default Report Options    │   │Replace...││
│                                                          │
│ ☑ Show report title │Micromeritics Instrument Corporation││
│ ☑ Show graphic   │Miclogo.emf              │ │Browse... ││
│        Width │ 2.000 │ in.  Height │ 0.250 │ in.         │
│                                                          │
│           Selected Reports                               │
│          ┌──────────────────────────────┐▲              │
│          │ ✓ Summary                    │█              │
│          │ ✓ Tabular Report             │█              │
│          │   BET Surface Area           │█              │
│          │ ✓ Langmuir Surface Area      │█              │
│          │   Total Pore Volume          │              │
│          │   Pulse Chemisorption        │    ┌───────┐  │
│          │   First Order Kinetics       │    │Edit...│  │
│          │   Loop Calibration           │    └───────┘  │
│          │   Analysis Log               │█              │
│          │ ✓ TCD Signal vs. Time        │█              │
│          │ ✓ Temperature vs. Time       │▼              │
│          │ ◄                          ► │              │
│          └──────────────────────────────┘               │
│                                                          │
│ Enter a value between 0.100 and 7.500.                   │
│          ┌──────┐              ┌──────┐                  │
│          │ Save │              │Close │                  │
│          └──────┘              └──────┘                  │
└─────────────────────────────────────────────────────────┘
```

4. Enter a description (up to 42 characters) in the **Report Options** field. Use a description that will help you identify the types of reports selected.

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 do not wish to have a title on your report.

> **If your company logo exists as a bitmap (bmp) or enhanced metafile (emf) and you would like to have it display in the report header, select *Show graphic*. Then click Browse to select the file. Use the Width and Height fields to specify the size.**

6. Select (or deselect) desired reports by: highlight the report and double-click. Alternatively, you can press the **Spacebar** after the report is highlighted. A report is selected when it is preceded with a check mark

7. Click **Edit** to edit report details if desired.

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

Creating Sample Information Files

A sample information file consists of information groups which, collectively, identify the sample, detail steps for the analysis, and specify the data reduction.

A sample file may be created in three different formats:

- **Advanced**
  Provides complete access to all parts of the sample file, allowing you to quickly edit parameters as required.

- **Basic**
  Presents a single window in which you select predefined parameter files for your analysis. You can also switch to the Advanced format to edit parameters if desired.

- **Restricted**
  Presents a single window in which you select predefined parameter files (identical to the Basic format). You cannot, however, switch to the Advanced format for editing purposes. This format is password-protected.
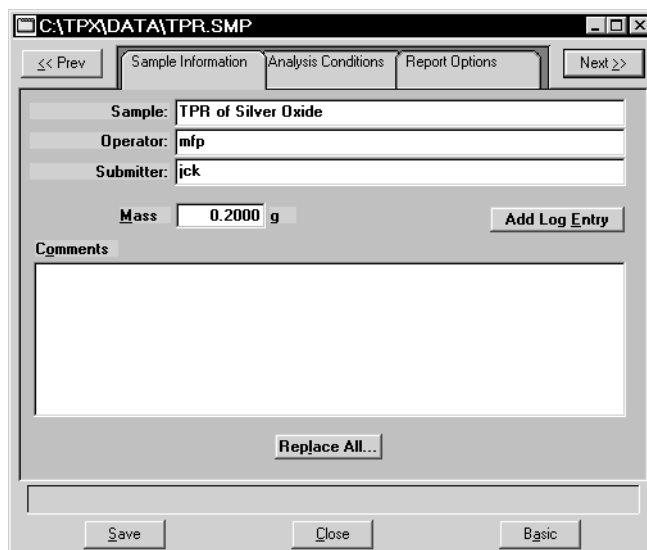
Select Option presentation from the Options menu to choose the format you wish to use.

Sample information files can be created in advance of the analysis or they can be created at the time of analysis, whichever is convenient for you.

Advanced Format

The Advanced format presents the sample information editor in a manner similar to that of an index card file. Simply click on the appropriate tab and enter the required information.

1. Select **Open**, **Sample information** from the File menu; the Open Sample Information dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field; you can use up to eight characters.

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

The values contained on the dialog are those specified in Sample Defaults. You can accept these defaults to create your file or continue with the subsequent steps to edit them.

4. In the **Sample** field, enter a description or add to the existing one. You can use up to 42 characters.

5. Edit the **Operator** and **Submitter** fields if desired. You can use up to 40 characters in these fields.

6. Use the **Comments** window to enter any comments about the sample or analysis conditions. Anything you enter in this field will display in the header of some reports.

7. Click the **Analysis Conditions** tab and define analysis conditions. Refer to **Defining Parameter Files** if you need assistance in completing this dialog.

8. Click the **Report Options** tab and specify desired report options. Refer to **Defining Parameter Files** if you need assistance in completing this dialog.

9. Click **Save**, then **Close**.

Basic Format

The Basic format allows you to create a sample file by choosing from predefined parameter files. The values in these files can be edited when needed by switching to the Advanced format.

1. Select **Open**, **Sample information** from the File menu; the Open Sample Information dialog is displayed.

2. Accept the next sequenced file number or enter a new name in the **File name** field; you can use up to eight characters.

3. Click **OK**, then **Yes** to create the file; the Basic Sample Information dialog is displayed.
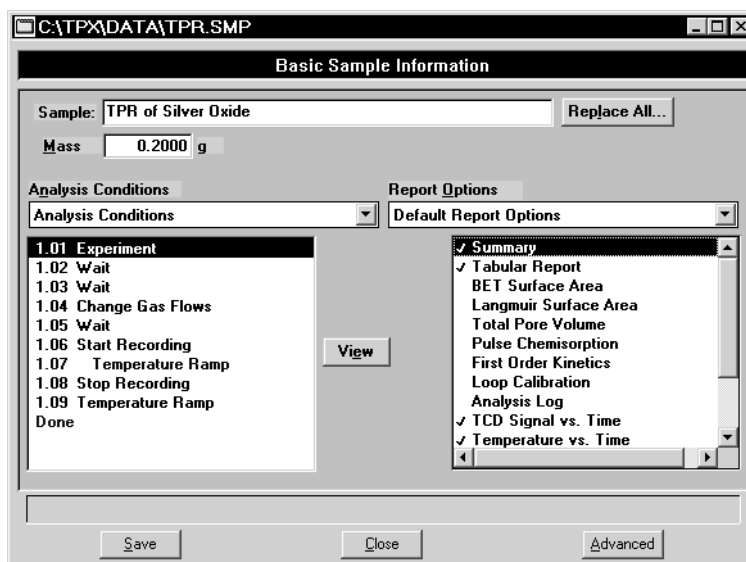
The values contained on the dialog are those specified in Sample Defaults. You can accept these defaults to create your file or continue with the subsequent steps to edit them.

4. In the **Sample** field, enter a description or add to the existing one. You can use up to 42 characters.

5. Click the down arrow to the right of the **Analysis Conditions** field and choose the file containing the appropriate analysis conditions. After you choose a file, its steps are displayed in the window below the **Analysis Conditions** field. You can view the details of the steps by highlighting the step and clicking **View**.

6. Click the down arrow to the right of the **Report Options** field and choose an appropriate file. You can select additional (or deselect current) reports by double-clicking on the report(s).

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

Restricted Format

Sample information files are created using the Restricted format in the same manner as with the Basic format. The name displayed in the title bar even displays as Basic Sample Information. However, the dialog for this format does not include the Advanced push button allowing you to switch to the Advanced format to edit parameters. Certain menu functions are also disabled with the Restricted format.
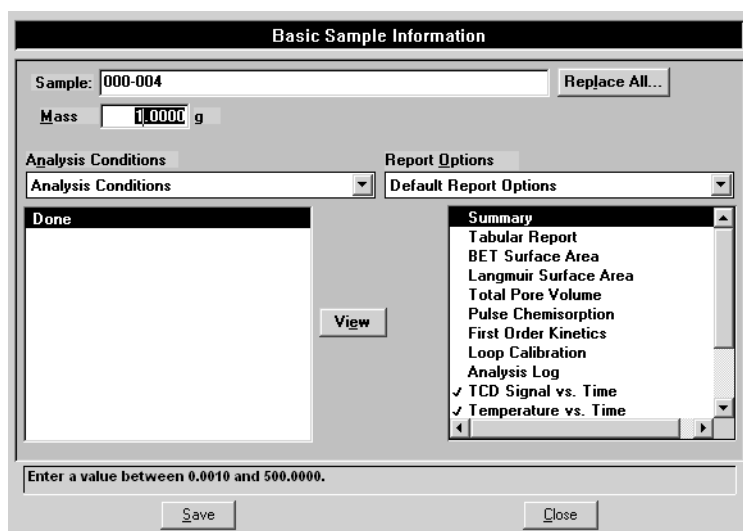
An Advanced push button is not included with the Restricted format.

Performing an Analysis

> ✏️ **Appendix F contains a tutorial for a typical multipoint BET analysis, including creating the sample file; performing the analysis; and editing peaks.**

Loading the Sample

1. Install the furnace on the analyzer (refer to Chapter 2 for instructions); open it fully.

2. Load the sample tube (containing the prepared sample) onto the sample port of the analyzer.

3. Carefully close the furnace around the sample tube and latch the furnace.

4. Insert the sample thermocouple into the desired position. You can insert the thermocouple inside the tube or on the outside of the tube.

Insert the thermocouple here if using outside of the tube.

Insert the thermocouple here if using inside of the tube.
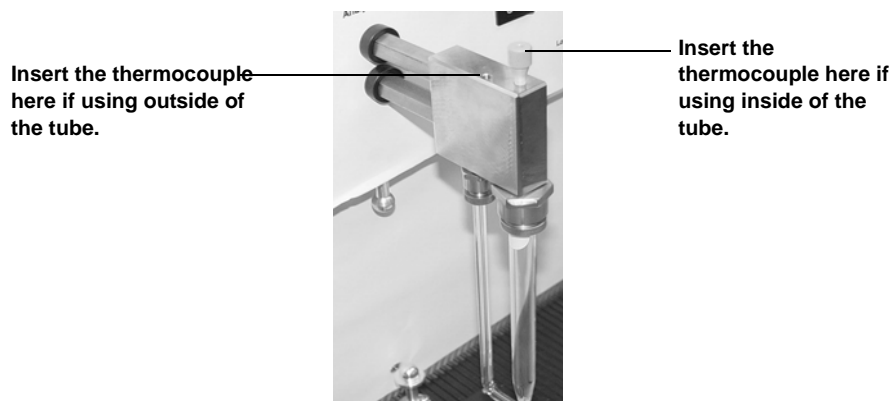
The best accuracy is obtained when the thermocouple is placed inside the sample tube; adjust the thermocouple so that the end is located in the sample or just above it.

If the thermocouple is placed outside the sample tube, make sure it is clamped against the sample tube (using the clamp provided in the accessories kit). Orient the thermocouple so that its tip is at the same level as the sample and that it does not extend below the point where the sample tube begins to taper.

Thermocouple

Clamp

Sample Tube
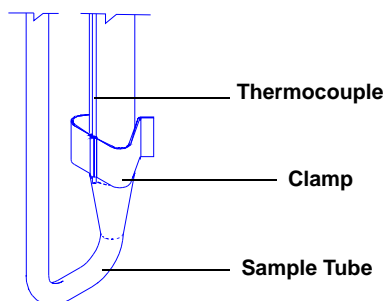
Programming the Eurotherm Controller

The Eurotherm controller must be idle before you can enter analysis parameters. The idle state is indicated when the Run/Hold lights are not illuminated. If either light is illuminated or flashing, press and hold the Run/Hold button for three seconds.

Since the controller will reset after a very brief idle period, it is best to have all information available so you can quickly complete the entries. Make a copy of the Worksheet provided in Appendix A, record the values, then enter them into the controller.

> **Quartz sample tubes may be used up to 1100 ⁰C. Borosilicate sample tubes must not be used above 500 ⁰C.**

1. Press ⬜ until **ProG LiSt** is displayed.

2. Press ⬜ until **SEG.n** is displayed; 1 defaults on line 2 of the display.

3. Press ⬜ to display **tYPE**.

4. Press ⬜ or ⬜ to select the type of segment; the ChemiSoft program uses only the following segments:

   rmP.r   Ramp to a new setpoint at a set rate

   dwEll   Dwell for a set time

> **When you see this symbol while performing the steps in the following table, refer to the appropriate column of your Controller Worksheet for the value.**

If you choose....	Perform the following steps....
rmP.r	Press ⬜ until **tGt** is displayed.
	Press ⬜ or ⬜ to set the target setpoint (ºC).
	Press ⬜ to display **rAtE**.
	Press ⬜ or ⬜ to set the ramp rate (ºC/min).
dwEll	Press ⬜ until **dur** is displayed.
	Press ⬜ or ⬜ to set the dwell time (min).

5. Press ⬜ ; **SEG.n** is displayed with 2 (representing the second segment) displayed on line 2.

6. Press ⬜ or ⬜ until **End** is displayed.

7. Press ⌨ until **End.t** is displayed on the top line and **dWEll** on the bottom line.

8. Press ▽ to choose **dWell**. This holds the furnace at the previously attained temperature.

9. Press ⌨ until **ProG LiSt** is displayed.

10. Press ▯ to return to the Home screen (the Home screen shows the current temperature on the top line and the current setpoint on the bottom line).

Starting the Analysis

1. Select **Start analysis** from the Unit menu; the Sample Analysis dialog is displayed with the Open Sample information dialog positioned on top.

2. Choose a file for your analysis (or accept the default to create a new one) and click **OK**; the Sample analysis dialog containing the parameters of the selected file (or the defaults) is displayed.
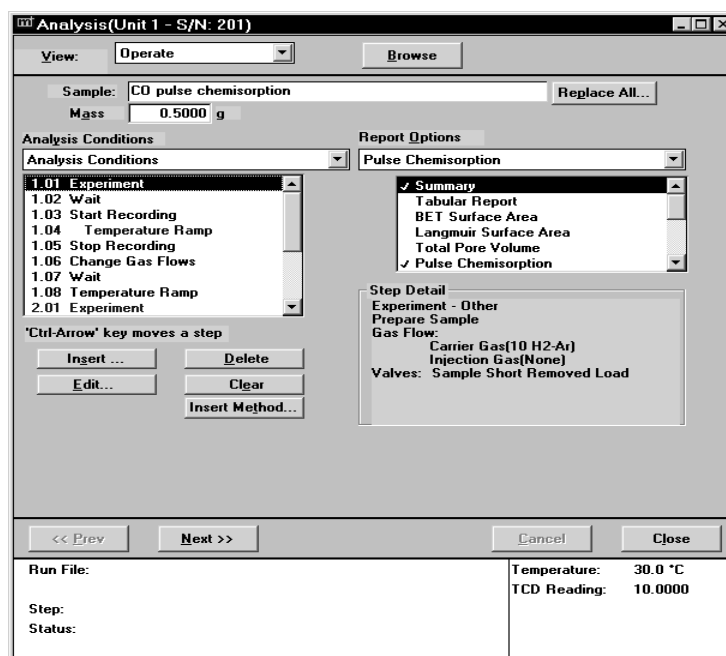
3. Review and verify analysis conditions and report options; edit if necessary.

4. Click **Next**; the second view of the analysis dialog is displayed.This view of the dialog displays the gases that are to be used in the analysis; ensure that appropriate gases are connected.

5. Click **Next**; this view of the dialog contains report output options. If you wish to have a report generated automatically after the analysis; select **Report after Analysis** and specify the destination.You can use the Start Report option on the Reports menu to generate reports if desired.

6.   Click **Next**; this view of the dialog displays safety information.
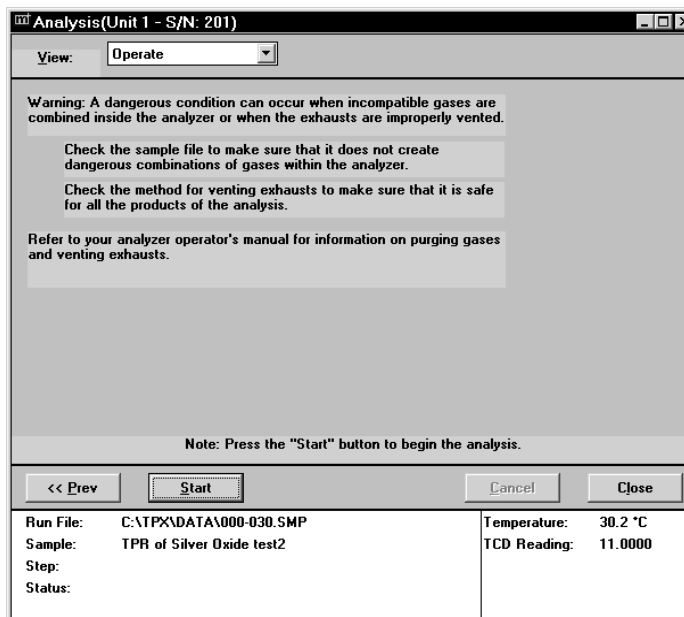
7.   Click **Start;** this view of the dialog, as well as subsequent dialogs, contains a **Prompt** section. Pay close attention to the information displayed at the Prompt and perform any requested action(s) accordingly. The information in this prompt enables you to interface manually with the analyzer to complete analysis.

Shows how the analyzer should be set up.

Lists the task(s) required before continuing with analysis.
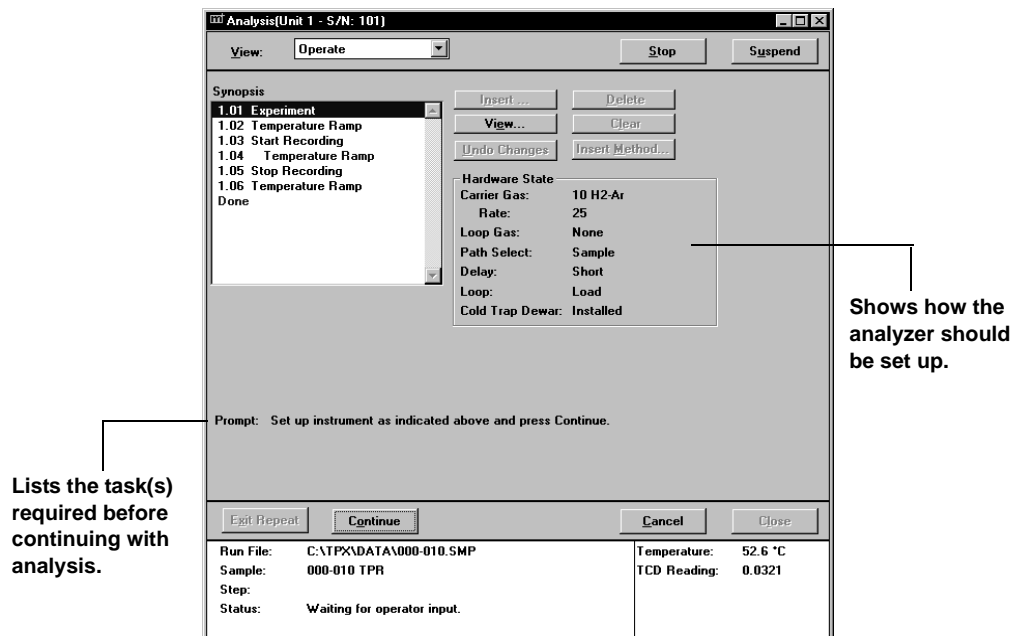
8.   After performing the task(s) listed, click **Continue**; this view of the analysis dialog instructs you to turn on the Eurotherm controller.

9. Press the **Run/Hold** button on the controller (the **Run** light illuminates), then click **Continue**.

10. Observe the **Run** light on the controller; when it begins to flash, the **End** step of the current step has been reached.

11. Press and hold the **Run/Hold** button for three seconds to return to its original programming mode; the **Run** light will turn off.

12. Continue to perform the task(s) as displayed, clicking **Continue** after each one.

Each time your analysis reaches a Temperature Ramp, program the Eurotherm controller (refer to Programming the Eurotherm Controller if you need assistance). Programming for each temperature ramp individually provides the best synchronization between the analysis conditions and the Eurotherm operation.

Generating a List

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

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

Perform the following steps to generate a list:

1. Select **List** from the File menu.

2. Select the type of file on which you wish information; a dialog similar to the one shown below is displayed.

The Status drop-down list does not display on a parameter file list dialog.
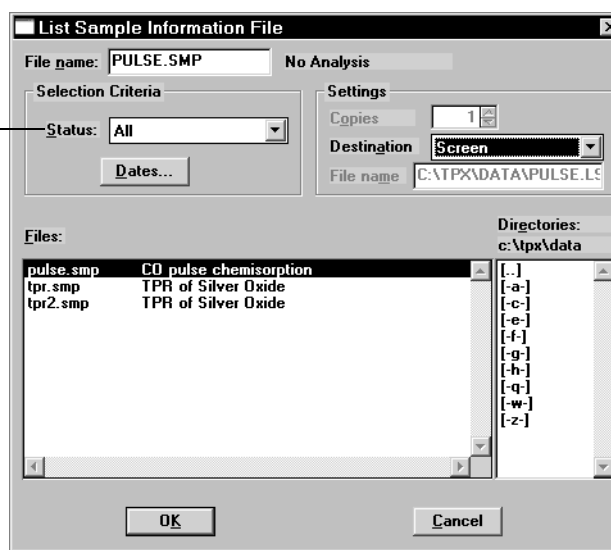
3. From the **Files** list box, choose the desired file(s). If you wish to include all files in the list, leave all files deselected.

4. At the **Destination** field, click on the down arrow and choose an output destination. If you choose **File** as the destination, enter a name in the **File name** field (or accept the default).

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

Exporting Data from a Sample Information File

Data from a sample information file can be exported in ASCII or Grams/32 format. ASCII data can then be imported into other applications, such as spreadsheets. Grams/32 data can be imported into the Grams/32 program (a peak-editing program used with early versions of Micromeritics' AutoChem software). Refer to Appendix D for a description of exported data.

Perform the following steps to export a sample information file:

1. Select **Export** from the File menu; the Export Sample File dialog is displayed.
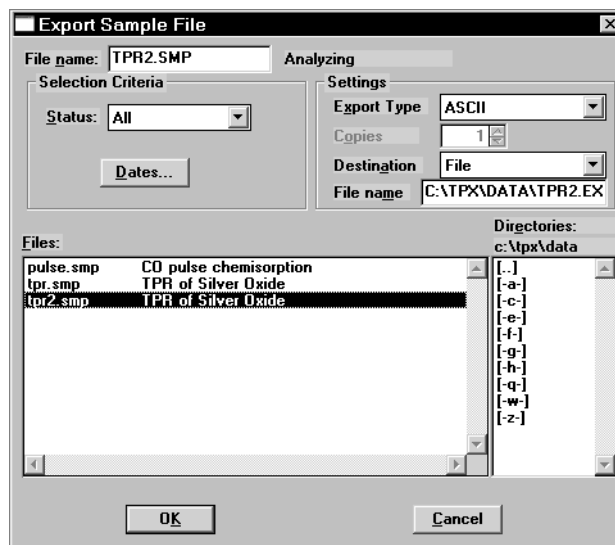
2. From the **Files** list box, choose the file(s) you wish to export. You can choose multiple files by holding down and clicking on the desired files.

3. From the Settings group box:

    a. Choose whether you are exporting in ASCII or Grams/32 format.

    b. Choose a destination for your exported file. If you choose **File**, enter a name in the **File name** field (or accept the default). If you choose **Printer** or **Printer/Plotter**, the **Copies** field is enabled allowing you to print up to 4 copies.

If you are exporting in Grams/32 format, you must choose File as the destination. Refer to Appendix D for additional information.

4.   Click **OK**; the Collected Data Export Options dialog is displayed.
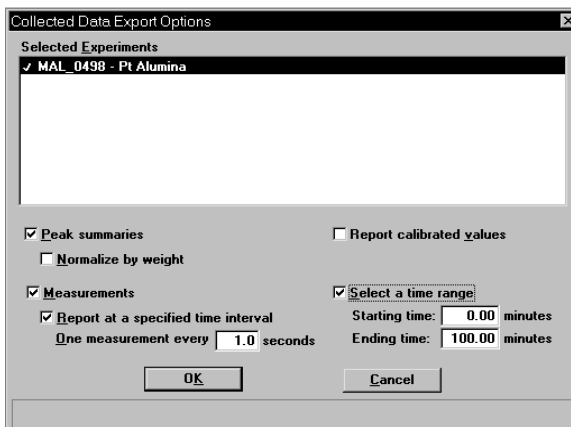
5.   Choose the experiments whose data you wish to export. Then select any other desired options. Refer to Chapter 5 for additional information on this dialog.

6.   Click **OK**; data are exported to the specified destination.

5. FILE MENU

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

Description

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

| **Open** | Opens an existing file or creates a new file. When you select this item, a drop-down menu is displayed containing three types of files: sample information, analysis conditions, and report options. Page **5-3**. |

Open   Opens an existing file or creates a new file. When you
                      select this item, a drop-down menu is displayed
                      containing three types of files: sample information,
                      analysis conditions, and report options. Page **5-3**.

Save   Saves the file in the active window. Page **5-44**.

Save As   Allows you to save the file that is in the active window
                      under a different name. It also can be used to save a
                      subset of a sample file as an independent parameter file.
                      Page **5-44**.

Save All   Saves all open files. Page **5-44**.

Close   Closes the file in the active window. Page **5-45**.

Close All   Closes all open files. Page **5-45**.

Print   Prints details of the selected file(s). Page **5-46**.

List   Generates a list of certain information for the selected
                                      sample file(s) or parameter file(s). Page **5-47**.

Import MS Signal   Allows you to import data collected on a Pfeiffer Vacuum
                                      (formerly Balzers) mass spectrometer which uses the
                                      Quadstar software. Page **5-49**.

Export   Exports a sample file in ASCII format or in a format
                                      compatible with the Grams/32 application (a peak editing
                                      program used previously with the AutoChem analyzer).
                                      Page **5-51**.

Exit   Exits the ChemiSorb program. Page **5-54**.

Open

Select Open to create a new sample or parameter file or to edit an existing one. Regardless of which file type you select, a dialog box similar to the one shown below is displayed.
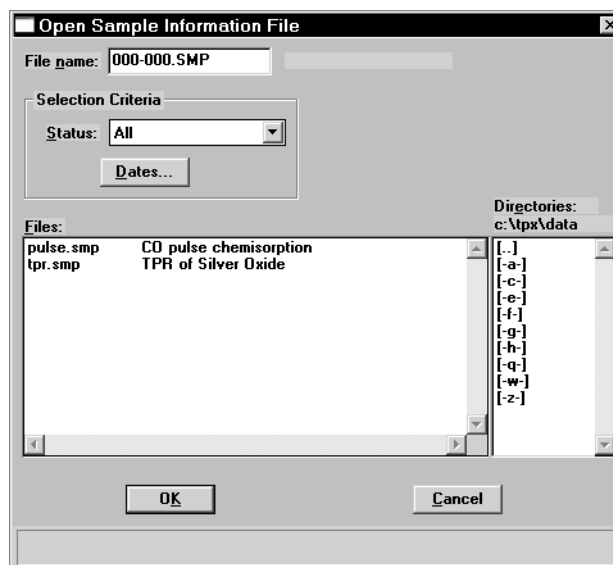
File name		For sample information files, this field contains the next sequenced file name generated by the system.
		For parameter files (analysis conditions and report options), the file name displayed includes the wild card (*) and the default extension as follows:
		*.ANC for analysis conditions *.RPO for report options
		If you are creating a new file, enter a name in the **File name** field. You can use up to eight characters. If you are editing an existing file, select a file from the list.
Status		This drop-down list allows you to choose the types of files to display in the **Files** window.
	All	Displays all sample information files in the specified directory and within the range of dates selected in the **Dates** dialog.
	Analyzing	Displays sample information files that are currently being used for analysis.

Complete	Displays sample information files that have been used in an analysis.
No Analysis	Displays sample information file that have been prepared but not yet used in an analysis.
Dates	Displays the Select Dates dialog.

This dialog enables you to specify a date range for the files you wish to display in the **Files** window.

Show All Dates	Displays all sample files of the type selected in the Status list, in the current directory.
Show Date Range	Enables the **From** and **To** fields allowing you to specify a range of dates in which to display the sample files. Use one of the following methods to select date ranges:

- Highlight the field and enter a date, simply by typing the date; you do not have to enter a slash (/).

- Double-click in the selected field to display a calendar from which you can select a date.

For convenience, the following shortcut keys are available to assist you:

F2 Clears the field
F3 Inserts the current date
F4 Displays a calendar from which to select a date

Files	This window displays the files available in the selected directory for the chosen status within the selected date range.

Sample Information

Sample Information files contain information used to control the analysis, as well as collected data. A sample information file consists of:

- Sample information
- Analysis conditions
- Report options
- Collected data (appears after analysis is complete)

Portions of the sample file can also exist as parameter files separate from the sample information file itself. These parameter files contain frequently used analysis conditions and report options that can be conveniently loaded into a new sample information file.

Sample information files are presented in three different formats, Basic, Advanced, and Restricted. You can select any one of these formats as your default. A convenient toggle button makes it easy to switch between Basic and Advanced formats, regardless of which is selected as your default. The Restricted format does not allow switching to the Advanced format.

- **Basic Format**
  Presents the entire sample information file in a single dialog box. Basic format makes it easy to select previously created, standard parameter files from convenient drop-down lists.

- **Advanced Format**
  Presents all parts of the sample information file as a group of dialog boxes with index card-type tabs (although the Collected Data tab does not appear until after analysis). Clicking on each tab opens the associated dialog. Use this format to create customized sample information files.

- **Restricted Format**
  Restricted presentation format is identical to Basic presentation format, except that you cannot switch to the Advanced presentation. Restricted presentation limits the operator's ability to change analysis conditions and report options.

Select Option presentation from the Options menu to specify your default format.

Basic Presentation

The Basic format displays all parameters of the sample information file on a single dialog. With the Basic format, you can quickly create a sample information file using predefined parameter files from the drop-down lists.
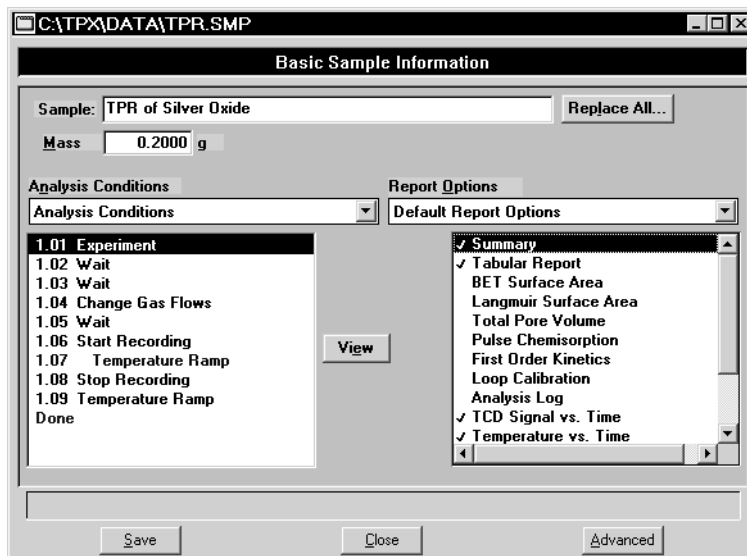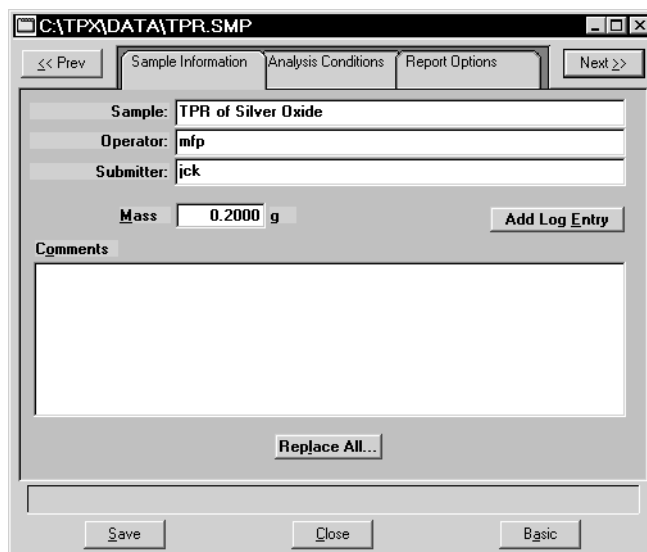
Sample	Contains the description of the current sample file.
	If this is a new file, this field contains the description you specified in **Sample defaults** (see Chapter 8). You can enter a new description or add to the existing one if desired.
	Range: 50 alphanumeric characters
Replace All	This push button allows you to replace the values for all parameters of the current file with the values from an existing file. Simply click the push button, choose the desired file, and click **OK**. This does not import the file itself; only the values are copied and imported. The file remains intact.
Mass	Enables you to enter the mass (weight) of the sample.

Analysis Conditions	Displays the name of the current Analysis Conditions file.
	If this is a new file, this field contains the name of the file you specified as the default. Click the down arrow to the right of the field to choose a different file. This list includes Analysis Conditions files supplied with the analysis program as well as those you create and save to the directory specified as the Parameter Files directory (Chapter 8). If new files are not saved to the specified directory, they are not included in the list.
	This list is disabled for files which have been used for an analysis.
View	Allows you to view details about the currently selected step in the analysis conditions set.
Report Options	Displays the name of the current Report Options file.
	If this is a new file, this field contains the Report options file you specified as the default. Click the down arrow to the right of the field to choose a different file. This list includes Report Options files supplied with the analysis program as well as those you create and save to the directory specified as the Parameter Files directory (Chapter 8). If new files are not saved to the specified directory, they are not included in the list.
Reports List Box	Displays a list of available reports. The checked reports are the ones requested for the current Report options file.
	If this is a new file, the checked reports are the ones you specified for the default(s). You can select (or deselect) reports by double-clicking on the desired report. Alternatively, you can highlight the report and press the **Spacebar**.
Save	Saves the current file.
Close	Closes the current file. If the file contains unsaved values, you are prompted to save before closing.
Advanced	Switches the presentation format to the Advanced presentation.

Advanced Presentation

The Advanced format displays the sample information file in a manner similar to that of an index card file. Each parameter has its own tab and is accessed by clicking the tab. Or, you can use **Next** and **Prev** to move through the dialogs. This format allows you to create customized sample information files.

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

Sample	Contains the description of the current sample file.
	If this is a new file, this field contains the description you specified in Sample Defaults (see Chapter 8). You can enter a new description or add to the existing one if desired.
