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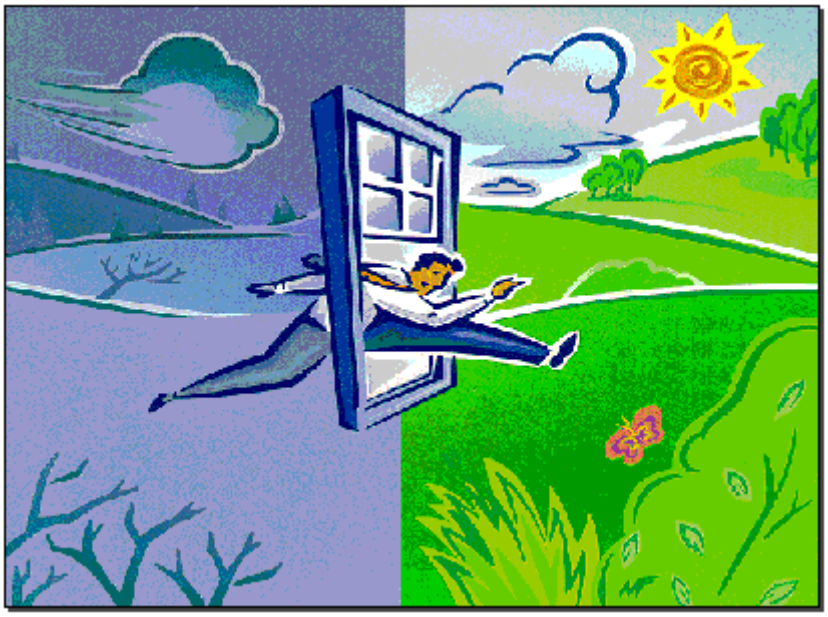
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Pyris Software for Windows



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Version 3.7

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Chapter 1
Using Pyris Help

The online help for Pyris Software for Windows is now in HTML help format. HTML help topics appear on the right side of the Help window, with scroll bars visible for further viewing. Navigation buttons are displayed across the top of the window. A special nagivation pane appears on the left side of the Help window. It displays tabs for the Contents, Index, and Search features.

Table of Contents

Most of Pyris Help is presented in the main help pane that you are looking at now. You can access other parts of the help system by using the Table of Contents to the left. The Table of Contents is a summary of the Pyris Help system with topics arranged by category. The Table of Contents' tree-like outline that displays topics in expandable/collapsible format is intended to provide you with a hierarchical view of the Help topics. The topics are organized by category which is indicated by a book. If you double click on a closed book, or the "+" sign next to the book, the book opens and the list of topics in the book is displayed. These topics are called pages. A topic that is new to the Pyris Help since the previous release is indicated by a red asterisk (*). The Table of Contents is synchronized with the Help so that if the topic you are in is a page in the Contents, that location is automatically highlighted in the Contents. Other forms of help in Pyris Software for Windows are context-sensitive help and Quick Help.

Index

Another feature of Pyris Help that helps you get around is the Index which visually displays an alphabetic list of all keywords associated with the Help system. The Index is visible while you are viewing the topic associated with that index entry.

Search

Pyris Help supports full-text searching. The left pane of the Help window contains a Search tab along with Contents and Index. To use the full-text search feature, click on the Search tab. Enter the word or phrase that you are looking for in the space at the top and click on **List Topics**. The Help system will go out and find all the topics that contain that word or phrase and will list the

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topic titles in the Topic list. Find the topic you want to display and either double click on it or highlight it and click on the **Display** button to display the topic in the right pane.

Context-Sensitive Help

Context-sensitive help lets you get information about any screen, menu command, dialog box, or toolbar button in Pyris Software for Windows. It provides specific information at precisely the place in Pyris where you want to find it. Pyris Software for Windows has "window-level help" or **Help** button or **F1** help. Window-level help provides an explanation of all the items in a dialog box or window within one Help topic. In addition to the **Help** button and **F1**, you can activate context-sensitive help by clicking on the **Context-Sensitive Help** button  in the Pyris toolbar. The cursor changes to an arrow with a question mark. Place the arrow on the screen, dialog box, menu command, or toolbar button about which you want information. A help topic describing that item appears in the help window.

Quick Help

Quick Help includes quick and easy procedures for using the Pyris Manager, calibrating analyzers, performing a DMA analysis, performing a purity analysis, determining lag or rate compensation, and creating play lists. Some topics contain videos that show what is happening on the screen while these steps are performed. Quick Help is displayed in its own window.

Multimedia Help

Multimedia Help comprises topics that include multimedia clips that are on the Pyris Software for Windows CD. The CD must be in the CD drive when you use Multimedia Help. The topics cover such topics as preparing samples for use with a DSC analyzer, how to install a hangdown wire in a TGA 7, and so on. The topics are useful when using Hardware Help.

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Chapter 2
Using Pyris Software

Getting Started

The Pyris Software for Windows group of items is accessible by selecting Programs from the Start menu in Windows 95 and NT. During installation you can elect to place a shortcut to the Pyris Manager on the Start Menu or on the Windows desktop. Items in the Pyris Software for Windows menu reflect how the software is organized. They are as follows:

Pyris Configuration

Use Pyris Configuration to dynamically configure the analyzers in your system. Pyris Configuration can be opened from the Pyris Manager **Start Pyris** button or from the Pyris Software for Windows program group.

Pyris Data Analysis Application

This application analyzes data collected by any analyzer. This application is not associated with a particular analyzer and can be used to analyze data and edit methods for any instrument attached to your thermal analysis system. More than one Data Analysis Application can be opened at a time.

Pyris Help

All of the documentation necessary for operating the Pyris Series Thermal Analysis System is provided online. Help contains complete descriptions of all of the software's features in addition to information on maintaining and optimizing your analyzers.

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Pyris Installation Help

Pyris Installation Help provides step-by-step procedures for installing all thermal analyzers and accessories supported by Pyris.

Pyris Manager

The Pyris Manager provides access to the Instrument Application for each configured instrument in your system. From the **Start Pyris** button in Pyris Manager, you can also access the Data Analysis application, monitor the system status, run Pyris Configuration, access Pyris Help, and close all Pyris-related windows.

Pyris Readme

This text file contains the latest information on the version of Pyris installed on your computer. It lists the new features included in the software and any information on the software or hardware that did not get included in the online Help.

Pyris Uninstaller

This tool is used to remove all Pyris Software for Windows files from your system. You must use this utility before installing a new version Pyris. It does not remove data, method, calibration, or play list files, however.

Temperature-Dependent Crystallinity

Temperature-Dependent Crystallinity is a third-party application that is used with Pyris data. It can be accessed either from the Calc menu in Data Analysis or from the Pyris Software for Windows menu. It has its own Help and Readme files.

Instrument Applications

Each thermal analyzer installed and configured in your Pyris software is represented by a button on the Pyris Manager. Clicking on a button opens the Pyris Instrument Application for that analyzer. The analyzer's name, assigned in the Configuration utility, is displayed in the title bar of the Instrument Application window.

In addition to the standard Windows components (see "Standard Windows Components section below), an Instrument Application screen comprises the following main parts (also see *Chapter 5: Instrument Applications*):

Method Editor

The Method Editor is used to create and set up methods for your sample runs. It comprises three pages — Sample Info, Initial State, and Program — each indicated by a tab at the top of the Method Editor window. Clicking on a tab displays that page.

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Instrument Viewer

This window displays a curve representing the real-time signal from the instrument. Depending on what you select from the Curves menu, you can display more than one curve at a time. Appropriate axes labels are automatically added to the display. The display of the real-time signal can be toggled on and off by selecting Monitor from the View menu or the **Monitor** button from the toolbar.

Data Analysis Window

You can open a Data Analysis window while in an Instrument Application by clicking on the **Data Analysis** button on the toolbar. It can also be opened from the Pyris Task menu seen by clicking on the **Start Pyris** button. When you select Data Analysis, a Select Data File dialog box appears in which you indicate the file you want to open. The selected data file is displayed on which you can perform various calculations, e.g., specific heat and purity. You can also display different types of curves from the data file, e.g., Heat Flow, Baseline Flow, and Sample Temperature. Also part of the Data Analysis window is access to Remote Monitor with which you can monitor a remote analyzer.

Pyris Player

You can open a Pyris Player window while in an Instrument Application by clicking on the **Pyris Player** button on the toolbar. When you select Pyris Player, the play list last used is displayed in the Edit Play List page. You can create a new play list, open another existing list, or edit the one displayed. The play lists seen here are analyzer-dependent, e.g., if you are in the DSC 7 Instrument Application, then the play list files will have a .dsp extension and can be used only with a DSC 7.

Control Panel

This is a dockable panel (i.e., it can be moved around the window) containing buttons with which you directly control the analyzer. The default position is the right-hand side of the screen. You can toggle the display of the panel by clicking on Control Panel in the View menu. A checkmark is displayed next to that item when the Control Panel is displayed.

Standard Tools Toolbar

This dockable toolbar contains buttons to help you create and edit methods and have access to the other parts of an Instrument Application. Some buttons are grayed out depending on what window is active, e.g., the New Method button is unavailable when the Instrument Viewer is the active window. The default position of the toolbar is across the top of the screen below the title bar. You can toggle the display of the toolbar by clicking on Toolbar in the View menu. A checkmark is displayed next to that item when the standard toolbar is displayed.

Rescale Tools Toolbar

This is a dockable toolbar that contains buttons to tools that optimize the data displayed in the Data Analysis and Instrument Viewer windows. The default position of the toolbar is across the top of the screen below the standard toolbar and above the Status Panel. You can toggle the

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display of the toolbar by clicking on Rescale Tools in the View menu. A checkmark is displayed next to that item when the Rescale Tools toolbar is displayed.

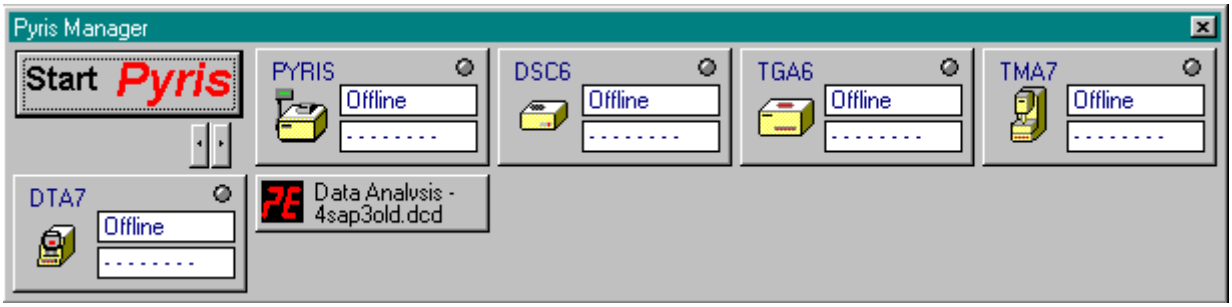
Status Panel

You can monitor the status of several analyzer parameters in this panel, whose default position is below the toolbars. You can toggle the display of the panel by clicking on Status Panel in the View menu. A checkmark is displayed next to that item when the Status Panel is displayed. The Status Panel comprises a user-defined number of boxes that are used to monitor the status of user-selected parameters of the analyzer. The current value of the parameter is displayed beneath the parameter name. Display the drop-down list of choices to select another parameter.

Data Analysis

The Data Analysis Application can be accessed by selecting **Pyris Data Analysis** either from the Pyris Software for Windows menu in the Programs menu or from the Pyris Manager Task menu displayed by clicking on the **Start Pyris** button in the Pyris Manager panel. When you access Data Analysis either of these ways, you have access to **all** data files, i.e., data from all attached analyzers. You can also access the analyzer-specific Data Analysis application by clicking on the **Data Analysis** button on the toolbar while in an Instrument Application. However, the only data files you can call up for analysis are those associated with the current instrument.

Pyris Software for Windows lets you analyze data collected from any thermal analyzer. Data are saved to the file that you specified in the Sample Info page in the Method Editor, to the file specified in a play list, or to the default file name specified in Preferences. You can open multiple Data Analysis Applications from the Pyris Manager as well as open Data Analysis windows inside an Instrument Application. You can switch from one Data Analysis window to another by clicking on the appropriate **Data Analysis** button in the Pyris Manager. As each Data Analysis window is opened, it is indicated by an additional button in the Pyris Manager bar, in addition to the instrument buttons.



In Data Analysis, you can perform many functions on a data file to manipulate the data. The menus available while in Data Analysis indicate the possibilities. The items in these menus can be used to rescale the displayed curves, display different types of curves for the data, perform calculations with the data, and other functions.

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Standard Window Components

The standard window components are as follows:

Title Bar

The title bar is located along the top of a window. It contains the name of the application and, in some cases, the active file. To move the window, click on and drag the title bar. You can also move dialog boxes by dragging their title bars.

Depending on the window, a title bar may contain the following elements:

- Application Control menu button
- Document Control menu button
- **Maximize** button
- **Minimize** button
- Name of the application
- Name of the document
- **Restore** button

Status Bar

The status bar is displayed at the bottom of the main application window. The left area of the status bar displays the function of a menu item when it is highlighted. It also shows messages describing the functions of the toolbar buttons as you "depress" them, before releasing them. If you do not wish to execute the button's command, release the mouse button while the pointer is off the toolbar button.

The right side of the status bar displays the tare reading when you tare the probe of the DMA 7e or TMA 7.

Maximize Button

The **Maximize** button  is on the right end of the title bar of a window. Click on the button to enlarge the window so that it fills the maximum area possible. Clicking on the **Maximize** button is the same as choosing Maximize from the application's Control menu.

Minimize Button

The **Minimize** button  is on the right end of the title bar to the left of the **Maximize** button. Click on the button to reduce the application window to an icon. Clicking on the **Minimize** button is the same as choosing Minimize from the application's Control menu.

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Vertical Scroll Bar

If a window in Pyris Software for Windows cannot display its entire contents on the screen, scroll bars are displayed. The vertical scroll bar on the right side of a window is used to access the top and bottom areas of a window by moving the scroll box in the bar. You can drag the scroll box using the mouse or click on the scroll bar arrows at the top and bottom of the bar to scroll the window vertically.

Horizontal Scroll Bar

If a window in Pyris Software for Windows cannot display its entire contents on the screen, scroll bars are displayed. The horizontal scroll bar is used to access the left and right areas of a window by moving the scroll box in the bar. You can drag the scroll box using the mouse or click on the scroll bar arrows at the left and right ends of the bar to scroll the window horizontally. The horizontal scroll bar is located at the bottom of a window.

Left Window Border

Resize the active window horizontally by dragging the left window border with the mouse.

Right Window Border

Resize the active window horizontally by dragging the right window border with the mouse.

Top Window Border

Resize the active window vertically by clicking on the top edge of the window (the cursor becomes a two-headed arrow) and dragging the border with the mouse.

Top Left Window Border

Resize the active window diagonally by clicking on the very edge of the top left border of the window (the cursor becomes a two-headed arrow) and dragging border with the mouse.

Top Right Window Border

Resize the active window diagonally by clicking on the very edge of the top right border of the window (the cursor becomes a two-headed arrow) and dragging the border with the mouse.

Bottom Window Border

Resize the active window vertically by clicking on the bottom edge of the window (the cursor becomes a two-headed arrow) and dragging the border with the mouse.

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Bottom Left Window Border

Resize the active window diagonally by clicking on the very edge of the bottom left border of the window (the cursor becomes a two-headed arrow) and dragging the border with the mouse.

Bottom Right Window Border

Resize the active window diagonally by clicking on the very edge of the bottom right border of the window (the cursor becomes a two-headed arrow) and dragging the border with the mouse.