	Range: 50 alphanumeric characters
Operator Submitter	Displays the Operator and Submitter names (if specified) of the current sample file.
	If this is a new file, these fields contain the names specified in Sample defaults. You can enter a different name in either (or both) field if desired. You can also edit the prompts for these fields or choose to have them omitted entirely (refer to Sample defaults in Chapter 8).
	Range: 40 characters

Mass   Allows you to enter the mass of the sample.

Add Log Entry   Displays a dialog box allowing you to enter pertinent
                                  information relating to the sample. Any information you
                                  enter here is printed as part of the Analysis Log report
                                  (select in Report Options).

Comments   Use this field to enter any comments about the sample or
                                  the planned analysis conditions that you wish to note.
                                  Comments entered here are printed in the header of some
                                  reports.

                                  Range: Up to 500 alphanumeric characters

Replace All   This push button allows you to replace the values for all
                                  parameters of the current file with the values from an
                                  existing file. Simply click the push button, choose the
                                  desired file, and click **OK**. This does not import the file
                                  itself; only the values are copied and imported. The file
                                  remains intact.

Save   Saves the current file.

Close   Closes the current file. If the file contains unsaved
                                  values, you are prompted to save before closing.

Basic   Switches the presentation format to the Basic
                                  presentation.

The Analysis Conditions and Report Options tabs of the Advanced presentation sample
information file are described in subsequent sections.

Restricted Presentation

A third format is provided to control access to some portions of the sample information file. Restricted format displays in the same manner as the Basic presentation format.
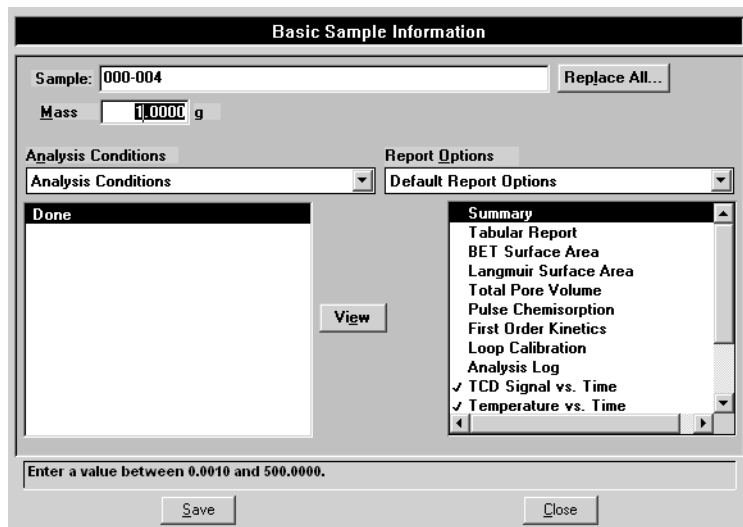
The Restricted presentation does not allow you to switch to the Advanced format. This format is also password-protected, preventing the operator from making changes to analysis conditions and report options sets. Chapter 8 provides additional information on this format.

Restricted presentation is ideal for laboratories in which standard analysis procedures are established by a lab manager, while one or more lab technicians actually perform the analyses. The lab manager can create independent analysis conditions and report options files containing standard sets of operating conditions, then the operator(s) can use Restricted mode for daily operations, selecting the standard parameter files from the drop-down lists.

Analysis Conditions

Analysis conditions files specify each step in the analysis, including temperatures, pressures, and gases. You can create an Analysis Conditions file as an independent parameter file or as part of a sample information file. You can also save the analysis conditions portion of a sample information file as an analysis conditions file.
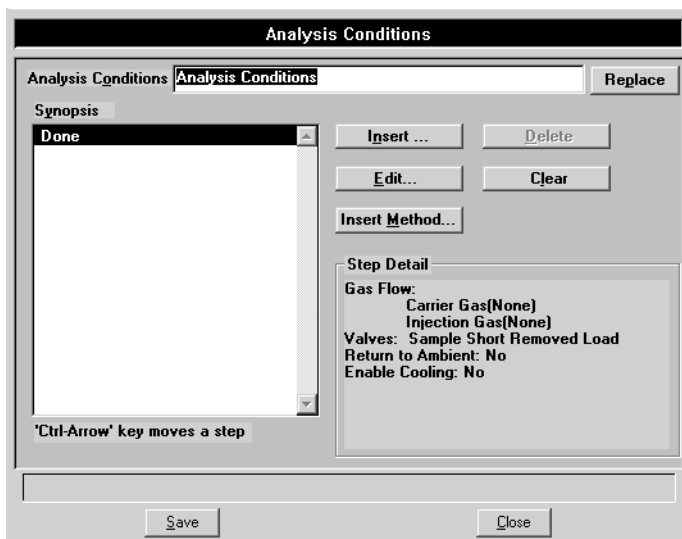
When creating parameter files to display in the drop-down list on the Basic and Restricted sample information dialogs, be sure to save them to the directory specified as the Parameter file directory (Chapter 8).

Analysis Conditions	If you are opening an existing analysis conditions file, this field contains the description.
	If you are creating an analysis conditions file, this field contains the description specified in Sample defaults. You can enter a new description or add to the existing one if desired. Use a description that will help you identify this set of analysis conditions in the future.
	Range: 40 alphanumeric characters.
Replace	Allows you to copy values from an existing file to the one you are creating. A dialog box is displayed allowing you to choose the desired file. You can then edit these values if desired; the file you copied them from will not be affected.

Synopsis   Displays a list of the steps the analyzer will follow during the analysis.

Steps within Experiment 1
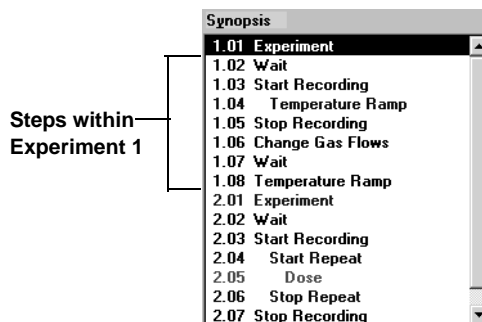
An analysis is a complete characterization of the sample; in other words, it is all of the experiments and steps within the Synopsis. It may consist of up to 99 separate experiments, such as TPR or TPO. Each experiment may consist of up to 99 steps.

Insert   Enables you to insert an experiment or a step within an experiment.

This dialog displays if you are inserting a new experiment.
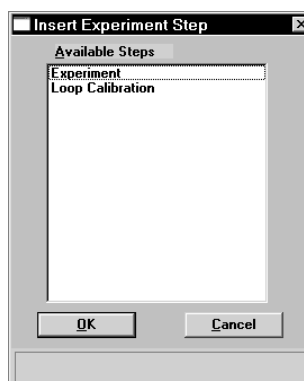
This dialog displays if you are inserting a step within an experiment.
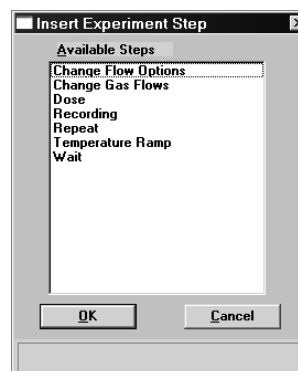
Insert
(continued)

At some points in the sequence of steps, all steps are available. At other points, certain steps cannot be performed. For example, you cannot start a new experiment inside a Repeat loop. The steps that appear in the list box are those which are currently available.

Highlight the desired experiment or step, then click **OK**; a related dialog allowing you to specify instructions is displayed. The dialogs for each experiment and step are discussed in subsequent sections.

When you insert a step, it is inserted above the one you have highlighted in the Synopsis.

You can easily move a step from one position in the list to another. Simply click on the step you wish to move, then use **Ctrl +** ↑ **(**Up Arrow**)** or **Ctrl +** ↓ (Down Arrow) to move the highlighted step.

Delete

Deletes the selected item from the synopsis list. If you click this button when an experiment is selected, the entire experiment (including all its steps) is deleted after a confirmation.

Edit

Enables you to edit the selected experiment or step; an associated dialog is displayed.

You can also edit a step or experiment during analysis. However, you must suspend the experiment before any editing can be accomplished; any temperature ramps will continue. The temperature ramps may be paused (if desired) using the furnace controller. You can edit only the steps or experiments following the current one; all completed steps will be disabled. After an analysis has been completed, **Edit** displays as **View**; steps may be viewed but not edited.

Clear

Clears the list of experiments and steps for this Analysis Conditions dialog. A confirmation message is displayed.

Insert Method

Allows you to insert the entire list of steps from another analysis conditions file. After you click this button, select an analysis conditions file from the list. All the steps in that file are inserted directly above the highlighted step. Inserted methods can be edited without altering the original analysis conditions file(s) from which they were copied.

Insert Method   For example, if you have three different standard sample
(continued)   preparation methods that you use under varying
                                   circumstances, create an analysis conditions file for each
                                   preparation method. Then use **Insert Method** to insert
                                   the appropriate preparation method in the analysis
                                   conditions file you create when you are ready to analyze
                                   a sample.

                                   You can also create several analysis conditions files
                                   containing individual experiments, then use **Insert
                                   Method** to combine them into a comprehensive analysis
                                   comprised of these individual analyses.

Step Detail   Displays information about the currently highlighted
                                   step in the Synopsis. The information displayed for each
                                   step is a summary of the information contained on that
                                   step's dialog.

Insert Experiments

An analysis set is created by inserting a series of experiments in the sample file Synopsis. You can insert up to 99 experiments. There are two types of experiments that you can insert in an analysis conditions set. The following dialog is displayed when you choose to insert an experiment:
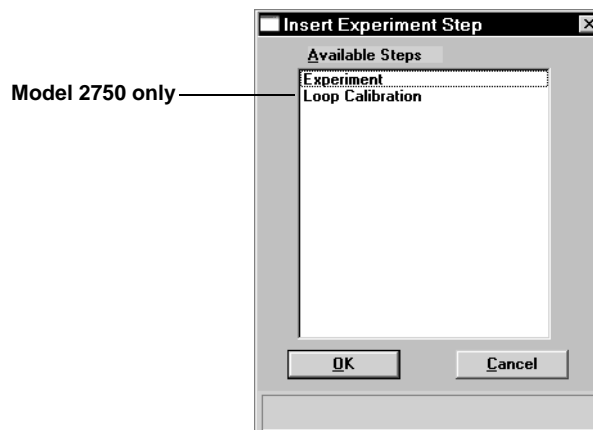
The first type is the customizable Experiment. You will insert into this Experiment the individual steps which control the reaction(s) in the analysis. When you insert an experiment, you specify the Initial Conditions, then insert individual steps. Each step can be customized in detail.

You can also perform a Loop Calibration if you have the Model 2750; the sequence of steps in the Loop Calibration cannot be altered.

Experiment

Inserts a basic experiment into the list of steps.
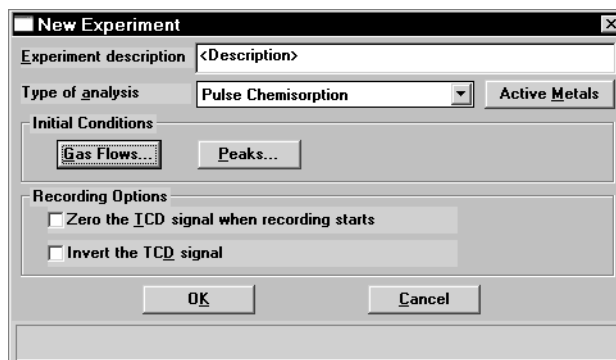
Experiment description   Allows you to enter a description or identifier for this experiment. The description you enter in this field appears in the legend of the report.

Range: 40 alphanumeric characters

Type of analysis   Select the type of analysis from the drop-down list.

Choices: Temperature-Programmed Oxidation
        Temperature-Programmed Reduction
        Temperature-Programmed Desorption
        Physisorption Surface Area
        Pulse Chemisorption
        Other

It is important to select the correct experiment type because the type is referenced during some other operations. For example, a Pulse Chemisorption report can only be generated for those experiments specifying Pulse Chemisorption as the type.

Active Metals   Opens the Active Metals Table dialog, allowing you to specify the percent sample mass of the active metals in the sample (in the far right column), and the stoichiometry factor.
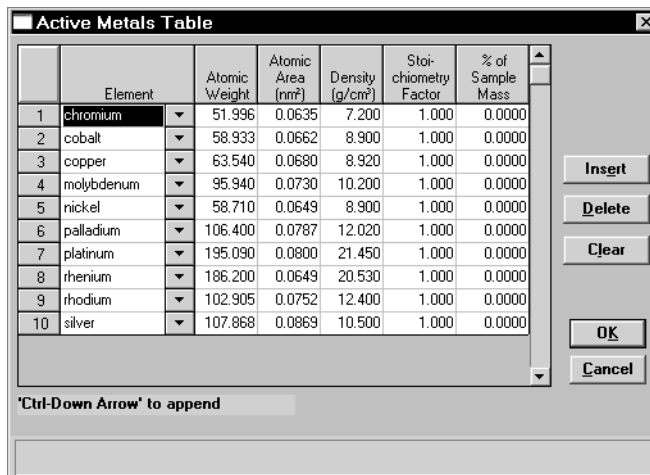
Enabled when you choose a Pulse Chemisorption experiment.

This table displays the defaults specified using the Active Metals Defaults option on the Options menu. After this sample file is created, this table becomes specific to the sample file. Therefore changes made in the current table do not affect default settings.

Insert   Inserts a new row into the table; the row is inserted above the selected one.

Delete   Deletes the selected row.

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

Gas Flows   Displays the Gas Flows dialog.
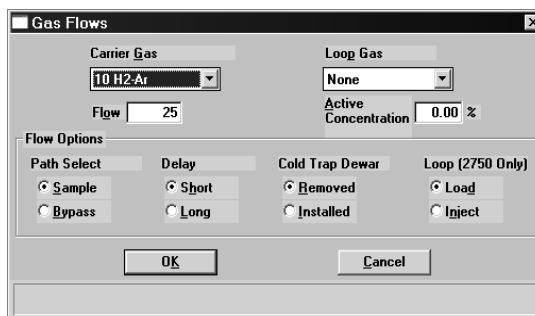
This dialog allows you to specify details for the carrier
and injection gases, and valves

Gases   The gases included in the drop-down lists for the Carrier
and Injection gases are the ones specified in Gas
Defaults on the Options menu.

Select **None** when you wish not to have gas flowing (the
flow rate is ignored).

When you start an analysis, you are prompted to verify
that the selected gases are connected to the appropriate
ports.

Carrier Gas   After selecting the desired gas, enter a flow rate in the
Rate field.

Injection Gas   After selecting the desired gas, ensure that the value in
the **Active Concentration** field is appropriate. The value
for this field should be the percent of the gas mixture
which is composed of the reactive gas versus an inert
filler. The value that defaults in this field is the one
specified in Gas Defaults on the Options menu.

This value is used for pulse chemisorption data
reduction.

When inserting multiple experiments into the Synopsis, consider the following when selecting gases:

• Avoid placing incompatible gases in the analyzer in immediate sequence.

When you change the gas flowing into the analyzer, the new gas enters the analyzer while the previous gas is still present.

You can "purge" the previous gas before starting to flow the new gas by selecting an inert gas for the initial conditions gas in an experiment step, or by inserting an extra **Change gas flows** step in which you flow an inert gas between the flows of incompatible gases. If you do so, you must also insert a **Wait** step specifying how long to flow the inert gas. You can specify a specific time period or select one of the other **Wait** step criteria. (Each of these step types is described later in this chapter.)

• Changes in the gas cause a brief disturbance of the flow rate.

This disturbance may be detected by the TCD and may be visible in the data (if data are being recorded when the gas flow is changed). Either disregard such disturbances in the data or stop recording before you change the gas flows, then insert a Wait for stable baseline step after the **Change gas flows** step. Resume recording after a stable baseline is detected. A suggested sequence of steps then might be as follows:

Start recording
Experiment steps as needed
Stop recording
Change gas flows
Wait for stable baseline
Start recording
Experiment steps as needed

Path Select	These valves enable flow of the gas stream from the sample or from the septum and loop to the detector. Typically, this selection should be **Sample**. Use **Bypass** when performing a loop calibration.
Delay	Serves to delay the arrival of desorbed gases at the detector system long enough for the flow rate to recover from the rapid desorption of adsorbed gases created when the cryogenic liquid is removed. Choose **Long** when performing physisorption analyses. For other operations, **Short** is sufficient.

Cold Trap Dewar   Use the **Installed** option when using a Dewar of
                             cryogenic liquid to trap water and/or condensable vapors
                             to prevent them from reaching the TCD. This option is
                             often useful for TPR analyses.

                             For other analyses, choose **Removed**.

Loop (2750 only)   Choose the initial position for the loop valve. The **Load**
                             position is recommended for the start of each analysis.

                             The loop is used for pulse chemisorption experiments.

Peaks   Displays the Peak Detection/Integration dialog.
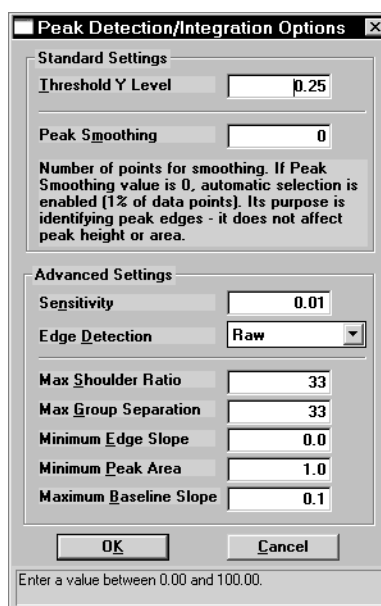
This dialog enables you to specify peak detection and
integration criteria for the analysis After analysis, you
can further control peak detection using the Peak Editor,
described in Chapter 7. Refer to Appendix E for
additional information on the fields of this dialog.

The TCD detects and records all deviations from
baseline, but only those which satisfy the criteria
established in this dialog are reported as peaks.

Zero the TCD signal when   Allows you to zero the TCD signal automatically the
recording starts   first time recording starts in an experiment.

Invert the TCD signal	Inverts the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.

Loop Calibration

Loop Calibration enables you to calibrate the gas injection loop (ChemiSorb 2750 only). This experiment verifies the exact volume of the loop for use in calculations on analyses in which the loop is used. Sample analysis data yield signal vs. temperature data and peak areas. Associating the sample information file with a loop calibration file makes it possible for the software to convert sample peak data to gas quantity values.

Description	Enter a description or identifier for this experiment. It is important to use a descriptive identifier here, in order to easily identify this data when displayed in sample reports.
Calibration Gases	Allow you to select the carrier and loop gases to be used in this calibration. The gases available are those specified in Gas defaults on the Options menu.
Flow Rate	Specify the flow rate for the carrier gas; typically, the default of 25 mL/min is appropriate.
Loop thermal equilibration delay	Allows you to specify the duration of the equilibration delay. This delay allows extra time for the instrument to stabilize thermally after the baseline has fully stabilized.
Injection Volume	Enter the physical volume of gas you will inject from the syringe.

Zero the TCD signal   Select this option to zero the TCD signal automatically
when recording starts   the first time recording starts in an experiment.

**Invert the TCD signal**   Inverts the TCD signal. For example, if you expect to
                                 obtain only negative peaks, this option can be used to
                                 record the peaks in the positive direction.

Insert Experiment Step

After you have set up the initial conditions for your basic Experiment, the Analysis Conditions dialog is again displayed showing the Experiment inserted. Clicking **Insert** now allows you to insert a step in the Experiment you just added to the Synopsis. You can insert steps in a wide variety of sequences. You can also use steps repetitively within the Experiment.
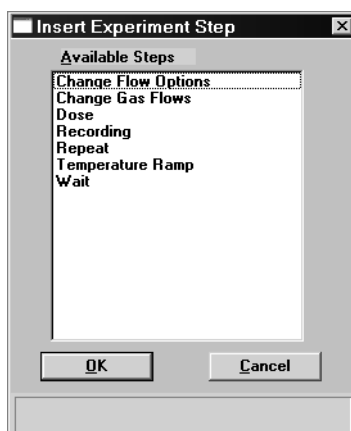
You cannot insert steps within a Loop calibration. The Loop calibration is preprogrammed with appropriate steps.

> **You cannot change the steps included in a loop calibration, but you can include a loop calibration in analyses that contain other experiments. For example, you can include a loop calibration experiment in an analysis that also contains a pulse chemisorption experiment.**

Change Gas Flows

Displays the Gas Flows dialog. This dialog is identical to the one that is opened when selected from the Insert Experiment Step dialog. A complete description of this dialog is found earlier in the Experiment section.

Change Flow Options

Displays the Change Flow Options dialog.
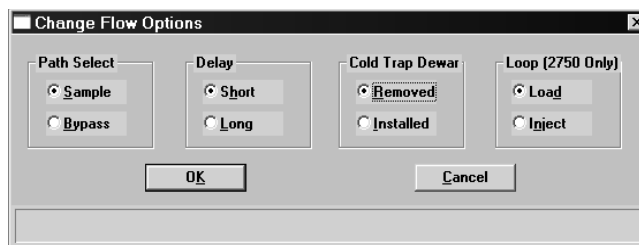
Path Select   These valves allow flow of the gas stream from the
                               sample or from the septum and loop to the detector.
                               Typically, this selection should be **Sample**. Use **Bypass**
                               when performing a loop calibration.

Delay   Serves to delay the arrival of desorbed gases at the
                               detector system long enough for the flow rate to recover
                               from the rapid desorption of adsorbed molecules created
                               when the cryogenic liquid is removed during physical
                               adsorption experiments. Choose **Long** when performing
                               physisorption analyses. For other operations, **Short** is
                               sufficient.

Cold Trap Dewar   Use the **Installed** option when using a Dewar of cryo-
                               genic liquid or a slush bath around the sample. For other
                               analyses, choose **Removed**.
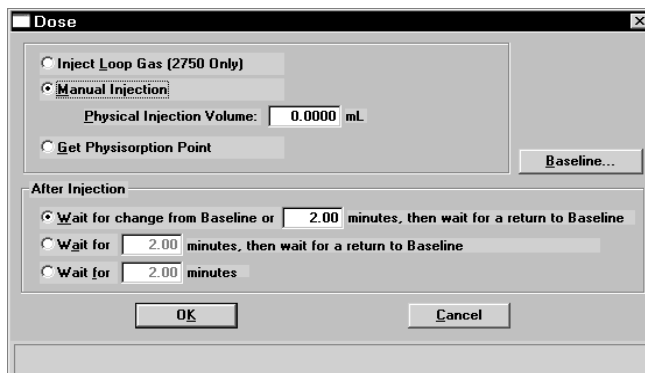
Loop (2750 only)   Choose the initial position for the loop valve. The **Load**
                               position is recommended for the start of each analysis.

                               The loop is used for pulse chemisorption experiments
                               and calibrations.

Dose

Displays the Dose dialog enabling you to specify dosing methods.

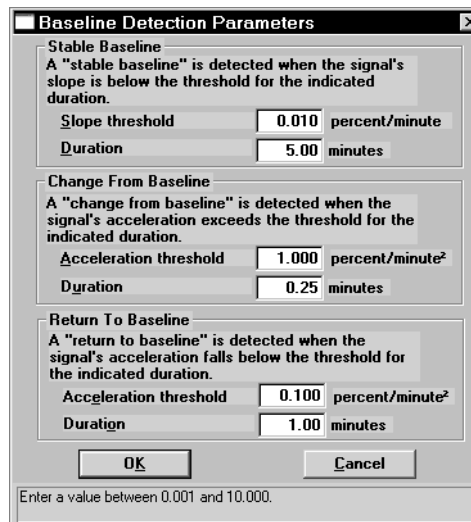| **Inject Loop Gas (2750 only)**	Injects the contents of the loop into the path that leads to the sample. The contents of the loop are pushed out of the loop by the Carrier Gas.
Manual Injection	Prompts you to inject a dose of gas into the septum using a syringe. The **Physical Injection Volume** field is enabled allowing you to enter the volume of gas injected.
Get Physisorption Point	Prompts you through the steps necessary to collect data for a physisorption point. These steps include: placing a Dewar of liquid nitrogen around the sample tube, waiting for a return to baseline, then replacing the Dewar with water at room temperature. You can take repeated points by inserting the **Dose** step within a **Repeat Loo**p (discussed later in this chapter).
After Injection	Enables you to choose the waiting method after injection of the gas.

Baseline

Displays the Baseline Detection Parameters dialog allowing you to specify baseline settings when a Wait step involving the baseline is selected.

Establish the **Slope** or **Acceleration threshold** and **Duration** for determining what constitutes a **Stable Baseline**, a **Change From Baseline**, and a **Return To Baseline**. These values control whether a particular change in the signal is significant to the current experiment; in other words, they define a stable baseline.

Some Wait steps are contingent upon the values you select here. For example, if your experiment contains a **Wait until Baseline is stable** step, the signal is compared to these values to determine if a stable baseline has been established. Lower slope/acceleration values and longer durations create a more rigorous definition of these factors than higher values and shorter durations. Normally the default values are satisfactory.

Ranges for each field are displayed in the information bar across the lower section of the dialog.

Recording

```
┌─────────────────────────────────────────┐
│ ■ Start Recording                     ☒ │
├─────────────────────────────────────────┤
│                                         │
│  One measurement every   [ 1.0 ] seconds│
│                                         │
│                                         │
│     ┌──────────┐      ┌──────────┐      │
│     │   OK     │      │  Cancel  │      │
│     └──────────┘      └──────────┘      │
├─────────────────────────────────────────┤
│ Enter a value between 0.4 and 10.0.     │
└─────────────────────────────────────────┘
```
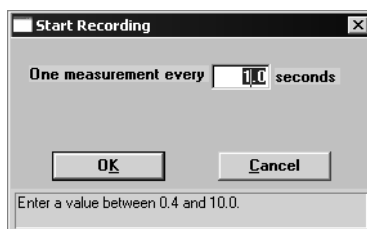
Enables you to specify how frequently the signal reading is recorded.

Recommended values are 1.0 for TPR, TPD, and TPO experiments, and 0.4 for pulse chemisorption.

The Start Recording dialog starts the recording of the signal for the analysis. A **Stop Recording** step is inserted in the list of steps automatically on the Analysis Conditions dialog when you insert a **Start Recording** step.

If a **Start Recording** step is immediately followed by a step that prompts an immediate peak, peak data are recorded before any baseline readings can be collected. To collect some baseline data before the first peak in such an experiment, insert a **Wait** (**Wait _____ minutes** option) step after the **Start Recording** step, but before the step which causes the peak.

Repeat

When you insert a Repeat step in your analysis conditions, the Start Repeat Sequence dialog opens allowing you to specify the duration of the repeat sequence. After you complete the Start Repeat Sequence dialog and click **OK**, a **Start Repeat** step and a **Stop Repeat** step are automatically inserted in the list of steps for this analysis. You may insert multiple experiment steps within the **Repeat** loop.
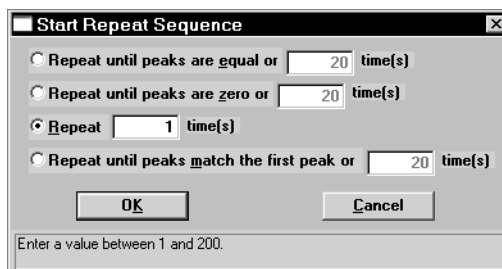
During the Repeat sequence, the values for peak areas are displayed at the **Prompt** on the operational window. Regardless of which option you choose, you will be given the opportunity to continue with the Repeat sequence or you can click Exit Repeat to exit the Repeat sequence.

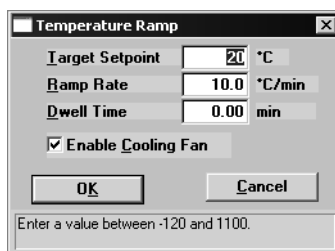|	
Repeat until peaks are equal or [n] times	Choose this option to display values for the last two peak areas, or you can enter a number for the repeat sequence.
	This option is useful when performing H_2 or CO pulse chemisorption on supported metal catalysts.
Repeat until peaks are zero or [n] times	Choose this option to display the values for the last peak area and the maximum peak area, or you can enter a number for the repeat sequence.
	This option is useful when performing an N_2O decomposition for characterizing copper catalysts.
Repeat [n] times	This option does not display peak area data; you will be presented with the opportunity to continue the repeat sequence or, as with the other options, you can exit the repeat sequence.
Repeat until peaks match the first peak or [n] times	Choose this option to display values for the first and last peak area, or you can enter a number for the repeat sequence.

Temperature Ramp

Insert this step to change the sample temperature.

|	
Target Setpoint	Enter the ending temperature for this ramping procedure.
Ramp Rate	Enter the ramp rate for this ramping procedure.
Dwell Time	Enter the amount of time to hold the temperature at the conclusion of this step.
Enable Cooling Fan	Select this option to enable the cooling fan to reduce the furnace temperature more rapidly during cool-down.

Wait

Enables you to specify a waiting routine.
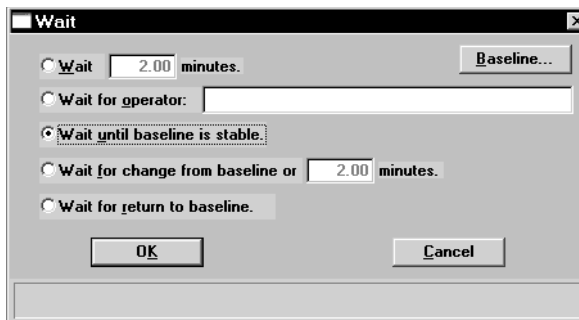
Wait [n] minutes.	Specify an exact number of minutes to wait.
Wait for operator	Enter a description of the operator's task; for example "Fill the dewar, then click OK."
	During an analysis, the message you enter is displayed on the monitor at the appropriate time. The analysis continues after you perform the task and click **OK**.
Wait until Baseline is stable	Waits until the baseline becomes stable. If you choose this option, click **Baseline** to specify the settings
Wait for change from Baseline or [n] minutes	Enter the number of minutes to wait, then click to specify the settings
Wait for return to Baseline	Waits for a return to Baseline. If you choose this option, click **Baseline** to specify the settings.

Done

This step is automatically inserted in every experiment. The defaults for this step are the ones specified for Sample defaults (Options menu). You can view and/or edit the details of this dialog by clicking **Edit** when Done is selected (highlighted); the Done dialog is displayed.
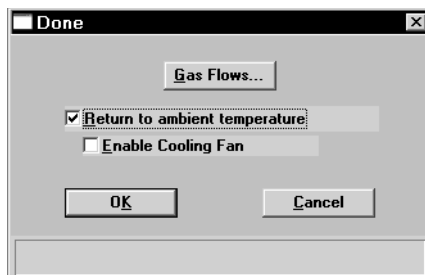
Gas Flows	Displays the Gas Flows dialog allowing you to view or edit current gasses and their flows. Refer to Experiment earlier in this chapter for information on this dialog.
Return to ambient temperature	Select this option to have the sample cooled to ambient temperature.
Enable Cooling Fan	Select this option to enable cooling fan during the cool-down operation.

Report Options

This dialog is used to edit existing report options, or to specify report options for a new file.

> **When creating parameter files to display in the drop-down list on the Basic and Restricted sample information dialogs, be sure to save them to the directory specified as the Parameter File directory (see Chapter 8).**

Report Options

| Report Options | Default Report Options | Replace... |

☑ Show report title — Micromeritics Instrument Corporation

☑ Show graphic — Miclogo.emf — Browse...

Width 2.000 in.   Height 0.250 in.

Selected Reports

A report is selected when preceded with a check mark.

- ✓ Summary
- ✓ Tabular Report
- BET Surface Area
- ✓ **Langmuir Surface Area**
- Total Pore Volume
- Pulse Chemisorption
- First Order Kinetics
- Loop Calibration
- Analysis Log
- ✓ TCD Signal vs. Time
- ✓ Temperature vs. Time

Edit...

Enter a value between 0.100 and 7.500.

Save   Close

Report options   If you are editing an existing file, this field displays the description or identification of the report options file. This description can be edited if desired.

If this is a new file, this field contains the description specified in Sample defaults. You can enter a new description or add to the existing one if desired. Use a description that will help you identify this set of report options in the future.

Range: 40 alphanumeric characters

Replace   Enables you to copy values from an existing file to the one you are creating. A dialog is displayed so that you may choose the file containing the values you wish to copy. Since the values are copied into the new file, you can edit them without affecting the source file.

Show report title

This option allows you to have a heading appear on your report and to enter a title for it.

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

Range: 40 characters

Show graphic

Select this option to have a graphic (bitmap or enhanced metafile) display above the report title. For example, you may wish to display your company logo. Click **Browse** to select the desired graphic, then enter the **Height** and **Width** in the fields provided. This image can also be edited from the report window.

Selected Reports

Contains a list of available reports. A report is selected when it is preceded with a check mark. Select reports by double-clicking the desired report(s). Alternatively, you can highlight the desired report and press **Spacebar**. Reports are deselected in the same manner. Certain reports can be edited by highlighting the desired report and clicking **Edit**.

Edit

Enables you to edit the highlighted report. The dialog(s) associated with each type of report are described in the following sections.

The Summary Report and the Analysis Log cannot be edited.

Tabular Report Option

Clicking **Edit** when Tabular Report is selected opens the following dialog:
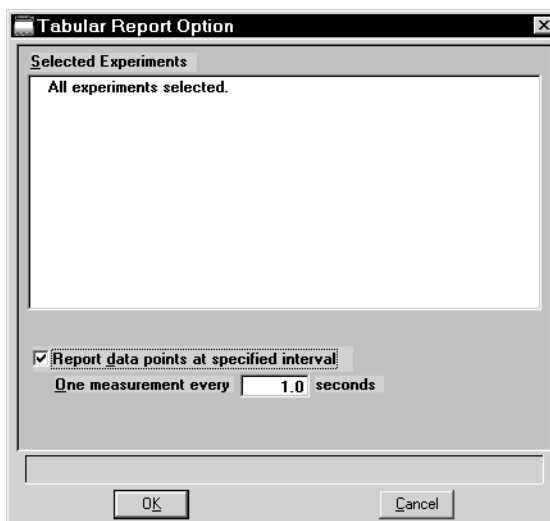
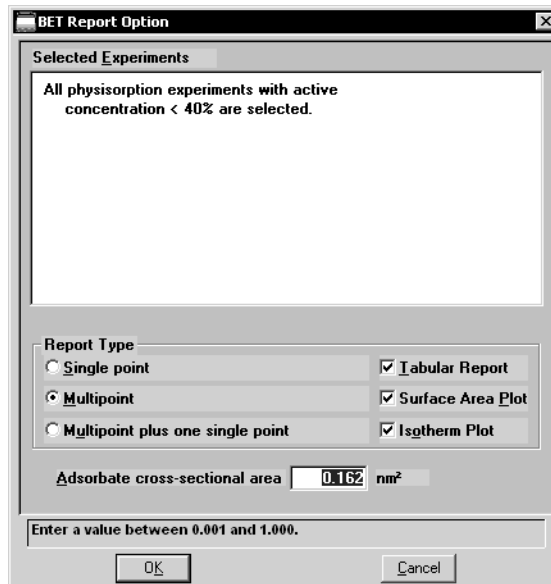|	
Selected Experiments	Displays all of the experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.