Context-Sensitive Help Button

When you select the Context-Sensitive Help button  the cursor changes to a question mark. Move the question mark cursor onto any part of the screen and click to display context-sensitive help about that part of the window, dialog box, or menu command.

Control Menu

The Control menu of a standard window is displayed by clicking on the upper-leftmost box. It contains the following Windows commands:

- Restore
- Move
- Size
- Minimize
- Maximize
- Close

Customizing Pyris

You can customize Pyris Software for Windows to suit your needs. You can change the way the screen looks, how curves are displayed, and the default values for many program parameters.

Pyris Main Frame

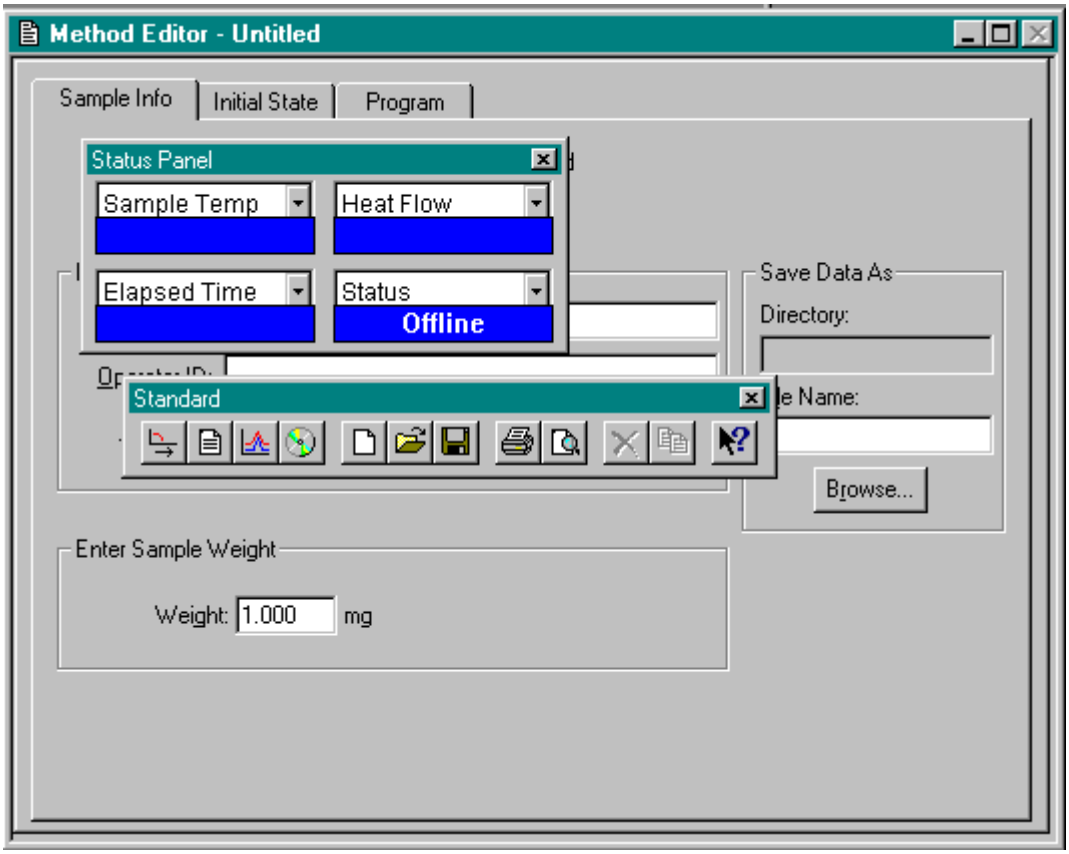
You can change the graphics display in the underlying main frame of Pyris by clicking on the right mouse button while the cursor is in the main frame area. This displays a popup menu from which you can select the Thermal Dragon bitmap, a pale gray screen, the normal Windows window, the default Pyris logo, or browse for another bitmap file.

Dockable Toolbars, Status Panel, and Control Panel

All toolbars – standard, Pyris, and Rescale Tools – and the status panel can be attached to any side of the Pyris window or they can “float” over the window, as seen in the figure below. The control

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panel, whose default position is the right-hand side of the Pyris window, can be attached to the left side of the Pyris window. When a toolbar, the status panel, or the control panel floats, it has a title bar. The graphic below shows the status panel and standard toolbar "floating" on the Pyris screen. Each one has a title bar.



Click on the background of the toolbar or panel and drag it to the desired location. When you drag a dockable toolbar or panel to any edge of the Pyris window, it becomes attached to that side.

Customizing the Status Panel

The Status Panel consists of boxes each of which contains a parameter name and that parameter's current value. Each box contains a drop-down list of parameters from which you select the parameter to be displayed in that box. As soon as you select a parameter, its current value is displayed in the lower part of the box. If you highlight the entry field in the status panel and then type the first letter of another parameter, e.g., for a DTA 7 type an "f"), then that parameter, or another parameter that begins with that letter, will be displayed. So for a DTA 7, if you continuously type "f" the parameters Furnace Cover, Furnace Lock, Furnace Status will be displayed. You can scroll through all parameters starting with that letter by continuously pressing that key.

The Status Panel must contain at least one parameter box. To resize the status panel, i.e., to eliminate or add a parameter box, first detach the status panel from the window frame (click on it and drag it away from the window frame), then click and drag the status panel border to include or exclude a box.

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Curves

The curves you initially see in the Instrument Viewer window as a run progresses depend on the selections made in the Real-Time Curves page of Preferences. There you indicate which curves you want displayed (up to four) and the order of display (e.g., Baseline Heat Flow, Sample Temperature, and Temperature for Pyris 1 DSC). In the Real-Time Curves page you can also indicate whether to use Time or Temperature for the X axis at the start of a run. You can change the types of curves displayed in Instrument Viewer and Data Analysis by making selections from the Curves menu.

In Instrument Viewer and Data Analysis, there are four places for positioning the cursor at which, when you click on the right mouse button, a popup menu appears: (1) on an axis label, (2) inside the graphical area, (3) on an existing curve label, and (4) on a curve. Each popup menu contains options that can be used to change the appearance of the axis line or label, the curve, the curve label, and the grid and graphical labels. These popup menu items are discussed below.

Active or Focused Curve

Regardless of the number of curves displayed in a Data Analysis window or Instrument Viewer, only one curve can be active at a time. The active curve, a thick solid line type, is the curve upon which all actions are performed. The Y axis of the active curve has a thick, solid line above the label, indicating that this is the active curve's Y axis.

To make a curve the active curve, position the cursor on any point on the desired curve and click the left mouse button. Another way to make a curve active is to select it in the Legend window.

Remove Curve

To remove the active curve from the Instrument Viewer or Data Analysis window, press the **Shift + Delete** keys simultaneously or select **Delete** from the Edit menu. All calculation results for that curve will be deleted along with the data. The data and results can be retrieved by opening the data file again. You can also right-click on the active curve and select **Remove Curve** from the popup menu.

Curve Colors

The curve's color initially depends on your selection in the Colors page of Preferences. Colors can be assigned to up to 12 files. The order in which you open data files determines which color is used to display that curve. All curves for a sample run (e.g., Sample Temperature and Program Temperature) displayed in the Instrument Viewer or Data Analysis are the same color. Each data file added to the window (by using **Add Data** from the File menu or the **Add Curve** button on the toolbar) takes the next color in order. Also in the Colors page, you can assign the color for the background and the grid lines. You can change the color of the active curve by right-clicking on the curve and selecting **Change Curve Color** from the popup menu. The standard Color dialog box is displayed from which you can select another color or create a custom color. When you click on **OK**, the color is applied to the focused curve.

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Add/Remove Curve Label

In Data Analysis, you can annotate the active curve by using the **Annotate** button on the toolbar or selecting **Annotate** from the Display menu. Enter label text, font, and orientation in the dialog box. You can also add a label by right-clicking on the active curve and selecting **Add Label to Curve** from the popup menu. Enter the label text, font, and orientation in the dialog box. You can remove a curve label by either clicking on it to display a box around it and then selecting **Delete** from the **Edit** menu, or by right-clicking and selecting **Remove Label** from the popup menu.

You can edit the label by right-clicking and selecting **Edit Label** from the popup menu. This displays the Define Label Properties dialog box.

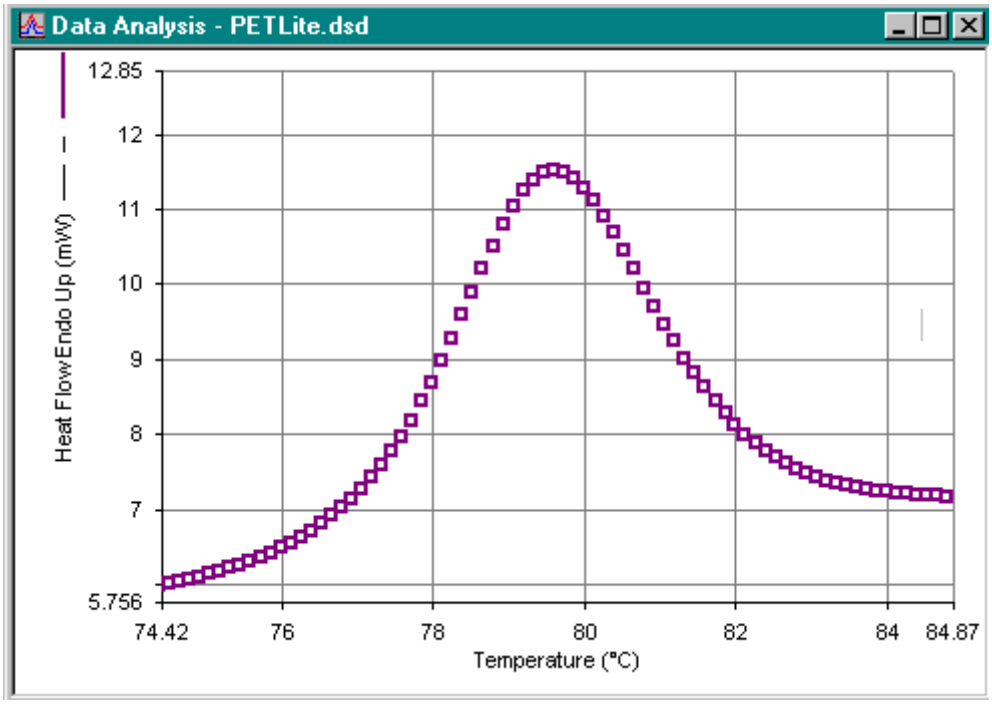
Change Line Style

There can be from one to four Y axes displayed at a time, referred to as Y1, Y2, Y3, and Y4. Each Y axis is assigned a default line type. You can change the assignment of line type in the General page of Preferences. All curves scaled on a particular axis will have that assigned line type. You can change the line type used to display the focused curve by right-clicking on the curve and selecting **Change Line Style** from the popup menu. The selections are Solid, Dotted, Dashed, Dashed Dot, and Dash Dot Dash.

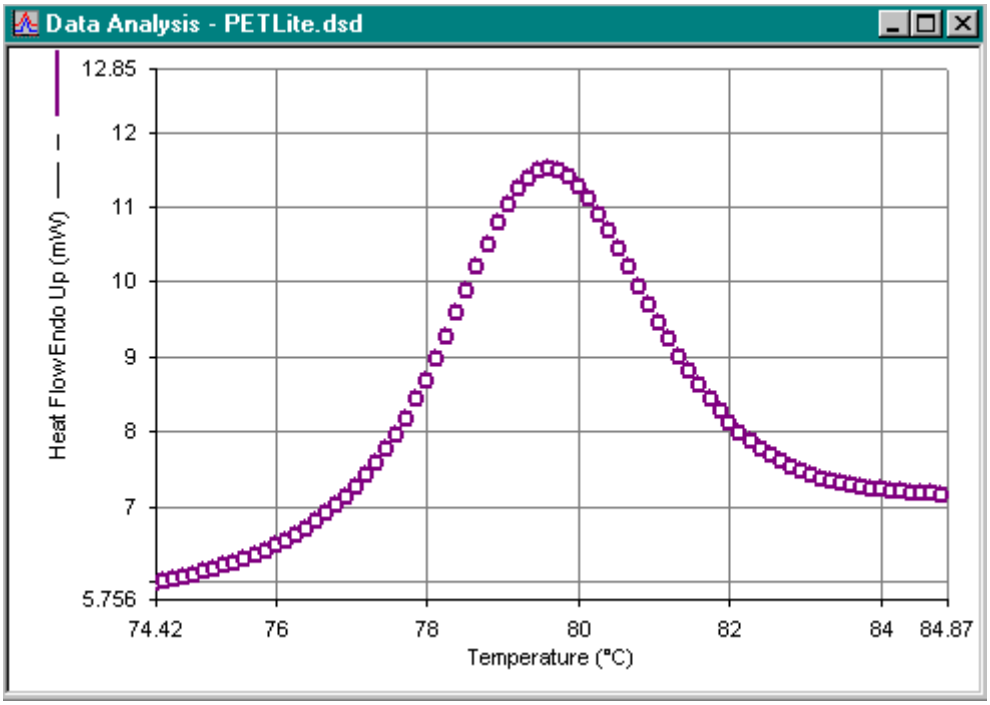
Plot Type

In addition to a line style, a curve also has a plot type. You can change the plot type for the active curve by right-clicking on the curve and selecting **Change Plot Type** from the popup menu. The selections are Line, Stick, Scatter, and Scatter Eclipse.

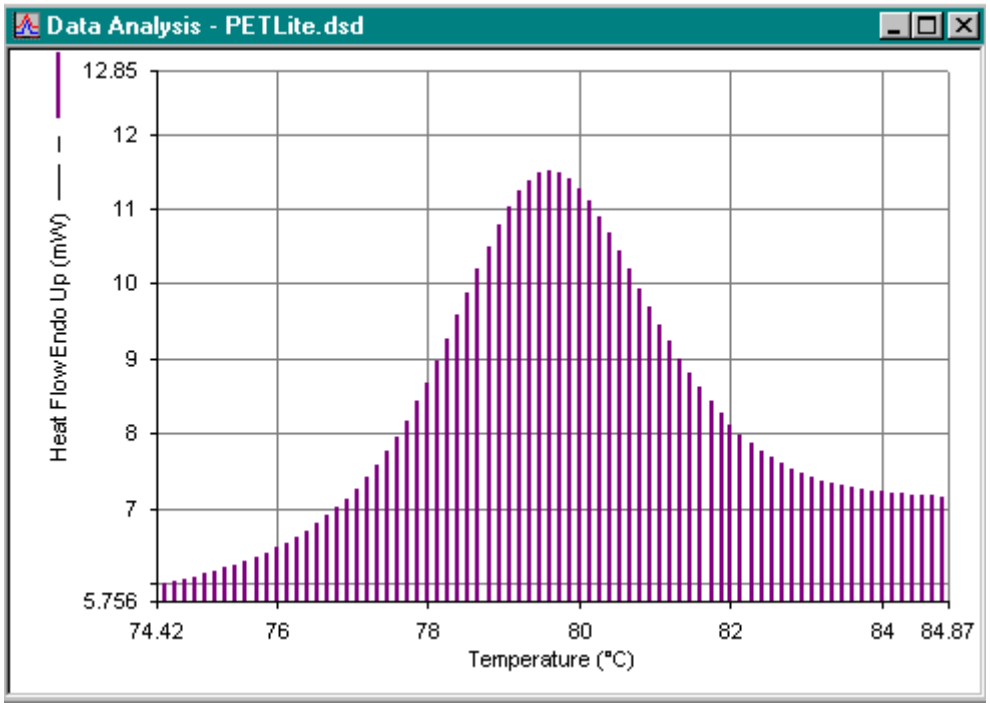
1. **Scattered Plot Type**



2. **Scattered Ellipse Plot Type**



3. **Stick Plot Type**



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Axes Labels and Lines

The axes of the Instrument Viewer and Data Analysis windows can be modified. With the cursor positioned near the axis label or title, right click on the mouse; the popup menu displayed contains options that affect the axis title, labels, and tick marks. The default axis label [e.g., Temperature (°C)] can be changed. Select **Change Axis Title** from the popup menu and the Define Label Parameters dialog box appears. You can change the words, color, orientation, and font of the title. Select Override Color and Override Font check boxes and then click on **Select Font/Color** button. In the Font dialog box, select the font, size, and color.