	If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Report data points at specified interval	Select this option to report data points at a specified interval, then specify the frequency of reported data (the number of seconds between reported data readings).
	This feature is useful if you anticipate a large amount of data to be collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, all points are reported, regardless of the length of the report.

BET Surface Area Report Option

Clicking **Edit** when BET Surface Area is selected opens the following dialog:

BET Report Option	☒
Selected Experiments	

All physisorption experiments with active
concentration < 40% are selected.

Report Type
- ○ **Single point**   ☒ **Tabular Report**
- ◉ **Multipoint**   ☒ **Surface Area Plot**
- ○ **Multipoint plus one single point** ☒ **Isotherm Plot**

Adsorbate cross-sectional area `0.162` nm²

Enter a value between 0.001 and 1.000.

OK   Cancel

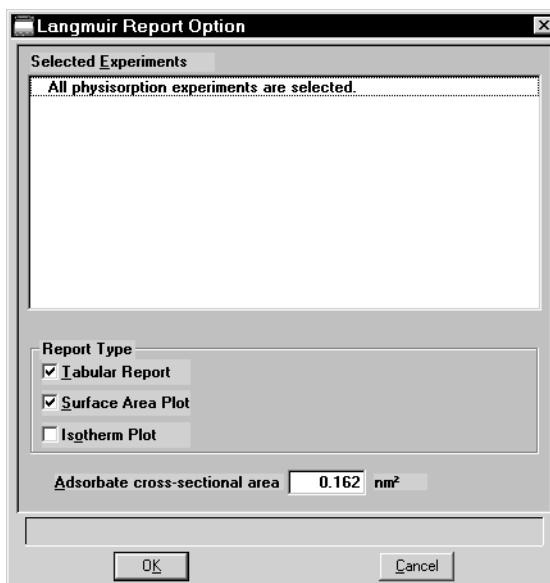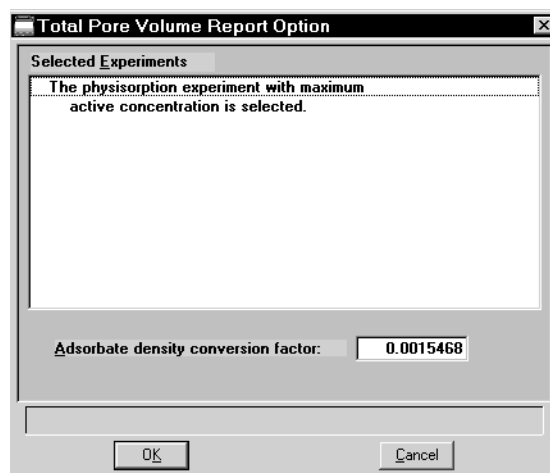|	
Selected Experiments	Displays all BET surface area experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.
	If this is a new file, no experiments are listed. Experiments are not included in the list until BET data are collected.
Report Type	From this group box, choose the type of report(s) desired. If you choose **Multipoint** or **Multipoint plus one single point**, the choices on the right side of the group box are enabled. You can choose as many of these options as desired.
Adsorbate cross-sectional area	Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface.

Langmuir Surface Area Report Option

Clicking **Edit** when Langmuir Surface Area is selected opens the following dialog:

Selected Experiments	Displays all Langmuir surface area experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.
	If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Report Type	From this group box, choose the type(s) of reports you wish to generate.
Adsorbate cross-sectional area	Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface

Total Pore Volume

Clicking **Edit** when Total Pore Volume is selected opens the following dialog:

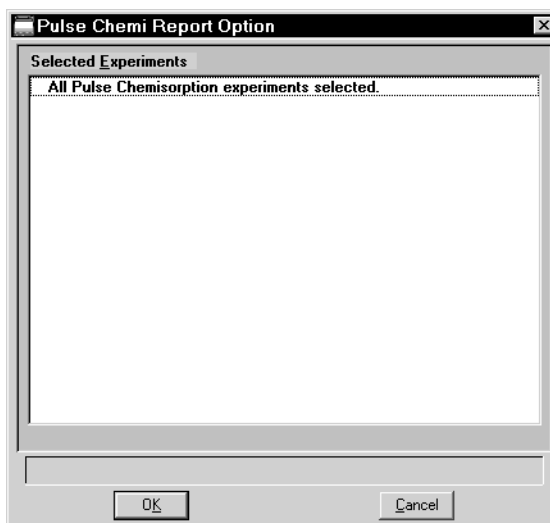| Selected Experiments	Displays all Total pore volume experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.
	If this is a new file, no experiments are listed. Experiments are not included in the list until data are collected.
Adsorbate density conversion factor	Enter the appropriate value for converting the gas volume to liquid volume. This value can be obtained by dividing the gas density by the liquid density at the adsorption temperature.

$$\text{Density conversion factor} = \frac{\rho_{gas}}{\rho_l} \cdot \frac{T_{BATH}}{T_{STD}}$$

Pulse Chemisorption Report Option

Clicking **Edit** when Pulse Chemisorption is selected opens the following dialog:

```
┌─────────────────────────────────────────────────────┐
│ ▪ Pulse Chemi Report Option                      ☒  │
├─────────────────────────────────────────────────────┤
│ Selected Experiments                                 │
│ ┌─────────────────────────────────────────────────┐ │
│ │ All Pulse Chemisorption experiments selected.   │ │
│ │                                                 │ │
│ │                                                 │ │
│ │                                                 │ │
│ │                                                 │ │
│ │                                                 │ │
│ │                                                 │ │
│ │                                                 │ │
│ └─────────────────────────────────────────────────┘ │
│ ┌─────────────────────────────────────────────────┐ │
│ └─────────────────────────────────────────────────┘ │
│      ┌────────┐              ┌────────┐             │
│      │  OK    │              │ Cancel │             │
│      └────────┘              └────────┘             │
└─────────────────────────────────────────────────────┘
```
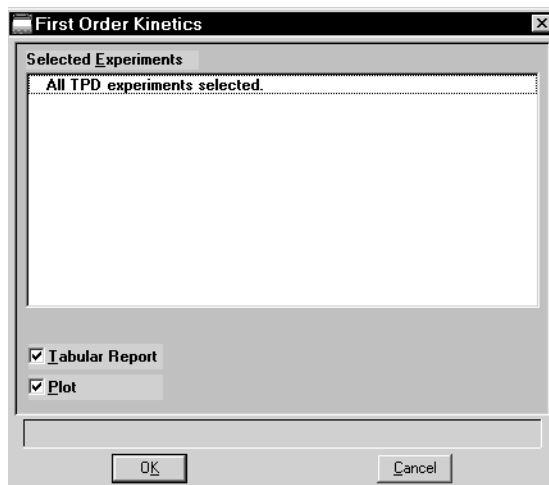
Selected Experiments   Displays all pulse chemisorption experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.

If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.

First Order Kinetics Report Option

Clicking **Edit** when First Order Kinetics (heat of adsorption) is selected opens the following dialog:

Selected Experiments	Displays all TPD experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report. However, you must include at least three experiments for the first order kinetics to be calculated.
	If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.
Tabular Report	Select this option to have a tabular (data in a table form) report generated.
Plot	Select this option to have the data plotted in a graphical format

Loop Calibration Report Option

Clicking **Edit** when Loop Calibration is selected opens the following dialog:
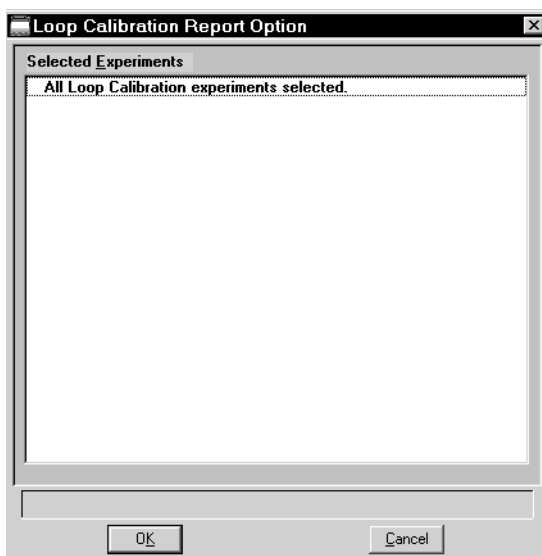
Selected Experiments   Displays all loop calibration experiments in the current
                              file. All experiments are selected by default; you may
                              deselect any that you do not wish to include in the
                              report.

                              If this is a new file, no experiments are listed. Experi-
                              ments are not included in the list until related data are
                              collected.

Analysis Log Report

When Analysis Log is selected, a log of analysis operations is included in the reports. The
Analysis Log cannot be edited.

Graph Report Options

All graph report options are similar and are edited in the same manner. Click **Edit** for a selected graph report; a dialog similar to the following is displayed:
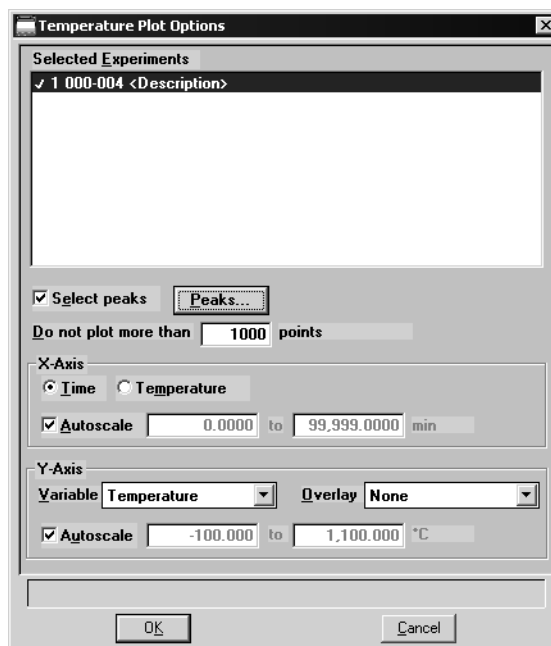
The detector signal may be plotted against time or temperature.

For color output to a monitor or printer, signals are displayed in different colors. For black and white output, different symbols are used.

Selected Experiments	Displays all related experiments in the current file. All experiments are selected by default; you may deselect any that you do not wish to include in the report.
	If this is a new file, no experiments are listed. Experiments are not included in the list until related data are collected.
Select peaks	Select this option to:

- display the areas and baselines on the graph
- draw a straight baseline between the selected peaks
- enable the **Peaks** push button, allowing you to specify the peaks to include in the experiment

This option is disabled for new files.

Do not plot more than _____ points	Allows you to specify a maximum number of points to plot. Typically, the default of 1000 is sufficient. You can specify up to 8000 if higher resolution and more detail is desired. The speed at which graphs are plotted on some computers may be affected when large numbers of points are requested.
Peaks	Opens a dialog that contains a list of all the peaks for the results of the experiment highlighted in the Experiments list box. All peaks are selected by default; remove the check mark next to any peak you do not wish to include on the graph.
X-Axis group box	The options in this group box enable you to specify X-axis criteria.
Time/Temperature	Select the variable you wish to have as the X-axis.
Autoscale	Select this option to have the X-Axis scaled automatically. As the default, this axis begins at zero, and the system uses the highest value collected during analysis as the ending point. The Axis **From** and **To** fields are disabled if you choose to autoscale data. If you choose not to autoscale data, you must enter a range. The value entered in the **To** field must be greater than the value entered in the **From** field.
Y-Axis	
Variable	Choose a variable for the Y-axis from the drop-down list.
Overlay	Use this option to overlay another variable with the current one. Select a variable from the drop-down list if desired.
Autoscale	Select this option to have the Y-Axis scaled automatically. As the default, this axis begins at zero, and the system uses the highest value collected during analysis as the ending point. The Axis **From** and **To** fields are disabled if you choose to autoscale data. If you choose not to autoscale data, you must enter a range. The value entered in the **To** field must be greater than the value entered in the **From** field.

Collected Data

The tab for Collected Data does not appear on the Advanced dialog presentation until after analysis.

Po of adsorbate	Displays the saturation pressure of the adsorbate.
Ambient temperature	Displays the room temperature.
Ambient pressure	Displays the room pressure.
Experiment	Defaults to the first experiment of the current file. Click the down arrow to choose other experiments in the file.
Physical injection volume	Displays the volume of the syringe used to perform the manual injections.
Active Concentration	Displays the percent of the gas mixture that is composed of the reactive gas versus an inert filler.
Peak table	Displays the peak summaries for the experiment shown in the Experiment field.
Use Fitted points	Select this option to report peak data obtained using the Fit peak option in the Peak Editor (refer to Peak Editor in Chapter 7).

Active Metals   Enabled for Pulse Chemisorption reports; displays the
                                            Active Metals Table dialog, allowing you to specify the
                                            percent of sample weight of the active metals in the
                                            sample (in the far right column), and the stoichiometry
                                            factor.

	Element		Atomic Weight	Atomic Area (nm²)	Density (g/cm³)	Stoi-chiometry Factor	% of Sample Mass	
1	chromium	▼	51.996	0.0635	7.200	1.000	0.0000	
2	cobalt	▼	58.933	0.0662	8.900	1.000	0.0000	
3	copper	▼	63.540	0.0680	8.920	1.000	0.0000	
4	molybdenum	▼	95.940	0.0730	10.200	1.000	0.0000	
5	nickel	▼	58.710	0.0649	8.900	1.000	0.0000	
6	palladium	▼	106.400	0.0787	12.020	1.000	0.0000	
7	platinum	▼	195.090	0.0800	21.450	1.000	0.0000	
8	rhenium	▼	186.200	0.0649	20.530	1.000	0.0000	
9	rhodium	▼	102.905	0.0752	12.400	1.000	0.0000	
10	silver	▼	107.868	0.0869	10.500	1.000	0.0000	

Buttons: Insert, Delete, Clear, OK, Cancel

'Ctrl-Down Arrow' to append

This table displays the defaults specified using the Active Metals Defaults option on the Options menu. After a sample file is created, the table becomes specific to the sample file. Therefore changes made in the current table do not affect default settings.

Insert   Inserts a new row into the table; the row is inserted
                                            above the selected one.

Delete   Deletes the selected row.

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

Save

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

Save As

Save As enables you to save the sample file or parameter file in the active window under a new name. Saving the file under a new name allows you to make modifications without changing the original file.

You can save a sample information file as another sample information file or as any parameter file. Parameter files, however, can be saved only as another parameter file of the same type. For example, you can save the analysis conditions portion of a Sample Information file as an analysis conditions file or the report options portion as a report options file. But an analysis conditions file can be saved only as another analysis conditions file.

Be sure to save parameter files to the directory specified as the Parameter Files directory (see Parameter Files Directory in Chapter 8) or it will not display in the drop-down list on the Basic Sample Information file editor. The default directory is PARAMS.

> **After you use the Save As function, the original file is still open, not the new one. To edit the new file, close the original file, then open the new file.**

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:

> **(path\file name) has been changed. Save changes before closing?**
>
> **Yes   No   Cancel**

- Click **Yes** to save the changes and close the dialog box
- Click **No** to discard the changes and close the dialog box
- Click **Cancel** to allow the file to remain active

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:

> **(path\file name) has been changed. Save changes before closing?**
>
> **Yes   No   Cancel**

- Click **Yes** to save the changes and close the dialog box
- Click **No** to discard the changes and close the dialog box
- Click **Cancel** to allow the file to remain active

Print

Print enables you to print details of a sample or parameter file; a dialog similar to the one shown below is displayed.
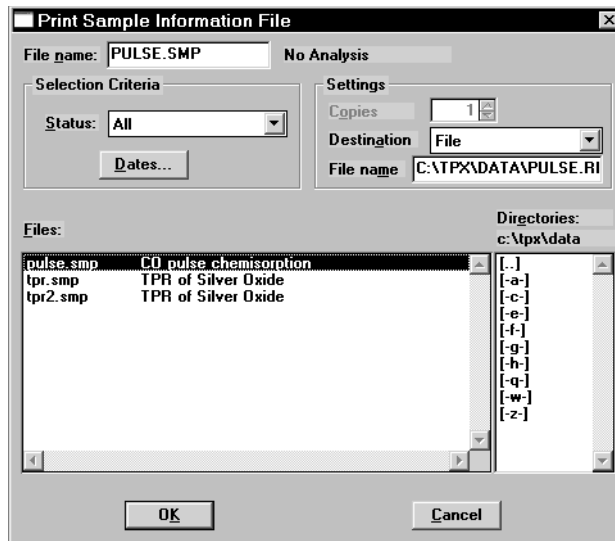
File name	The name of the file you select from the **Files** list window is copied to this field. If choosing multiple files, the last name chosen is displayed.
Status	Enables you to choose the type of files to display in the **Files** window.
Dates	Enables you to choose a date range for the files to display in the **Files** window.
Settings group box	The **Destination** field contains a list of the available print destinations.

If you select **Printer** or **Printer/Plotter**, the **Copies** field is enabled allowing you to enter up to four copies.

If you select **File**, 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 tools. The **File name** field is also enabled allowing you to enter a name (or you can accept the default).
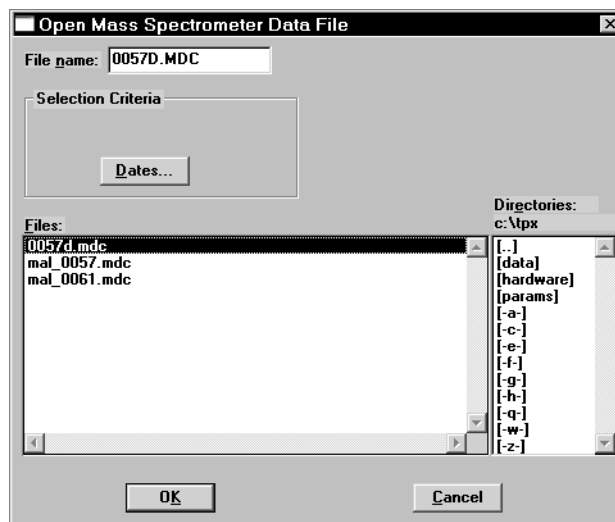
List

List enables you to generate a listing of the following information on a selected file, or on multiple files:

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

Regardless of which file type you select, a dialog similar to the one shown below is displayed.
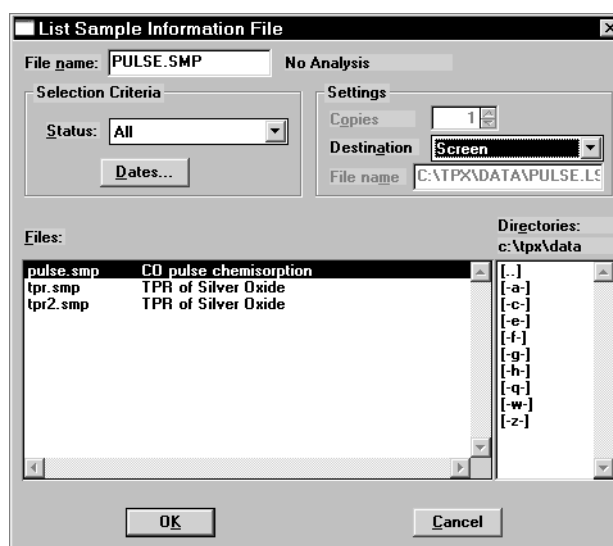
| File name | If you select only one file from the **Files** list window, the name is copied to this field. If multiple files are selected, the last one selected displays.

To select multiple files, hold down the **Ctrl** key while selecting the files. |
| **Status** | Enables you to choose the type of files to display in the **Files** window. |
| **Dates** | Enables you to choose a date range for the files to display in the **Files** window. |

Settings group box   The **Destination** field contains a list of the available
                                           print destinations.

                                           If you select **Printer** or **Printer/Plotter**, the **Copies** field
                                           is enabled allowing you to enter up to four copies.

                                           If you select **File**, 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 tools. The **File
                                           name** field is also enabled allowing you to enter a name
                                           (or you can accept the default).

Import MS Signal

Import MS Signal allows you to import data collected by Pfeiffer Vacuum's ThermoStar mass spectrometer (which uses QuadStar software).

> **Analog signals must be configured in the QuadStar software before they can be imported properly. Refer to Configuring Analog Signals on the following page.**

Select **Import MS Signal** from the File menu; the Open Mass Spectrometer Data File dialog is displayed.

File name	Displays the wildcard (*) with an MDC extension. This is the file extension used by QuadStar software.
Dates	Enables you to choose a date range for the files you wish to display in the **Files** window.
Files list	Displays the files available in the current directory.

After you select the desired file and click **OK**, the Import Mass Spectrometer Signal dialog is displayed.
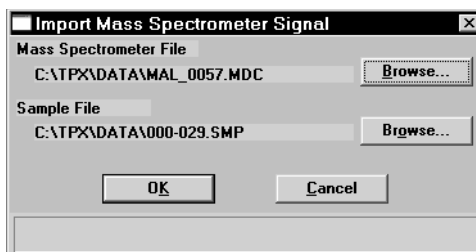
Mass Spectrometer File	Displays the mass spectrometer file you selected from the previous dialog to import into the ChemiSoft program.
Sample File	Displays the sample file into which the mass spectrometer file will be imported.
	If you have a sample file open, its name is displayed in this field and the data will be imported into this file when you click **OK**.
	If you do not have a sample file open, the next sequenced file number is displayed in this field. When you click **OK**, the data will automatically import and assume the next sequenced number; you will not be asked if you wish to create the file.
Browse	This push button is provided for each type of file so that you can choose a different file from the one(s) displayed.

Configuring the Analog Signals

The import utility reads both the mass spectrometer data and any analog signals that are collected by the ThermoStar. The analog signals must be configured for them to be imported properly. This is accomplished through the Parset program, which is part of the Quadstar software.

1. Launch the Parset program; using Windows Explorer, double-click on Parset.exe.

2. Refer to the **Instrument Configuration** section of the ThermoStar operator's manual and configure the characteristic curves as follows:

   A1 = T
   A0 = TCD

Export

Export allows you to copy data in a sample information file and reformat it in ASCII format or Grams/32 format. ASCII data can be imported into other applications, such as spreadsheets. Grams/32 data format can be used with the Grams/32 program (a peak editing program used in earlier versions of the Micromeritics AutoChem analyzer). You may select multiple files by holding down and clicking the left mouse button once on each file. See Appendix D for a description of exported data.

Select **Export** from the **File** menu; the Export Sample File dialog is displayed.

File name	The name of the file you select from the **Files** list box is copied to this field.
Export Type	You can export the data from a sample information file in an ASCII or Grams/32 format.

- Choose ASCII to create a data file that can be imported into an application such as a spreadsheet program.

- Choose Grams/32 to create a data file that can be used with the Grams/32 program.

Status	Enables you to choose the type of files to display in the **Files** window.
Dates	Enables you to choose a date range for the files to display in the **Files** window.

Settings group box   The **Destination** field contains a list of the available
                                print destinations.

                                If you select **Printer** or **Printer/Plotter**, the **Copies** field
                                is enabled allowing you to enter up to four copies.

                                If you select **File**, 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 tools. The **File
                                name** field is also enabled allowing you to enter a name
                                (or you can accept the default).

> **A sample information file exported as a Grams/32 type can only be generated to a
> File destination. When you export a Grams/32 type, five separate files (which can
> be read only the Grams/32 software) are generated.**

When you click **OK**, the Collected Data Export Options dialog is displayed.

**Only this portion of the
dialog displays when
you choose Grams/32
as the type of export.**
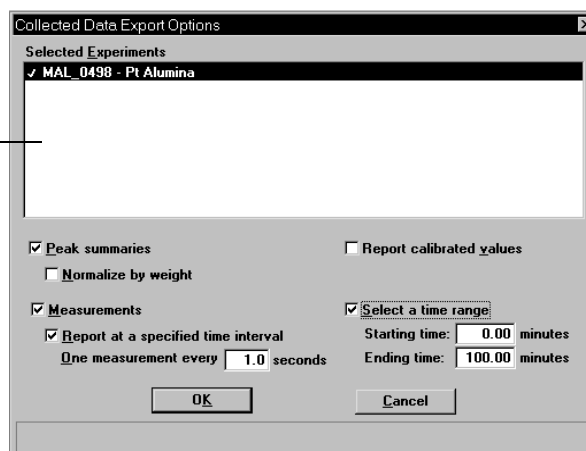
Selected Experiments   Displays a list of the experiments included in this sample
                                file. All experiments are selected by default, you may
                                deselect any you do not wish to include in the export
                                operation.

                                This portion of the dialog is the only field displayed if
                                you are exporting a file as a Grams/32 type.

Peak summaries   Exports a summary of the peaks contained in the
                                selected experiment(s); enables the **Normalize by
                                weight** field.

Normalize by weight   Divides the TCD signal by the sample weight to correct
                                the signal strength for different sample sizes.

Report calibrated values   Exports the calibrated values for the TCD signal if calibration data are available.

Measurements   Select this option to report measurements; the related fields are enabled.

Report at a specified time interval   Select this option to report measurements at a specified interval; a field is enabled so that you may enter an interval (the number of seconds between reported data readings). You may wish to use this feature if a large amount of data was collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, points are reported as follows:

- Up to 500 lines per experiment if destination is to the screen or to a printer.

- Up to 10,000 total lines if destination is to a file.

Select a time range   Select this option to specify relative starting and ending times of the measurements you wish to export. This feature allows you to specify a region of interest to be extracted from a large data set.

Exit

Exit enables you to exit the ChemiSoft program. Select **Exit** from the File menu.

- If a window containing unsaved changes is open, the following message is displayed:

> **(path\file name) has been changed. Save changes before closing?**
>
> **Yes   No   Cancel**

Click **Yes** to save the changes and exit the program
Click **No** to discard the changes and exit the program
Click **Cancel** to allow the file to remain active

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

> **2547- An instrument is busy. Exiting the applicaiton will result in termination of the analysis. Continue with Exit?**
>
> **Yes   No**

Click **Yes** if you are not concerned with terminating the analysis.
Click **No** to allow the analysis to finish.

- If a report is in progress or a report window is open, the following message is displayed:

> **A report is currently printing. Exiting the application now will cancel the report. Continue with the termination?**
>
> **Yes   No**

Click **Yes** to cancel the report(s) and exit the program.
Click **No** to allow the program to remain active.

6. UNIT MENU

The Unit menu contains options that enable you to perform operations relating to the analyzer.

Description

Listed below are brief descriptions of the Unit menu options. Detailed descriptions follow this section.
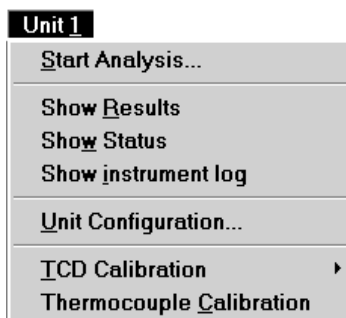
Start Analysis   Opens a series of dialog boxes that enable you to start an analysis. Page **6-2**.

Show Status   Displays only the Status Bar of the operational window. Page **6-9**.

Show Instrument Log   Displays a log of instrument operations. Page **6-10**.

Show Results   Shows data collection for the current analysis. Page **6-12**.

Unit Configuration   Displays the software versions for the analysis program and controller, the analyzer model and serial numbers.

                                Also allows you to enter the loop (if applicable) volume. Page **6-14**.

TCD Calibration   Displays a series of dialogs allowing you to create, edit, and manage calibration files. Calibration files enable the software to convert sample signal, temperature, and peak area data to volume data. Page **6-15**.

Thermocouple Calibration   Enables you to calibrate the sample thermocouple. Page **6-20**.

Start Analysis

When you select Start Analysis, the Analysis dialog is displayed with a file selection dialog positioned on top. You can either select a sample file that has been previously created, or you can create a new sample file using the next sequenced number.

Which option you choose depends on the method you prefer for your laboratory. The analysis process is the same either way. For example, your lab may prefer to create numerous sample files in advance, then analyze the samples when they become available. Alternatively, your lab may prefer to create sample files for each sample as you begin the analysis.

After you choose a sample file and click **OK**, the Analysis dialog becomes totally visible.

> **The software is unable to determine if the wrong gas is physically connected to the port, or selected with the proper push button on the analyzer front panel. Be sure that appropriate gases are connected as requested in the sample file.**

This group of push buttons does not display in the Basic and Restricted presentations.

The Analysis dialog for all presentation modes are identical except for the group of push buttons positioned just below the Analysis Conditions window. This group of push buttons is used for inserting and editing steps (if needed) and is available only in the Advanced mode.

View	Allows you to choose from four views to display in the current window
Operate	Displays the dialogs associated with the current operation.
Results	Displays analysis results as they are collected.It is best to use the **Show Results** command so that you can allow the operation window to remain viewable.
Sample log	Displays a log of each step as it is completed, as well as any error messages or manual operations that occur during the analysis.
Browse	Enables you to choose a different sample file for the current analysis.
Sample	Displays the description for the current file.
Replace All	Allows you to replace the contents of the current file with those of another one. After the values have been copied into the current file, you may edit them in any way you wish. Editing the values will not affect the file from which they were copied.
Mass	Enables you to edit the value for the mass of the current sample.
Analysis Conditions	Displays the current Analysis Conditions file. You can choose a different file (if desired) from the drop-down list. The files in the drop-down list are predefined parameter files created and saved to the parameter files directory (some also were included with the ChemiSoft program).
	The window directly below this list contains the conditions specified in the Analysis Conditions file. If you are using the Advanced format for the current analysis, you can edit the conditions using the push buttons provided below the Analysis Conditions window. Refer to Chapter 5 for operation of these push buttons.

Analysis Conditions
(continued)

You can also edit analysis conditions after the analysis has begun. Click **Suspend** to suspend the analysis; this enables the editing push buttons. You can edit only the steps or experiments following the current one. After editing is complete, resume the analysis.

Report Options

Displays the current Report Options file. This drop-down list contains predefined parameter files, some of which are supplied with the analysis program. For newly created files to display in this list, they must be saved to the directory specified as the parameter files directory (see Chapter 8). Click the down arrow to the right of the field to choose a different file.

The window directly below this list contains the reports requested for the current Report Options file. You can select additional reports or deselect currently selected ones if desired. A report is selected when it is preceded with a check mark. Simply double-click to select (or deselect) a report. Alternatively, you can highlight the report and press **Spacebar**.
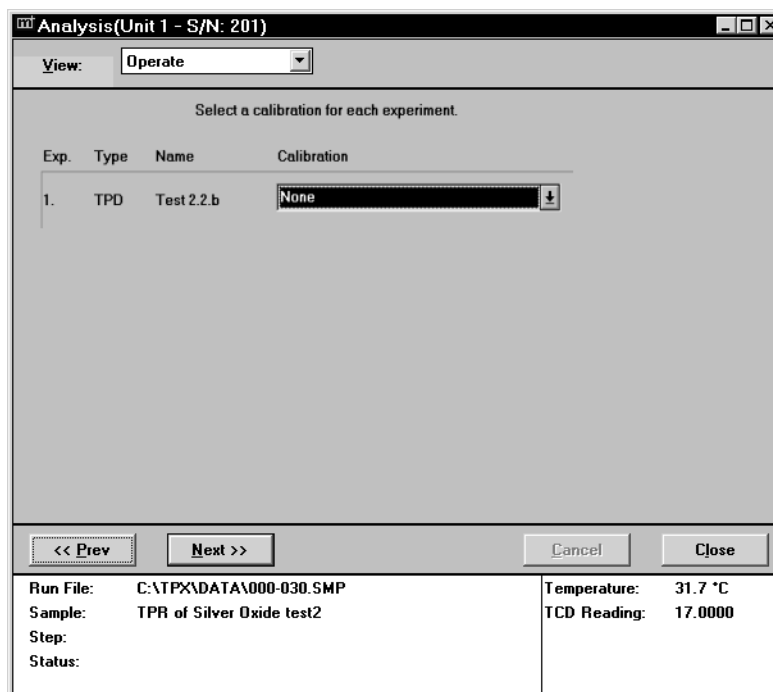
Next

Advances you to the second view of the Analysis dialog. This view lists the gases required for the current analysis.
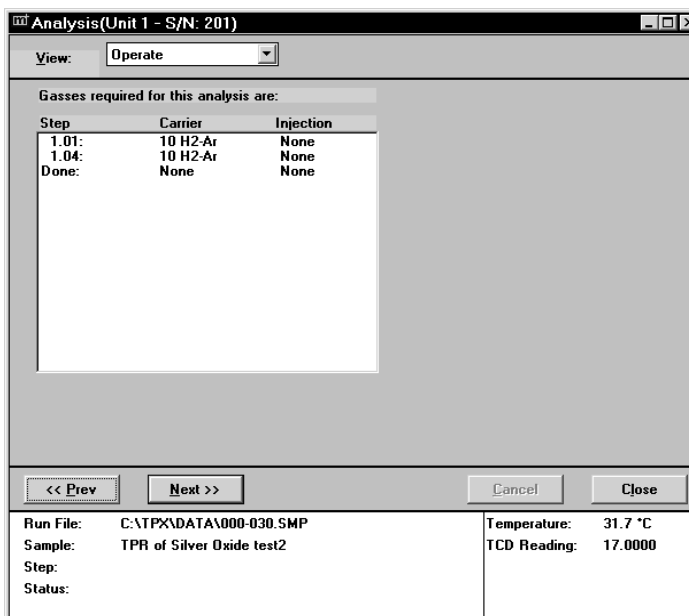
Click **Next** to advance to the next view of the Analysis dialog. This view enables you to choose a TCD calibration for each experiment in this sample's analysis conditions set. A TCD calibration allows the ares of the peaks recorded by the instrument to be converted into volumes. A TCD calibrating is not required by all analysis types, nor is it necessary to select the calibration from this dialog. A calibration can be associated with an experiment during peak editing (after analysis has ended).

The default(s) for the calibration file(s) are those specified using the TCD Calibration Defaults option on the Unit menu (discussed later in this chapter).

Click **Next** to advance to the next view of the Analysis dialog. This view enables you to generate reports automatically when the analysis is complete. The reports generated are those selected in the report options portion of the sample information file.

Report AfterAnalysis	Choose this option to have the reports specified in the sample file generated automatically after the analysis is complete.
Number of Reports	Enabled when **Printer** or **Printer/Plotter** is selected as the report destination. You can print up to four copies.
Destination	Allows you to choose a destination for the reports.
	Choices: File, Printer, Printer/Plotter, Screen
	If you choose **Screen** for the destination, many options are available for manipulating and customizing your reports.
File name	Enabled when **File** is selected as the report destination, allowing you to enter a file name (or you can accept the default).

Click **Next**; a dialog instructing you to verify the following is displayed:

- Verify in the sample file that the combinations or sequence of gases in the analyzer are not dangerous, and that the sample will not react with any of the gases in a dangerous way.

- Verify that the current method for venting the analyzer's exhausts is adequate and safe for all products of the analysis.

After you have ascertained that all conditions are safe for the analysis, click **Start** to begin the analysis; this view of the analysis dialog is displayed.
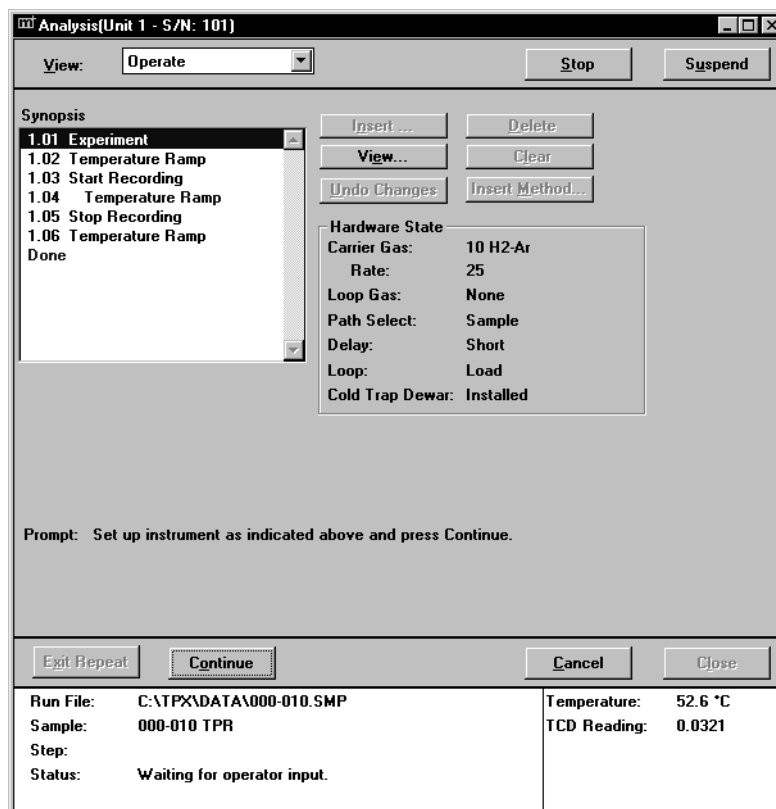
| **Synopsis** | Shows the list of experiment steps in the sample information file. Upcoming steps are shown in blue or black. The current step is highlighted and shown in light blue; completed steps are shown in light green.

You may scroll through the list to review the analysis steps. You can also edit steps that have not been started; the analysis must be suspended to do this (see Suspend below). |

| **Step Detail** | Shows detailed information about the current step. |

Stop   Stops operation of the current step, displaying of the
                                 prompts, and suspends the analysis. Click **Resume** to
                                 restart the analysis and the operating prompts. The anal-
                                 ysis resumes at the beginning of the next step. This
                                 button is used to skip the remaining portion of the cur-
                                 rent step.

Cancel   Cancels the analysis; the current step is not completed.
                                 After confirmation, all operating prompts cease to
                                 display, and the analysis ends. The analysis cannot be
                                 resumed.

Suspend   Suspends the analysis. This button is a toggle button; it
                                 reads **Resume** when the analysis is suspended.

                                 You may wish to suspend the analysis to edit steps in the
                                 list or add steps to the list. Steps can only be added or
                                 edited in the portion of the list that has not yet been
                                 executed; such editing is available only if you are using
                                 Advanced presentation format. The current step cannot
                                 be edited. Click **Resume** to continue the analysis after
                                 editing has been completed.

> **During analysis, analysis conditions can be edited only using this view of the Start
> Analysis dialogs. Analysis conditions cannot be edited in the sample file during
> analysis. The analysis must be in a suspended state.**

Prompt   Displays information about the step and any actions
                                 required from the operator before the analysis can
                                 continue. If an action is required, the message displays in
                                 blue.

Continue   Click this push button after you have completed the
                                 task(s) displayed at the **Prompt**.

Exit Repeat   Enabled during a **Stop Repeat** step, enabling you to exit
                                 the Repeat sequence.

Show Status

Select this option to display the status bar of the operation window. The bottom portion of the Analysis dialog sequence displays the instrument's Status window. This option allows you to display only the status portion of the operational window.

Status (Unit 1 - S/N: 101)			
Run File:	C:\TPX\DAVE\000-071.SMP	Temperature:	873.1 °C
Sample:	High Temp Programmed Reduction - iron	TCD Reading:	0.2607
Step:			
Status:	Waiting for operator input.		
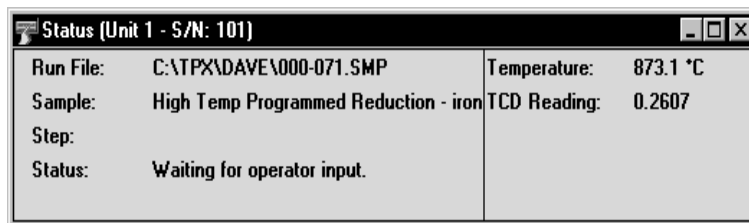
The Status window displays:

Run File   Gives the name of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.

Temperature   Gives the sample's temperature, as measured by the sample thermocouple.

TCD Reading   Displays the current detector reading (0.0000 if disabled).

During analysis, the Status window also displays:

Sample (Identifier)   Gives the identifier of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.

Step   Shows the step currently being performed. When no analysis is in progress, Status: Idle is displayed instead.

Duration   Shows the amount of time that has elapsed since the current step was started. If the analysis is waiting for a fixed period of time, the time left to wait is shown in parentheses next to the time.

Repeat Count   Shows how many times the current Repeat loop has been entered.

Show Instrument Log

Select this option to display a log of instrument tasks and messages.
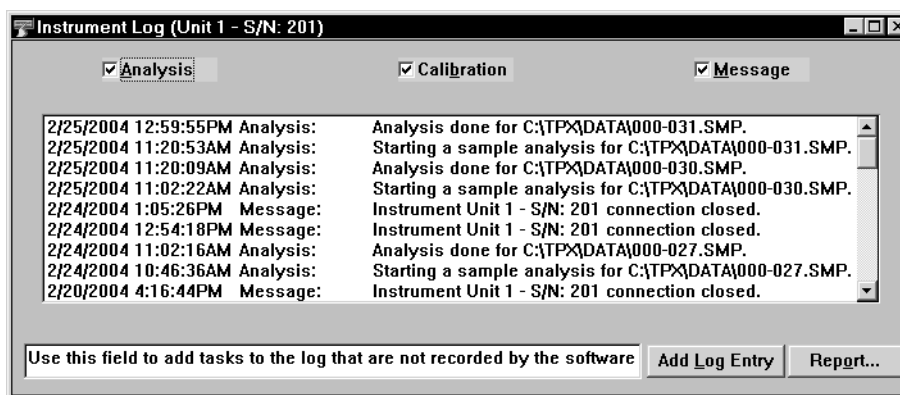
Analysis
Calibration
Message

Use these options to choose the type of information you wish to display in the window. For example, you may wish to select only the **Analysis** option to display analyses only.

Add Log Entry

Enables you to add an entry to the instrument log. For example, you may wish to enter some type of maintenance you performed on your analyzer. This entry displays in the **Message** category.

Report

Displays the Log Report dialog enabling you to generate a report on the contents of the Instrument Log.
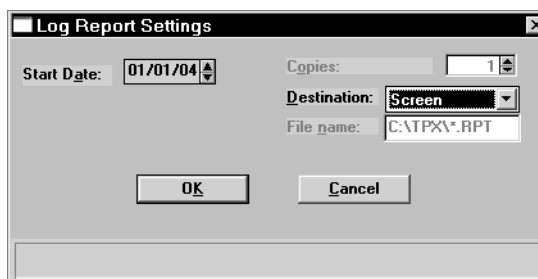
Start Date

Use this field to specify a date at which to start the log entries. You can enter a date or double-click in the field to display a calendar from which to choose a date.

Copies

Enabled when you choose Printer or Printer/Plotter as the destination, allowing you to print up to four copies of the report.

Destination   Presents destination options; Printer, Printer/Plotter, Screen, or File name.

File name   Enabled when you choose **File** as the print destination, allowing you to enter a name for your file.

Show Results

Select this option to view data collection for the current analysis.
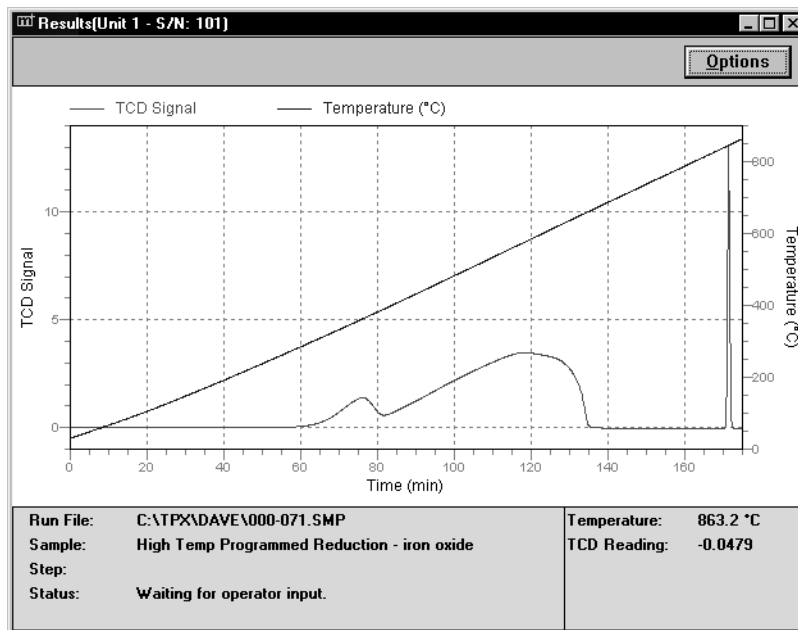
This feature enables you to view collection of data as it progresses and leave the operational window open so that you can respond to prompts requesting user interaction. If you choose **Results** from the **View** drop-down list on the operational window, you cannot view the requested actions of the prompts.

Click **Options** to customize the presentation of data in the **Results** view. You can change the axes and select or deselect the data that are presented. If you specify a value for Starting at (X-Axis), only those data collected after that point are shown.

Y-Axis   Displays Y-axis choices.Use the Scaling push button to choose autoscaling or to specify a range for scaling

X-Axis   Allows you to choose the X-Axis variable. You can use the **Starting at** field to specify at what point you wish to start data collection. If you enter a value greater than the experiment duration, all data are shown

Smoothing Options   When detector signals are weak and noise or other irregular signals are present, judicious use of these options can often suppress noise and enhance data.

Unit Configuration

Select this option to display the configuration for the analyzer on which the software is
being used.

```
┌─ Unit configuration (Unit 1 - S/N: 101) ──────────────────── [X] ─┐
│ ┌─ Software Versions ───────────────────────────────────────────┐ │
│ │   Application:  ChemiSoft TPx V1.00                            │ │
│ │   028 Controller:   109                                       │ │
│ │                                                               │ │
│ │   Model:      2750          Loop Volume [  0.0000 ] mL        │ │
│ │   Serial #:   101                                             │ │
│ │            [    OK    ]          [   Cancel   ]               │ │
│ └───────────────────────────────────────────────────────────────┘ │
│ Enter a value between -1000.0000 and 1000.0000.                   │
└───────────────────────────────────────────────────────────────────┘
```
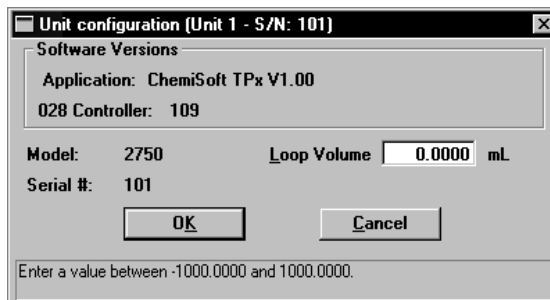
Software Versions   Displays the name and version number of the ChemiSoft
                              program and the data acquisition module.

Model   Displays the model and serial numbers of the instrument
Serial #   on which the ChemiSoft program is being used.

Loop Volume   Allows you to enter the volume of the gas injection loop
                              that is attached to the instrument. Three standard-volume
                              loops are provided with your instrument. The loop
                              volume is determined by performing a loop calibration.
                              Loop calibration is performed as an experiment step
                              (refer to **Loop Calibration** in Chapter 5)

TCD Calibration

Analyses yield data on signal reading, peak area, temperature, and time. These data are sufficient for many applications, but in some cases, actual gas volume data are desired as well.

> **In applications where volume data are not required, it is not necessary to perform a calibration. If volume data are desired, calibration may be performed either before or after the analysis.**

Unit 1

Start Analysis...

Show Results
Show Status
Show instrument log

Unit Configuration...

TCD Calibration ▶   Defaults...
Thermocouple Calibration   New...
   Open...
   Report...
   List...
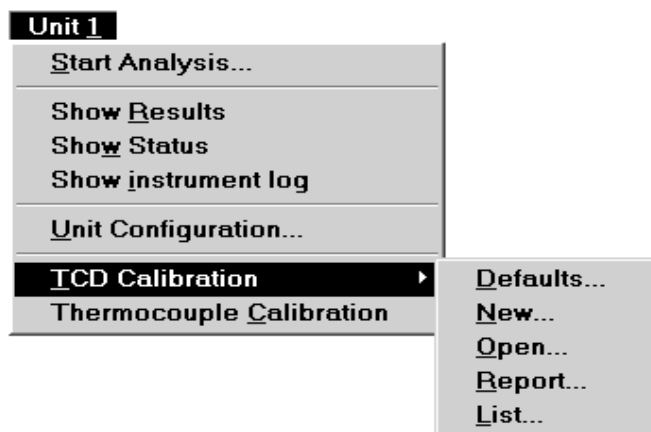
A group of calibration routines is provided in the form of specialized experiment steps. A calibration run is an analysis using one of these experiment steps. The calibration run can be performed before or after the sample analysis. A calibration experiment can be included as a step within your sample analysis, or it can be performed as a separate analysis.

After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example, TPR yields peak area and the temperature(s) at which maximum reduction occurs. To obtain the volume of gas uptake, however, you must select a calibration file to associate with the analysis file. Then you can create reports in which the area data are converted to volume data.

You can associate a calibration file with a sample file in three different ways:

- Select TCD calibration, **Defaults** on the Unit menu and specify a default calibration file for each experiment type.

- Choose **Set Calibration** from the Peak Editor (described in Chapter 7). This choice allows you to select a calibration file that was created after the sample file was used in an analysis.

Defaults

Allows you to specify default calibration files for each type of analysis. An additional default can be specified for analyses you have customized, using the **Other** drop-down list. Setting default calibrations can save you time when you frequently perform analyses using the same gases.
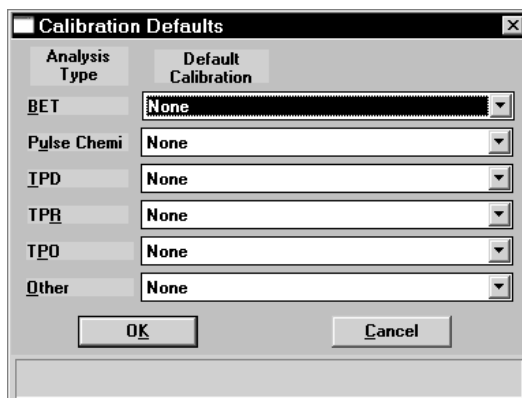
The calibration files that appear in the lists are those that are available for the instrument.

You can select a different calibration file using **Set Calibration** on the Peak Editor dialog.

New

Allows you to create a new TCD calibration file; the TCD Calibration dialog is displayed.
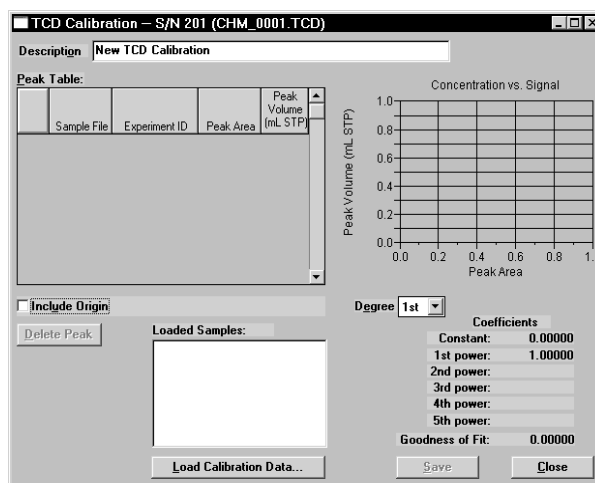
Click **Load Calibration Data** to load the values from an existing TCD file, then proceed with editing as desired. The fields on this dialog are identical to the ones on the Open TCD dialog (refer to the next section for a description of the fields).

Open

Allows you to open an existing calibration file for editing; the Edit Existing TCD Calibration dialog is displayed.
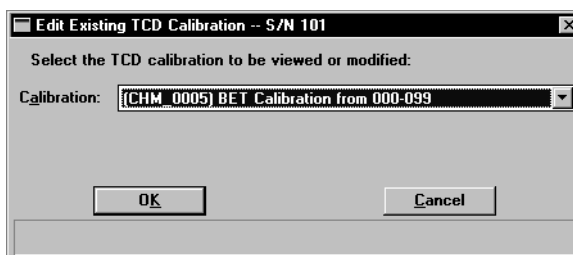
This dialog contains a drop-down list of the existing TCD calibration files for this instrument. After choosing the desired file, click **OK**; the TCD Calibration dialog is displayed.
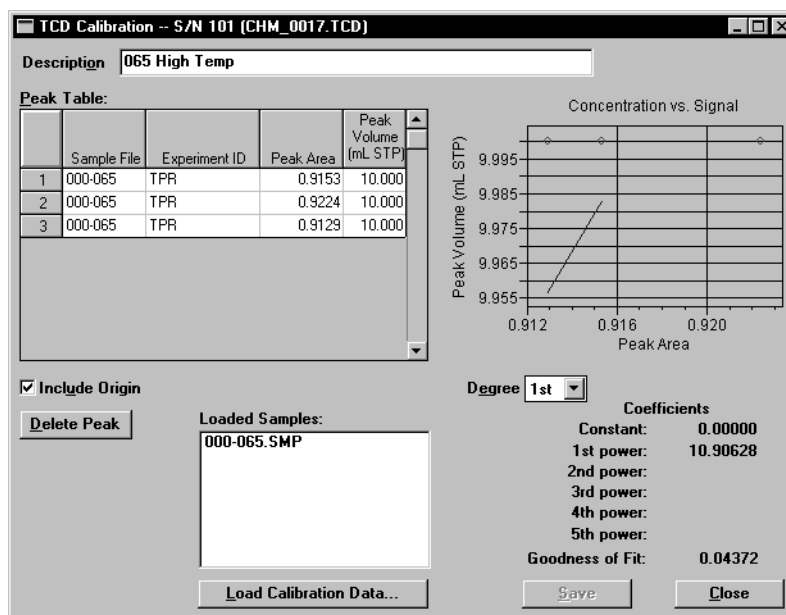
Peak Table	This table displays the sample file number, the experiment identification, the peak area, and the peak volume.The values in the table are graphically depicted in a plot adjacent to the table.
Delete Peak	Deletes the selected peak.
Load Calibration Data	Enables you to load another calibration file. The calibration file you choose must have been created with the current instrument. If you select a file created on a different instrument, an error message is displayed

Degree	Allows you to choose a different degree of polynomial; usually the 2nd degree is adequate.
Coefficients	Shows the fit of data to the polynomial.
Description	Displays the name of the current calibration file. This field can also be sued to enter a new name when changes have been made.
Save	Enabled when the current file as been edited, allowing you to save the changes. If you wish to save it under a different name, enter a name in the **Description** field.
Close	Closes the dialog box.

Report

Select this option to print a report of a calibration file.
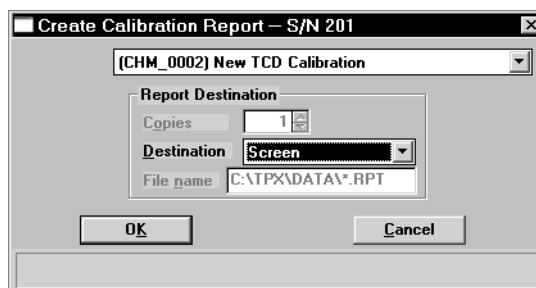
Calibration	From the drop-down list, select the file for which you wish to generate a report.
Copies	Enabled when you choose **Printer** or **Printer/Plotter** as the report destination. You can print up to four copies.
Destination	Allows you to choose a destination for the report. *Choices: File, Printer, Printer/Plotter, Screen*
File name	Enabled when **File** is selected as the report destination, allowing you to enter a file name (or accept the default).

List

Select this option to display a list of the files in the Calibration Directory, including details such as the calibration file's identifier (as entered in the sample information file) and the date the file was created (or last edited).
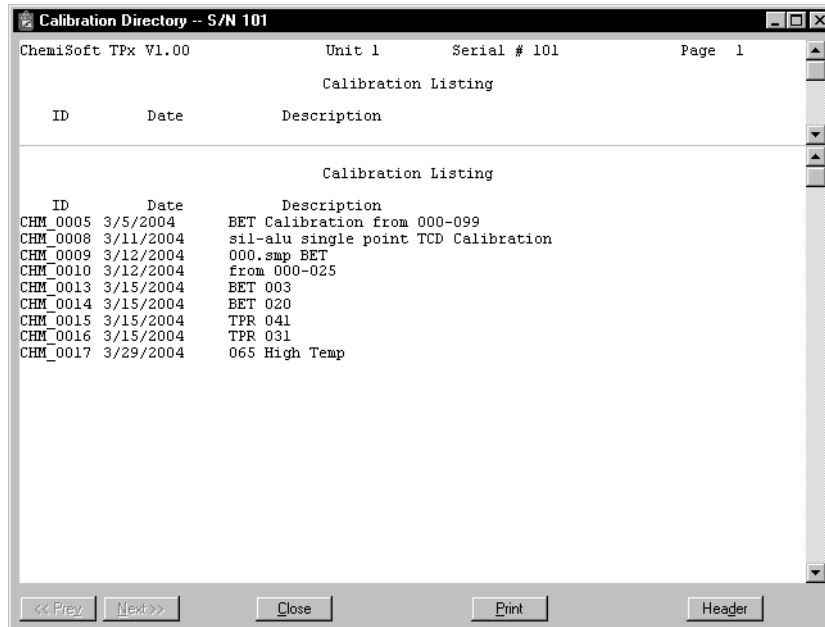
Next/Prev	Allow you to view the next or previous page of the list. Enabled only when the report contains multiple pages.
Close	Closes the window.
Print	Prints the list.
Header	This toggle button hides/shows the header of this list. If the header is selected, it appears on every page of the list when it is displayed and/or printed.

Thermocouple Calibration

This option displays the Thermocouple Calibration dialog.

The sample and furnace thermocouples are precalibrated and typically are appropriate for all applications. However, this option has been included if modifications are desired.

Heat Zones

Displays information on the sample and furnace thermocouples. If you choose to recalibrate either thermocouple using an external reference, simply double-click in the appropriate **Low/High Cal** field to enter the value you obtain; a dialog similar to this one is displayed.

The date you enter the new value will automatically be inserted into the appropriate **Calibrated** field.

Load from File

Allows you to load a different calibration file.

Save to File   Allows you to save the current calibration settings to a file. This function is useful in protecting your calibration data. It is wise to save the current settings before attempting a recalibration. Be sure to use the next sequenced file name generated by the system.

Accept   Replaces the existing calibration values with those you have entered in this session. After a confirmation, the software gives you the opportunity to save the existing values. The Calibrate Hardware dialog is closed after you accept the new values.

Cancel   Cancels any changes made to the current calibration settings. A warning message is displayed before the dialog closes

Reset   Enabled when a change has been made to any field on this dialog. Click this push button to reset any changed value(s) to its last saved state.

7. REPORT MENU

The Report menu contains options which allow you to initiate and close reports. This menu also allows you to access the Peak Editor, a flexible and powerful tool for processing results.

This chapter describes the options contained on the Report menu, as well as a few examples of generated reports.

Description

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

Start Report   Allows you to generate a report on a sample which has been analyzed or on one which is being analyfzed. Page **7-2**.

Close Reports   Closes all open report window(s). Page **7-5**.

Peak Editor   Allows you to view peak data, combine and overlay results from multiple analyses, change the calibration files selected, and edit the data. Page **7-14**.

Start Report

Select this option to generate a report on a sample analysis. The Start Report dialog is displayed.
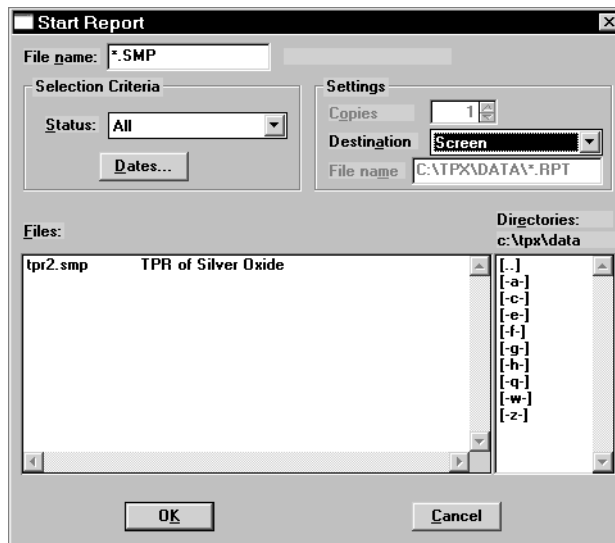
File name	The name you select from the Files list box is copied to this field. If you select multiple files, the last one selected is displayed. If you have a file open, its name is displayed.
Status	This drop-down list determines the type of sample files displayed in the **Files** list box.

Choices: All, Analyzing, Complete |
All	Displays all sample information files in the specified directory and within the range of dates selected in the **Dates** dialog.
Analyzing	Displays sample information files that are currently being used for analysis.
Complete	Displays sample information files that have been used in an analysis.
No Analysis	Displays sample information file that have been prepared but not yet used in an analysis.

Dates   Displays the Select Dates dialog.

                                       This dialog enables you to specify a date range for the
                                       files you wish to display in the **Files** window.

Show All Dates   Displays all sample files of the type selected in the
                                       Status list, in the current directory.

Show Date Range   Enables the **From** and **To** fields allowing you to specify
                                       a range of dates in which to display the sample files. Use
                                       one of the following methods to select date ranges:

                                       • Highlight the field and enter a date, simply by typing
                                         the date; you do not have to enter a slash (/).

                                       • Double-click in the selected field to display a calendar
                                         from which you can select a date.

                                       For convenience, the following shortcut keys are
                                       available to assist you:

                                       F2 Clears the field
                                       F3 Inserts the current date
                                       F4 Displays a calendar from which to select a date

Copies   Enabled when the **Printer** or **Printer/Plotter** destination
                                       is chosen, enabling you to print up to four copies of the
                                       selected report(s).

Destination   Displays a list of output destinations.

                                       Choices: File, Printer, Printer/Plotter, Screen

                                       If you select **Printer** or **Printer/Plotter**, requested
                                       reports are printed to your default printer and/or plotter.

                                       If you select **Screen**, many options are available for
                                       manipulating and customizing reports. Refer to **Printed
                                       Reports** later in this chapter for manipulation methods
                                       of onscreen reports.

Destination   If you select **File**, the tabular reports of the requested file
(continued)   are converted to a text file which can be viewed with a
                             text editor or other text file manipulation tools. Graphi-
                             cal reports cannot be generated to a file.

File name   Enabled when you select **File** as the destination. Allows
                             you to enter a name, or you may accept the default.

Files list   Displays a list of the available sample files for the choice
                             shown in the Status field.

Directories   Displays a list of available drives and directories. The
                             drive and directory last accessed is displayed
                             immediately above the Directories: list box.

If you choose a single sample file and click **OK**, the Select Reports dialog is displayed so
that you may select the desired reports. The reports selected are the ones you specified in
the sample file. This dialog allows you to deselect reports or select additional ones. If you
choose more than one file, this dialog is not displayed; the reports selected in each file are
generated.
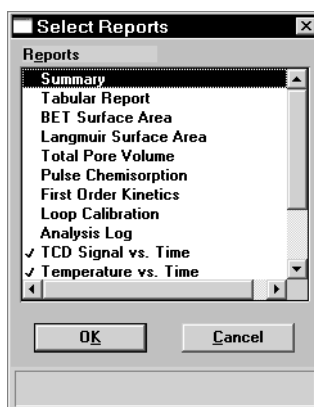
Double-click on the report(s) you wish to select (or deselect). A check mark is placed
before each selected report. Pressing the **Spacebar** (when the report is highlighted)
performs the same function. After you make your report selection and click **OK** , the
requested reports are sent to the specified destination.

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.

> **Some operating systems may cause the main menu bar to grey-out and appear nonfunctional when a Report window is closed. Simply click on the menu bar to return it to its normal state.**

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.

  Also included in the Tabular and graphical report headers are notes of any changes to the sample file which occur after analysis.

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

Onscreen Reports

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

- a tool bar
- shortcut menu options
- 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; reports 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 generate.

Header

Lists the types of report generated.

Toolbar options

Displays graphical (or tabular) data
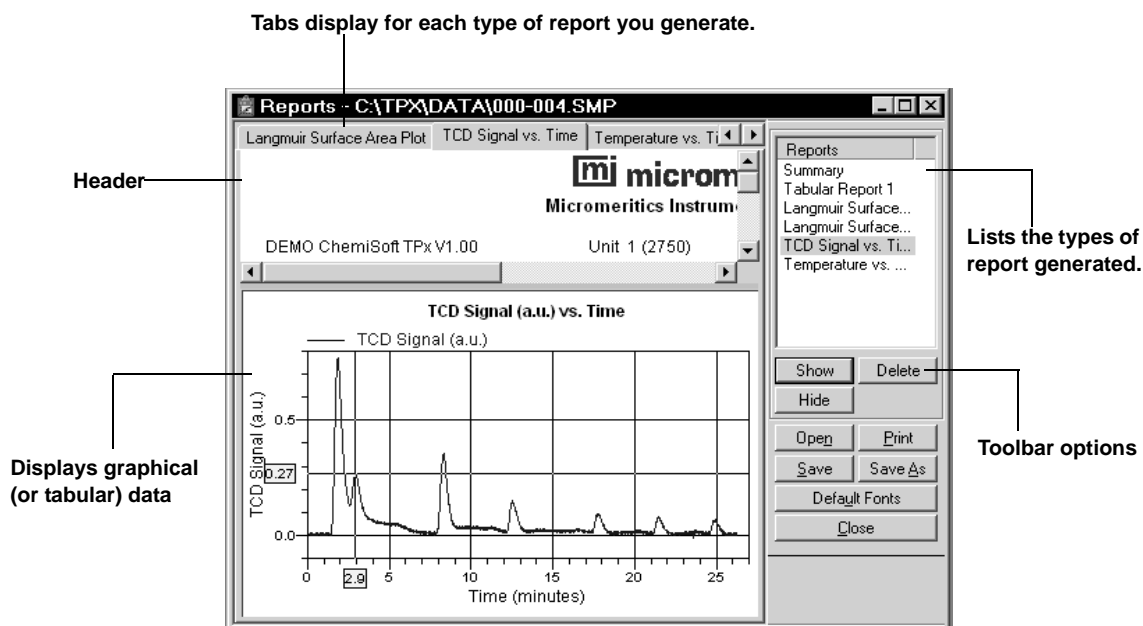
Tool Bar
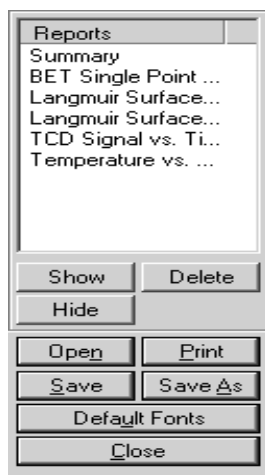
Reports   Contains a list of all requested reports.

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

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

Hide   Hides (removes) the selected report from the report window. The report's associated tab is also removed.

Open   Allows you to open a previously saved report file.

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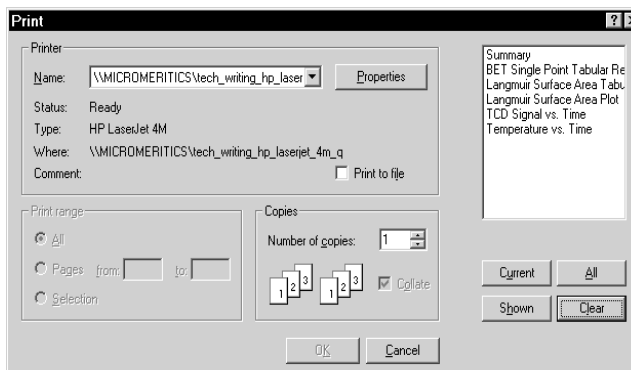
Print
(continued)

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

Current selects the report displayed in the report window.

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

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

Clear clears all selections.

Default Fonts

Displays the Default Fonts dialog so that you can edit report fonts.
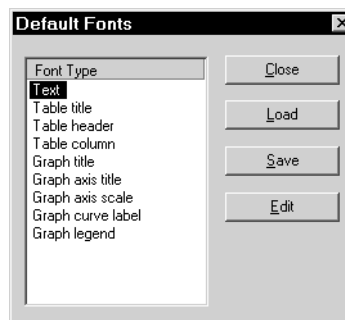
The window on the left lists the items for which the font can be edited.

Close

Closes the dialog.

Load

Loads the default font for the selected item.

Save

Saves the changes as the defaults. If you do not click **Save**, the changes will apply to the current report set only; subsequent reports will revert to the default font

Edit

Enables you to edit the font for the selected item; a font dialog is displayed so that you can specify the desired font and attributes.