You can also select **Change Axis Label Color** (i.e., the numbers along the axis line) and **Change Axis Line Color**. When these two options are selected, the standard Windows Color dialog box is displayed from which to select a new color or you can define a custom color. When the axis labels and title are displayed, the default, there is a checkmark next to the **Has Axis Labels + Title** item in the menu. Axis ticks are also displayed by default and can be turned off by selecting **Has Axis Ticks**. The checkmark is removed.

Adding Curves to the Display

When data are collected from a run, different signals are saved, e.g., Sample Temperature, Program Temperature, and Heat Flow for a DSC. Multiple data curves can be displayed in a single Data Analysis window and multiple Data Analysis windows can be displayed at any time.

Add Curves from the Current Data File

Use the commands in the Curves menu or the Math menu to add curves from the active data file to the display. The rules for displaying additional curves from the same data file in one Data Analysis window are as follows:

1. If there are less than four Y axes displayed and
   - if the data to be displayed is on a different scale than any of the displayed curves, a new Y axis is added.
   - if the data to be displayed is on the same scale as another displayed curve, the new curve is displayed using the existing scale.
2. If there are four Y axes currently displayed and
   - if the data to be displayed is on a different scale than any of the displayed curves, the new curve is displayed without an axis displayed.
   - if the data to be displayed is on the same scale as another displayed curve, the new curve is displayed using the existing scale.

Add a Data File to an Open Data Analysis Window

Select the **Add Data** command in the File menu or click on the **Add Curve** button  on the toolbar, then select a data file in the Add Data dialog box. The new data file selected will be added to the display. The last curve added is the active curve.

To add a data file to an open Data Analysis window and **clear all other curve**s, use the **New Data** command in the File menu or click on the New button  in the toolbar. All curves in the active window are cleared and you can select a new data file to display.

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Open a New Data Analysis Window

Use the **Open Data** command or click on the **Open** button , then select a data file in the Open Data dialog box. The data file selected will be the first file in a new Data Analysis window. All other open Data Analysis windows remain open.

Grid Display

In both the Instrument Viewer and the Data Analysis windows, you can turn the display of a grid (vertical and horizontal lines) on and off by either selecting **Grid** from the View menu or the **Grid** button from the toolbar. Either way activates or deactivates both horizontal and vertical lines. You can also right-click within the window to display a popup menu. Here you can select **Horizontal Grid** or **Vertical Grid** from the **Display Grid** option to activate or deactivate the individual grid lines.

In addition to turning the vertical and horizontal grid display on and off, you can select the color of the grid lines from the right-click menu by selecting **Grid Color**. The standard Windows Color dialog box is displayed from which you can select a new color for the grid lines or create a custom color.

Change the color of the background of the curve display by selecting **Background Color** from the right-click menu in Data Analysis. The standard Windows Color dialog box appears from which to select a different color or to create a custom color.

Graph Title

You can add, edit, or delete a title for the Data Analysis window. Display the popup menu by right-clicking in the graphical area; the menu contains the **Add/Edit Graph Title** with which you can enter a new title or edit the existing title for the graph of your data. You can also select **Delete Graph Title** to remove the label. The label is associated with the entire display; it is not associated with a particular curve.

Copy Image

You can copy the image of the curves to the clipboard by selecting Copy Image either from the Edit menu or from the menu displayed when you right click within the Data Analysis or Instrument Viewer window. The image does not include peripheral items such as the toolbar or control panel that you get when you perform a screen capture. The image contains the curves, axes, and axes labels. The pasted object can then be manipulated in another application, i.e., resized, and its aspect ratio is maintained.

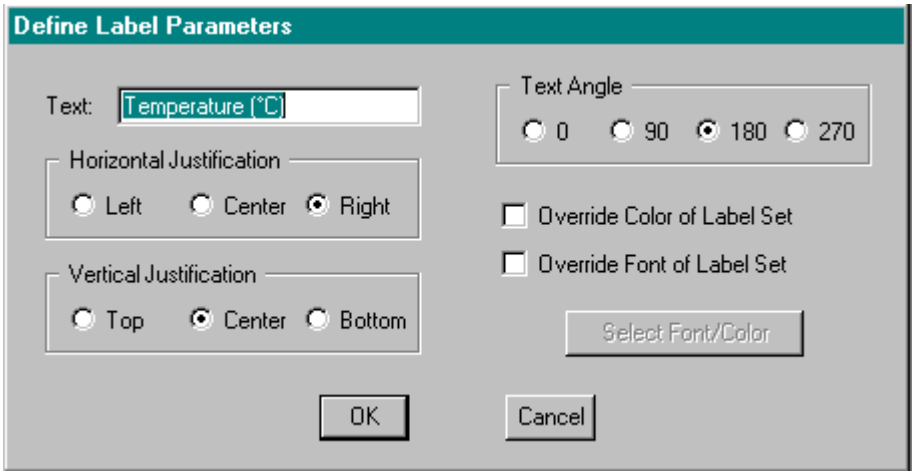
Edit Label

You can change a graph title's color and font by right clicking on the existing label and selecting **Edit Label** from the popup menu. This displays the Define Label Parameters dialog box. Select the Override Color and Font by clicking in the check boxes and then select the **Select Font/Color** button to display the Font dialog box. In that box select the new font, type, size, and color.

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Define Label Parameters Dialog Box

The Define Label Parameters dialog box appears when you select **Change Axis Title** from the right click menu for axis labels and graph titles and the **Edit Label** selection from the right click menu seen when you click on a graph title.



4. **Text**

   The text of the label that you want to edit. The existing text is displayed as the default.

5. **Horizontal and Vertical Justification**

   Click the radio button for the position you want for the label. The default positions are centered.

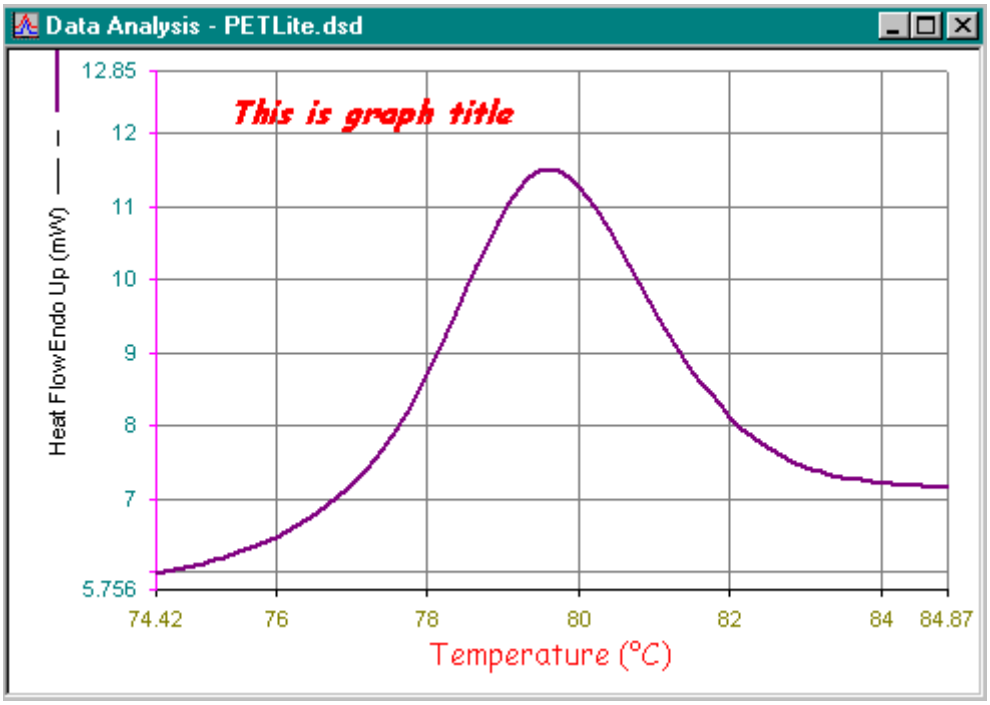
6. **Text Angle**

   This selection is useful for the Y axis label. You can have the label turned 90° so it is parallel to the Y axis.

7. **Override Color and/or Font of Label Set**

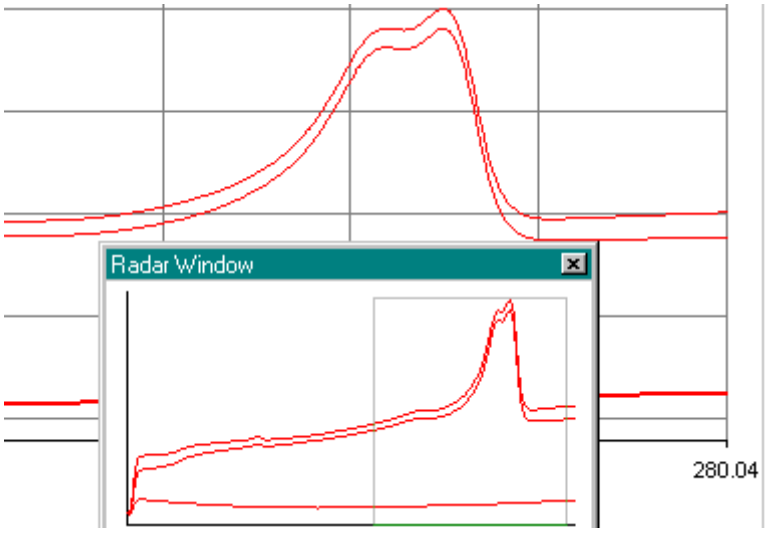
   Click on one or both check boxes to display the Windows Font dialog box. You can select a different font, style (bold, regular, italic, bold italic), type size, and color.

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Using the Radar Window

The Radar window is a movable, sizable window that shows at full scale all of the curves that are using the active Y axis. The current scale (i.e., the area displayed in the Instrument Viewer or the Data Analysis window) is shown as a boxed area in the Radar window.



Only one Radar window can be open for a Data Analysis Application or for the Instrument Viewer. If there is more than one Data Analysis window open, the Radar window is associated with the active window.

Open the Radar window by selecting the **Radar** button  in the Rescale Tools toolbar or use the **Radar** command in the View menu. This command toggles the Radar window display on and off.

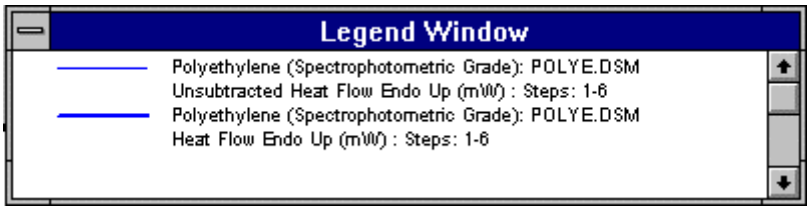
To graphically rescale both axes in the active Data Analysis window or in the Instrument Viewer using the Radar window, click on and hold down the left mouse button and then drag the cursor inside the Radar window to draw a box around the desired area. This defines the new scale. When you release the mouse button, both axes will be rescaled. You can also drag one side or one corner of the existing box to resize and rescale.

If you rescale directly in the Data Analysis or Instrument Viewer window by drawing a box around the desired area of the curves, the Radar window will automatically display a box that reflects the new scale.

Legend

The Legend command on the View menu displays the Legend window. The Legend window shows a description of each of the curves displayed. You can toggle the Legend window on and off by clicking on the **Legend** button  on the toolbar, selecting **Legend** from the View menu, or typing the **Ctrl + F1** key combination.

The Legend window is a small movable and sizable window containing information about the curves displayed. The curves are listed in the legend in the order in which they are opened. You can use the Legend window to quickly view important characteristics about a curve as well as make a curve active.



The Legend window contains the following information about all curves displayed:

- **Line Type and Color** displayed on the left side of the window. These are determined in the General Preferences page and Color Preferences page.
- **Sample ID and Data File Name** displayed on the first line next to the line type.
- **Step ID and Step Number** displayed on the second line, with Total Number of Steps if there is more than one step in the method used to collect the data.
- **Units** displayed next to Step ID in parentheses.

You can make a curve the active curve by positioning the dotted box around the curve description in the Legend window and clicking. The curve is then displayed as a thick solid line.

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Using the Pyris Manager

You can access the Pyris Manager panel when you select **Pyris Manager** from the Pyris Software for Windows group in the Programs menu displayed from the Start menu. You may have selected to have the Pyris Manager icon on the Desktop screen when Pyris was installed. Double-click on the icon to load the Pyris Manager panel. You could also have elected during installation to have the Pyris Manager load automatically when you turn on your computer.

The Pyris Manager is the main location from which to operate the Pyris Series Thermal Analysis System. The Pyris Manager is used to start instrument applications and Data Analysis applications can be initiated from there. Analyzers can be added to and removed from the system dynamically with the Configuration utility which is accessed from the Pyris Manager task menu. The status of each analyzer in the system can be monitored in Pyris Manager as well.

The Pyris Manager can be displayed horizontally or vertically on the screen. If it is horizontal and you want it displayed vertically, just click on and drag the Pyris Manager down into the middle of the screen; it will redisplay vertically. To change from vertical to horizontal display, place the cursor in the Pyris Manager (but not on an instrument button or the **Start Pyris** button), click on and hold down the left mouse button, and start moving the mouse to the left, to move the Manager from across the top to down the left-hand side of the screen. Or start moving the mouse to the top and the right to move the Pyris Manager from the left side up to across the top of the screen. If the Autohide feature is activated, the bar shrinks to a smaller area as it is moved. Just the **Start Pyris** button is displayed.

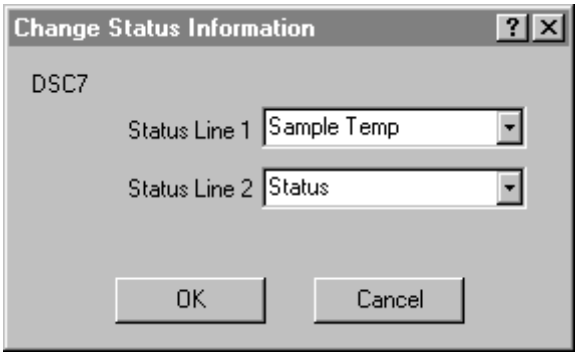
The parts of the Pyris Manager are described below.

Instrument Button

An Instrument button opens the Instrument Application for the analyzer shown on the button or, if the Instrument Application is already open, jumps to it. There is a button for each analyzer configured on your system. The name of the analyzer that appears on the button is specified in Configuration. The button also indicates the current state of the analyzer and information about the current step in the method (if one is running).

Change Status Information

If you place the cursor within an instrument button on the Pyris Manager, but not in a display field, and click on the right mouse button, the Change Status Information dialog box appears:

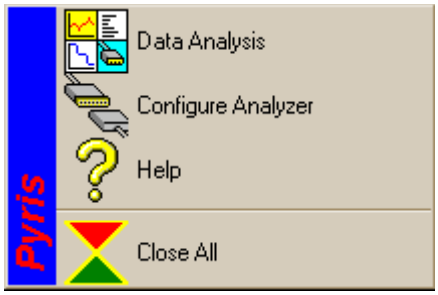


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Here you can change the information displayed on the instrument button. Display the drop-down menus for both status lines and select the parameter you want displayed. The list of available items for both status lines is instrument-dependent.