Shortcut Menus

Shortcut menus are accessed when you right-click on the tabular or graphical portion of a report.

For Tabular Reports

<u>R</u>esize column
R<u>e</u>name column
<u>M</u>ove column ▶
<u>A</u>lign column ▶
<u>H</u>ide column ▶
<u>S</u>how column ▶
Column f<u>o</u>nt
Header f<u>o</u>nt
Edit <u>t</u>itle...
<u>C</u>opy table as text

Resize column	Displays a dialog so that you can specify the width of the selected column (in inches).
Rename column	Displays a dialog so that you can edit the name of the selected column. Use **Ctrl** + **Enter** to insert line feeds.
Move column	Allows you to move the location of the selected column to the left or to the right.
Align column	Enables you to right-align, left-align, or center the data in the selected column.
Hide column	Displays a list of all columns, enabling you to select the one you wish to hide.
Show column	Displays a list of all hidden columns, enabling you to select the one you wish to have shown again.
Column font	Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.
Header font	Displays a Font dialog, allowing you to change font attributes for column headers in the current report.

Edit title   Allows you to edit the table title, as well as its font.

Copy table as text   Enables you to copy the entire table (column headers and data) and then insert it into another program. Columns are tab-delimited, allowing easy alignment.

For Graphs

Autoscale	
Redraw	
Show Curve	▶
Hide curve	▶
Edit curve	▶
Edit axis	▶
Edit legend	
Edit title	
Copy as metafile	
Copy as text	

Autoscale   Sets the axis boundaries to autoscale, presenting the signal in full scale.

Redraw   Sets axis boundaries to its original view, allowing easier editing of peaks

> **In many cases, the AutoScale and Redraw options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.**

Show curve   Allows you to show curves that have been hidden. This option is disabled (greyed) if no curves have been hidden.

Hide curve   Allows you to hide (remove) unwanted curves.

Edit curve   Displays the Curve Properties dialog, allowing you to edit curve properties.
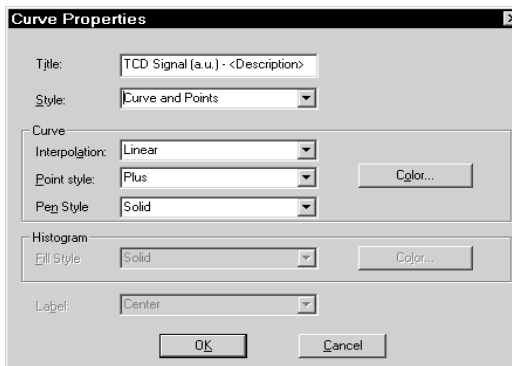
Title   Displays the title of the curve you are editing.

Style   Drop-down list containing styles in which collected data can be displayed.

Choices: Curve, Histogram, Points, Curve and Points

Curve group box   These options are enabled when you choose Curve, Points, or Curve and Points as the style. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color.

Histogram group box   These options are enabled when you choose Histogram as the style, allowing you to specify the type of fill as well as the color

Edit axis   Displays the Axis Properties dialog, allowing you to edit axis properties.
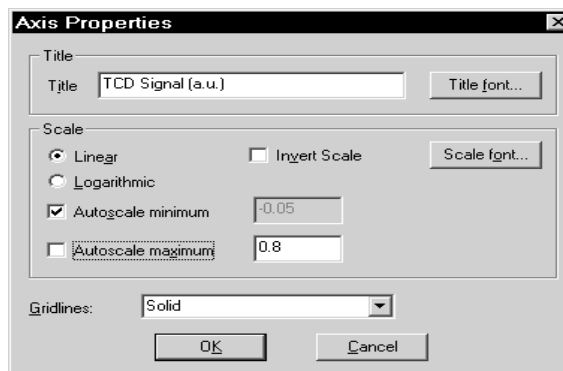
The options in this dialog enable you to edit the axis title, scaling, and graph grid lines.

Edit legend   Displays the Legend Properties dialog, allowing you to
                           edit the placement of the legend.
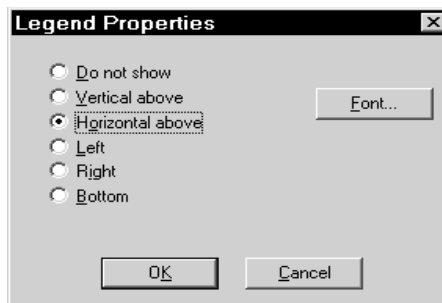
Edit title   Displays the Title Properties dialog, allowing you to edit
                           the current graph's title and font.
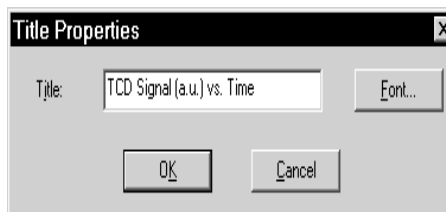
Copy as metafile   Copies the graph and places it on the clipboard, allowing
                           you to paste it into other applications accepting
                           Windows metafiles.

Use Copy as metafile to capture graphics for use in reports and presentations.

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 detail of the distribution. 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. You can return to the
normal view by right-clicking on the graph and selecting **Redraw**.

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.
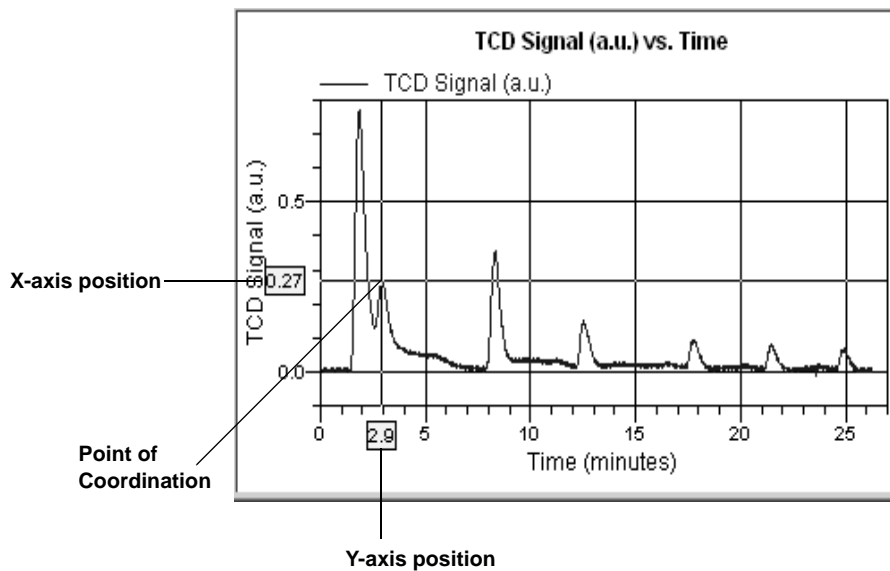
You can remove cross-hair lines and return to the normal view by right-clicking on the graph and selecting **Redraw**, or clicking out of the graph area.

Peak Editor

The Peak Editor allows you to view and edit data from sample files. You can view the peaks in a stacked or overlaid manner. You can also edit the peaks and delete undesirable ones.

When you select the **Peak Editor** option, the Edit Sample Peaks dialog allowing you to select the desired file(s) is displayed. The fields on this dialog function in the same manner as other dialogs allowing you to select files. The Peak Editor allows you to view up to 16 experiments at one time. If you choose files containing more than 16 experiments, a dialog displaying the experiments available in the chosen files is displayed.
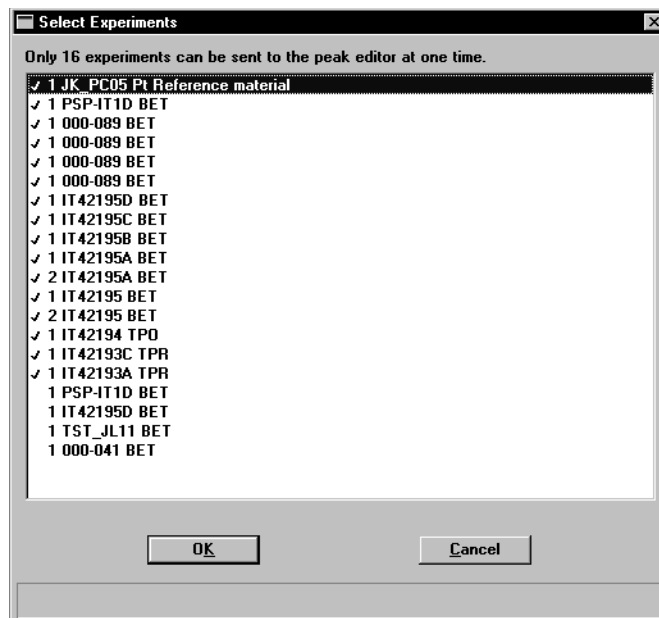
This dialog allows you to choose the 16 experiments you wish to view and defaults with the first 16 experiments selected. You may deselect and select as desired. This is accomplished by double-clicking on the file, or by highlighting the file and pressing the space bar (the same method used in selecting reports).

After you select your file(s) or your experiments (whichever is relevant) and click **OK**, the Peak Editor window is displayed. This window contains five tabs. the first three tabs — Experiments Stacked, Experiments Overlay, and Signals View — provide different views of the data; the last two tabs — Edit Peaks and Fit Peaks — provide the TCD vs. time (or temperature) and tabular information for the integrated peaks.

Features of the Peak Editor Window

The Peak Editor window provides the following tools for viewing and editing peaks:

- Shortcut menus
- Shortcut keys
- Zoom in on the TCD signal
- Cross-hairs

Shortcut Menu

A shortcut menu is available for each view of the Peak Editor window. This shortcut menu contains the available functions and is accessed by right-clicking in the graph area. For example, when you right-click in the Experiments view, the following menu is displayed:

```
Autoscale   Ctrl+A
Redraw      Ctrl+D
View                 ▶

Insert
Remove
Save As...

Close       Ctrl+F4
```
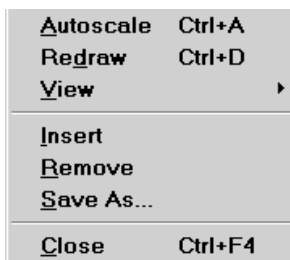
The options on the menu displayed for each view are explained in subsequent sections.

Shortcut Keys

Shortcut keys are provided for many of the functions in the Peak Editor. These shortcut keys have been assigned function-relevant characters enabling you to remember them more quickly. For example, **Ctrl+A** is assigned to Autoscale. Refer to Table 7-1 for a complete list of the shortcut keys you can use while operating the Peak Editor.

Table 7-1. Shortcut Keys for the Peak Editor

Key(s)	Function
Ctrl + **A**	Sets the axes to autoscale (full scale view)
Ctrl + **D**	Returns the Peak Editor window to its original view
Ctrl + **M**	Shows or Hides Peak Marks
Ctrl + **T**	Displays the X-axis by Time or Temperature
Ctrl + **F**	Copies graph(s) as a Metafile, allowing you to paste it into another application
Ctrl + **C**	Copies data to the clipboard, allowing you to paste it into another application
Ctrl + **N**	Advances you to the next peak
Ctrl + **P**	Returns you to the previous peak
Ctrl + **L**	Marks the left edge of the peak
Ctrl + **R**	Marks the right edge of the peak
Ctrl + **S**	Saves the changes made to the current file

Zoom Feature

A zoom feature is included with the Peak Editor allowing you to zoom in on peaks. This feature functions in the same manner as the zoom for reports; 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. If you have multiple graphs displayed, the area you define on the selected graph is enlarged for each graph. You can return to the normal view by right-clicking on the graph and selecting **Autoscale**, or you can use the shortcut keys **Ctrl + D**.

Cross-Hairs

When you click the mouse in the graph area, locator cross-hairs are displayed. The exact values associated with the intersection of the cross-hairs are displayed along the axes. This makes it easy to determine exact values for points on the second Y-axis, or for the point on the time/temperature trace that corresponds with the signal/temperature trace.

Experiments

The Experiments View shows curves for all experiments within the selected file(s). You can display the curves in a stacked fashion or in an overlaid fashion. You can display up to 16 experiments; each experiment is displayed in a different color.

Stacked
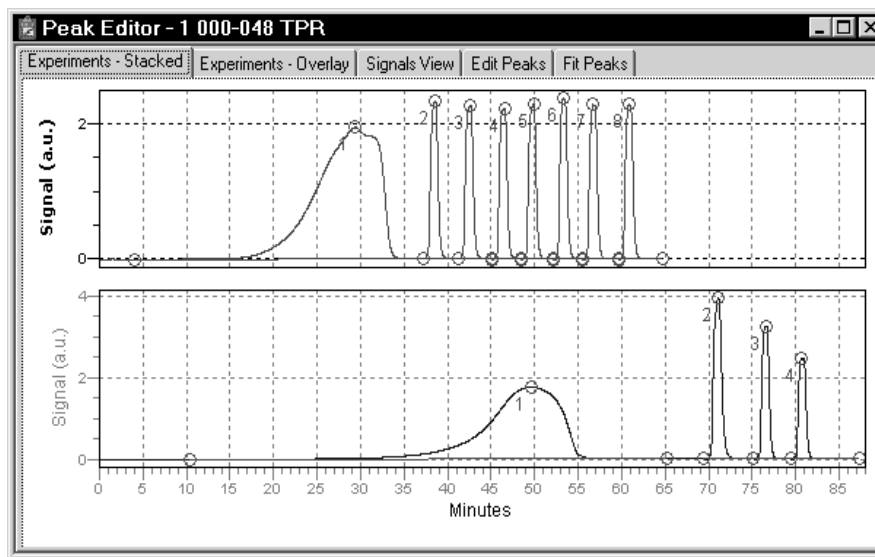
The Stacked view displays up to 16 experiments, stacked one after the other. In this view, you can select the individual experiments to obtain information. When you select an experiment, its name is displayed in the title bar of the window. Cross-hairs are also displayed to show exact vales in the selected area.

When you right-click in the graph area, the following shortcut menu is displayed:

Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 in this chapter for a list of the shortcut keys available with the peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale   Sets the axis boundaries to autoscale, presenting the signal in full scale.

Redraw   Sets axis boundaries to its original view, allowing easier editing of peaks.

> **In many cases, the AutoScale and Redraw options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.**

View   Displays a cascading menu allowing you to:

- Show or hide the peak marks

- Specify X-axis in Time or Temperature

- Copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application

Insert   Enables you to insert experiments from another sample file.

Remove   Removes the selected experiment from the Peak editor window; the data are not deleted from the sample file.

Save As   Allows you to save the experiments in the window as a new sample file. For example, if you have a file opened containing five experiments but are only interested in two of them, you can remove the three of no interest and save the file with the remaining two.

Close   Closes the Peak Editor window.

Overlay

The Overlay view displays up to 16 experiments, overlaid each on top of the other. This view does not allow you to select an experiment; you must switch to the Stacked view to select experiments.
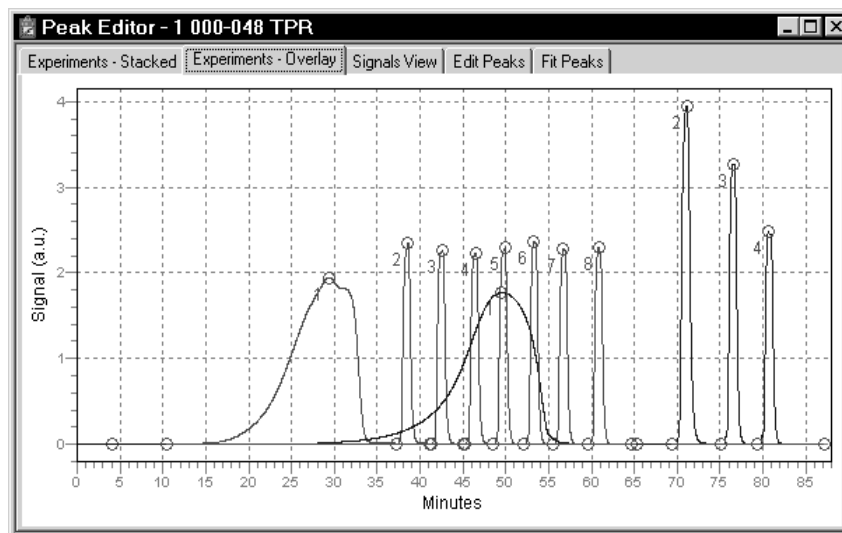
When you right-click in the graph area, the following shortcut menu is displayed:
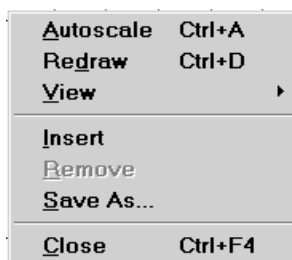
The options on this menu are identical to the ones available in the Stacked view. Remove is disabled on this menu because individual experiments cannot be selected in the Overlay view.

Signals View

The Signals view displays all signals stacked one on top of the other. This allows you to view the relationship of peaks to temperature and auxiliary signal conditions that occurred during data collection.
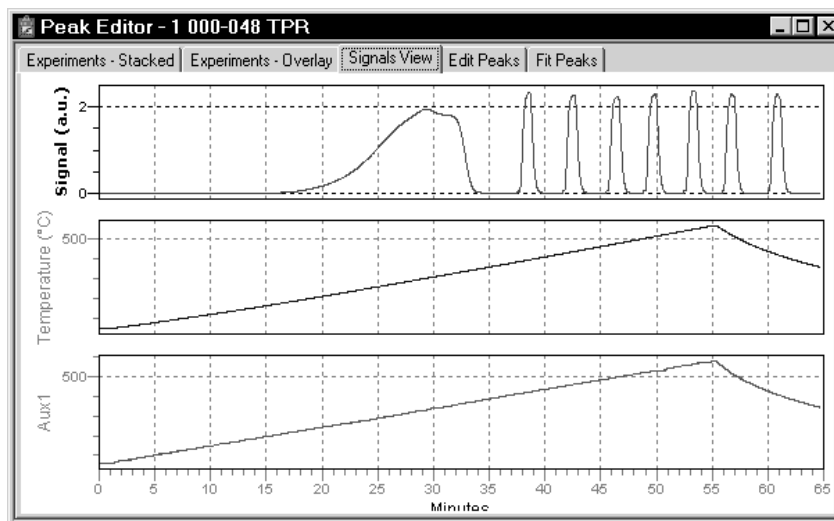
When you right-click in the graph area of the Signals view, the following shortcut menu is displayed:

Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 in this chapter for a list of the shortcut keys available with the peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale   Sets the axis boundaries to autoscale, presenting the signal in full scale.

Redraw   Sets axis boundaries to its original view, allowing easier editing of peaks

.

In many cases, the Autoscale and Redraw options will show very little difference. Both options are also used to return the view to tis original presentation after zooming a report or to remove cross-hairs.

X-Axis   Enables you to display the X-axis by Time or Temperature.

Copy   Enables you to copy the graph area to the clipboard as a metafile or as text, then paste it into another application.

Close   Closes the Peak Editor window.

Edit Peaks

Edit Peaks allows you to edit the peaks of the selected experiment; the Peak Table dialog listing peak information for all peaks of the selected experiment is displayed.
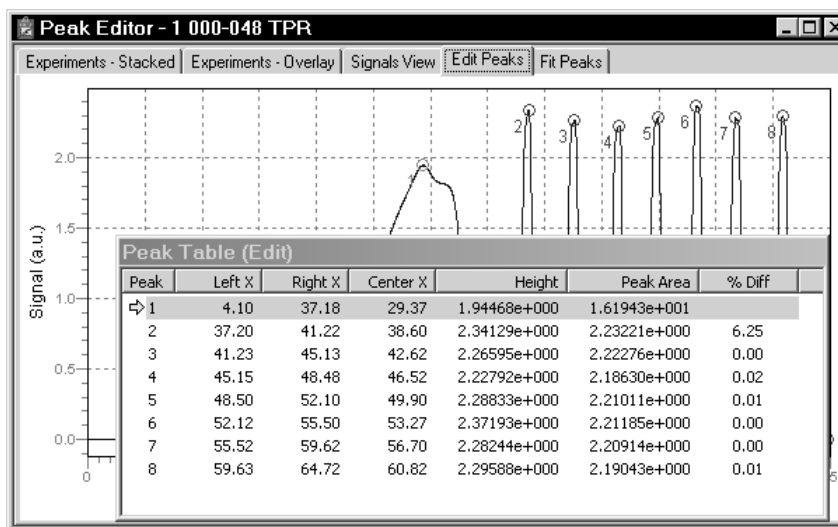
The peak table remains on the screen until you select a different view; you cannot minimize or close the peak table until you switch views. You can move the peak table over to the side, top, or bottom or your screen.

Use one of the following methods to select a peak:

• Highlight the desired peak in the Peak Table

• Access the shortcut menu and select **Next Peak** or **Previous Peak** until you navigate to the desired peak

• Use **Ctrl + N** (advance to the next peak) or **Ctrl + P** (return to the previous peak) to navigate to the desired peak

When a peak is selected, its peak marks display in a light green color; the peak marks for unselected peaks display in red.

Peaks are edited by accessing functions from the shortcut menu which is displayed when you right-click in the graph area.

<u>A</u>utoscale	Ctrl+A
Re<u>d</u>raw	Ctrl+D
<u>N</u>ext Peak	Ctrl+N
<u>P</u>revious Peak	Ctrl+P
<u>I</u>nsert New Peak	Ins
Mark <u>L</u>eft Edge	Ctrl+L
Mark <u>R</u>ight Edge	Ctrl+R
<u>F</u>ind All Peaks	
<u>D</u>elete Peak	Del
Delete All Pea<u>k</u>s	
<u>V</u>iew	▶
<u>S</u>ave	Ctrl+S
Set <u>C</u>alibration	
Peak Integration <u>O</u>ptions	
<u>C</u>lose	Ctrl+F4

Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 in this chapter for a list of the shortcut keys available with the peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale   Sets the axis boundaries to autoscale, presenting the signal in full scale.

Redraw   Sets axis boundaries to its original view, allowing easier editing of peaks.

In many cases, the AutoScale and Redraw options will show very little difference. Both options are also used to return the view to tis original presentation after zooming a report or to remove cross-hairs.

Next Peak   Advances you to the next peak.

Previous Peak	Returns you to the previous peak.
Insert New Peak	Click in the graph area where you wish to insert a new peak; the peak is inserted where the cross-hairs meet.
Mark Left Edge	A peak is automatically marked by the program; this option allows you to redefine the left edge. Simply place the mouse cursor where you wish to have the left edge marked, right-click, and select this option.
Mark Right Edge	A peak is automatically marked by the program; this option allows you to redefine the right edge. Use the same method used for marking the left edge.
Find All Peaks	This option is used to find all peaks automatically in the Peak Editor window. This is a rapid technique for integrating pulse chemisorption data. This option is most effective for narrow peaks with good baseline separation.
Delete Peak	Removes the selected peak from the peak table. Deletes all peak information for a single peak.
Delete All Peaks	Clears the Peak table of all data and removes all markings from the peaks.
View	Displays a cascading menu allowing you to:

- Show or hide the peak marks

- Specify X-axis in Time or Temperature

- Enable or disable smoothing, and specify smoothing options if desired, as well as zero and/or invert signal options

- Copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application

Save	Saves the peak table to the sample file.

Set Calibration Allows you to choose a TCD calibration that is appropriate for the selected experiment.
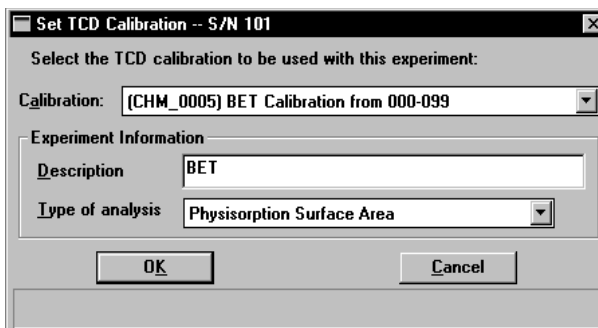
Peak Integration Options Displays the Peak Integration dialog.
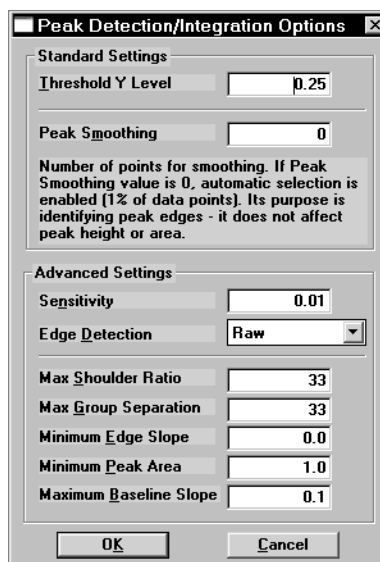
This dialog allows you to adjust the parameters enabling you to optimize the results you obtain using the **Find All Peaks** option. Refer to Appendix E for additional information on the fields in this dialog.

Fit Peaks

Fit Peaks allows you to separate overlapping peaks by fitting curves to the data, and save them as a new peak set in the Fit Peak table.
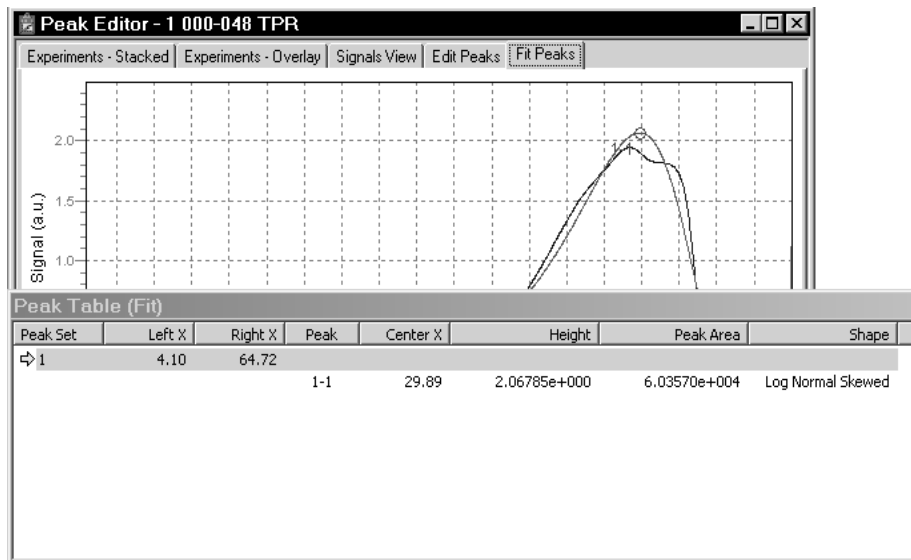
Right-click in the graph area to access the shortcut menu for creating the table.

Some functions are assigned shortcut keys allowing you to perform the action without having to access the shortcut menu. Refer to Table 7-1 in this chapter for a list of the shortcut keys available with the peak Editor. You may wish to make a copy of this page and keep it close to your keyboard.

Autoscale	Sets the axis boundaries to autoscale, presenting the signal in full scale.
Redraw	Sets axis boundaries to its original view, allowing easier editing of peaks.

> **In many cases, the AutoScale and Redraw options will show very little difference. Both options are also used to return the view to tis original presentation after zooming a report or to remove cross-hairs.**

Insert New Peak Set	Enables you to define a region of the TCD signal that contains multiple peaks. The peak fitting and optimization are constrained to this region of the TCD signal.
	To insert a new peak set: left click at the point you wish to insert the peak set, then right-click and choose this option.
Insert New Peak	Allows you to insert a new peak into the selected peak set. If you are inserting a peak outside of a peak set, a peak set is automatically created. You cannot insert a peak in a negative area.
Mark (Peak Set) Left Edge	Changes the left edge of the peak set.
Mark (Peak Set) Right Edge	Changes the right edge of the peak set.
Change Peak Shape	Allows you to choose the type of peak shape.
Log Normal Skewed	This is a 4-parameter, log-normal shape that allows for skewed peaks, and is the default peak.
Gaussian	This is the traditional symmetrical shape.
View	Displays a cascading menu enabling you to copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application
Delete Peak	Removes the selected peak from the peak set table.

Delete Peak Set	Deletes the selected Peak set from the table.
Delete All Peaks	Removes all peaks and peak-set information from the table.
Import Standard Peaks	Imports the saved peak parameters from the Peak Edit screen and uses them for the initial estimates for the peak set.
Optimize Peaks	Locates the best peak height, width, and skewness after initial peak estimates have been given.
Auto Fit	Locates the best number of peaks and parameters from the Peak Set without user estimates.
Display	Allows you to enable or disable smoothing, and specify smoothing options if desired, as well as zero and/or invert signal options
Save	Save the current peak set.
Close	Closes the Peak Editor window.

Report Examples

This section contains examples of some of the types of reports that may be generated by the ChemiSoft program. You can customize portions of some reports, such as the title and the axes of graphs, using the Report Options tab of the sample information file (Advanced format only). If reports are printed to the Screen, there are many ways in which they may be manipulated and customized.

Temperature-Programmed Desorption

TCD Signal vs. Time

TCD Signal vs. Temperature

mi micromeritics®

Micromeritics Instrument Corporation

| ChemiSoft TPx V1.00 | Unit 1 (2750) | Serial # 101 | Page 3 |

Sample: Calcium Oxalate TPD
Operator:
Submitter:
File: C:\TPX\DATA\CA-OX-1.SMP

Started: 3/23/2004 2:56:24PM   Sample Mass: 0.0404 g
Completed: Report Time: 4/14/2004 9:13:05AM

TCD Signal (a.u.) vs. Temperature

TCD Signal (a.u.) - Calcium oxalate
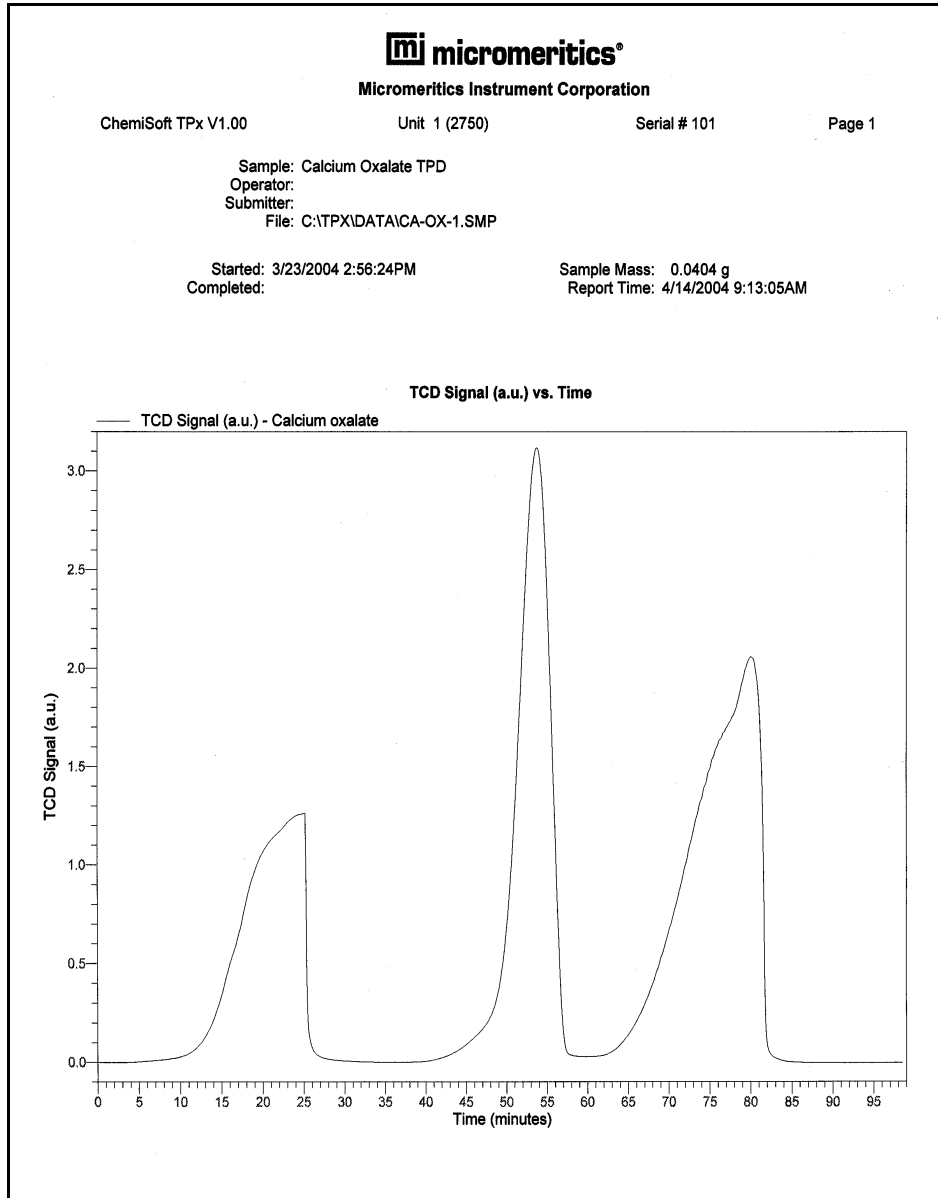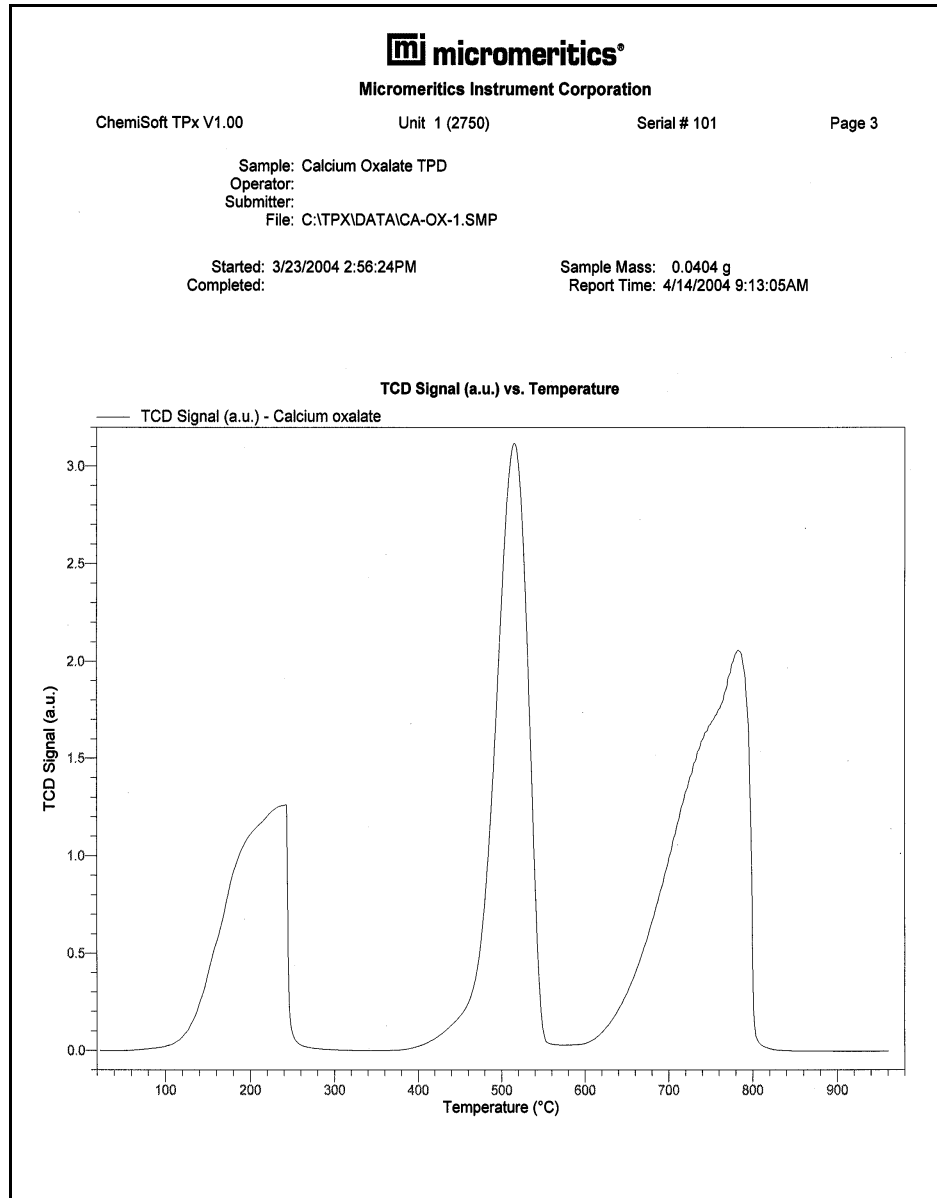
Temperature vs. Time
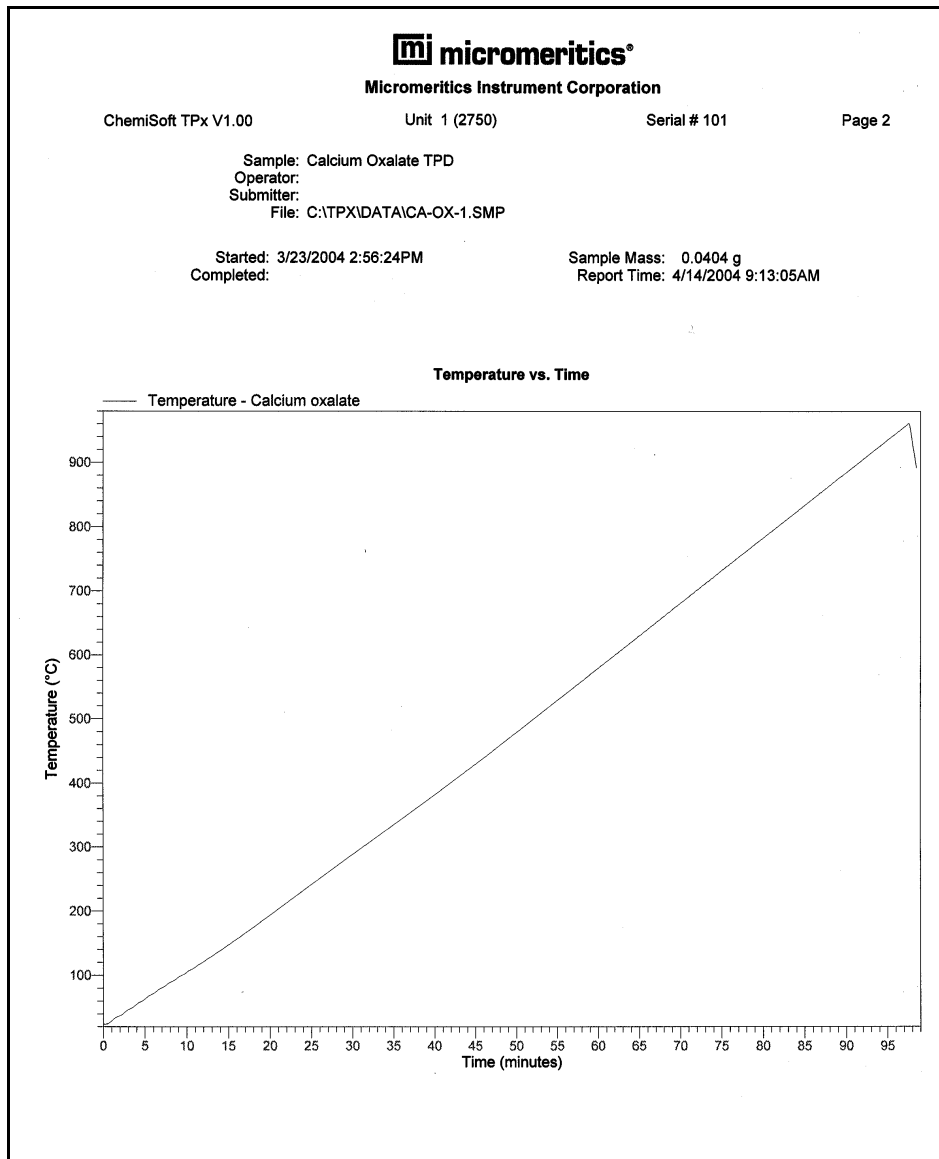
Pulse Chemisorption

Report

ⓜ micromeritics®

Micromeritics Instrument Corporation

ChemiSoft TPx V1.00   Unit 1 (2750)   Serial # 101   Page 2

Sample: 0.5% Pt on Alumina
Operator:
Submitter:
File: C:\TPX\DATA\JK-PC1.SMP

Started: 3/25/2004 8:53:41AM   Sample Mass: 0.6201 g
Completed: 3/25/2004 11:17:44AM   Report Time: 4/14/2004 9:10:03AM

Pulse Chemisorption Report

Experiment 1: 0.5 Pt

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 20.00 mL STP/min
Signal Offset: 0.00000
Signal Inverted: Yes

Peak Table

Peak Number	Temperature at Maximum (°C)	Volume Adsorbed (mL/g STP)	Cumulative Volume (mL/g STP)
1*	24.6	0.03937	0.12128
2	25.5	0.03381	0.15509
3	25.3	0.00841	0.16350
4	25.7	0.00229	0.16579
5	26.4	0.00066	0.16645
6	25.4	0.00046	0.16691
7	26.4	0.00000	0.16691

* 2 doses were completely adsorbed with a cumulative volume of 0.08191 mL/g.

Pulse Chemisorption Analysis Summary

Element	Percent of Sample Mass (%)	Atomic Weight	Stoichiometry Factor	Atomic Cross-Sectional Area (nm²)	Density (g/cm³)
platinum	0.5000	195.090	1.000	0.0800	21.450

Active Loop Volume at 25.0 °C: 0.02540 mL STP
Cumulative Volume: 0.16691 mL/g STP
Metal Dispersion: 29.0554%
Metallic Surface Area: 0.3588 m²/g sample
Metallic Surface Area: 71.7620 m²/g metal
Active Particle Diameter: 3.8979 nm

TCD Signal vs. Time

Summary

<div style="border:1px solid black; padding:1em;">

◫ micromeritics®

Micromeritics Instrument Corporation

ChemiSoft TPx V1.00   Unit 1 (2750)   Serial # 101   Page 1

Sample: 0.5% Pt on Alumina
Operator:
Submitter:
File: C:\TPX\DATA\JK-PC1.SMP

Started: 3/25/2004 8:53:41AM   Sample Mass: 0.6201 g
Completed: 3/25/2004 11:17:44AM   Report Time: 4/14/2004 9:10:03AM

Summary Report

Experiment 1: 0.5 Pt

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 20.00 mL STP/min
Signal Offset: 0.00000
Signal Inverted: Yes

Peak Number	Temperature at Maximum (°C)	Area	Peak Height
1	24.6	5.41039	0.03327
2	25.5	24.38880	0.03425
3	25.3	111.11912	0.04208
4	25.7	132.03966	0.04431
5	26.4	137.60569	0.04447
6	25.4	138.27116	0.04470
7	26.4	139.84907	0.04480

</div>

Loop Calibration

Report

micromeritics®

Micromeritics Instrument Corporation

ChemiSoft TPx V1.00   Unit 1 (2750)   Serial # 101   Page 1

Sample: 0.25 ml Loop Calibration
Operator:
Submitter:
File: C:\TPX\DATA\JK-LC-5.SMP

Started: 3/25/2004 3:36:00PM   Sample Mass: 1.0000 g
Completed: 3/25/2004 4:23:52PM   Report Time: 4/14/2004 9:14:01AM

Loop Calibration Report

Experiment 1: loop cal
Analysis Type: Loop Calibration

Syringe Injections

Peak Number	Start Time (min)	Stop Time (min)	Area
1	0.00	4.58	0.10386
2	4.59	7.64	0.10417
3	7.65	10.32	0.10297

Loop Injections

Peak Number	Start Time (min)	Stop Time (min)	Area
1	10.33	13.24	0.06643
2	13.56	16.32	0.06636
3	16.73	18.51	0.06638

Loop Temperature: 25.0 °C
Ambient Temperature: 25.0 °C
Ambient Pressure: 760 mmHg
Physical Syringe Volume: 0.4000 mL
Nominal Syringe Volume: 0.36646 mL STP
Average Syringe Injection Area: 0.10366
Average Loop Injection Area: 0.06639

Computed Loop Volume at 25.0 °C: 0.23470 mL STP
Computed Loop Volume at 0 °C: 0.25618 mL STP

TCD Signal vs. Time
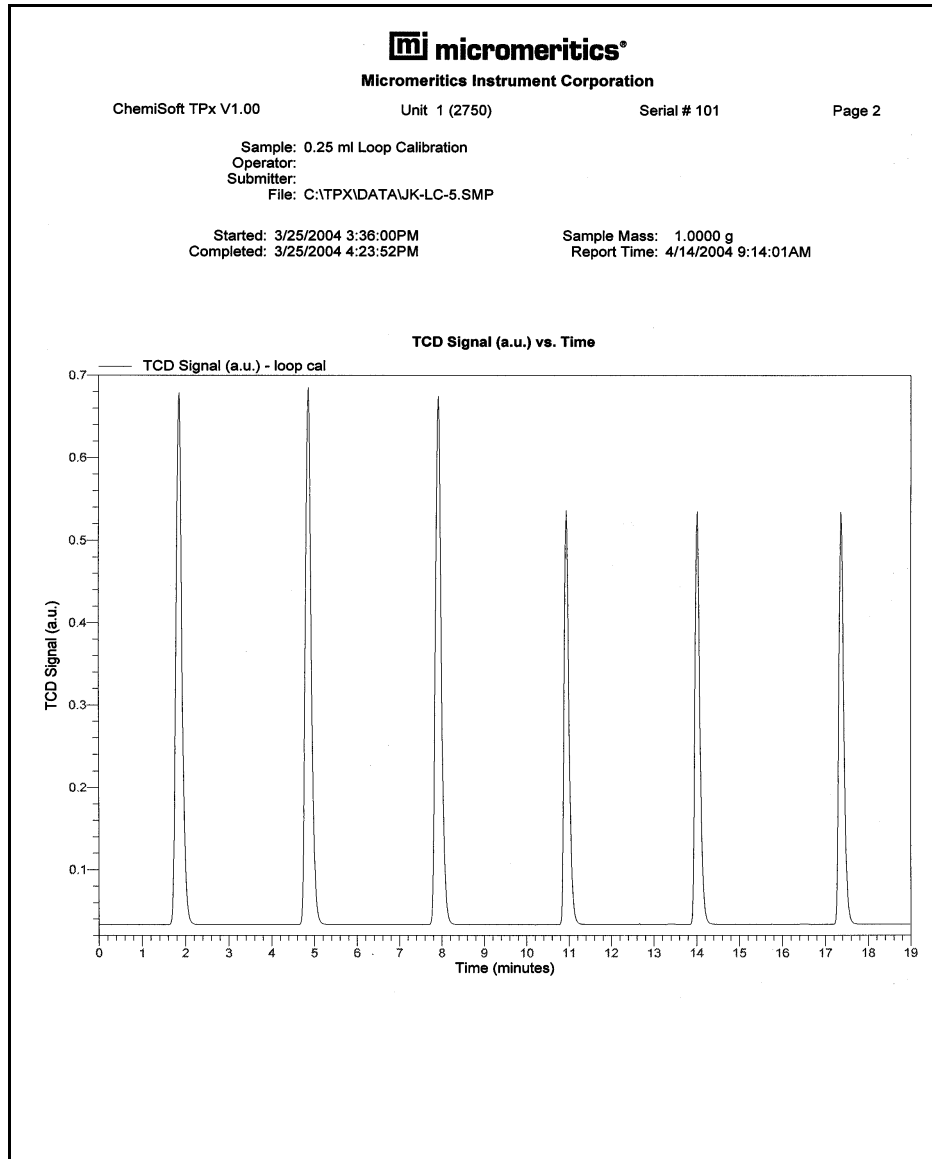
8. OPTIONS MENU

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

- Select a default for the sample file presentation
- Specify or edit program defaults
- Specify data presentation modes
- Specify printer/plotter options
- Specify a directory for predefined parameter files
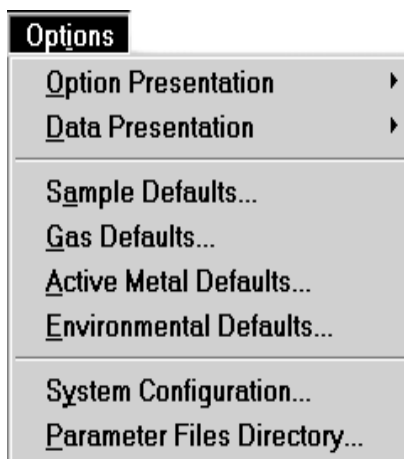
Description

Listed below are brief descriptions of the Options menu contents. Detailed descriptions are found in this chapter.

Option Presentation   Allows you to set the default for the sample information file presentation format. Choose between Basic, Advanced, or Restricted format. Page **8-3**.

Data Presentation   Allows you to specify the way in which data are presented on the screen and in printed reports. Page **8-6**.

Sample Defaults   Allows you to specify default values for sample and parameter files. Page **8-7**.

Gas Defaults   Provides a table defining characteristics of gases used for analysis, to be used in calculations. Page **8-14**.

Active Metal Defaults   Provides a table defining characteristics of metals
                               contained in samples, to be used in calculations.
                               Page **8-15**.

Environmental Defaults   Allows you to enter the current environmental
                               conditions, so that their effect on analyses can be
                               accounted for in calculations. Page **8-16**.

System Configuration   Allows you to specify the printer and/or plotter to which
                               you wish to send reports. Page **8-17**.

Parameter Files Directory Allows you to specify a directory for predefined
                               parameter files displayed in the drop-down lists of the
                               Basic sample information dialog. Page **8-18**.

Option Presentation

Sample information file dialogs may be presented in three formats: Basic, Advanced, and Restricted.

Advanced Format

Presents all four parts of the sample information file as a group of dialog boxes with index card-type tabs (the Collected data tab does not appear until after analysis). Clicking on each tab opens an associated dialog.

The sample information file for the Basic and Advanced format contains a toggle button for conveniently switching back and forth between the two formats.

Use this format when you wish to edit the parameter files and create customized Sample Information files, rather than using standard parameter files. Just click on the tab of the parameter you wish to edit, or use **Next** and **Prev** to navigate through the dialogs.

Basic Format

Presents the entire sample information file in a single dialog box.
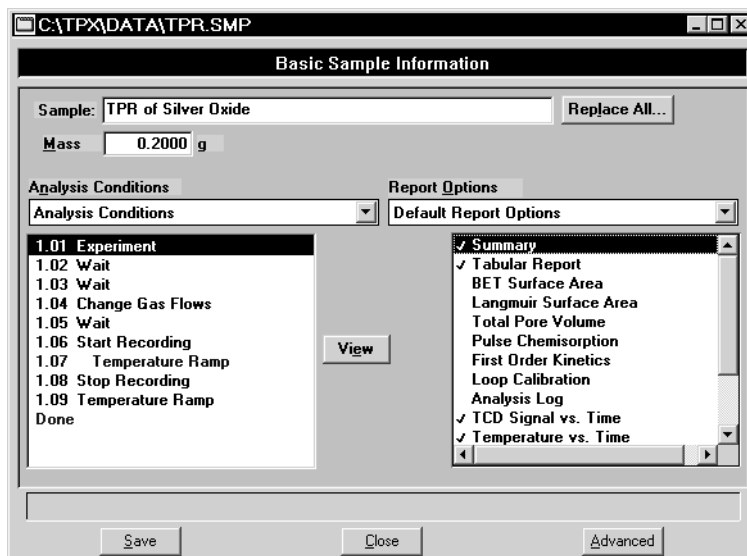
Basic format makes it easy to create sample files using previously defined parameter files in convenient drop-down lists. Each time you select a file from the list, its conditions are listed in the window directly below. If editing is required, you can easily switch to the Advanced format by clicking **Advanced**.

Restricted Format

Restricted format is identical to the Basic format, except that certain menu options are disabled. You also cannot switch to the Advanced format. This format is password-protected and typically is used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.
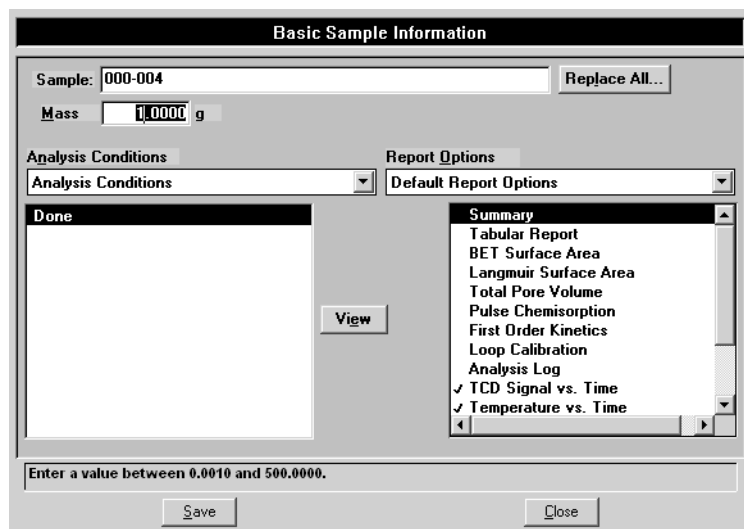
When you choose the Restricted format from the Options menu, the Option Presentation Password dialog is displayed.

You can enter any password (up to 31 characters) to enable the Restricted format. The same password must be used to exit the Restricted format. For example, if you enter "password" to enable the restricted format, then you must enter "password" to exit. If you forget the password, open the system INI file and navigate to the Private section. The password is shown immediately following "OptionPresentationPassword." Make a note of the password, exit the INI file, and enter the password where prompted. Deleting the password from the INI file will not disable the Restricted mode; you must enter the password using the Password dialog to exit the Restricted format.

Data Presentation

The options on the Data presentation drop-down menu allow you to specify how you wish tabular and graphical data to be displayed onscreen and in printed reports.
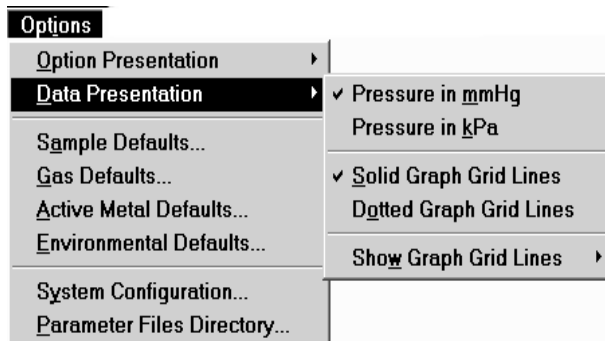
Pressure in mmHg
Pressure in kPa

Allow you to choose how data are displayed on the screen and in printed reports.

Solid graph grid lines
Dotted graph grid lines

These options allow you to choose whether you wish to have grid lines display on your report.

Show graph grid lines

Allows you to specify whether or not you wish to have grid lines display on graphs and, if so, what type. You can choose to have Horizontal, Vertical, Both, or None.

Sample Defaults

This option allows you to specify default parameters for the dialogs used to create sample information files.

Sample defaults are the values you see in the sample information editors when you create a new sample file. For efficiency, it is best to specify defaults for materials you most commonly analyze. You can always edit the values in the sample file when it is created.

The dialog used for specifying default parameters is presented in the same format you have selected as your default for sample information files. Sample defaults cannot be specified using the Restricted format. The default values that appear in the Restricted sample information presentation are the same as specified for the Basic presentation.

Basic
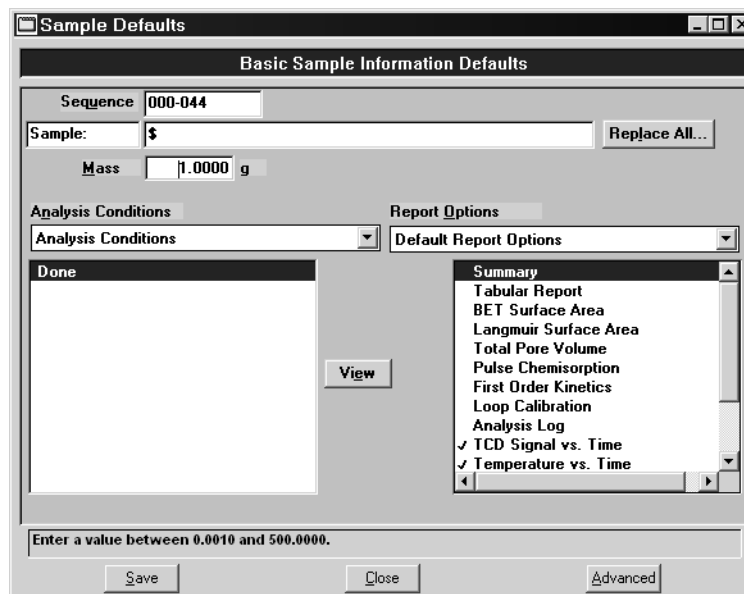
Sequence	Allows you to specify a default sequence for the file name. The number you enter is incrementally sequenced each time you create a file. It is the number that appears in the **File name** field when you select File, Open, Sample Information.

- Use numbers, letters, or other printable characters, such as dashes.

- Include at least three numbers

- Use up to eight characters.

- Do not use characters such as * or ?. Refer to your system operating manual for more information.

Sample   Allows you to enter an additional identification that
                                provides more information than the sequence number.

                                You can edit the label for the prompt using the field on
                                the left. For example, you may prefer to use *Test* or
                                Material. You can use up to 20 characters.

                                The field on the right is used to specify a format for the
                                sample identification.

                                • Use numbers, letters, or other printable characters,
                                  such as dashes.

                                • Use up to 42 characters, plus the $ symbol.

                                • Include the automatically generated file name as part
                                  of the identification by using the $ symbol where you
                                  want the sequence number to appear

                                  For example, if the sequence number is 000-001, enter
                                  the sample identification as follows:

                                      Lab #25 - $

                                  The resulting sample identification for the first
                                  Sample Information file would be:

                                      Lab #25 - 000-001

                                  and for the second one:

                                      Lab #25 - 000-002, and so on.

Replace All   Click this button to copy the values from an existing
                                sample information file into the default file you are
                                creating. A dialog is displayed so that you can choose
                                the file containing the values you wish to copy. Click
                                OK; the values are copied into the defaults automatically.
                                You may then edit the values in the default file without
                                altering the sample information file from which they
                                were copied.

Mass   Enter a default value for the sample mass. This value can
                                be edited in the sample file if needed.

Analysis Conditions   From the drop-down list, choose a default analysis conditions file. The list contains the analysis conditions files included with the software, as well as any you have created and stored in the parameter file directory (selected on the Options menu).

Each step in the analysis is listed in the Synopsis (directly below the drop-down list) and its parameters can be viewed by clicking **View**.

Report Options   From the drop-down list, choose a default report options file. The list contains the report options files included with the software, as well as any you have created.

Directly below the drop-down list is a list of the reports available. The reports preceded with a check mark are the ones selected for the file you choose. Select or deselect or desired.

Save   Saves the defaults you have selected.

Close   Closes the dialog. If there are any unsaved changes, you are prompted to save before the dialog closes.

Advanced   Changes the presentation to the Sample Defaults dialog to the Advanced presentation.

Advanced

When you select Sample Defaults while in the Advanced format, the Sample Defaults dialog is displayed in this manner.

Sequence   Allows you to specify a default sequence for the file name. The number you specify is incrementally sequenced each time you create a 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.

- Do not use characters such as * or ?. Refer to your system operating manual for more information

Replace All   Click this button to copy the values from an existing sample information file into the default file you are creating. A dialog is displayed so that you can choose the file containing the values you wish to copy. Click **OK**; the values are copied into the defaults automatically. You may then edit the values in the default file without altering the sample information file from which they were copied.

Sample   Allows you to enter an additional identification that provides more information than the sequence number.

In the field on the left, you can edit the prompt for the sample identification. For example, you may prefer to use *Test* or *Material*. You can use up to 20 characters.

In the field on the right, you can specify a format for the sample identification.

• Use numbers, letters, or other printable characters, such as dashes.

• Use up to 42 characters, plus the $ symbol.

• Include the automatically generated file name as part of the identification by using the $ symbol where you want the sequence number to appear

For example, if the sequence number is 000-001, enter the sample identification as follows:

Lab #25 - $

The resulting sample identification for the first Sample Information file would be:

Lab #25 - 000-001

and for the second one:

Lab #25 - 000-002, and so on

Operator
Submitter

These fields contain the name (or other identification) of the operator performing the analysis, and the department, customer, etc, that submitted the sample for analysis.

- In the fields on the left, you can edit the prompts if desired; you can enter up to 20 characters.

- In the fields on the right, you can specify default names or titles; you can use up to 42 characters (including 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. Refer to the example in the previous section.

- Select **Omit** to prevent the fields from appearing on the sample information dialog.

Mass

Enter a default value for the sample mass. You can edit this value in the sample file if needed.

Comments

Use this window to enter pertinent information. Any information you enter in this window is printed in the report header. You can edit the comments in the sample file if needed.

Analysis Conditions Defaults

The default settings for analysis conditions files are set using the Advanced format sample information file defaults, regardless of whether you plan to create independent analysis conditions files, or complete analysis conditions dialogs as part of the sample information file.

From the Options menu, choose **Sample Defaults** when Advanced is selected as the default format. The Advanced presentation of the Sample Defaults dialog is displayed; click the Analysis Conditions tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described in Chapter 5.

> **If you repeatedly use the same sample preparation techniques, then vary the analysis conditions, you may wish to make your default analysis conditions the preparation steps alone. Then the preparation steps will always be in place when you create a new file, ready for you to add analysis steps as needed.**

> **When the first experiment is inserted into an analysis conditions file, its initial values are set by the first experiment step in the default analysis conditions. Including a single "default" experiment step in your default sample file will ensure that new experiments are initialized properly.**

Report Options Defaults

The default settings for report options files are set using the Advanced format sample information file defaults, regardless of whether you plan to create independent report options files, or complete report options tabs as part of the sample information file.

From the Options menu, choose **Sample Defaults** when Advanced is selected as the default format. The Advanced presentation of the Defaults dialog is displayed; click the Report Options tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described in Chapter 5.

Gas Defaults

In order to characterize the gases used in an analysis, a symbol and the % of the gas that is active concentration must be specified. A table is provided containing default values; these values can be edited simply by highlighting the field and entering the desired value.

	Gas	Symbol	% Active Concentration
1	10 H2-Ar	10H2-AR	10.000
2	2 O2-He	2O2-HE	2.000
3	30 N2-He	30N2-HE	30.000
4	Ammonia	NH3	100.000
5	Argon	AR	0.000
6	Carbon Monoxide	CO	100.000
7	Helium	HE	0.000
8	Hydrogen	H2	100.000
9	Nitrogen	N2	0.000
10	Oxygen	O2	100.000

'Ctrl--Down Arrow' to append
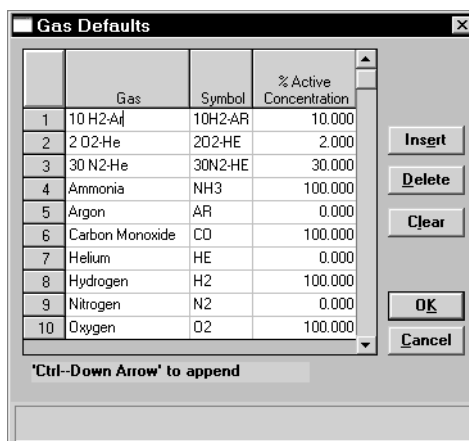
Symbol	Contains the standard symbol (mnemonic) for each gas listed.
% Active Concentration	Displays the percent of the gas mixture that is composed of the reactive gas versus an inert filler.
Insert	Enables you to insert a new gas into the table above the selected line.
Delete	Deletes the selected gas from the table.
Clear	Clears the table of all gases except one; one is required.

Active Metals Defaults

In order to characterize the active metals in a sample, certain properties of the metals must be defined. A table containing default values for the following properties is provided with the ChemiSoft program; however, you can edit the contents as desired.

The values defined in this table are the defaults used when a new entry is inserted into the metal table of the Pulse Chemisorption report options. Changing this table does not affect the tables within the report options files.

Atomic Weight	The atomic weight of the element.
Atomic Area	The atomic cross-sectional area of the element.
Density	The density of the element.
Stoichiometry Factor	A factor which expresses the ratio between the number of active metal atoms and the number of adsorbate molecules. This number varies, based on the adsorbate used. The most common value should be entered into the defaults table. Within the Pulse-Chemisorption report options, the value for a specific analysis can be set.
Insert	Enables you to insert a new element and its values into the table above the selected line.
Delete	Deletes the selected element from the table.
Clear	Clears the table of all gases except one; one is required.

Environmental Defaults

To ensure accurate loop calibration and BET surface area data, you should enter current environmental conditions every day, or whenever there is a significant change in one or more of the environmental conditions. These values are used in some calculations to account for the effect of environmental conditions on the analysis. When you select this option, the Environmental Defaults dialog opens.
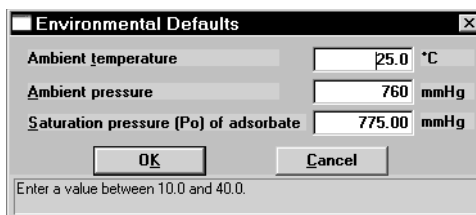
As an analysis is started, these parameters are saved in the Collected Data portion of the sample information file. (Collected Data can be viewed in the sample file in Advanced format only.)

Ambient temperature	Enter the temperature of the room in which the analyzer is located.
Ambient pressure	Enter the atmospheric pressure of the room in which the analyzer is located.
Saturation pressure (Po) of adsorbate	Enter the saturation pressure of the adsorbate gas in the room in which the analyzer is located. Disregard this field if the saturation pressure of the adsorbate gas is not pertinent to your analysis.

System Configuration

This option allows you to specify printer and plotter destinations for data output. When you select this option, the System Configuration dialog is displayed.

System Configuration

Printer name: \\MICROMERITICS\tech_writing ☑ **Default**

Plotter name: \\MICROMERITICS\tech_writing ☑ **Default**

OK   Cancel
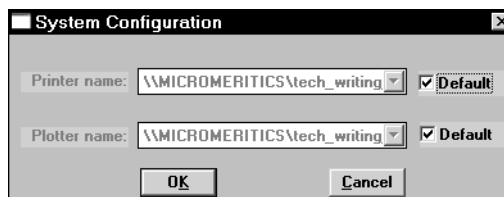
Printer name
Plotter name

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 of installed printers (plotters).

Parameter Files Directory

This option allows you to specify a location for the predefined Analysis conditions and Report options files displayed in the drop-down lists on the Basic and Restricted Sample Information dialogs.
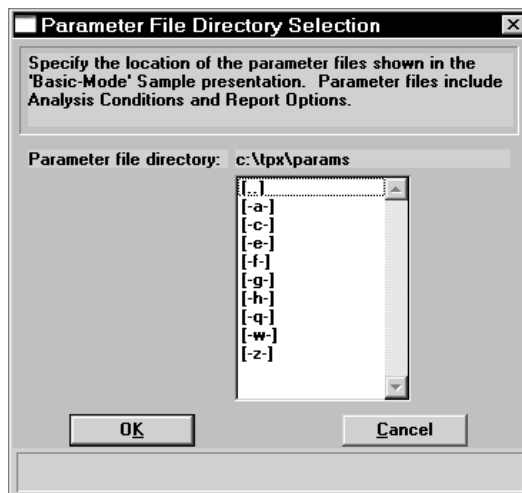
The current directory is displayed above the drives/directory window. The default directory is params and includes several parameter files supplied with the Chemisoft program. If you specify a different directory, these files will not be included in the drop-down lists unless you copy (or move) them to the new directory.

If you wish to continue using the params directory for parameter files, be sure to save any files you create to this directory; otherwise they will not be included in the drop-down lists(s).

9. ORDERING INFORMATION

Accessories for the ChemiSoft TPx option can be ordered using one of the following method:

- Call our Customer Service Department at (770) 662-3636
- Access our web site at www.micromeritics.com
- Contact your local sales representative

When ordering, please use the information provided in this chapter to place your order:

Part Number	Item and Description
003-51134-00	Fuse; 3AG Slow-Blow, 5 A, 250 V
003-51208-00	Fuse; 5 x 20 mm Slow-Blow, 2.0 A, 250 V
004-32187-00	Gloves, cotton
027-32600-00	Insulating cover, right side
027-32600-01	Insulating cover, left side
291-53904-00	Thermocouple, sample
200-60845-00	Extension cord, for sample thermocouple
291-53901-00	Thermocouple, furnace
291-63700-00	Cable, thermocouple

A. FORMS

This appendix contains the following form:

- Programmer worksheett

Copy and use this form as needed.

mi micromeritics®

Programmer Worksheet

This worksheet is used to record target setpoints, ramp rates, and dwell times when programming the controller for your analysis. (Use this as your original, making a copy as needed.)