Start Pyris Button

The **Start Pyris** button is on the Pyris Manager. Beneath the **Start Pyris** button are left and right arrow buttons if the Pyris Manager is horizontal and up and down arrow buttons if the Pyris Manager is vertical. These are used to scroll the instrument buttons left or right or up and down if there are more buttons than can be displayed on the screen. Click on the **Start Pyris** button to display the **Pyris Manager Task menu**.



Data Analysis

Opens a new Data Analysis Application window. Any analyzer's data can be used in this Data Analysis Application; it is not analyzer-specific.

Configure Analyzer

Displays the Pyris Configuration dialog box with which you add or remove an analyzer to your system or edit an existing analyzer's configuration.

Help

Click on this item to load the online help.

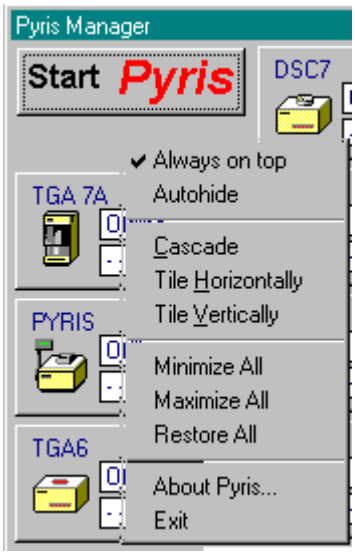
Close All

Closes all Instrument and Data Analysis Application windows, prompting you to save any files that have been changed before closing. It also closes the Pyris Manager panel.

Pyris Manager Popup Menu

If you place the cursor inside the Pyris Manager, but not within an Instrument button, and click the right mouse button, a popup menu containing the following options is displayed:

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Always On Top

Keeps the Pyris Manager displayed on top of all other open Pyris and non-Pyris windows. You can relocate the panel anywhere on the screen by clicking on the title bar and dragging. This button works in conjunction with Autohide.

Autohide

Causes the Pyris Manager to "hide" below or above the screen or off the side. Always On Top must be activated in order for this feature to work. Drag the panel to the bottom or top of the screen until it redisplays in horizontal form; or drag the panel to the side until it redisplays in vertical form. Move the cursor into the window and click. The panel rolls off of the screen and will reappear when you bring the cursor to the bottom or top of the screen.

Cascade

Displays all the Pyris windows in a cascade format. Make sure all of the windows that you want to cascade are open. Closed or minimized windows can not be displayed.

Tile Horizontally

Displays all the Pyris windows tiled horizontally, i.e., they are full width and variable height. Make sure all of the windows that you want to tile are open. Closed or minimized windows can not be displayed.

Tile Vertically

Displays all the Pyris windows tiled vertically, i.e., they are full height and variable width. Make sure all of the windows that you want to tile are open. Closed or minimized windows can not be displayed.

Minimize All

Minimizes all open Pyris windows.

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Maximize All

Maximizes all open Pyris windows.

Restore All

Restores all open Pyris windows to their original size and position before any resizing and repositioning occurs.

Navigating in Pyris Software for Windows

It is easy to navigate around Pyris Software for Windows' components. You can go from Instrument Viewer to Method Editor to Data Analysis and other parts by a click of the mouse on the standard toobar. The first four buttons on the standard toolbar are used to navigate between the four major parts of Pyris.

When a window such as Pyris Player or Calibration displays tabs, indicating that there are pages that make up the window, click on the tab to display the page on top of the other pages.

Another way to navigate is the use of the menu bar. Each menu item contains a specific drop-down list dependent on the analyzer and the window displayed.

Menus and toolbars are discussed in detail in Chapter 6.

Standard Toolbar

The standard toolbar is displayed across the top of the screen, beneath the title bar of the Pyris window. It is a dockable toolbar. The buttons available for selection depend on where you are in the software: Instrument Viewer, Method Editor, Data Analysis, and Pyris Player. To use the toolbar, just click on the button that corresponds to the option you want to use. If you position the cursor in the upper-right-hand corner of a button, a tool tip will be displayed indicating what the button is. For example, if the cursor is positioned in the upper-right-hand corner of the first button on the Method Editor's toolbar, the tool tip displayed would read: "Instrument View."

The following is the standard toolbar seen when the Method Editor is the active window. If the button is gray, it is not active in the Method Editor.



The standard toolbar for Data Analysis windows is seen below.



The standard toolbar for Instrument Viewer is seen below. If the button is gray, it is not active in Instrument Viewer:



The standard toolbar seen when the Calibration window is displayed is as seen below:

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The standard toolbar seen with the Pyris Player is open. This is displayed above the Player Control bar.



Tools for Support of Validation and Compliance

Good Laboratory Practice (GLP) requirements place great emphasis on proper method development and auditing. Pyris Software for Windows directly addresses this issue by providing a number of features and tools to help the integration of thermal analysis products into your quality system. Please note that if compliance and validation are important issues in your laboratory, it is recommended that Windows NT be your operating system.

Validation and compliance tools and support features available in Pyris Software for Windows are the following:

Simplified Method Development

Method generation has been simplified with the Method Editor of Pyris. Point-and-click interaction, scroll bars, spin buttons, and easy selection of options from lists of choices make method creation easier than ever. The use of pages to logically separate different portions of the method further simplifies method development. Method creation can be performed on the computer directly attached to the instrument or you can develop a method elsewhere, such as in the method development area, and then transfer it via network to the workstation at the instrument.

Raw Data and Method Storage

Raw data are permanently stored with the method used to collect it under a file name entered by the operator. The raw data and the validated method cannot be modified, with the exception of Sample ID, Operator ID, Comment, Sample Weight, and Sample Dimensions. In addition, calibration file information is stored with the data file. Because the data, method information, and calibration information cannot be modified, a reliable audit trail is available, supporting regulatory and compliance requirements. The parameters saved with the data file include:

Raw Data:	Temperature, time, ordinate signal
Method:	Complete method used to collect the data
Calibration:	Name of the calibration file used

Method Validation

The **Method Validation Stamp** placed on a method indicates that it has been created under validated rules by qualified personnel (e.g., the System Administrator). The stamp is transferred with the method to any work site. When data are collected using a validated method, they are clearly “stamped” as such since the method is stored with the data. Any change made to the method (except to sample-specific information, such as weight/dimensions, sample ID, and operator ID) will automatically remove the validation mark. Lab managers can easily execute an audit trail and be assured that data were collected using a validated method, adhering to GLP and

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other regulatory requirements. Those users that do not have administrator status (from the Windows NT login) do not have the ability to validate methods.

Security

Microsoft Windows NT operating system provides a complete security system for desktop computers (C2-level security as defined by the United States National Security Agency). This allows you to set up specific user accounts and to password-protect these accounts. In addition, the level of security protection can be defined, providing further system security. Note that this security facility is available in Windows NT but not in Windows 95.

Pyris Software for Windows Compliance

To assist you in complying with laboratory standards, Pyris Software for Windows is developed under the formal requirements of the Perkin Elmer Quality System. The integrity of the Perkin Elmer Quality System is routinely audited and is certified by British Standards Institution as meeting the requirements of ISO 9001, the internationally recognized standard for Quality Assurance.

Remote Monitor

A powerful feature of the Pyris Software for Windows is that it can expand to a broad network of users. With the Remote Monitor optional software, you can monitor the real-time status of and stop a run on an analyzer on a remote computer via a network connection. In the remote computer's Remote Access page in Preferences, the user must select the amount of access to his analyzer(s) that a remote user is allowed. You can open a Remote Monitor window for each analyzer configured on the remote computer. In order to use the Remote Monitor feature, the security permission bits must be transferred from the Pyris Application Add On button to the Pyris Main security button. This is done at installation by your Service Engineer. Remote Monitor is part of the Data Analysis application. There is one Remote Monitor window per Data Analysis application. Once installed, the Remote Monitor option appears in the Tools menu.

PC Page

The PC page appears when you first open the Remote Monitor from the Tools menu. The page contains the following fields:

Enter a PC Name

Displays a list of computers available for monitoring from your computer via the Remote Monitor software. Only those computers whose names have been entered are displayed. To add a computer to the list, type the name of the computer in the entry field and click on the **Connect** button.

Delete from List

Removes the highlighted computer from the list of available PCs for monitoring.

Connect

Makes the connection to the selected PC and displays the Select Instrument page.

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Instrument Page

The Instrument page appears after you select a PC to monitor in the Select PC page or when you select the Instrument page tab. After you select the instrument and click on **OK**, the Instrument Viewer for the remote analyzer is displayed.

The Instrument page contains the following fields:

List of available instruments

Displays a list of all Pyris analyzers that are available on the computer selected in the PC page. Highlight an analyzer in the list or use the up and down keys to scroll the list. Grayed out items are currently unavailable.

OK closes the dialog box and enables the Remote Monitor for the highlighted analyzer.

Remote Instrument Viewer

The Instrument Viewer window in Remote Monitor displays the real-time curves of a run taking place on the remote analyzer that you selected in the Select Instrument page. If you have permission, given by the user of the remote computer to which the remote analyzer is attached, to stop a run on that analyzer, click on the **Stop Run** button. This is equivalent to the **Stop Run** button on the control panel.

The status panel displayed shows the status of the remote analyzer's parameters. You can change the items displayed just as you would in your own instrument's status panel.

Starting the Remote Monitor

To start the Remote Monitor for a Pyris analyzer:

8. **Open a Data Analysis Application**

   At your Pyris computer, open a Data Analysis application either by selecting **Data Analysis** from the **Start Pyris** Task menu, by clicking on the Data Analysis icon on the toolbar, or by selecting it from the Pyris Software for Windows group in the Programs menu displayed from the **Start** button. At the Open Data File dialog box select any data file in order to open the Data Analysis application.

9. **Open the Remote Monitor**

   Click on the **Remote Monitor** command in the Tools menu. The Select PC dialog box is displayed.

10. **Select a Computer to Monitor**

    With the PC tab selected, display the drop-down list of names of computers on the network that you can monitor. The first time you open the Remote Monitor, the name of only your computer is listed. This is the name you assigned when you installed Windows. Add a computer to the list by typing its name in the Name field. The next time you open the Remote Monitor, the computer name you entered will be displayed in the drop-down list. After entering the PC, click on **Connect**.

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11. **Select an Instrument to Monitor**

    After you select a computer to monitor, select the Instrument tab. All Pyris analyzers configured on the selected computer are listed in the drop-down list. Select the analyzer you want to monitor, then click on **OK**. Only analyzers that are connected and running can be selected. Analyzers that are configured but not running are grayed out and cannot be selected.

Viewing the Instrument Monitor and Status Panel

After selecting and connecting to the remote analyzer that you want to monitor, the Status Panel for that analyzer appears and the Instrument Viewer displays the remote analyzer's real-time curve. The Status Panel on your computer screen is exactly the same as what you see on the screen of the computer to which the monitored analyzer is attached. In addition, the Status Panel contains a **Stop Run** button at the left-hand side. If you have permission, you can stop a run on the remote analyzer by clicking on **Stop Run**. Permission is set in the Remote Access page of Preferences.

The Status Panel is dockable. The current values of several analyzer parameters, read from the analyzer, are displayed in the Status Panel. You can customize the Status Panel to display different parameters.

Display in the Instrument Monitor

1. If Remote Monitor is activated when the monitored analyzer is running, the curve(s) displayed will include data starting from the beginning of the run.
2. If Remote Monitor is activated when the monitored analyzer is not running, the curve(s) displayed will include data only from the time the remote connection is made.
3. If Remote Monitor is already on when a run begins on the monitored analyzer, the data is cleared and data from the beginning of a run is displayed.

Turning the Instrument Monitor and Status Panel Off

1. When both the Status Panel and the Instrument Monitor are on and then the Status Panel is toggled off, the remote connection is maintained. The Status Panel can be redisplayed by clicking on **Status Panel** in the View menu.
2. When both the Status Panel and the Instrument Monitor are on and then the Instrument Monitor is toggled off, the remote connection is maintained. The Instrument Monitor can be redisplayed by clicking on **Monitor** in the View menu.
3. When the Status Panel is displayed and the Instrument Monitor is off and then the Status Panel is toggled off, the connection to the remote analyzer is lost. The connection can be reestablished by using **Remote Monitor** in the Tools menu.
4. When the Instrument Monitor is displayed and the Status Panel is off and the Instrument Monitor is toggled off, the connection to the remote analyzer is lost. The connection can be reestablished by using **Remote Monitor** in the Tools menu.

Stopping a Run in the Remote Monitor

Select the **Stop Run** button in the Remote Monitor Status Panel to stop the sample run you are monitoring. When you select this button, a confirmation message appears; select **No** if you change your mind and do not want to stop the run.

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The **Stop Run** button may be inhibited by the permission setting on the PC that is running the analyzer. Permission is set in the Remote Access page in Preferences. Permission settings are (1) no access to the analyzer, (2) status monitoring only and curve display, and (3) status monitoring, curve display, and ability to stop a run remotely.

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Chapter 3
Configuration

Pyris Configuration

Before you can use Pyris Software for Windows to run experiments with your Pyris analyzers, you must configure the instruments. Configuration is dynamic, that is, when you add or remove an analyzer, you do not have to reboot the system in order for the configuration to take effect. The instrument's button is automatically added to or removed from the Pyris Manager panel.

You can run Pyris Configuration either during software installation or later, after Pyris software has been installed. You must run Pyris Configuration whenever you remove an analyzer from or add an analyzer to your system, or if you remove or add an accessory to the system.

You can open the Pyris Configuration utility by selecting Pyris Configuration in the Pyris Software for Windows folder in the Programs menu or by selecting Configure Analyzer from the **Start Pyris** button's Task menu. The utility must be able to detect the analyzer in order to configure it so the analyzer must be connected to the computer and powered on before you start configuration. Configuration is performed using the Pyris Configuration dialog box:

Analyzers/Ports Lists

Lists all configured analyzers and their respective COM ports. After you add an analyzer to your Pyris system, it appears in this list. The maximum number of analyzers permitted on a system is 10 if your computer has a multiport serial card installed. To edit or remove an analyzer, you must highlight it in this list and select the appropriate button.

Add Analyzer

Displays the Add Analyzer dialog box which is used to add an analyzer to your system's configuration. If you already have the maximum number of analyzers permitted on your system when you select **Add Analyzer**, you receive an error message.

Edit

Displays the selected instrument's Configuration dialog box in which you can add or remove accessories or change the instrument's name. The accessories available for that particular analyzer

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are listed with check boxes in addition to the available purge gas accessories in a drop-down menu. Select the gas accessory from the menu and then click in the following boxes to select or deselect that accessory.

Remove

Removes the highlighted analyzer from your system and the analyzer's entry from the Analyzers list. It also frees up the associated port. A confirmation message appears before the analyzer is removed.

Add Analyzer Dialog Box

When you add an analyzer to your system using Pyris Configuration, the system is automatically reconfigured so you do not have to close and then reboot your system in order for the change to take effect. Also, a button representing that instrument is added to the Pyris Manager panel.

To add an analyzer to your system, the instrument must be connected properly and powered on. Access the Pyris Configuration dialog box and select the **Add Analyzer** button. This displays the Add Analyzer dialog box. Select the communication port to which the analyzer is connected. The drop-down list will display all of the available ports on the PC, excluding those that have already been assigned to other instruments. You can configure one analyzer for each communication port on your computer. (If you have a 8-port serial card in your PC, COM3: refers to port 1 of the multiport card.)