Type of Analysis:_____

SEG.n (Segment)	rmP.r		dwEll	End
	tGt (Target Setpoint) ºC	rAtE (Ramp Rate) ºC/minute	dur (Dwell Time) minutes	

B. ERROR MESSAGES

This appendix contains a numerical listing of the error messages associated with the Chemi-Soft program.

> **The 1000-series error messages (used primarily for software testing) are not included in this appendix. These errors should not occur during normal operation. If you receive a 1000 series error message or an error message not listed in this appendix: record the error message, make backup copies of any files involved, then contact your service representative.**

2201- Cannot execute report subsystem.
2202- Cannot execute Peak Editor subsystem.
2203- Cannot execute 028 controller subsystem.

> *Cause:*   The software for the report subsystem has been corrupted and cannot be started.

> *Action:*   Reinstall the software.

2401- FATAL ERROR: (Error message)

> *Cause:*   An internal processing and/or hardware error has occurred.

> *Action:*   Contact your service representative if you continue to receive this error message.

2430- Error accessing file (file name), error code = (number)

> *Action A:*   Hard disk may be damaged.

> *Action A:*   Run diagnostics.

> *Action B:*   A software error occurred when the file was accessed.

> *Action B:*   Contact your service representative.

2431-   Error writing file (file name), error code = (number).

Cause: The hard disk does not have enough space left to perform the opera-
                  tion.

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 files (sample information, analysis conditions, or report
                  options) have been added to this directory by some function other than
                  the AutoChem program.

Action: Click **Yes** to update the directory information with data from each new
                  file. This operation may take a few minutes.Click **No** if you do not
                  want to spend the time updating the directory information.

                  This option may be feasible if a large number of files has 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.

2436-   Path specification (path name) is invalid.

Cause: You entered an invalid path name and/or extension.

Action: Enter a valid path name (including the proper extension) and press
                      Enter.

2437-   File (file name) does not exist.

Cause: You entered an invalid file name.

Action: Enter the name of an existing file or select a file name from the Files
                      list window.

2438-   Disk drive (letter): is inaccessible.

Cause: You selected a disk drive that is presently inaccessible.

Action: Ensure that the disk in the disk drive is not write-protected

2439-   Could not register file.
2440-   Subset not found.
2441-   Seek within file failed.
2442-   Had 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 operating system or Windows manual and reenter the name.

2450-   Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: You are in the process of initiating an automatic analysis (an analysis in which sample files are created using the defaults) while editing the defaults.

Action: Finish your edit session of the defaults and close the dialog. Then restart the automatic analysis.

2452-   Attempt to write MICATTR.DIR in read only mode. File (file name).

2453-   Attempt to append MICATTR.DIR in read only mode. File (file name)

Cause: The Read-Only attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file and disable the Read-Only option.

2454-   Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

2455-   Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: You selected too many files for this operation.

Action: Select only the number of files specified in the message.

2456-   Insufficient file handles available. Application cannot continue.

Cause: You have more than 50 files open at the same time.

Action: Refer to the manual for your operating system and set the limit for open files to 50 or greater.

2457-   Results cannot be displayed. More than (number) windows are currently displaying or printing results.

Cause: You have too many windows open in the application.

Action: Close some of the open windows.

2458-   An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: You 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.

2461-   No instruments are in operation. This application will unconditionally terminate.

Cause: An analyzer must be active for the application to operate. The initialization of the analyzes configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the analyzer's failure to initialize. See the instructions for that message.

Action B: Ensure that the analyzer is attached to the computer. Verify that the analyzer has the power switch in the ON position. If the application continues to fail in its attempts to initialize the analyzer, contact your service representative.

2478-   Error copying sequential data segment.

Cause: An internal processing and/or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors; for example, you may wish to use a utility such as ScanDisk.

Contact your service representative if you continue to receive this message.

2480-   File (name) cannot be analyzed. It is currently being edited.

Cause: You attempted to start an analysis using a file that is open for editing.

Action: Finish editing the file, save and close it; then restart 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 diagnostics.

2482-   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

2483-   An analysis cannot be performed on (file name). It is open for editing and contains errors.

Cause: You attempted to start an analysis using a file that contains errors and is open for editing.

Action: Finish editing the file, save and close it; then start the analysis.

2484-   The edit session for (file name) must be saved before the analysis. Save changes and proceed with the analysis?

Cause: You attempted to start an analysis using a file that contains unsaved changes and is still open.

Action: Click Yes to save the changes and proceed with the analysis. Click No to cancel the analysis and continue editing the file.

2486-   Cannot construct (name) report type. Program will terminate.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your service representative if you continue to receive this message.

2487-   Cannot 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 message.

2488-   File (file name) cannot be opened for editing. It is already in use.

Cause: The file you specified is already open for editing.

Action: Check the windows list to locate the other edit session.

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 AutoChem Setup program (located on the applications CD), select Change the number of units and enter the pertinent information.

2491-   Highlighted fields contain errors. Please correct the errors before closing.

Cause: The highlighted fields contain invalid entries. You will not be able to close the dialog box until you correct the errors.

Action: Check the entries, correct the errors, and close the dialog box.

2492-   This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493-   An entry is required for this field.

Cause: This field contains an invalid entry.

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 a value within the indicated range. The valid range is shown in the information bar across the bottom of the dialog.

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 a value between the range indicated.

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 data) could not be done.

Action: Contact your service representative if you continue to receive this 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 files 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 specified file sequence number.

Action: Select Options, Sample defaults form the main menu and enter a new sequence number.

2502-   Error writing to file (name) during print. Error code: (number).

Cause: An error occurred in the file being written to during a print operation.

Action: Ensure that there is sufficient space on the drive containing the file.

2503- Error converting file (name). Could not create DIO intermediate file.

Cause A: Insufficient space is available on the hard disk. The DIO file is placed in the directory specified by the TEMP environment variable.

Action A: Determine if there is sufficient space on the drive where the TEMP directory is located.

Cause B: An internal processing and/or hardware error has occurred.

Action B: Contact your service representative.

2504-   Cannot create output file for sample (name).

Cause: Insufficient space may be available on the hard disk.

Action: Ensure that sufficient space is available. Contact your service representative if you continue to receive this 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.

  Action: Select a different output device in the System Configuration dialog. Install the device using the Control Panel, Printers operation.

2507-   Error opening file (name) for printing. Error code: (number).

  Cause: An error occurred in the selected file for print output.

  Action: Ensure that sufficient space is available on the drive containing the file.

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, possible due to insufficient disk space.

Action B: Check the space available on the had 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 form the disk. You will require at least five
                  megabytes of free space for normal operation.

2512-   Print job (name) has been canceled.

Cause: The requested print job was canceled at your request.

Action: None required.

2513- Unable to read the calibration file (file name).

Cause: The calibration file selected for loading is not valid or cannot be read.

Action: Confirm the media containing the alleged calibration file has no prob-
                  lems.

2514- Unable to write the calibration file (file name).

Cause: An attempt to Save calibration data has failed due to possible media problems

Action A: Confirm the media containing the alleged calibration file has no problems.

Action B: Choose an alternate media to Save the calibration data.

2534- Error opening file (name) for printing. Error code: (number)

Cause: An error occurred in the selected file for print output.

Action: Ensure that sufficient space is available on the drive containing the file.

2547- The instrument is busy. Exiting the application will result in the termination of the analysis. Continue with program exit? (Yes, No)

Cause: You attempted to exit the application while an analysis is in progress.

Action: If you are not concerned with cancellation of the analysis, click **Yes** to continue the exit; otherwise select **No** and allow the analysis to finish.

4200- At least two experiments are needed for this report.

Cause: The report options for this report do not include the selection of at least two experiments.

Action: Open the sample file and choose at least two experiments for this report.

4209- New steps cannot be inserted in the synopsis at any point that precedes the step that is currently being executed.

Cause: The analysis has already progressed past the point at which you attempted to insert a step; therefore the step would not be performed.

Action: Insert the new analysis step after the step that is currently being executed.

4210- No new steps may be inserted. Analysis is complete.

Cause: The analysis for this sample file has ended. You cannot insert a step in a sample file whose analysis is complete.

Action: The analysis conditions for a completed sample cannot be changed. You can, however, create a new sample file and use the Replace button to copy the completed file. Then, add the desired step to the new sample file. A new sample can be analyzed using the new (edited) sample file.

4214-   This analysis exceeds the maximum allowed length.

Cause: The current analysis conditions script contains the maximum number of allowed steps (1000). You cannot add another step.

Action: Divide the analysis steps into two or more sample information files. Allow the sample tube to remain undisturbed on the analyzer while you repeatedly start analyses using these sample information files in sequence. When you divide the steps between sample information files, plan your divisions so that the ending conditions of one file and the initial experiment conditions of the next file facilitate the continuing analysis. The data from the various sample files can be integrated using the Peak Editor since multiple sample files can be edited at once. Note that the maximum number of experiments that can be loaded into Peak Editor at one time (regardless of the number of sample files) is 16.

4219-   At least one experiment must be selected and at least one box checked.

Cause: You attempted to export the collected data from a sample file, but not all of the required fields on this dialog have been completed correctly.

Action: Choose at least one experiment for export and select either Peak summaries, Measurements, or both.

4220-   Unexpected I/O error accessing data for an experiment in sample (name).

Cause: An unexpected disk error occurred while you were attempting to access experiment data.

Action: The hard disk drive may have failed or may be corrupted. Run diagnostics.

4221-   Cannot append an experiment to Sample file (name). Maximum size may be exceeded.

Cause: You selected Save As from the experiment editor and elected to append the loaded experiments to an existing file. Either the existing sample file already contains the maximum number of allowed experiments or the hard disk is full.

Action: Verify that the sample file contains fewer than 20 experiments and that there is at least 20 megabytes of disk space available on the target disk drive.

4222-   The Active Metal Table Options dialog box does not specify a stoichiometry factor for element (name). One (1.0) was substituted for this value in the calculations.

Cause: The Pulse Chemisorption report was selected and requires a valid stoichiometry factor for calculations. The Active Metal Table does not contain a valid stoichiometry factor for the specified element.

Action: Open the sample file in Advanced presentation. Click on the Report Options tab. Select the Pulse Chemisorption report, then click Edit. Specify a valid stoichiometry factor for the metal named in the error message. Save and close the file, then print the report.

4223-   No element in the Active Metals Table Options dialog box has a '% of Sample Weight' value greater than zero.

Cause: The Pulse Chemisorption report was selected. None of the metals in the Active Metal Table are designated as being part of this sample.

Action: Edit the Active Metal Table in the Pulse Chemisorption report options and specify a % of Sample Weight greater than zero for at least one metal.

4224-   The first peak is larger than the last. This report assumes inverse chemisorption.

Cause: This message appears on the Pulse Chemisorption Report when an inverse chemisorption experiment is reported.

Action: None required; informational only. If this experiment is not actually an inverse chemisorption experiment, it is inappropriate for this type of data reduction.

4225-   No reports were selected.

Cause: A report was requested, but there are not reports selected in the sample information file.

Action: Open the sample information file and edit the report options. Make sure that at least one report type is selected (double-click on a report to select it; a check mark appears next to selected reports).

4226-   No experiments were selected for inclusion in this report.

Cause: The report type that was selected for printing requires that you specify which experiments should be included in the report. No experiments were selected for this report.

Action: Open the sample file in advanced presentation. Click on **Report Options**, then on the selected report type. Click **Edit** and choose the experiments you wish to include in this report. If none of the experiments within the sample file are applicable to this type of report, deselect the report type from the list shown on the Report Options dialog.

4227-   The data requested on this report are not available.

Cause: The data requested on a report, such as the Auxiliary 1 signal, are not contained within any of the selected experiments.

Action: Open the sample file in advanced presentation. Click the **Report Options** tab and deselect this report type. Or, click the report type and click **Edit**. Change the options so that they request data contained within the file.

4228- This sample file does not contain an experiment which can be used in a (name) calibration.

Cause: You attempted to load a sample file that does not have valid peaks for this type of calibration into the calibration editor. A Gas Concentration calibration requires a TCD calibration experiment. A Spread Peaks calibration requires a Vapor Calibration experiment. A User-Defined Peaks calibration will accept any experiment type with valid peaks defined.

Action: Select a sample file that contains the appropriate experiment type.

4229- The calibration data for experiment (name) may already be loaded. You may delete duplicates after loading.

Cause: You attempted to load calibration data from an experiment that was already loaded.

Action: Delete any duplicate peaks from the peak table and continue with the calibration.

4230- The destination of a Grams/32 export must be a file.

Cause: You attempted to export data to the Grams/32 application using an invalid destination. Data exported to the Grams/32 application must be printed as a disk file.

Action: Repeat the export operation and choose File as the destination.

4231- An error occurred exporting experiment (number) from the sample '(file name)'.

Cause: You attempted to export data to the Grams/32 application when an error occurred. Either the disk file required by the export could not be created or the named experiment contains invalid data.

Action: Ensure that sufficient disk space (at least 20 megabytes) to contain the exported file is available. Also examine the selected experiment with the Peak Editor to ensure that it contains valid data.

4232-   At least two peaks are required to determine heat of desorption.

Cause: The heat of desorption is determined by fitting data obtained at different temperature ramp rates. At least two data points must be present for the fit to be performed.

Action: Repeat the analysis with at least two experiments that have different ramp rates. Refer to Appendix J in the operator's manual for a tutorial on performing a TPD analysis.

4233-   Default mass spectrometer 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.

4234-   File (name) is not a mass spectrometer data file.

Cause: You selected a file that does not contain appropriate mass spectrometer data.

Action: Choose a file that contains data collected by the Pfeiffer Vacuum (formerly Balzers) mass spectrometer which uses the QuadStar software.

4235-   The mass spectrometer file (file name) cannot be opened for reading.

Cause: A file-access error occurred when you attempted to access the mass spectrometer file.

Action: Ensure that you have selected a valid file name, and that you have the required permissions to read mass spectrometer files.

4236-   The calibration data file (name) was not created with this instrument.

Cause: You attempted to load a calibration file containing data collected on an instrument different from the current one.

Action: Choose a sample file containing calibration data that were collected on the current instrument.

4237-   Peak processing cannot be performed. The mass spectrometer data cannot be imported.

Cause: The mass spectrometer data did not include identifiable peaks.

Action: Review the mass spectrometer data; select a different mass spectrometer data file.

4238-   Peak Editor cannot be started.

Cause: You attempted to open the Peak Editor, but one of its components is missing.

Action: Reinstall the software.

4239-   A calibration from instrument S/N (number) is required to calibrate experiment (name).

Cause: You attempted to associate a calibration file with a sample file, but were unable to complete the operation. In order to associate a calibration file with a sample file, the files' analyses must have been performed on the same instrument. The instrument must be connected to the computer for its calibration files to be available.

Action: Ensure that the instrument with the designated serial number is connected to the computer and is active.

4240- Error writing calibration information. The calibration of experiment (name) could not be changed.

Cause: The calibration information for an experiment could not be changed because of an error writing to the disk file.

Action: The hard disk drive may have failed or it may be corrupt. Run diagnostics. Check that no other application has write access to the sample file.

4241- TCD Calibration is required for the calculations of this report.

Cause: This report requires that TCD Calibration be applied to the sample file.

Action: BET, Langmuir, and Total Pore Volume reports require inclusion of a TCD Calibration. Use the Peak Editor command on the Reports menu to set a TCD Calibration for the file (done on the Edit Peaks view).

4243- Ambient Temperature should be > 10º C.

Cause: The value for the ambient temperature is out of range; the sample file may be corrupt.

Action: Contact your Micromeritics service representative.

4244- Average Syringe Area should be > 0.

Cause: There is no peak data for the Loop calibration.

Action: Using the Peak Editor, edit the peaks; insert new peaks if possible.

4245- The peak table contains [nn] peaks. Exactly 6 peaks are required for loop calibration.

Cause: You do not have six peaks in the loop calibration; six are required.

Action: Using the Peak Editor, insert new peaks until there is a total of six.

4375-   Cannot insert peak with negative height.

Cause: You attempted to insert a peak below the peak set's baseline.

Action: Insert the new peak above the peak set's baseline and/or adjust the baseline.

4376-   Auto Fit cannot be executed unless a valid peak-set is selected.

Cause A: You attempted to auto-fit a peak set, but there is no peak set current active.

Action A: Click on the desired peak or peak set in the peak-fit table to select a peak set.

Cause B: You attempted to auto-fit, but there are no peak sets in the experiment.

Action B: Insert a new peak set into the experiment. Then choose Auto Fit to find all peaks in the set.

4377-   This operation cannot be performed on a negative peak set.

Cause A: You attempted to insert a peak in a negative peak set.

Cause B: You attempted to move the boundaries of a negative peak set.

Cause C: You attempted to change the shape of a peak found within a negative peak set.

Cause D: You attempted to optimize the peaks of a negative peak set.

Cause E: You attempted to auto-fit the peaks of a negative peak set.

Action: Invert the graph to make the peak set positive, then repeat the operation.

6002-   File cannot be opened for writing. It is already in use.

   Cause: You attempted to access a file that is in use by some other part of the
                     application.

   Action: Close the window or application that is currently using the desired file
                     and try again.

6003-   Time-out on wait. The wait on a single event or wait on multiple events timed out.

6004-   Requested device is not online or it is missing. The device cannot be detected.

6005-   A buffer overrun occurred. The acquisition transfer buffer located in PC memory has overrun.

6006-   Invalid scan frequency specified. Could not program the unit to scan or update at requested rate.

6007-   The driver cannot properly communicate and/or initialize the device.

6008-   Invalid digital I/O device type.

6009-   Could not allocate the needed memory. Memory could not be allocated by the driver.

6010-   Invalid device handle.

6011-   Communications failure. System will shut down.

   Cause: An internal processing error has occurred.

   Action: A   Exit the ChemiSoft program and disconnect the USB cable from the
                     controller. Then reconnect the cable, and restart the application. If the
                     problem persists, try Action B.

   Action: B   Restart the computer, then restart the application. If the problem per-
                     sists, contact your Micromeritics service representative.

6012-   The sample has an invalid status and cannot be used for this analysis.

   Cause: Only sample files with a status of **No analysis** can be used for analysis.
                     The sample file you have selected has a status other than **No analysis**.

   Action: Select a sample with a status of **No analysis**.

6227-   This instrument has no TCD calibration file to edit.

Cause: You are attempting to edit a TCD calibration file. No TCD calibration files currently exist for this instrument.

Action: Select **TCD, New** from the Unit menu to create a TCD calibration file.

C. CALCULATIONS

Volume at STP

Volume at standard reduction of gas data to Standard Temperature Pressure (STP) conditions (273.13K and 760 mmHg).

$$V_{STP} = V_a\left(\frac{273.15}{273.15 + T_a}\right) \times \left(\frac{P_a}{760\ mmHg}\right)$$

V_{STP} = Volume at STP, mL
V_a = Volume at ambient temperature and pressure, mL
T_a = Ambient temperature, °C
P_a = Ambient pressure, mmHg

Injection Loop Calibration

The Injection Loop calibration applies to the Pulse ChemiSorb 2750 only. These equations are used during calibration of a dose loop

$$V_{S_{STP}} = V_S\left(\frac{273.15}{273.15 + T_a}\right) \times \left(\frac{P_a}{760\ mmHg}\right) \times \frac{C_A}{100} \tag{1}$$

$$V_1 = \frac{A_1 \times V_{S_{STP}}}{A_S} \tag{2}$$

$V_{S_{STP}}$ = Volume of syringe at STP, mL
V_S = Physical volume injected from syringe, mL
T_a = Ambient temperature, °C
P_a = Ambient pressure, mmHg
V_l = Effective loop volume (at loop temperature), mL
A_l = Average peak area of loop injections
A_S = Average peak area of syringe injections
C_A = % of active gas, typically 100%

Injection Loop Volume – Temperature Change

This equation is used to correct the apparent loop volume when the temperature of the loop heat zone is changed.

$$V_n = V_a \left(\frac{T_o + 273.15}{T_n + 273.15} \right)$$

V_n	=	New Effective Loop Volume, mL
V_o	=	Old Loop Volume, mL
T_o	=	Old Loop Temperature, °C
T_n	=	New Loop Temperature, °C

Calibration Error (Goodness of Fit)

This equation is a means to determine whether the calibration curve is acceptable.

$$E_\% = \left[\sum_{i=0}^{N} \frac{(Q_{m_i} - Q_{c_i})}{N(-1)} \right]^{\frac{1}{2}}$$

$E_\%$	=	Percent error
Q_m	=	Quantity measured
Q_c	=	Quantity calculated
N	=	Number of measurements

Percent Dispersion

This equation is used in a Pulse Chemisorption Analysis to calculate the percent dispersion. Some understanding of the chemistry is required to select the proper stoichiometric factor and gram molecular weight.

$$\%\mathrm{D} \; = \; 100\left(\frac{V_S \times SF_{calc}}{F_N \times SM \times 22414}\right) GMW_{calc}$$

%D	=	Percent dispersion
V_s	=	Volume sorbed (mL at STP)
SF_{calc}	=	Calculated stoichiometry factor
SM	=	Sample mass (g)
F_N	=	Fraction of sample mass of metal N
GMW_{calc}	=	Gram Molecular Weight (g/g-mole)

BET Surface Area

Single-Point

These equations are used to determine the BET surface area. The default value for surface area of the N_2 molecule is $16.2 \times 10^{-20} m^2$.

$$V_{STP} = \frac{V_a}{SM} \times \left(\frac{273.15K}{273.15K + T_a} \right) \times \left(\frac{P_a}{760 \; mmHg} \right) \tag{1}$$

$$V_m = V_{STP} \left(1 - \frac{P}{Po} \right) \tag{2}$$

$$SA = \frac{V_m}{22414} \times (6.023 \times 10^{23}) \times A_{N_2} \tag{3}$$

V_a	=	Volume sorbed at ambient conditions, mL
V_{STP}	=	Volume sorbed at STP, mL/g of sample
V_m	=	Volume of the monolayer, mL
T_a	=	Ambient temperature, °C
P_a	=	Ambient pressure, mmHg
P	=	Absolute pressure of N_2 (estimated by $\%N_2/100$ x P_a), mmHg
SA	=	Surface area, m^2/g
A_{N_2}	=	Surface area of the N_2 molecule, m^2/molecule
SM	=	Sample mass (g)
P_0	=	Saturation pressure of N_2 (estimated by $P_a + 15$ mmHg), mmHg
		(The default value for P_0 is used in data reduction unless youcalculate a new value and enter it on the Collected Data tab of the sample information file.)

Volume Sorbed

This equation is for loop or syringe injections to calculate volume removed from the injections by the sample.

$$V_S = V_I \times \Sigma \left(1 - \frac{A_i}{A_p} \right)$$

V_s	=	Volume sorbed, mL
I	=	Number of injections
V_I	=	Volume per injection, mL
A_i	=	Injection peak area from Volume V_1
A_p	=	Peak area at saturation

Peak Area Volume

This is the basic data reduction equation. It converts the area under a peak to mL of gas.

$$V_{pa} = A_p \times F_c$$

V_{pa}	=	Peak area volume, mL
A_p	=	Peak area
F_c	=	Calibration factor

Stoichiometry Factor

The calculated stoichiometry factor is a weighted average. It is dependent on both the individual stoichiometry factor and number of moles of each active metal.

$$SF_{calc} = \left[\left(\frac{F_1 \times SF_1}{W_{atomic1}} \right) + \left(\frac{F_2 \times SF_2}{W_{atomic2}} \right) + \dots + \left(\frac{F_N \times SF_n}{W_{atomicN}} \right) \right] GMW_{calc}$$

SF_{calc}	=	Calculated stoichiometry factor
SF_N	=	Stoichiometry factor for metal N
F_1	=	Fraction of sample mass for metal N
$W_{atomic1}$	=	Gram molecular weight of first metal (g/g-mole)
GMW_{calc}	=	Gram molecular weight (g/g-mole)

Gram Molecular Weight

The calculated Gram Molecular Weight is a weighted average of the number of moles of each active metal.

$$GMW_{calc} = \cfrac{1}{\left(\cfrac{F_1}{W_{atomic1}}\right) + \left(\cfrac{F_2}{W_{atomic2}}\right) + \ldots + \left(\cfrac{F_N}{W_{atomicN}}\right)}$$

GMW_{calc}	=	Gram molecular weight (g/g-mole)
F_N	=	Fraction of sample mass for metal N
$W_{atomicN}$	=	Gram Molecular Weight of first metal (g/g-mole)

Cross-Sectional Area

$$SA_{calc} = \left(\frac{F_1 \times SA_1}{W_{atomic1}}\right) + \left(\frac{F_2 \times SA_2}{W_{atomic2}}\right) + \ldots + \left(\frac{F_N \times SA_n}{W_{atomicN}}\right) \times GMW_{calc}$$

SA_{calc}	=	Calculated cross-sectional area
F_N	=	Fraction of sample mass for metal N
SA_N	=	Cross-sectional area for metal N
$W_{atomicN}$	=	Gram molecular weight of first metal (g/g-mole)

Metal Density

$$D_{calc} = \left(\frac{F_1 \times D_1}{W_{atomic1}}\right) + \left(\frac{F_2 \times D_2}{W_{atomic2}}\right) + \ldots + \left(\frac{F_N \times D_n}{W_{atomicN}}\right) \times GMW_{calc}$$

D_{calc}	=	Calculated density
F_N	=	Fraction of sample mass for metal N
D_N	=	Density for metal N
$W_{atomicN}$	=	Gram Molecular Weight of first metal (g/g-mole)

Metallic Surface Area

This equation calculates the effective metallic surface area per gram of sample.

$$SA_{metallic} = \left(\frac{V_S}{SM \times 22414}\right) \times (SF_{calc}) \times (6.123 \times 10^{23}) \times (SA_{calc})$$

$SA_{metallic}$	=	Metallic surface area (m²/g of metal)
V_s	=	Volume sorbed (mL at STP)
SF_{calc}	=	Calculated stoichiometry factor
SA_{calc}	=	Calculated specific surface area
SM	=	Sample mass (g)

Active Particle Size

This equation calculates the active particle size for a hemispherical particle.

$$APS = \frac{6}{D_{calc} \times \left(\dfrac{M_s}{GMW_{calc}}\right) \times (6.023 \times 10^{23}) \times (SA_{calc})}$$

APS	=	Active particle size
D_{calc}	=	Calculated metal density (g/mL)
M_s	=	Sample mass, g
GMW_{calc}	=	Gram molecular weight (g/g-mole)
SA_{calc}	=	Calculated specific surface area (per gram of metal)

First Order Kinetics

This series of steps calculates the heat of desorption. At least two experiments must be run — typically three experiments are run. The ramp rate must be different for each run. The data are plotted and the slope determined.

The general equation is:

$$\ln\left(\frac{\beta}{T_p^{\ 2}}\right) = -\frac{E_a}{RT_p} + \ln\left(\frac{E_d Vm}{Rk_o}\right)$$

Determine the slope s by plotting $2 \ln T_p$ - $\ln \beta$ vs. $\frac{1}{T_p}$ then determine E_d using:

$$E_a = s \times R$$

β =   Sample ramp rate, degrees/min
E_a =   Activation energy, K joules/(mole K)
R =   Gas constant
T_p =   Temperature at peak max
s =   Slope
Vm   =   Monolayer volume
k_o =   Arrhenius pre-exponential constant

D. FORMAT OF EXPORTED DATA

Data collected using the ChemiSoft program can be exported to a file for use in other applications. Data may be exported as an ASCII or Grams/32 (a peak-editing program used with early versions of Micromeritics' AutoChem software) format. Refer to Chapter 4 for instructions on exporting data.

ASCII Format

Data are exported in the following format when **ASCII** is chosen as the export type:

Header

<sample id>
<operator>
<submitter>
<weight>
<loop volume>
<room temperature>
<ambient pressure>
<N2Po>

Remaining Data

The remaining data are displayed based on your choice of **Peak Summaries** or **Measurements** in report options.

Peak Summaries

The peaks for each selected experiment in the file are summarized. Data are exported as follows:

<experiment number>, <start time>, <end time>
<start temp>, <end temp>,
<peak height>, <peak area>

Measurements

The data points for each checked experiment in the file are reported. Data are exported as follows:

> \<experiment number\>, \<start time\>, \<end time\>
> \<temperature\>, \<tcd signal,\> \<event flag\>, \<aux1\>, \<aux2\>

The tcd signal may be either calibrated or not calibrated based upon the selections made when the export operation was performed.

The event flag is either 1 or 0. The state of the event flag switches when a pulse chemisorption dose is started.

Grams/32

When Gr:ams/32 is chosen as the export type, five files are created for each experiment selected for export. Grams/32 is a peak editing program used in early versions of Micromeritics' AutoChem analyzer. The data in these files can be read only by the Grams/32 software. Table D-1 lists the names and contents of these files.

Table D-1. Grams/32 Export File Names

File Name	Contents
xxxnn.CGM	Calibrated TCD signal vs. time (same as xxxSGnn.SPC when no calibration is specified)
xxxSGnn.SPC	Uncalibrated TCD signal vs. time
xxxTMnn.SPC	Sample temperature vs. time
xxxT1nn.SPC	Auxiliary signal one vs. time
xxxT2nn.SPC	Auxiliary signal two vs. time

Where **xxx** is the first three characters of the input sample file name and nn is the number of the experiment within the sample.

For example, the second experiment of the sample file ABC.SMP would be exported into the following Grams/32 files:
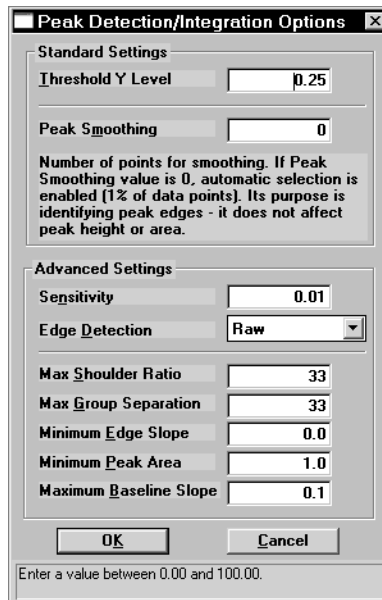
> ABC02.CGM
> ABCSG02.SPC
> ABCTM02.SPC
> ABCT102.SPC
> ABCT202.SPC

E. PEAK DETECTION/INTEGRATION OPTIONS

Peak detection parameters can be customized using the Peak Detection/Integration
Options dialog. If you customize this dialog when you create the sample file, you can
control peak detection during analysis. You can also control peak detection after analysis
by customizing this dialog in Peak Editor. The defaults for the parameters on this dialog
are usually acceptable, but they may be changed if you wish.

- To open this dialog while you are creating the sample file:
  With the sample file open in Advanced presentation, click on the Analysis
  Conditions tab. Click **Insert**, then select **Experiment**. Click **Peaks**.

- To open the dialog while you are in Peak Editor:
  Select the **Edit Peaks** tab. Then right-click in the graph area and choose **Peak
  Integration Options**.

The TCD detects and records all deviations from baseline, but only those which satisfy the
criteria established in this dialog are reported as peaks.

> **Peak detection is accomplished through a combination of noise, height, and area
> thresholds.**

Threshold Y Level

Sets the minimum height for peaks to be identified and included in the peak table. This value is expressed in terms of the trace's Y-axis units. Use a value of 0 (zero) to include all peaks.

Smoothing

This feature allows the software to average the points before using them, so that noise spikes are ignored. Specify the number of points you wish the software to average into a single value during the peak picking process.

The smoothing parameter can be turned off by setting the value at 1 or 0 (zero). A setting of 1 disables smoothing, and the peak edges are interpolated to the best X-axis value. A setting of 0 also disables smoothing, but the peak edges (the points where the peak begins and ends) are not interpolated. Instead, the nearest data point is used as the peak edge.

Sensitivity

Sets the noise rejection level for identifying the peaks in a trace. Use a value from -100 to 100%. For example, a sensitivity level of 5% means that 5% of all local maxima in the trace are greater than the noise and are, therefore to be considered as peaks. A value of 100% identifies all local maxima (or minima for traces with transmission Y units) as peaks. One possible exception to note is the combination of other rejection parameters (such as Minimum Peak Area and Threshold Y Level) which can reject peaks even when the sensitivity is set to 100%.

The sensitivity can also be set to negative values to define a specific noise level (in Y units) for peak rejection. For example, a sensitivity setting of -2.5% sets the noise rejection to 2.5 V. This means that maxima with an amplitude of 2.5 V or less will be considered as baseline noise instead of as peaks. (As opposed to the Threshold Y Level rejection parameter which eliminates refined peaks by using their height above the baseline.)

A setting of 0 (zero) automatically sets a default noise level for the trace.

Edge Detection

Determines the peak edge-detection algorithm used by the software.

These algorithms can be classified into two general categories: noise-level-based detection and slope-based detection.

Algorithm	Description
Noise-Based Detection	
Raw	The "Raw" algorithm performs noise-level-based edge detection without further edge refinement. The peak edges are detected at the local minimum on either side of the peak. This is the fastest method of peak edge detection.
Tangent	The Tangent algorithm performs noise-level-based edge detection with "tangent baseline" peak edge refinement. The raw edges are first detected at the local minimum on either side of the peak. These edges are then refined by finding a line that is tangent to the trace on either side of the identified peak. The peak edges are defined where the tangent line touches the trace. This tangent line then becomes the baseline used for calculating the peak height and area.
Slope-Based Detection	
Slope	The Slope algorithm performs slope-based edge detection without further edge refinement. The peak edges are detected on either side of a peak where the absolute value of the slope exceeds a minimum slope value. his minimum detectable slope is defined by another parameter, Minimum Edge Slope. Normally, a Smoothing setting greater than 1 must also be used.
Normal	The Normal algorithm performs slope-based edge detection with "tangent baseline" peak edge refinement. The raw edges are first detected on either side of a peak where the absolute value of the slope exceeds a minimum slope value. (This minimum detectable slope is defined by the Minimum Edge Slope parameter described later in this chapter.) These edges are then refined by finding a line that is tangent to the trace on either side of the identified peak. The peak edges are defined where the tangent line touches the trace. This tangent line then becomes the baseline used for calculating the peak height and area.