After selecting the port, click on the **Add** button. The specific analyzer's Configuration dialog box appears in which you name your analyzer and configure accessories. The system detects the type of analyzer that is attached and displays the default name and other information. The Configuration dialog box is also displayed when you select **Edit** in the Pyris Configuration dialog box for a previously configured analyzer.

Pyris 1 DSC Configuration

The Pyris 1 DSC Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the Pyris 1 DSC analyzer is highlighted in the Analyzers list. It is also displayed when there is a Pyris 1 DSC attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a new Pyris 1 DSC, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

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Serial Number

You can enter the serial number of the analyzer for further identification; it is not a required entry.

Accessories

Lists the available accessories for the Pyris 1 DSC. The first selection is for the purge gas accessory. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

The next four accessories are

CryoFill: Click in the box if your Pyris 1 DSC is attached to the CryoFill LN2 Cooling System for subambient operation.

DPA 7 Photocalorimetric Accessory: This selection is grayed out because the accessory is not available for use with the Pyris 1 DSC at this time.

High-Pressure Cell: Select this accessory if you are going to use the high-pressure cell on the Pyris 1 DSC.

Autosampler: Select this accessory if the Pyris 1 DSC Autosampler is installed on your analyzer. If you connected the autosampler directly into the COM1 or COM2 port on the computer instead of to the Autosampler port on the Pyris 1 DSC, indicate that port in the **Port** field. Test the configuration by clicking on the Test button. The message "An autosampler was found" should be displayed if the autosampler is connected properly.

Firmware Version

Displays the version of firmware in the analyzer.

Update Flash EPROM

Select this button when you want to update the EPROM in the analyzer. This would occur if you receive an updated version of the software from Perkin Elmer. Clicking on the button displays the Pyris Flash ROM Utility dialog box in which you select the ROM file to use for updating the Pyris EPROM. Data are then transferred from the file to the analyzer's memory.

NOTE: Do not update the firmware at the same time that you add the analyzer to the configuration list. If you need to add the analyzer **and** update the firmware, first add the analyzer and close the Configuration dialog box. Then reopen the Pyris Configuration dialog box, highlight Pyris 1 DSC's name, and click on the **Edit** button. Click on **Update Flash EPROM**.

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Pyris Flash ROM Utility

Enter the path to the location of the update ROM file. The default directory where the current ROM file is located is displayed. Use the **Browse** button to select the A drive where the update floppy disk is to be placed. The ROM file on the floppy disk will be displayed; double click on it. That path will be displayed in the field. To quit without updating the flash ROM, click on **Cancel**. To begin the utility, click on the **Next** button. You will receive the message:

The instrument: PYRIS

will now be updated with the file:
***<path>\<filename.>*ROM>**

If this is correct, click on Finish to continue.

Click on the **Back** button if you want to change the path to the update file. Click on **Finish** to begin transfer of data from the disk to the ROM. The progress of the transfer is shown in percent. You can cancel the update at any time by clicking on the **Cancel** button.

Pyris 1 DSC High Pressure Cell

This accessory is used for performing heating, cooling, and isothermal measurements at elevated pressures. The measuring system operates with two independently controlled, platinum-iridium alloy sample holders that are incorporated into the Pyris 1 DSC.

The sample enclosure block is pressure-sealed by use of a high-pressure cell cover. The pressure of the accessory is 0 – 4200 kPa while the temperature range of the cell is 40°C – 500°C. Allowable purge gases for the system include oxygen, argon, helium, carbon dioxide, air, and any other inert or reactive gas. An ambient liquid cooling system can be attached to the sample holder.

DSC 7 Configuration

The DSC 7 Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the DSC 7 analyzer is highlighted in the Analyzers list. It is also displayed when there is a DSC 7 attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a DSC 7, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

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Serial Number

You can enter the serial number of the analyzer for further identification; it is not a required entry.

Accessories

Lists the available accessories for the DSC 7. The first selection is for the purge gas accessory. Click on the drop-down menu arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

The other accessories available for selection by clicking on the check box are

CCA 7 Controlled Cooling Accessory: The CCA 7 allows low-temperature operation of the DSC 7 to –140°C (and to –70°C with a DSC 7 Robotic System).

High-Pressure Cell: This accessory is used for performing DSC experiments at elevated pressures. It has built-in connections for a liquid cooling system such as tap water or a circulating system.

DPA 7 Photocalorimetric Accessory: The DPA 7 allows samples contained in the DSC 7 to be exposed to full spectrum or individual wavelength UV light. Pyris-controlled shutter provides reproducible exposure times. Multiple lamp source capability allows selection of the appropriate UV lamp for experiments. Selection of this accessory is reflected in the Program page of the Method Editor where the **Gas Change** button changes to **Lamp Change**.

Autosampler: Select this accessory if you are using the DSC 7 Robotic System. If you connected the autosampler directly into the COM1 or COM2 port on the computer via an RS 232 cable instead of to the AUX port on the TAC 7/DX, indicate that port in the **Port** field.

Firmware Version

Displays the version of firmware in the analyzer.

Pyris 6 DSC Configuration

The Pyris 6 DSC Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the Pyris 6 DSC analyzer is highlighted in the Analyzers list. It is also displayed when there is a Pyris 6 DSC attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a Pyris 6 DSC, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name

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identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

Serial Number

You can enter the serial number of the analyzer for further identification; it is not a required entry.

Accessories

Lists the available accessories for the analyzer. For the Pyris 6 DSC, the only accessory is for purge gas. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

No Gas Accessory

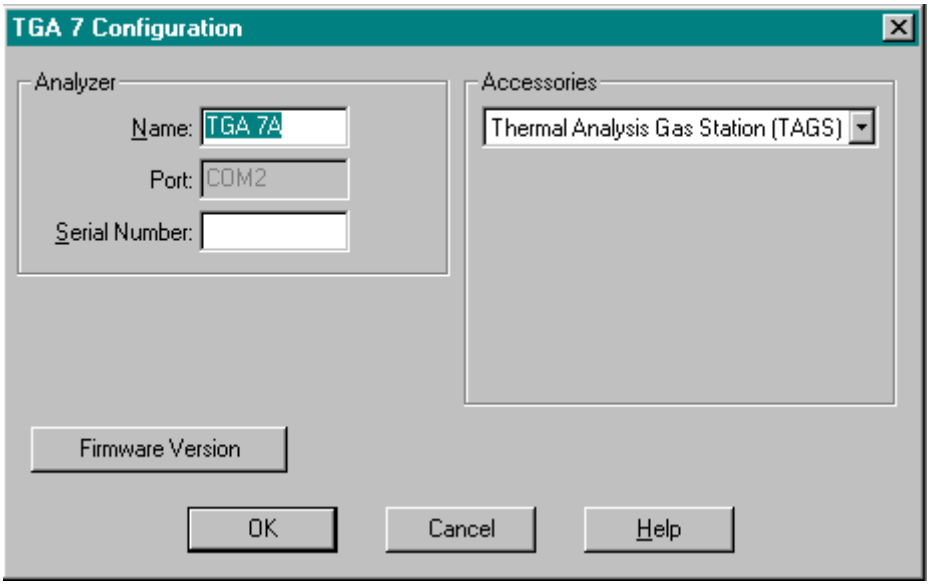
Firmware Version

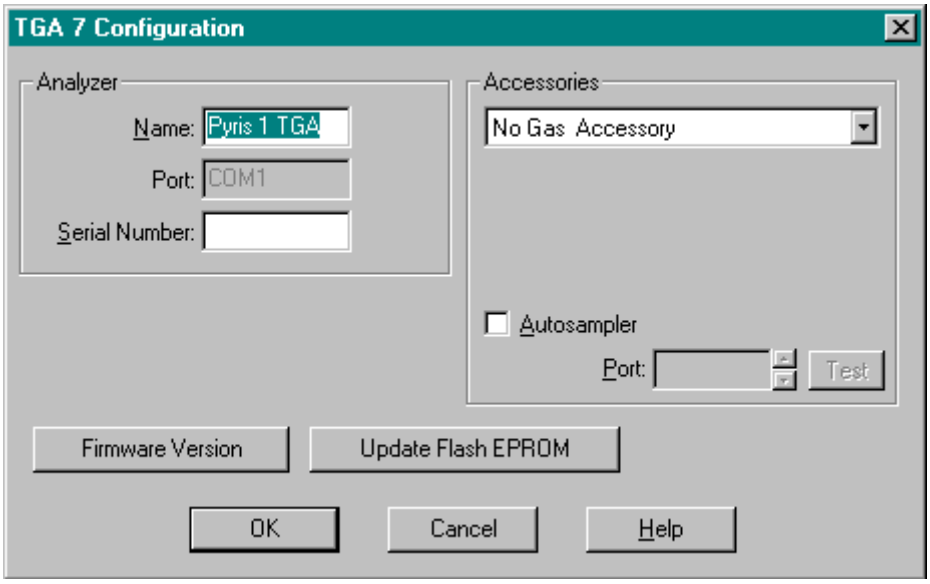
Displays the version of firmware in the analyzer.

TGA 7 and Pyris 1 TGA Configuration

The Configuration dialog box for a TGA 7 or a Pyris 1 TGA appears when you select the **Edit** button in the Pyris Configuration dialog box when either analyzer is highlighted in the Analyzers list. It is also displayed when there is a TGA 7 or a Pyris 1 TGA attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

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Name

If you are adding a TGA 7, the system will display the default name TGA 7 in this field. For a Pyris 1 TGA, it assigns Pyris 1 TGA. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port that you selected in the Add Analyzer dialog box to which the analyzer is attached.

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Serial Number

You can enter the serial number of the analyzer for further identification; it is not a required entry.

Accessories

Lists the available accessories for the analyzer. For the TGA 7 and Pyris 1 TGA, the only accessory in the list is for purge gas. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

Autosampler: This box appears for a Pyris 1 TGA. Click in the check box if an autosampler is attached to the analyzer. The **Port** field should be the same as that for the analyzer. Click on the **Test** button to have the system check for the presence of the autosamper. A clock cursor will be displayed while the system goes out and locates the autosampler. A message stating that it was found should be displayed.

Firmware Version

Displays the version of firmware in the analyzer.

Update Flash EPROM

Select this button when you want to update the EPROM in the analyzer. This would occur if you receive an updated version of the software from Perkin Elmer. Clicking on the button displays the Pyris Flash ROM Utility dialog box in which you select the ROM file to use for updating the Pyris EPROM. Data are then transferred from the file to the analyzer's memory.

NOTE: Do not update the firmware at the same time that you add the analyzer to the configuration list. If you need to add the analyzer **and** update the firmware, first add the analyzer and close the Configuration dialog box. Then reopen the Pyris Configuration dialog box, highlight Pyris 1 TGA's name, and click on the **Edit** button. Click on **Update Flash EPROM**.

Pyris 6 TGA Configuration

The Pyris 6 TGA Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the Pyris 6 TGA analyzer is highlighted in the Analyzers list. It is also displayed when there is a Pyris 6 TGA attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

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Name

If you are adding a Pyris 6 TGA, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

Serial Number

You can enter the serial number of the analyzer for further identification; it is not a required entry.

Accessories

Lists the available accessories for the analyzer. For the Pyris 6 TGA, the only accessory is for purge gas. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

No Gas Accessory

Firmware Version

Displays the version of firmware in the analyzer.

DMA 7e Configuration

The DMA 7e Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the DMA 7e analyzer is highlighted in the Analyzers list. It is also displayed when there is a DMA 7e attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a DMA 7e, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

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Accessories

Lists the available accessories for the DMA 7e. The first selection is for the purge gas accessory. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

Force Motor

The linear force motor in the DMA 7e provides precise control of all forces applied to the sample. The high resolution of the force motor allows for precise motor controls. There are two force motors available. Click on the appropriate radio button to indicate the force motor in your DMA 7e. The force motor in your DMA 7e cannot be changed.

Furnace Size

Two types of low-mass furnace are available for the DMA 7e and each can be programmed for high and low temperatures. When combined with the appropriate cooling accessories, you can program the DMA 7e to –170°C. The low mass furnace design permits a rapid cool down at the end of the experiment. Select the size of the furnace currently installed in your DMA 7e. You can change the furnace when necessary but you must edit the configuration by changing the furnace size in this box.

Firmware Version

Displays the version of firmware in the analyzer.

TMA 7 Configuration

The TMA 7 Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the TMA 7 analyzer is highlighted in the Analyzers list. It is also displayed when there is a TMA 7 attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a TMA 7, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

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Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

Accessories

Lists the available accessories for the TMA 7. The first selection is for the purge gas accessory. Click on the drop-down arrow to display the available choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

Force Motor

The linear force motor in the TMA 7 provides precise control of all forces applied to the sample. The high resolution of the force motor allows for precise motor controls. There are two force motors available. Click on the appropriate radio button to indicate the force motor in your TMA 7. The force motor in your TMA 7 cannot be changed.

Furnace Size

Two types of low-mass furnace are available for the TMA 7 and each can be programmed for high and low temperatures. When combined with the appropriate cooling accessories, you can program the TMA 7 to –170°C. The low mass furnace design permits a rapid cool down at the end of the experiment. Select the size of the furnace currently installed in your TMA 7. You can change the furnace when necessary but you must edit the configuration by changing the furnace size in this box.

Firmware Version

Displays the version of firmware in the analyzer.

DTA 7 Configuration

The DTA 7 Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the DTA 7 analyzer is highlighted in the Analyzers list. It is also displayed when there is a DTA 7 attached to the computer and it is detected by the Pyris software when you select the **Add** button in the Add Analyzer dialog box. The analyzer must be powered on when you configure it into the system; otherwise, it will not be recognized when you select the **Add** button. The fields in this dialog box are as follows:

Name

If you are adding a DTA 7, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the

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analyzer in Pyris Software for Windows; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port

Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box.

Accessories

Lists the available accessories for the DTA 7. The only selection is for the purge gas accessory. Click on the drop-down arrow to display the list of choices:

Thermal Analysis Gas Station (TAGS): Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one gas to another at selected times or temperatures. The flow rate can also be changed.

GSA 7 Gas Switching Accessory: Select this accessory if you are using a GSA 7. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the GSA 7 switch the purge gas at a selected time.

No Gas Accessory

Firmware Version

Displays the version of firmware in the analyzer.

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Chapter 4
Pyris Files

All methods, collected data, analyzed data, calibration values, and play lists are stored by Pyris Software for Windows as files on your computer or network. You can open and close, save, delete, rename, copy, or move any of the files you use with Pyris Software for Windows. These are the four types of Pyris files: methods, data, calibration, and play list.

Method Files

A method contains all of the experimental parameters for a sample run. It is stored on your computer or network as a file and can be recalled for later use. To use a method for a sample run, select **Open Method** from the File menu in Instrument Application. Choose the desired method file from the displayed dialog box. You can then edit the method, create a new method based on the open method, and start a run using the current method. You can save your method files anywhere on your computer or network.

The default directory for method files is C:\Program Files\Pyris\Methods. During installation you can accept the default directory for software files, i.e., C:\Program Files\Pyris, or you can select another location. If you put the Pyris software at a location other than the default, then the Methods subdirectory will also be located under that new location. You can also change the default directory for the Methods in the Save page of Preferences.