Max Shoulder Ratio

There can also be "shoulder peaks" (also called combination peaks) within a group. (For a discussion of peak groups, see the description of Max Group Separation below.) Shoulders are usually small peaks that are overlapped on the front or the tail of a larger peak. These peaks can also be called "leaders" and "followers," respectively. As with baseline groups, the areas of these peaks can be calculated incorrectly. If the larger "parent" peak has a long tail with a much smaller peak riding on it, most of the area under the trace belongs to the parent peak. However, if the area of these peaks was determined using baseline grouping, the smaller peak would be calculated by using vertical drop lines at the edges. This would give the parent peak too little area, and the rider peak too much.

The software can detect these shoulder peaks. The areas of shoulder peaks are calculated by drawing a skimmed baseline from the leading edge to the trailing edge. Either an exponential or a straight skim line can be used. The skim type is specified by a secondary method parameter and the default is exponential skimming. The remaining area between the shoulder peak baseline and the group baseline is considered to be part of the parent peak.

The Max Shoulder Ratio parameter is used to specify whether the peaks that are overlapped in the front or the tail of much larger peaks should be identified as shoulder peaks. To use shoulder peak detection, use a non-zero value for the Max Shoulder Ratio parameter. After a baseline group has been identified, the software looks for peaks within the group that satisfy the following shoulder peak criteria:

Shoulders must have a significantly higher Y value at one edge than the other. More importantly, the height of the shoulder above the common value must be much smaller than the height of the "parent" (larger) peak above the same valley. It must be smaller by the Max Shoulder Ratio setting. For example, a setting of 33 implies that shoulders must be smaller than 33% of their parents in terms of height above the common valley. The areas for shoulder peaks are calculated by drawing a skimmed baseline from the left edge to the right edge of the peak. The remaining area between the shoulder peak baseline and the group common baseline is considered to be part of the parent peak.

Shoulder peaks can only be calculated within a group of peaks. (See the description of peak groups in the section below on Max Group Separation.) If the Max Group Separation parameter is set to 0 (no groups), a Max Shoulder Ratio parameter value is not used. Use a value of zero to specify no shoulder peak detection.

A value of 33% works well with most data. Use a setting of zero to treat shoulder peaks with a perpendicular drop to the common group baseline instead of a skimmed baseline.

If the software detects unwanted baseline noise peaks, try increasing the Sensitivity setting. Conversely, if some peaks are not detected, decrease the value.

Max Group Separation

The software normally calculates peak areas by drawing a "valley-to-valley" baseline from the leading edge to the trailing edge of every identified peak. However, in many traces, the valleys between peaks do not always drop back to the original baseline. If a valley-to-valley baseline is used for this type of peak, the calculated area does not accurately reflect the true area under the peak.

The software provides a parameter that allows the calculation of "Baseline Groups." A group of peaks is defined by a common baseline that extends from the leading edge of the first peak in the group to the trailing edge of the last. The areas of grouped peaks are calculated by dropping vertical lines from the peak edges down to the group baseline. (Note there may also be "Report Groups" defined by the method and assigned group letters from A through Z. Unlike these Baseline Groups, the Report Groups need not be next to one another. The two types of groups are not related.)

The Max Group Separation parameter is used to determine which peaks in a trace have a common baseline. When using peak grouping, the software compares the width (actually double the largest half width) of every identified peak in a trace to the width of the following peak. The Max Group Separation parameter specifies a percentage of the smallest of these two widths in X units. If the edges of two adjacent peaks differ by less than this value, the two peaks constitute a group and are given a common baseline. For example, if two adjacent peaks in the trace have largest half widths of 1 and 1.5 respectively, and the Max Group Separation parameter is set at 20%, then a difference of less than 0.4 X units between the adjacent edges of these peaks would make them a group with a common baseline. If the same two peaks have adjacent edges that are greater than 0.4 X units apart, they do not define a group, and each peak has its own separate baseline.

The areas for grouped peaks are calculated by drawing imaginary vertical lines from the peak edges to the common baseline. Any peaks that share common edges are automatically considered a group and are given a common baseline for any Max Group Separation setting greater than 0. To specify no peak grouping (each identified peak has its own baseline), use a setting of zero. A value of 33% for this parameter works well with most data. Use a setting of zero to force all baselines to be drawn from peak valley to valley.

Minimum Edge Slope

This parameter is used by slope-based peak edge detection algorithms to differentiate between peaks and baseline. It specifies the minimum slope threshold in terms of your trace's Y-axis units divided by the X-axis units (e.g., millivolt/minutes). Sections of the trace with slopes exceeding this value are considered peaks, while all other areas are considered baseline.

To have the software automatically determine the best value for the "Minimum Edge Slope" parameter, use a setting of 0.

If the software incorrectly locates the trailing edges of your peaks, as evident by improperly drawn baselines, decrease the setting by typing in a smaller value. If the trace contains a significant amount of baseline noise, you may also need to adjust the Smoothing parameter.

Minimum Peak Area

This parameter sets the minimum area required for refined, processed peaks to be recognized, identified, and included in the peak table. Any peak with a calculated peak area smaller than the current setting is not detected. Values for this parameter are expressed in terms of the trace X-axis units multiplied by the Y-axis units (e.g., millivolt-minutes).

Setting the Sensitivity parameter to large values (greater than 20%) can cause noise "spikes" (or "dips") on the sides of major peaks to be identified as peaks themselves. If the areas of the peaks are smaller than the Minimum Peak Area parameter, major peaks in the trace may not be identified at all. Exercise caution when using high Sensitivity settings with the Minimum Peak Area parameter.

If peaks are not detected by the software, lower the setting with a smaller value. You must adjust this parameter in conjunction with the Peak Sensitivity and/or Threshold Y parameters above. Peak rejection is accomplished through a combination of peak height and peak area rejection parameters. If the software is not detecting your peak(s) of interest, you probably need to decrease both parameters.

F. TUTORIAL

This tutorial will guide you through a typical calibration, analysis, and peak-editing session for a multipoint BET analysis. The process involves:

- creating a sample information file, in which you set up parameters for your analysis
- performing the analysis
- creating the calibration file
- assigning the calibration file to the sample data
- editing peaks

Creating a Sample Information File

This sample file will contain steps for three calibrations and three analyses.

Calibration Steps

1. Select Open, Sample Information from the File menu; the Open Sample Information dialog is displayed.

2. Click **OK**, then **Yes** to create the file; the sample information dialog is displayed.

3. Enter an appropriate description in the **Sample** field.

4. Enter the operator and submitter names (if applicable).

5. Enter the sample mass in the **Mass** field.

6. Use the **Comments** window (if desired) to enter pertinent information about your sample or analysis conditions. Anything you enter in this window displays in the header of the report.

7. Click the Analysis Conditions tab; the Analysis Conditions dialog is displayed.
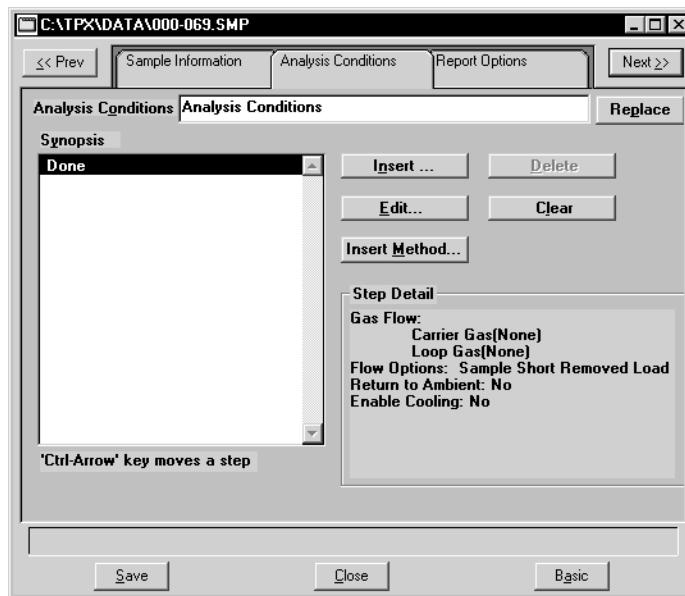
8. Click **Insert**; the Insert Experiment dialog is displayed.

9. Choose **Experiment**, then click **OK**; the New Experiment dialog is displayed.

10. Enter a description in the **Experiment description** field; the first experiment is a calibration.
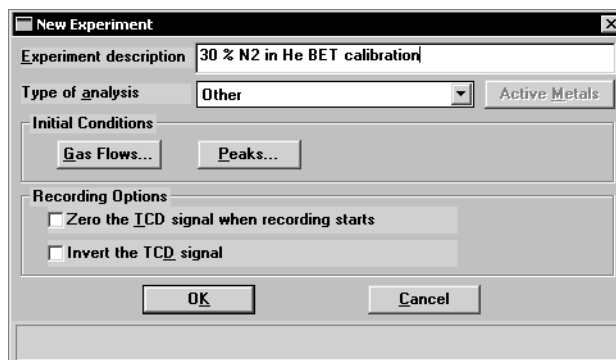
11. Click **Gas Flows**; the Gas Flows dialog is displayed.
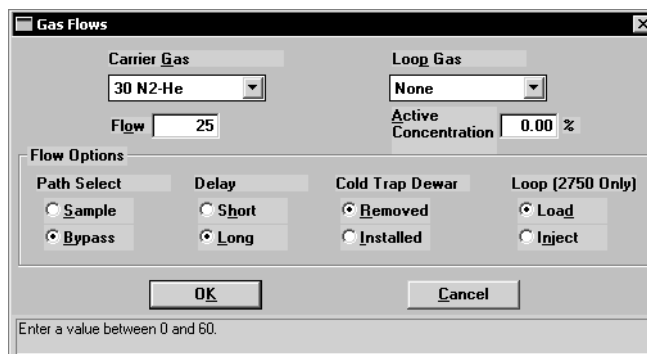
12. Choose your Carrier gas from the drop-down list; this example shows 30 N2-He. The flow rate of 25 is appropriate.

13. In the **Flow Options** group box, choose:

   • Bypass for the path
   • Long delay

   The Cold Trap Dewar and Loop options are not applicable in this tutorial.

14. Click **OK** to return to the New Experiment dialog, then again to close the dialog.

15. Now that you have defined your experiment (calibration), you must define its associated steps; click **Insert** to display the Insert Experiment Step dialog.

16. Select **Wait**, then click **OK**; the Wait dialog is displayed.
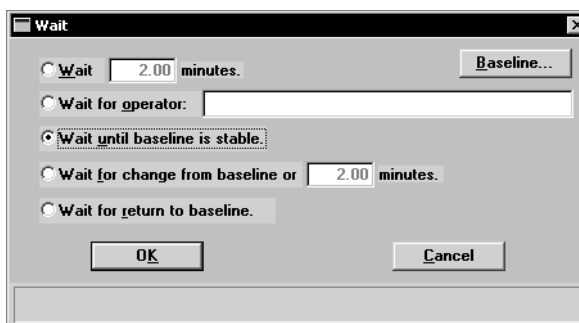
17. Click **Wait until baseline is stable**, then click **OK** to close the dialog.

18. Click **Insert**; the Insert Experiment Step dialog is displayed.

19. **Select Recording**; the Start Recording dialog is displayed.

```
┌─────────────────────────────────────────┐
│ ■ Start Recording                    ✕  │
├─────────────────────────────────────────┤
│                                         │
│  One measurement every    0.5  seconds  │
│                                         │
│                                         │
│       ┌────────┐      ┌────────┐        │
│       │   OK   │      │ Cancel │        │
│       └────────┘      └────────┘        │
├─────────────────────────────────────────┤
│ Enter a value between 0.4 and 10.0.     │
└─────────────────────────────────────────┘
```
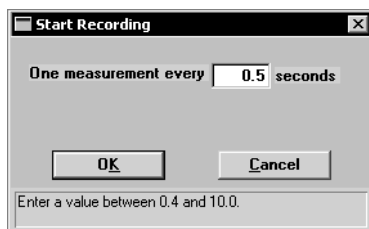
20. Enter how often you wish to record a measurement, then click **OK** to close the dialog; the Analysis Conditions dialog is displayed.

21. This experiment contains three calibration peaks, so repeat steps 8 through 20 two more times.

This completes the steps for the calibrations.

Analysis Steps

1. Click **Insert**; the Insert Experiment dialog is displayed.

2. Choose **Experiment**, then click **OK**; the New Experiment dialog is displayed.

```
┌──────────────────────────────────────────────────────────────┐
│ ■ New Experiment                                          ✕  │
├──────────────────────────────────────────────────────────────┤
│ Experiment description  30 % N2 in He BET                    │
│                                                              │
│ Type of analysis        Physisorption Surface Area    ▼  │ Active Metals │
│ ┌─ Initial Conditions ──────────────────────────────────┐   │
│ │      Gas Flows...        Peaks...                      │   │
│ └────────────────────────────────────────────────────────┘   │
│ ┌─ Recording Options ───────────────────────────────────┐   │
│ │  ☐ Zero the TCD signal when recording starts           │   │
│ │  ☐ Invert the TCD signal                               │   │
│ └────────────────────────────────────────────────────────┘   │
│           ┌────────┐         ┌────────┐                      │
│           │   OK   │         │ Cancel │                      │
│           └────────┘         └────────┘                      │
├──────────────────────────────────────────────────────────────┤
│                                                              │
└──────────────────────────────────────────────────────────────┘
```
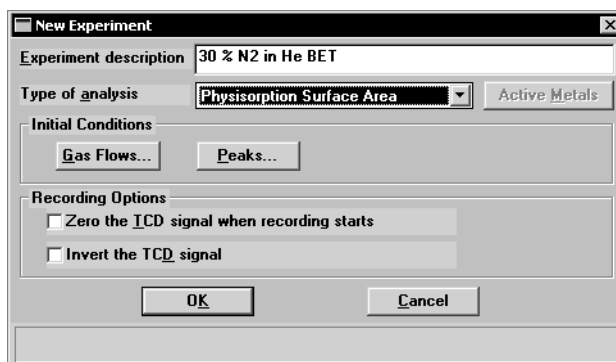
3. Enter a description in the **Experiment description** field.

4. Click the down arrow to the right of the **Type of analysis** field and select **Physisorption Surface Area**.

5.   Click **Gas Flows**; the Gas Flows dialog is displayed.

Gas Flows	☒

Carrier Gas   **Loop Gas**

| 30 N2-He ▾ | | None ▾ |

Fl**o**w   25   **Active Concentration**   0.00 %

Flow Options

Path Select	Delay	Cold Trap Dewar	Loop (2750 Only)
⊙ **S**ample	○ **S**hort	⊙ **R**emoved	⊙ **L**oad
○ **B**ypass	⊙ **L**ong	○ **I**nstalled	○ I**n**ject

| **OK** | | **Cancel** |
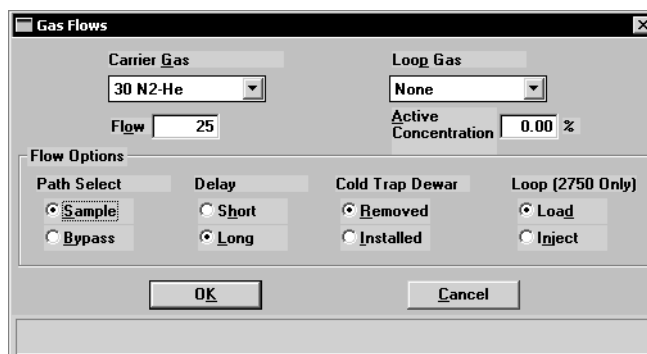
6.   In the **Flow Options** group box, choose:

• Sample for the path
• Long delay

The Cold Trap Dewar and Loop options are not applicable in this tutorial.
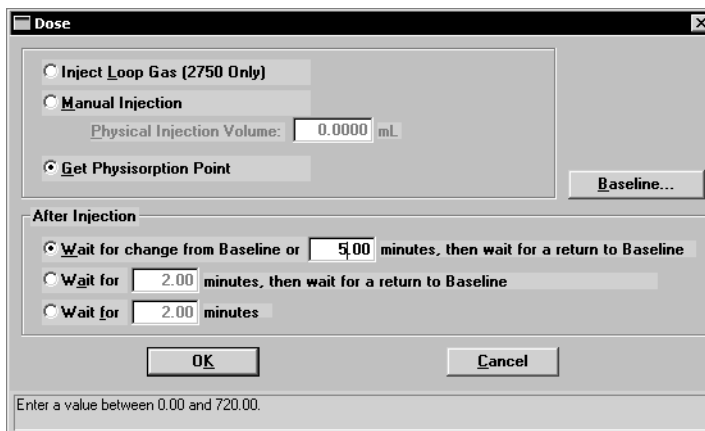
7.   Click **OK** to return to the New Experiment dialog, then again to close the dialog.

8.   Click **Insert**; the Insert Experiment Step dialog is displayed.

9.   Select **Wait**, then click **OK**; the Wait dialog is displayed.

Wait	☒

○ **W**ait   2.00 minutes.   **Baseline...**

○ Wait for **o**perator: _____

⊙ Wait **u**ntil baseline is stable.

○ Wait **f**or change from baseline or   2.00 minutes.

○ Wait for **r**eturn to baseline.

| **OK** | | **Cancel** |
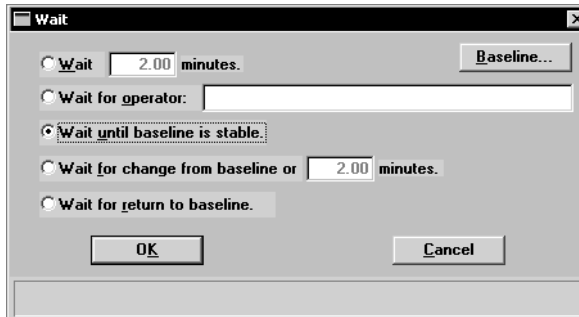
10. Click **Wait until baseline is stable**, then click **OK** to close the dialog.

11. Click **Insert**; the Insert Experiment Step dialog is displayed.

12. Select **Recording**; the Start Recording dialog is displayed.

Start Recording	☒

One measurement every   0.5 seconds

| **OK** | | **Cancel** |

Enter a value between 0.4 and 10.0.
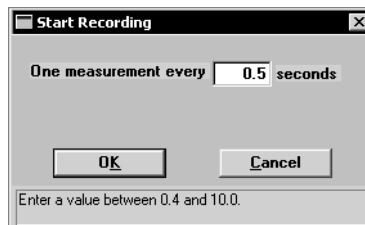
13. Enter how often you wish to record a measurement, then click **OK** to close the dialog.

14. Click **Insert**; the Insert Experiment Step dialog is displayed.

15. Select **Dose** and click **OK**; the Dose dialog is displayed.

16. Select **Get Physisorption Point**, then click **OK** to return to the Analysis Conditions dialog.

Select File, Print, and choose your sample file to print the entire contents of the file. This allows you to verify that all parameters are correctly entered. You can also check parameters by clicking on the step and viewing the contents in the Step Detail window.

17. Perform steps 1 through 16 two more times, entering two additional experiments. Be sure to change the description accordingly. For example, the second experiment will be on 20% concentration so indicate 20% in the description field of the second experiment.

18. Click the Report Options tab to specify report options.

19. Select the following reports (double-click on each, or highlight and press the spacebar):

   • Summary

   • BET Surface Area; edit this report and select **Multipoint plus one single point**, **Tabular Report**, **Surface Area Plot**, and **Isotherm Plot**

   • Langmuir Surface Area; edit this report and select **Tabular Report**, **Surface Area Plot**, and **Isotherm Plot**

20. Click **Save**, then **Close**.

Performing the Analysis

Now that you have created your sample file and loaded your prepared sample, it is time to perform the analysis.

1. Click **Start analysis** from the Unit menu; the Analysis dialog with the Open Sample Information dialog positioned on top is displayed.

2. Select the file you created and click **OK**; the Analysis dialog containing the parameters specified in your sample becomes fully visible.
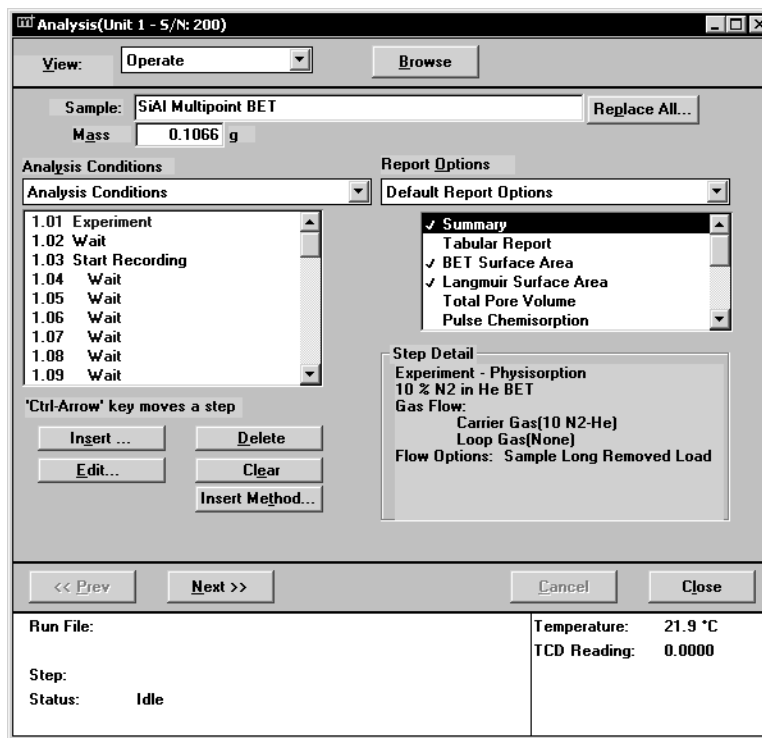
3.   Click **Next**; a dialog displaying the gases required for the experiments is displayed.

4.   Click **Next**; a dialog allowing you to choose a calibration file for your analysis is displayed.

Calibration experiments can be performed before analysis if desired and applied to the analysis using this dialog. This tutorial, however, does not show this method. Calibrations for this analysis are being performed in the first experiment and will be applied during the peak-editing procedure.

5. Click **Next**; a dialog enabling you to have a report generated automatically after analysis is displayed. You can select this option if desired, or you can have the report generated later by selecting Start Report from the Reports menu.

6. Click **Next**; a warning dialog about incompatible gases is displayed.

7. Click **Start**; this view of the dialog is displayed.

This prompt displays actions required to complete the analysis.
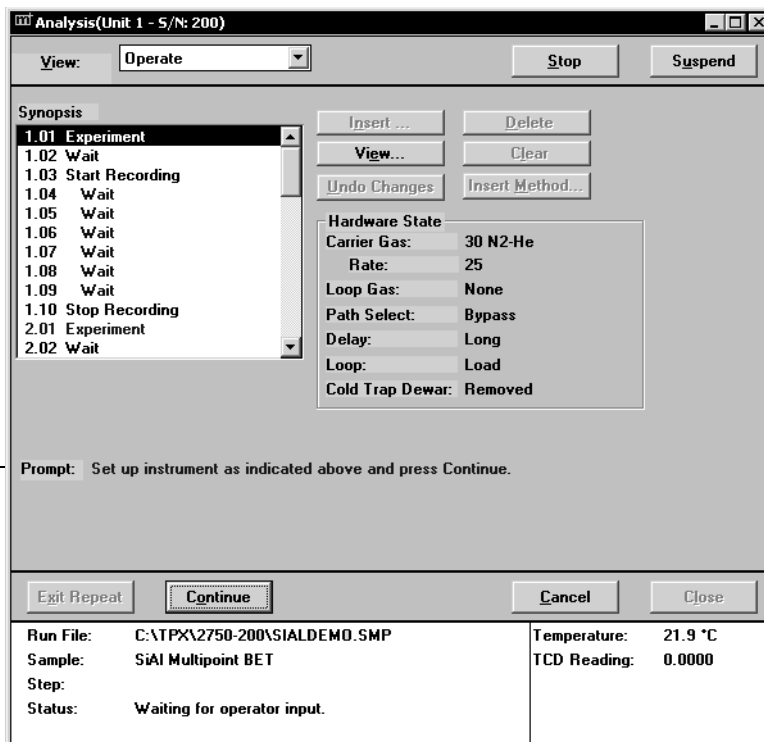
Notice that this view of the dialog contains a prompt just below the synopsis. This prompt displays interaction requirements with the analyzer. All remaining analysis dialogs will contain this prompt.

8. Perform the action requested at the prompt, then click **Continue**. Repeat this step for the remaining analysis dialogs.

9. Click **Show Results** on the Unit menu to view the data as collected. This enables you to view the data as collected and allows the analysis window to remain onscreen so that you can respond to the prompts.

After you have completed your experiments, edit your peaks and assign your calibration files (next section). It is best to do this before you generate your report(s).

Editing the Peaks

1. Select Peak Editor from the Reports menu.

2. Choose the file for the analysis we just completed, then click **OK**; the Peak Editor, Stacked view dialog is displayed. This view of the dialog displays all calibrations and experiments contained in the file, stacked one after the other.

3. Click a calibration experiment; the name of the experiment displays in the title bar of the dialog. This is why it is important when creating files, that you enter an intuitive description for your experiment.

4. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected calibration are displayed.
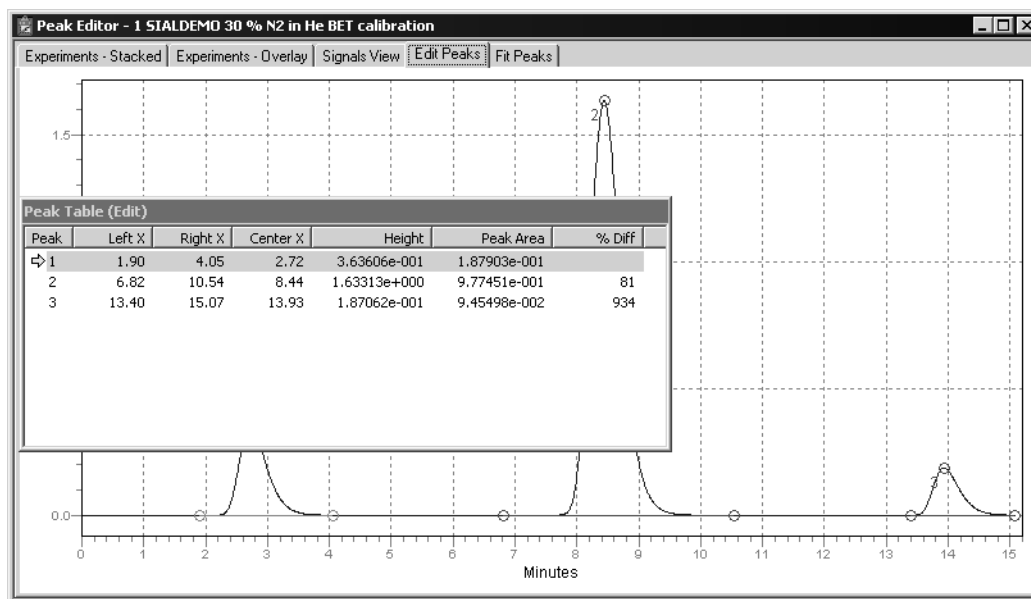
> **Peaks should be in a positive position before integrating. If you have any negative peaks, right-click in the graph area, choose View, Display and click the Invert option.**

5. Right-click in the graph area and select **Find All Peaks**; this option finds all peaks and marks them.

> **You can further define peaks if desired. Select the peak (the baseline area of the peak displays in green when selected), right-click, and use the Mark Left Edge and Mark Right Edge options.**

6. Right-click and select **Save**.

Creating the Calibration File

After you have defined your calibration peaks, you can create your calibration file.

1. Select **TCD Calibration**, **New** from the Unit menu; the TCD Calibration dialog is displayed
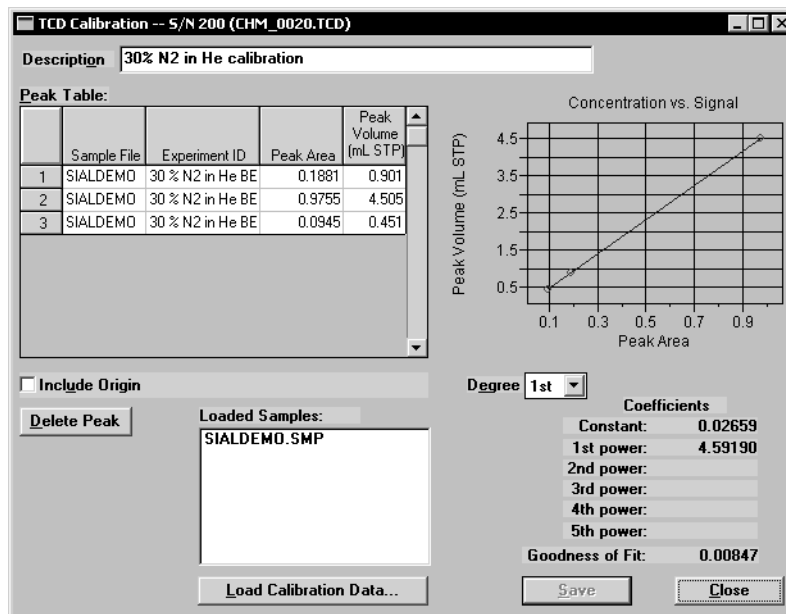
2. Click **Load Calibration Data**; the Load Calibration Sample File dialog is displayed.

3. Select the sample file, then click **OK**; all peak data are imported into the Peak Table.

4. Enter an intuitive description into the **Description** field. It is important that you use a description that you will easily recognize.

5. Delete from the Peak Table any data not associated with your calibration experiment.

6. Using the formula given below, calculate the peak volume for each peak in the table; enter the results into the appropriate fields

$$V_{syrinj} \; x \; \frac{273.15}{T_{amb} + 273.15} \; x \; \frac{P_{amb}}{760\,mmHg} = V_{Peak}$$

Example: Calculations for 1 mL injection, ambient temperature of 22 °C, ambient pressure of 740 mmHg

$$1 \; x \; \frac{273.15}{22 + 273.15} \; x \; \frac{740}{760\,mmHg} = 0.901\;mLSTP$$

7. Click **Save** to save the calibration file, then **Close** to close the dialog.

Repeat steps 1 through 7 for each gas concentration in the calibration. For example, if you have gas concentrations of 10%, 30%, and 20%, you should mark your calibration peaks and create calibration files for all three concentrations; a total of three files.

Assigning the Calibration File to the Experiment

Now you must assign the calibration to the appropriate experiment.

1. Click back into the Peak Editor dialog and select the **Experiments-Stacked** tab.

2. Click a sample experiment. Remember, you can view the experiment name in the title bar.

3. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected sample experiment are displayed.
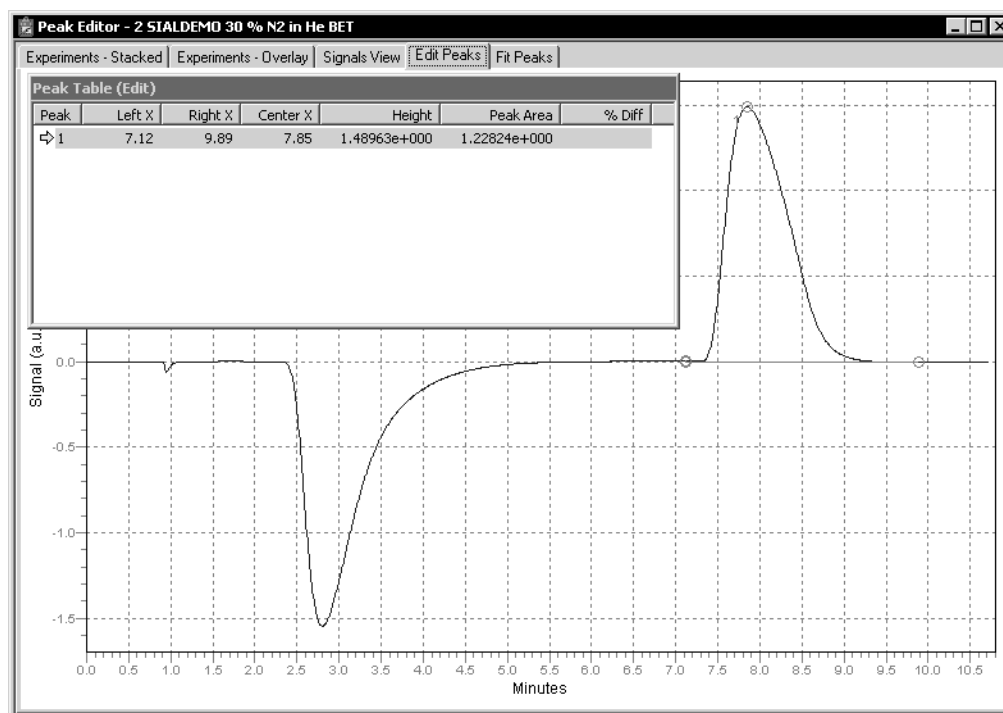
4. Right-click in the graph area and select **Find All Peaks**.

5. You should have only one peak in the table; if multiple peaks are included, delete all but the desorption peak. The desorption peak typically is the last one in the table.

6. Right-click in the graph area and select **Set Calibration**; the Set Calibration dialog is displayed.
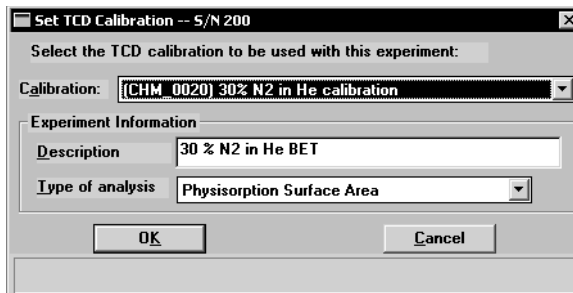
7. Click on the down arrow to the right of the **Calibration** field and choose the Calibration file you created for the concentration for the selected experiment. Be sure that the **Experiment** and the **Calibration** match.

8. Right-click in the graph area and select **Save**.

Now you are ready to generate your report; proceed with the next section.

Generate the report

1. Select Start Report from the Reports menu; The Start Report dialog is displayed.

2. Choose your sample file and click **OK**; the Select Reports dialog is displayed.

3. Ensure that appropriate reports are selected and click **OK**; requested reports are sent to the destination specified in the sample file.

> **Most users generate reports to the screen. This allows you to view parameters and data, and customize the reports in other ways. You can also generate the reports to a printer from the onscreen report window.**

INDEX

X

X-axis, 5-41, 7-21

Y

Y-axis, 5-41

Z

Zeolites, 1-7
Zoom feature
    peak editor, 7-16
    reports, 7-12