Method files have the following three-character file name extensions:

DSC 7 Methods:	**.dsm**
DDSC Methods:	**.ddm**
Pyris 1 DSC Methods:	**.dcm**
Pyris 6 DSC Methods:	**.d6m**
TGA 7 Methods:	**.tgm**
High Temperature TGA 7 Methods:	**.thm**
Pyris 6 TGA Methods:	**.t6m**
Pyris 1 TGA Methods:	**.tg1m**
High Temperature Pyris 1 TGA Methods:	**.th1m**
DMA 7e Methods:	**.dmm**
DMA 7e (28-mm Furnace) Methods:	**.d8m**

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TMA 7 Methods:	**.tmm**
TMA 7 (28-mm Furnace) Methods:	**.t8m**
DTA 7 Methods:	**.dtm**

Open Method Command

Use this command on the File menu to open an existing method. When you select this command, the Open Method dialog box appears. You can also access the Open Method dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Save Method Command

Use this command on the File menu to save the method currently loaded in the Method Editor. If the method is new and has not yet been saved, the Save As dialog box appears. You can save the method quickly by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Print Command

Use this command in the File menu to print the current method file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box by typing **Ctrl + P** or by clicking on the **Print** button on the toolbar: 

Calibration Files

The values used to calibrate a Pyris analyzer can be stored on your computer or network as a calibration file and recalled for later use. The default directory for calibration files is C:\Program Files\Pyris. Multiple calibration files can be saved for different analyzer conditions.

Calibration files have the following three-character file name extensions:

DSC 7 Calibration:	**.dsc**
DDSC Calibration:	**.ddc**
Pyris 1 DSC Calibration:	**.dcc**
Pyris 6 DSC Calibration:	**.d6c**
TGA 7 Calibration:	**.tgc**
High Temperature TGA 7 Calibration:	**.thc**
Pyris 6 TGA Calibration:	**.t6c**
Pyris 1 TGA Calibration:	**.tg1c**
High Temperature Pyris 1 TGA Calibration:	**.th1c**
DMA 7e Calibration:	**.dmc**
DMA 7e (28-mm Furnace) Calibration:	**.d8c**
TMA 7 Calibration:	**.tmc**
TMA 7 (28-mm Furnace) Calibration:	**.t8c**
DTA 7 Calibration:	**.dtc**

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Open Command

Use this command on the File menu to open an existing calibration file. When you select this command, the Open dialog box appears. You can also access the Open dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Save Command

Use this command on the File menu to save the current calibration file. When you select this command, the file is saved automatically and will overwrite the existing file. If the calibration is new and has not yet been saved, the Save As dialog box appears. If you want to save the calibration file under a different file name, use **Save As**. You can also save the file by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Print Command

Use this command to print the current calibration file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box by typing the shortcut keys **Ctrl + P** or by clicking on the **Print** button on the toolbar: 

Data Files

Data collected during a sample run is stored on your computer or network as a data file. The name and location of the data file are specified in the method used for that sample run. The default directory for data files is C:\Program Files\Pyris\Data. To analyze previously collected and stored data, activate Data Analysis by selecting the **Data Analysis** button on the toolbar, by selecting it from the Start Pyris Task menu, or by selecting Data Analysis from the Pyris Software for Windows folder in the Programs menu. Select a specific data file from the dialog box displayed. You can open another data file by selecting **Open Data** from the File menu.

Data files have the following three-character file name extensions:

DSC 7 Data:	**.dsd**
DDSC Data:	**.ddd**
Pyris 1 DSC Data:	**.dcd**
Pyris 6 DSC Data:	**.d6d**
TGA 7 Data:	**.tgd**
High Temperature TGA 7 Data:	**.thd**
Pyris 6 TGA Data:	**.t6d**
Pyris 1 TGA Data:	**.tg1d**
High Temperature Pyris 1 TGA Data:	**.th1d**
DMA 7e Data:	**.dmd**
DMA 7e (28-mm Furnace) Data:	**.d8d**
TMA 7 Data:	**.tmd**
TMA 7 (28-mm Furnace) Data:	**.t8d**
DTA 7 Data:	**.dtd**

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File Name Format

If a method in a Pyris Player play list is used for more than one sample, or if you use the Sample List feature in Pyris Player, e.g., in a Sample Group for an autosampler, enter a file name with the format <*user-defined name*>###. The ### tells the software to append a number, starting with 000, to the end of the file name. The software pads the number with zeros to the left as needed. If you enter a file name in the File Name field in the Sample Info page of a method or in a Pyris Player play list and use these symbols, then each time the method is run, the software increments the file name by 1. If you do not enter # signs at the end of the user-defined part of a file name and the method is used in a play list, or even when used in a stand-alone manner, the software will append a date – time stamp to the user-defined file name to create a unique file name for each run. The format of the file name is <*user-defined name*>@YYMMDDHHMMSS.xxD, where YY is the year, MM the month, DD the day, HH the hour (on a 24-hour clock), MM minutes, and SS seconds.

File Location

You can save your data files anywhere on your computer or network. During software installation, you can accept the default directory for the software files, i.e., C:\Program Files\Pyris. This means that data files are stored in C:\Program Files\Pyris\Data. You can identify a different default directory during installation of Pyris Software for Windows. The default directory can be changed in the Save page of Preferences.

Open Data Command

This command on the File menu is used to open an existing data file in a **new** Data Analysis window. All other open Data Analysis windows remain open. From the Open Data File dialog box, select the new data file you wish to display. You can also access the Open Data File dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the standard toolbar: 

Save Data Command

Select this command from the File menu to save the active curve and all derived curves, results, constructs, and annotations using the current file name. The data file is saved automatically without requesting confirmation, so be sure you want to save the file as is. If you want to save the data under a different file name, select **Save Data As**. You can also save the data file quickly by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Print Command

This command from the File menu is used to print the data file. For example, if you are in the Data Analysis window, the Instrument Viewer, or Remote Monitor and select **Print**, the loaded data file will be printed. The standard Print dialog box appears when you select this command. You can also access the Print dialog box by typing **Ctrl + P** or clicking on the **Print** button on the toolbar: 

Play List Files

The play lists you create in Pyris Player can be stored on your computer or network as play list files and recalled for later use or editing. A play list contains a sequence of commands that automate runs on instruments with autosamplers. Play lists also can be used with analyzers without an autosampler for automating postrun data analysis.

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You can save your play list files anywhere on your computer or network. During software installation, you can accept the default directory for the software files, i.e., C:\Program Files\Pyris. This means that play list files are stored in C:\Program Files\Pyris\Player Lists. You can identify a different default directory during installation of Pyris Software for Windows. The default directory can be changed in the Save page of Preferences.

You can also import data files from 7 Series/UNIX instruments or from PC Series instruments with the data conversion software.

Play list files have the following three-character file name extensions:

DSC 7 Play List:	**.dsp**
DDSC Play List:	**.ddp**
Pyris 1 DSC Play List:	**.dcp**
Pyris 6 DSC Play List:	**.d6p**
TGA 7 Play List:	**.tgp**
High Temperature TGA 7 Play List:	**.thp**
Pyris 6 TGA Play List:	**.t6p**
Pyris 1 TGA Play List:	**.tg1p**
High Temperature Pyris 1 TGA Play List:	**.th1p**
DMA 7e Play List:	**.dmp**
DMA 7e (28-mm Furnace) Play List:	**.d8p**
TMA 7 Play List:	**.tmp**
TMA 7 (28-mm Furnace) Play List:	**.t8p**
DTA 7 Play List:	**.dtp**

Open Player Command

Use this command to open an existing play list file. When you select this command, the Open dialog box appears. You can also access the Open dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Save Player Command

Use this command to save the current play list file. When you select this command, the file is saved automatically and will overwrite the existing file. If the play list is new and has not yet been saved, the Save As dialog box appears. If you want to save the play list file under a different file name, use the Save As command. You can also save the file by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Print Command

Selecting **Print** from the File menu or the Print button on the toolbar from any Pyris Player page initially displays a dialog box from which you choose the type of printout you want: summary or detailed. The summary printout includes the main-level items Prepare Sample, Data Analysis, and Sample Group which includes the Sample List and the Data Analysis List entries. The detailed printout includes the same information as a Summary printout but also includes additional information on the Sample List entries in a Sample Group.

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When **Detail** is selected in the Print dialog box for View History, the same information as given in Summary is given in addition to the date and time each event occurred.

History Summary yields a printout that includes just one-line entries for each time the play list was played back. The entry gives the date and time of the run.

In all cases, click on the **Print** button in the dialog box to display the standard Print dialog box.

You can also access the initial printing dialog box by typing the shortcut keys **Ctrl + P** or by clicking on the **Print** button on the toolbar with any of the six pages displayed: 

Data File Conversion

Data collected by 7 Series/UNIX analyzers can be imported to your Pyris system and converted for analysis by using the Pyris Import and Export utilities. To convert non-Pyris data files to Pyris data files, perform the following steps:

- Install the Export utility on the 7 Series/UNIX Workstation
- Run the Export utility
- Transfer the ANF files to floppy disks
- Transfer the ANF files to the Pyris Software for Windows workstation
- Convert the ANF files to Pyris data files

Install the Export Utility on 7 Series/UNIX Workstation

NOTES: The 7 Series/UNIX workstation must have SCO UNIX ODT version 2.0. This procedure needs to be performed only **once**.

1. From the blue menu in the 7 Series/UNIX software, select **Enter UNIX operating system** and press **Enter**.
2. Insert the Export floppy disk into the floppy drive of the UNIX computer.
3. At the TA: prompt, type

   cd /usr/tas7/run and press **Enter**.
4. At the TA: prompt, type

   tar -xv6 and press **Enter**.
5. Remove the Export disk from the floppy drive.

Run the Export Utility

1. From the blue menu in the 7 Series/UNIX software, select **Enter UNIX operating system**.
2. At the TA: prompt, type

   cd /usr/tas7/run and press **Enter**.
3. At the TA: prompt, type

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chmod +x export

export and press **Enter**.

4. If you want to export any DSC data files, at the prompt for DSC data files, type

   y and press **Enter**.

   NOTE: Files previously converted are not removed. The system will prompt you to remove them; answer "Y" to remove the previously converted files.

   If you do not want to export any DSC data files, type

   n and press **Enter**.

5. If you elected to export DSC files, you are prompted as follows for each data file:

   DSC 7 file <filename> [c=convert s=skip a=all q=quit]:

   Type **c** to convert the file.

   Type **s** to skip the file.

   Type **a** to convert all of the DSC files for which you have not yet been prompted.

   Type **q** to stop converting any more data files and quit.

6. At the prompt for the other data file types, follow the same procedure as followed for the DSC data files.

   NOTE: ANF files are stored in the /usr/tas7/anf directory, in subdirectories for each instrument type.

Next you have to transfer the .ANF files to floppy disks in order to copy them on to the Pyris PC.

Transfer the ANF Files to Floppy Disks

1. Insert a DOS-formatted floppy disk into the 7 Series/UNIX computer's disk drive.
2. At the TA: prompt, type the following lines; press **Enter** at the end of each line.

   cd /usr/tas7/anf

   dos

   e:

   cd \dos

   backup c: a: /s

3. Follow the instructions on the screen.

   NOTES: If more than one disk is required, you will be prompted to insert additional disks. Make sure disks are numbered to indicate the order of backup.

   You can ignore the message "Warning! Files in the target drive a:\root will be erased."

4. When complete, return to the 7 Series/UNIX Main Menu by typing the following lines, ending each by pressing **Enter**.

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> cd..

> quit

> quit

Now you have to copy the files on the floppy to the Pyris PC.

Transfer ANF Files to Pyris Workstation

NOTE: This procedure needs to be done only once.

1. Select **Command Prompt** from the Programs group in the Start menu of Windows 95 or NT.
2. Type the following lines at the DOS prompt:

   > md \usr and press **Enter > restore a: c:\ /s** and press **Enter**
3. Insert floppy disks in the disk drive when prompted.

   NOTE: If you have more than one disk, disks must be entered in the order of the backup.
4. When complete, type Exit to close DOS.

Finally, you can convert the .ANF files to Pyris data files.

Converting ANF Files to Pyris Data Files

In a Data Analysis Application, you can load in data files that were obtained on non-Pyris analyzers. These files must first be run through the Export utility on the non-Pyris analyzer. This creates ANF files. These files are copied to floppy disks which are used to copy the ANF files to the Pyris computer.

If you want to use an ANF file from a non-Pyris analyzer that has been imported to your Pyris computer:

1. Select the **Convert ANF File** command from the Tools menu in a Data Analysis Application. Alternatively, select **Open Data** from the File menu. The Open Data File dialog box is displayed.
2. In the Files of Type list box, select ANF Data (*.anf) to display files with the ANF extension.
3. Select the file to convert and click on **OK**.

The conversion occurs automatically; the converted file has the data file extension associated with the instrument that collected it. The file is placed in the same directory as the .ANF file. The curve is displayed in the Data Analysis window. If you used the File/Open Data route to conversion, the files will be displayed after conversion is complete. If you opened a Data Analysis application while in an Instrument Application, the ANF file that you convert must have been collected on the same type of instrument.

Note that 7 Series/UNIX results and Advanced Calculations are **not** imported, but all the data and method information is converted.

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PC Series Data File Conversion

In a Data Analysis Application, you can load in data files that were obtained on a PC Series system. The PC Series data files that can be converted are DSC 7 Temperature (.D7), Isothermal (.I7), and Specific Heat (.C7), TGA 7 (.G7), and TMA 7 (.T7). The data files from such a system are in ASCII form and are easily converted to Pyris-compatible format. Select **Convert PC-Series File** from the Tools menu in a Data Analysis Application. The Convert PC Series Data File dialog box is displayed. In the Files of type list, select the type of file you wish to convert. If necessary, use the **Browse** button to find where the PC Series data files are located (e.g., a floppy disk or on the network). Select the file to convert and click on **OK**. The conversion occurs immediately; the converted file has the extension associated with the instrument that collected it. The file is saved in the same directory as the PC Series file. You must retrieve the file using **Open Data** or **Add Data** from the File menu.

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Chapter 5
Instrument Applications

Pyris Software for Windows has two main parts: Instrument Applications and Data Analysis. Each instrument on your system has its own Instrument Application, which is accessed by clicking on the analyzer's button on the Pyris Manager bar. The main parts of an Instrument Application are

- Instrument Viewer
- Method Editor
- Data Analysis
- Pyris Player
- Status Panel
- Control Panel

To move from one part of the Instrument Application to another, you can use the toolbar buttons:


Instrument Viewer


Method Editor


Data Analysis


Pyris Player

Instrument Viewer

The Instrument Viewer is a window that displays the real-time signals from the analyzer as data are collected during a run. You can select the curves (signals) for display from the Curves menu. If there is no run in progress, the Instrument Viewer can be used to monitor the status of the instrument by activating **Monitor** from the View menu. If you click on the **Reset Monitor** button in the control panel, the display in the Instrument Viewer is cleared and monitoring begins again. The display of the curves can be optimized by using the rescale tools on the Rescale Tools toolbar.

The Instrument Viewer window contains a title bar, control box with the standard Control menu, **Minimize** and **Maximize** buttons, and a border that can be used for resizing. The curves displayed in the window can be changed by selecting items on the View, Display, and Curves menus.

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Method Editor

The method is a set of parameters that determine the conditions under which the system will collect data on a sample. Any number of methods can be created and stored so that the parameters can be optimized to handle different analyses. Methods are also used in play lists. The play list can be set up to run many samples using the same method via the sample group. This is especially convenient for use with an autosampler. The Method Editor comprises the following pages, each of which contains its own set of parameters:

- Sample Info Page
- Initial State Page
- Program Page

Each of these pages are described in detail for the individual instrument since the parameters displayed in them are instrument specific.

Another feature of the Method Editor is the **Thermal Program Window.** The Thermal Program window appears when you select the **Thermal Program** command in the View menu of the Method Editor. It displays the current method program graphically on a time – temperature plot. The Thermal Program display is automatically updated as you edit the current method program steps. The Thermal Program window stays on top of the other windows; move it away from the Program page by clicking on the title bar and dragging it to a new position. To close the Thermal Program window, double click on the control box of the window or select the **Thermal Program** command again.

Thermal Program Window

The Thermal Program window appears when you select the **Thermal Program** command in the View menu of the Method Editor. It displays the current method program graphically on a time – temperature plot. The Thermal Program display is automatically updated as you edit the current method program steps. The Thermal Program window stays on top of the other windows; move it away from the Program page by clicking on the title bar and dragging it to a new position. To close the Thermal Program window, double click on the control box of the window or select the **Thermal Program** command again.

Data Analysis

The Data Analysis window is opened one of two ways: First, you can select Data Analysis from the **Start Pyris** button Task menu or from the Pyris Software for Windows group in the Programs menu. The window displays the last data file displayed there. Use the options on the File menu to display data files collected by different analyzers.

Second, the Data Analysis window is opened while in the Instrument Viewer or Method Editor by selecting the **Data Analysis** button  from the toolbar. From the Open Data File dialog box select the data file you want displayed. Use the items in the File menu to open, add, close, and save a data file, or to print the display.

The View, Display, Curves, Math, and Calc menus are used to change the display and the type of curve displayed and to perform calculations on the data for additional information.

The Data Analysis window also contains the standard window items: title bar, control box with Control menu in the upper-left-hand corner, **Minimize** and **Maximize** buttons, and a border that can be used for resizing.

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Pyris Player

The Pyris Player is used to create play lists or sequences of commands that automate data collection and postrun data analysis functions. For an instrument without an autosampler, you can create a play list that prompts you through the steps necessary to run a sample or a series of samples (i.e., Load Sample, Start Method, Remove Sample) and then performs a series of analyses on the data files, displays the results, and prints the results. If your analyzer has an autosampler, the play list's Sample Group feature, which includes a Sample List and a Data Analysis List, automates running samples in the autosampler's tray. If your analyzer is a Pyris 1 TGA with an autosampler or a Pyris 6 TGA with an AS 6 autosampler, Pyris Player can be used to tare all or specific sample pans and weigh all or specific sample pans in the autosampler tray. The Sample List is a good way to group similar samples together to be analyzed using the same method. The Sample List can run the samples in the autosampler in any order. The items in the Data Analysis List can take the data file from the current run, preceding play list items, and existing files and perform the same procedures that are available in regular Data Analysis in a play list.

While a play list is running, you can perform other functions on your computer or pause the play list and edit the entries below the current line. A complete history file is generated that contains the record of success or failure or each item in the list. If an error occurs, a reason is listed. There also is a sample history page that displays past and current information on the samples in the sample groups in the current play list.

The parts of Pyris Player are

- Setup Page
- Edit Play List Page
- View List Page
- View Sample List Page
- View History Page
- Sample History Page
- Player Toolbars

Each of these pages is discussed in the Pyris Player chapter.

Status Panel

The status panel is a dockable panel in an Instrument Application that displays the real-time status of selected parameters of the analyzer. For example, if you click on the **Zero Height** button on the DMA 7e control panel, the reading is displayed in the Probe Position box in the status panel.

The panel is resizable so that you can display as many parameter boxes as desired. You can toggle the status panel on and off by selecting **Status Panel** from the View menu. Each parameter is displayed in a box that contains the parameter name in a drop-down list and the current value.

The parameters available for display depend on the analyzer you are using. To select a parameter to display in the status panel, you can highlight the display field and then type in the first letter of the parameter. This will display the first parameter that begins with that letter. Continue to type that letter to scroll through all parameters that begin with that letter. For example, for the DTA 7, if you type the letter “f” when a status panel display field is highlighted, you will scroll through Furnace Cover, Furnace Lock, Furnace Status, and Furnace Temperature. You can also select a parameter by displaying the drop-down list and highlighting the desired item.

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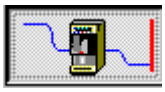
Control Panels

Each analyzer has is its own control panel. However, some of the buttons on the control panels are common to all analyzers. These buttons are listed below. Analyzer-specific buttons are then discussed.

Start Method Button



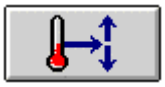
Stop Method Button



The **Start Method** button is an icon of the instrument with a red vertical line on the left side. Click on the button to start the run using the method loaded in the Method Editor. When the run starts, the Instrument Viewer becomes the active window, the run begins, and the button changes to the **Stop Method** button. The **Stop Method** button has the red vertical line on the right side. When you click on the **Stop Method** button, the method's end condition is executed, the data are saved, the curve(s) is redrawn in the Instrument Viewer (if Auto-Rescale was selected in the Display menu), and the button changes back to **Start Method.**

If the instrument is not powered on or not connected to the computer, the **Start/Stop Method** button is replaced by the following:



Go To Temperature Button 

This button programs the analyzer to the user-entered temperature displayed in the Go To Temperature entry field. If a run is in progress when you select this button, the run will terminate and the analyzer will heat or cool. For a DMA 7e or TMA 7, the stresses (or forces) and frequency will remain the same. If a run is not currently in progress when this button is selected, then the analyzer will heat or cool to the temperature entered in the Go To Temperature entry field.

Go To Temperature

Enter the temperature in °C to which the analyzer will be programmed when the **Go To Temperature** button is selected.

Go To Load Button 

This button on the control panel automatically initiates heating or cooling the analyzer to the Load temperature specified in the Instrument page of Preferences. For a DMA 7e or TMA 7, the Load conditions (stress or force and frequency) are also applied. The Load Static and Dynamic Forces and Load Frequency are also entered in the DMA 7 page in Preferences.

If a run is in progress, selecting this button will terminate the run and heat or cool the analyzer to the Load temperature and apply the Load forces and frequency. If the default stresses or forces are set to zero, clicking on **Go To Load** turns off the motor.

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Hold Temperature Button 

This button automatically holds the analyzer at the current temperature. If a run is in progress when this button is selected, the run is terminated and the analyzer is held at the current temperature. For a DMA 7e or a TMA 7, the stresses or forces and frequency will remain the same. The stresses or forces and frequency will remain the same.

Reset Monitor Button 

Clears the display in the Instrument Viewer window and restarts the monitor. When there is no run in progress, the monitor displays the baseline signals from the analyzer. When a run is in progress, the monitor displays the real-time signals as the data are collected.

Purge Gas

Use the drop-down list to switch the purge gas. This is available when you have a GSA 7 or a TAGS accessory attached to your analyzer and configured in the system. (Only the TAGS is available for the Pyris 6 DSC and Pyris 6 TGA.) The gases available are entered in the Purge Gases page of Preferences. If you are not using a gas switching accessory, the Purge Gas field is blank. Execute the purge gas change by clicking on the **Apply** button. The purge gas will flow at the displayed rate which was set in Preferences. It cannot be changed here.

Apply Button

After selecting a purge gas from the Purge Gas drop-down list on the control panel, execute the change by clicking on the **Apply** button. The output from the GSA 7 or TAGS will automatically be switched to the selected purge gas.

Purge Gas Flow Rate

This is the flow rate for the purge gas selected in the Purge Gas field. This is the Initial Flow Rate value that was entered in the Purge Gas page of Preferences and cannot be changed here.

 Pyris 1 TGA Control Panel

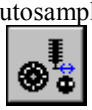
In addition to the common control panel buttons listed above, the Pyris 1 TGA control panel contains the following controls:

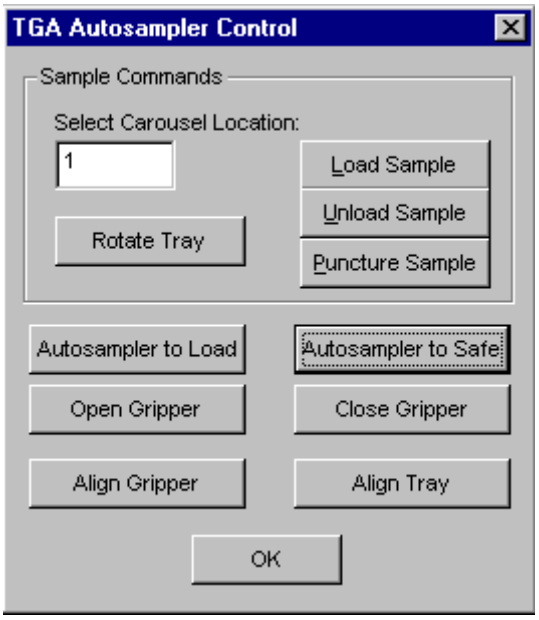
Autosampler Control Button 

Displays the Autosampler Control dialog box if an autosampler is installed on the Pyris 1 TGA. You can use the commands in the dialog box to control certain movements of the autosampler to handle an individual sample. If no autosampler is present, the button is grayed out.

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Pyris 1 TGA Autosampler Control Dialog Box

The Autosampler Control dialog box is displayed when you click on the Autosampler Control button  on the Pyris 1 TGA control panel. Use this dialog box to control the autosampler when there is an error during a run and you need to move the autosampler or access the sample pans in the tray. Through this dialog box you can also perform gripper and tray alignment, which should be done when you change a hangdown wire. Close the dialog box by either clicking on **OK** or the **Close** button in the title bar.



Load Sample

When you click on **Load Sample**:

1. The autosampler moves from the Safe to the Load position.
2. The tray rotates to position the specified location beneath the hangdown wire.
3. The sample pan is loaded onto the wire.
4. The autosampler moves back to the Safe position.

Unload Sample

When you click on **Unload Sample**:

1. The autosampler moves from the Safe to the Load position.
2. The tray rotates to position the specified location beneath the hangdown wire. Make sure that the position is empty.
3. The sample pan is returned to the tray.
4. The autosampler moves back to the Safe position.

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Puncture Sample

This button is used when your Pyris 1 TGA has the puncture device installed. The autosampler will position the specified carousel location underneath the puncture device's pin and rise up so that the pin punctures the sealed sample pan. You can then proceed to run the sample in a play list or separately.

Rotate Tray

This command rotates the tray so that the carousel location entered in the Select Carousel Location field faces the hangdown wire. The command works with the autosampler in the Load or the Safe position. Rotate Tray is convenient way to access position 1, for example, to load a new sample into the crucible. Just enter 11 into the Location field and click on Rotate Tray. The autosampler rotates 180° so that position 1 faces out for easy access.

Autosampler to Load

When the autosampler is in the Safe position (to the side and away from the hangdown wire), you can move it to the Load position, which is underneath the hangdown wire using this button. The default position is with location 1 underneath the hangdown wire.

Autosampler to Safe

Move the autosampler away from the furnace's path by clicking on this button. The autosampler moves to the right and away from the hangdown wire.

Align Gripper

Click on this button to display the first screen of the Gripper Alignment wizard (see Chapter 7). The wizard is a series of screens that walk you through aligning the gripper with the hangdown wire. This procedure should be performed when you change the hangdown wire or when it appears that the gripper is not grasping the hangdown wire properly.

Align Tray

Click on this button to display the first screen of the Tray Alignment wizard (see Chapter 7). The wizard screens walk you through the procedure for aligning the handle of each crucible in the tray with the hangdown wire so that it is picked up and unloaded properly.

Clean Furnace Button 

Executes the Clean Furnace procedure for the Pyris 1 TGA. If a run is in progress when you select this button, the run ends immediately. Pyris 1 TGA also has a Clean Furnace selection from its control display.

The clean furnace procedure for the Pyris 1 TGA involves lowering the furnace to expose it to the air and then heating it to approximately 900°C to burn off any materials coating the furnace surface.

WARNING: Since the furnace will be programmed to 900°C, make sure that the protective plastic visor is in the down position. DO NOT touch the furnace during this procedure.

With the control panel displayed, click on the **Clean Furnace** button. The furnace moves to the lowered position and the temperature is programmed to 900°C. When the cleaning procedure is complete, the furnace moves to the cool position and cools down to 30°C.

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Sample Weight Button 

Tells the analyzer to read the weight of the sample in the sample pan and display it in the Weight field in the Sample Info page in the Method Editor. This button is disabled during a run.

Zero Weight Button 

Tells the analyzer to read the weight of the empty sample pan; this is the offset weight which is subtracted from the total weight to obtain the weight of the sample in the pan. The weight is displayed in the Zero field in Sample Info page in the Method Editor. This button is disabled during a run.

Raise Furnace Button 

Moves the furnace to the Raised or Run position. If a run is in progress when you select this button, the run continues.

Lower Furnace Button 

Moves the furnace to the Lowered position in order to access the sample pan for loading or removing samples. Use this button to move the furnace from the cooling position when the furnace has cooled down. If a run is in progress when you select this button, the run ends immediately.

Cool Furnace Button 

Moves the furnace to the Cooling position. The cooling fan is above the furnace. If a run is in progress when you select this button, the run ends immediately. You can also move the furnace to the cool position by using the **Cool Furnace** button on the Pyris 1 TGA's front panel.

Antistatic Device Button 

Controls the state of the static shield. Pyris 1 TGA's static shield or antistatic device creates an invisible curtain of charged particles that prevent static cling between the sample pan and furnace wall. Static can be created by the glass furnace rubbing against the O ring around the upper ball that the furnace engages. If there is static, the sample pan will be pulled toward the furnace wall and the readings will be inaccurate. The device can be On, Off, Auto-Enabled, or Auto-Disabled. The status is displayed in the status panel. When the status is On, the device is on. The furnace travels all the way up to the ball joint without stopping. When the status is Off, the device is always off. The device is Off when the furnace is in the Cooling position. When the status is Auto-Enabled, the device is on when the furnace is stationary in the Lowered position, traveling between the Lowered position and the Raised position, or traveling to the Cooling position. Auto-Disabled is the same as Off, i.e., the device is always off. This control panel button can toggle the status of the antistatic device. It can also be controlled from the instrument's panel.

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Upper Fan Button 

The upper fan of the Pyris 1 TGA is used to create a gradient for the furnace to help it attain ambient temperature. The temperature gradient is needed in order for the motor to stay on and the furnace to run. The fan blows on the outside of the furnace tube when it is in the raised position. The upper fan is controllable from the control panel. There are two states: Auto and Off. The status is displayed in the status panel.

NOTE: The upper fan should be turned off when using the autosampler.

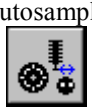
Pyris 1 DSC Control Panel

In addition to the common control panel buttons listed above, the Pyris 1 DSC control panel contains the following controls:

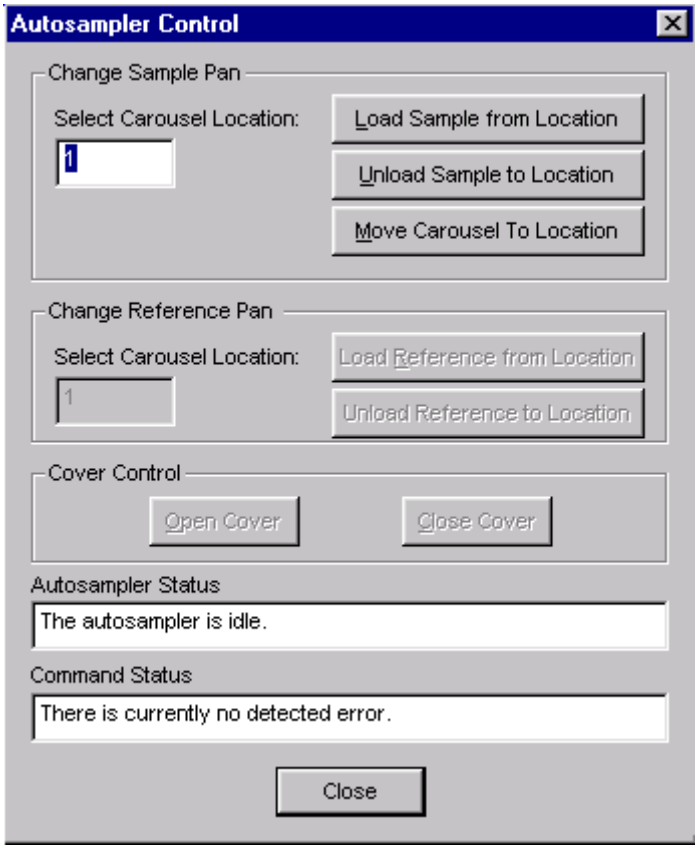
Autosampler Control Button 

Displays the Autosampler Control dialog box if an autosampler is installed on the Pyris 1 DSC. You can use the commands in the dialog box to control certain movements of the autosampler to handle an individual sample. If no autosampler is present, the button is grayed out.

DSC Autosampler Control Dialog Box

The Autosampler Control dialog box is displayed when you click on the **Autosampler Control** button  on the Pyris 1 DSC control panel. Use this dialog box to control the autosampler when there is an error and you need to access the autosampler or when you want to run an individual sample. Through this dialog box you can move samples to and from the sample tray and open and close the sample holder cover. (Note that the dialog box shown below is for a DSC 7 Robotics System, therefore, some of the items available for the Pyrid 1 DSC are grayed out.)

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Since you control the autosampler, you must be aware if a sample pan is already in the sample holder when you select **Load Sample from Location** in the dialog box. The autosampler will not check for the presence of a sample pan before loading the pan from a specified carousel location.

Change Sample Pan

Select Carousel Location

Enter the location in the tray that will be used when you click on one of the three buttons used to change the location of the sample pan: load, unload, and move.

Load Sample from Location

When you click on **Load Sample from Location**, the autosampler does **NOT** check for the presence of a sample pan in the sample holder. Click on the **Open Cover** button to see if a pan is in the sample cell. The system also does **NOT** check for the presence of a platinum lid in the lid receptacle before starting the sequence below. If there is no lid, the autosampler will retrieve the sample pan it just placed in the sample holder and return it to its location in the tray.

When you click on **Load Sample from Location** button in the Autosampler Control dialog box, the following sequence occurs:

1. If the sample holder cover is closed, the cover opens.
2. The sample tray transports the magazine containing the selected location to the sampling position. In the sampling position, the magazine is facing the sample holder.
3. The robot arm picks up the sample pan and carries it to the sample cell in the sample holder.

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4. The robot arm releases the sample pan into the sample cell.
5. The robot arm goes to the platinum lid receptacle, picks up the platinum cover, and carries it to the sample cell in the sample holder.
6. The robot arm releases the platinum cover onto the sample cell.
7. The sample holder cover closes.
8. The robot arm moves to the platinum cover receptacle and is lowered. The working gas is switched off.

Unload Sample to Location

When you select **Unload Sample to Location**, the system performs the steps below. It does **NOT** check the selected location for the presence of a sample pan before starting this sequence. Make sure that there is no pan already in the selected location.

1. The robot arm moves from over the platinum lid receptacle to over position 3 of the autosampler tray.
2. If the sample holder cover is closed, the cover opens.
3. The robot arm moves to the sample cell and picks up the platinum cover.
4. The robot arm carries the platinum cover to its receptacle and releases it.
5. The robot arm moves to the sample cell and picks up the sample pan.
6. The robot arm carries the sample pan to the carousel.
7. The robot arm places the sample pan in the selected location in the tray.
8. The robot arm positions itself over position 3 of the autosampler tray.
9. The cover remains open.

Move Carousel to Location

Commands the sample tray to rotate so that the magazine containing the specified location faces the front of the instrument. Now the magazine changing tool can be inserted to remove the magazine.

Change Reference Pan

Select Carousel Location

Enter the location in the tray that will be used when you click on one of the two buttons used to change the location of the reference pan: load or unload.

Load Reference from Location

When you select **Load Reference from Location**, the system does **NOT** check for the presence of a pan in the reference cell of the sample holder. If the cover is closed, click on the **Open Cover** button to see if a pan is in the reference cell.

When you click on **Load Reference from Location**, the following sequence occurs:

1. If the sample holder cover is closed, the cover opens.
2. The sample tray transports the magazine containing the selected location to the sampling position. In the sampling position, the magazine is facing the sample holder.
3. The robot arm picks up the reference pan and carries it to the reference cell in the sample holder.

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4. The robot arm releases the reference pan into the sample cell.
5. The robot arm goes to the platinum lid receptacle, picks up the platinum cover, and carries it to the reference cell in the sample holder.
6. The robot arm releases the platinum cover onto the reference cell.
7. The robot arm positions itself over position 3 of the autosampler tray.
8. The sample holder cover closes.

Unload Reference to Location

When you select **Unload Reference to Location** in the Autosampler Control dialog box, the system performs the steps below. It **DOES** check the selected location for the presence of a sample pan before starting this sequence.

1. The sample holder cover opens.
2. The robot arm checks the selected location to make sure it is empty.
3. The robot arm moves to the reference cell and picks up the platinum cover.
4. The robot arm carries the platinum cover to its receptacle and releases it.
5. The robot arm moves to the reference cell and picks up the reference pan.
6. The robot arm carries the reference pan to the tray.
7. The robot arm places the reference pan in the selected location in the tray.
8. The robot arm returns to position 3 of the autosampler tray.
9. The cover remains open.

Cover Control

Open Cover

Opens the sample holder cover.

Close Cover

Closes the sample holder cover.

Autosampler Status

This shows a descriptive message when there is an error with the autosampler.

Command Status

This shows the status of the commands being sent to the autosampler from the software.

Close

Closes the dialog box.

Clean Furnace Button 

Executes the Clean Furnace procedure for a DSC. This button is disabled during a run.

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NOTE: You must open the sample holder cover and expose the furnaces to air so all organic materials are removed during the cleaning process.

WARNING: Before cleaning the furnaces, remove all samples and pans.

When the **Clean** button is selected , a dialog box appears with a message. If you select **Continue**, the run begins. If you select **Cancel**, no further action takes place and the DSC goes to the load temperature. During the Clean procedure, the Status line displays the Cleaning status.

The Clean procedure heats the DSC sample holder to 600°C and holds it there for several minutes. This temperature is high enough to burn off any organic residue that may have been deposited in the sample holder.

Cover Heater Button 

Turns on or off the heater in the sliding sample holder enclosure cover. This is a toggle button. When the cover heater is on, the housing of the sample holder is heated to provide condensation-free and frost-free operation. This should be turned on only when subambient temperatures are in use. You can also turn the cover heater on or off by using the adjustable control panel on the Pyris 1 DSC.

AirShield Button 

Activates or deactivates the AirShield feature. If you have turned the AirShield on, the AirShield is activated when you slide the sample holder enclosure cover back to expose the sample holders. The AirShield is a thin curtain of dry gas that flows over the sample and reference holders when using subambient temperatures. It employs laminar flow to keep the sample holder region dry and frost-free when it is exposed to room temperature by sliding back the enclosure cover in order to change samples. You can also activate the AirShield by using the Adjustable Control Panel menu on Pyris 1 DSC.

CryoFill Button 

Activates or deactivates the CryoFill Liquid Nitrogen Filling System. When this feature is on, a sensor in the LN2 dewar in the Pyris 1 DSC detects the level of the LN2. If it drops below a certain level, the system will automatically bring in LN2 from the supply tank until it reaches the designated level. The level is held to within a few millimeters regardless of the pressure or flow rate from the LN2 supply tank. You must turn the CryoFill off when you are going to disconnect the supply in order to refill the supply tank. You can also turn CryoFill on or off by using the adjustable control panel.

 DSC 7 Control Panel

In addition to the common control panel buttons listed above, the DSC 7 and the DSC 7 Robotic System control panels contain the following items (the same control panel is seen for the DDSC):

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Clean Furnace Button 

Executes the Clean furnace procedure for the DSC 7 and the DDSC. This button is disabled during a run.

NOTE: You must open the sample holder cover and expose the furnaces to air so all organic materials are removed during the cleaning process.

WARNING: Before cleaning the furnaces, remove all samples and pans.

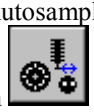
When the **Clean** button is selected, a dialog box appears with a message. If you select **Continue**, the run begins. If you select **Cancel**, no further action takes place and the DSC goes to the load temperature. During the Clean procedure, the Status line displays the Cleaning status.

The Clean procedure heats the DSC sample holder to 600°C and holds it there for several minutes. This temperature is high enough to burn off any organic residue that may have been deposited in the sample holder.

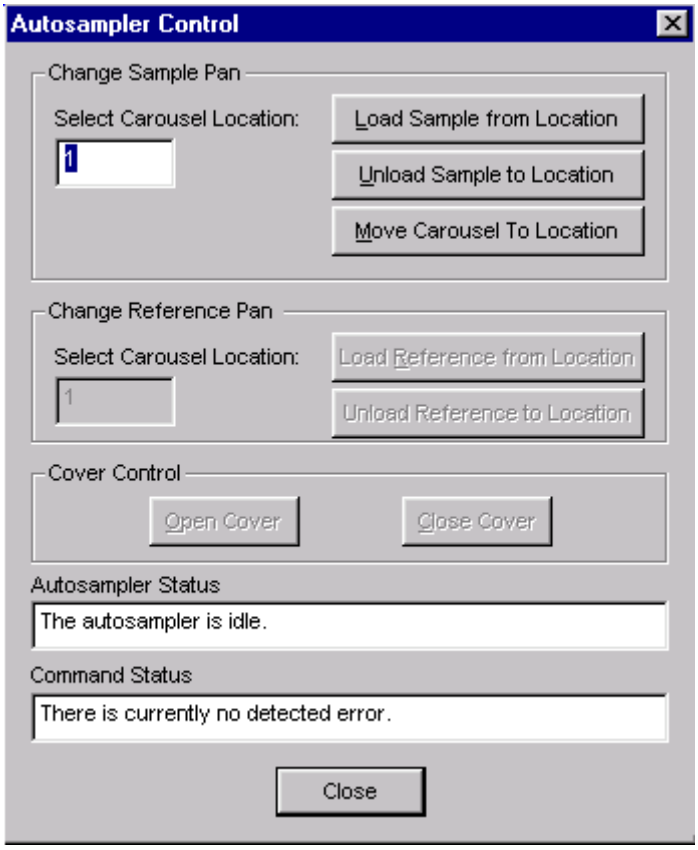
Autosampler Control Button 

Displays the Autosampler Control dialog box if a Robotics System is configured into your system. If there is no Robotics System present, the button is grayed out.

DSC Autosampler Control Dialog Box

The Autosampler Control dialog box is displayed when you click on the **Autosampler Control** button  on the DSC 7 control panel. Use this dialog box to control the autosampler when there is an error and you need to access the autosampler or when you want to run an individual sample. Through this dialog box you can move samples to and from the sample tray.

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Since you control the autosampler, you must be aware if a sample pan is already in the sample holder when you select **Load Sample from Location** in the dialog box. The autosampler will not check for the presence of a sample pan before loading the pan from a specified carousel location. The Autosampler Control dialog box is the same for both the Pyris 1 DSC and the DSC 7 except that for a DSC 7 Robotics System, you cannot load or unload a reference pan and open and close the sample holder cover from the dialog box. You must perform these functions from the DSC 7 Robotics System keypad.

Select Carousel Location

Enter the location in the tray that will be used when you click on one of the three buttons used to change the location of the sample pan: load, unload, and move.

Load Sample from Location

When you click on **Load Sample from Location** in the Autosampler Control dialog box for the DSC 7 Robotics System, the robot performs the following steps:

1. Before loading the sample, the robot arm is in its starting position, i.e., above the tray position 3. That is the third position from the center of the carousel of the magazine closest to the sample holder.
2. The sample holder cover opens.
3. The robot arm moves to the lid receptacle and verifies that the platinum cover is located in the receptacle.
4. The robot arm moves to the sample cell in the sample holder and verifies that the sample holder is empty.

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5. The tray transports the magazine containing the designated location, entered in the Select Carousel Location field, to the sampling position. In the sampling position, the magazine is facing the sample holder.
6. The robot arm picks up the sample pan and carries it to the sample cell in the sample holder.
7. The robot arm releases the pan into the cell.
8. The robot arm moves to the platinum lid receptacle, picks up the platinum lid, and carries it to the sample cell in the sample holder.
9. The robot arm places the platinum lid onto the sample cell.
10. The sample holder cover closes.
11. The robot arm moves to the platinum cover receptacle where it is lowered and the working gas is switched off.

Unload Sample to Location

When you select **Unload Sample to Location** in the Autosampler Control dialog box for the DSC 7 Robotic System, the system performs the following steps:

1. The sample holder cover lid is opened.
2. The working gas switches on.
3. The robot arm moves to the selected tray position, entered in the Select Carousel Location field, and checks for the presence of a sample pan. It should empty.
4. The robot arm moves to the sample cell and picks up the platinum lid.
5. The robot arm carries the platinum lid to its receptacle and places in the receptacle.
6. The robot arm moves to the sample cell and picks up the sample pan.
7. The robot arm carries the sample pan to the magazine and releases it into the selected position.
8. The robot arm moves to the start position, i.e., over the third position of the magazine closest to the sample holder.
9. The cover remains open.

Move Carousel to Location

Commands the sample tray to rotate so that the magazine containing the specified location faces the front of the instrument. Now the magazine changing tool can be inserted to remove the magazine.

Autosampler Status

This shows a descriptive message when there is an error with the autosampler.

Command Status

This shows the status of the commands being sent to the autosampler from the software.

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Pyris 6 DSC Control Panel

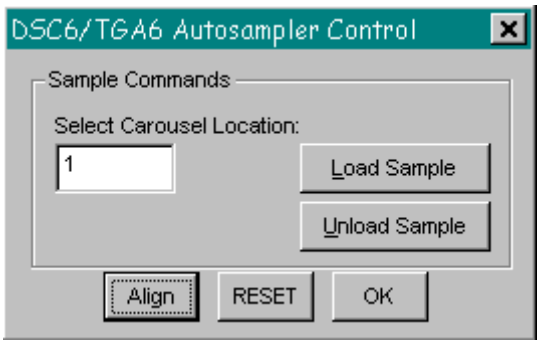
In addition to the common control panel buttons listed above, the Pyris 6 DSC control panel contains the following controls:

Autosampler Control Button

Displays the Autosampler Control dialog box if an autosampler is installed on the Pyris 6 DSC. You can use the commands in the dialog box to control certain movements of the autosampler to handle an individual sample. You can also access the gripper alignment wizard which is used to realign the gripper after removing and replacing the autosampler for installing a reference pan. If no autosampler is present, the button is grayed out.

AS 6 Autosampler Control Dialog Box

The Autosampler Control dialog box is displayed when you click on the Autosampler Control button on the Pyris 6 DSC control panel. Use this dialog box to control the autosampler when there is an error during a run and you need to unload a sample or reset the autosampler. Through this dialog box you can also perform gripper alignment, which should be done when you install a reference pan into the Pyris 6 DSC. Close the dialog box by either clicking on the **OK** button or the **Close** button in the title bar.



Load Sample

When you click on **Load Sample,** note that the system does **NOT** check for the presence of a sample pan in the furnace:

1. The autosampler moves from the home position to the furnace and removes the upper lid. It puts the lid on the ring.
2. The gripper removes the lower lid and place it in its position to the left of the furnace access hole.
3. The gripper goes to the specified carousel location and lifts the sample pan.
4. The sample pan is placed within the furnace.
5. The lower lid is returned to the furnace.
6. The upper lid is returned to the furnace.
7. The gripper returns to the home position.

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Unload Sample

When you click on **Unload Sample**:

1. The gripper moves from the home position to the furnace to remove the upper lid and place it on the ring.
2. The gripper removes the lower lid and place it on the ring.
3. Gripper retrieves the sample pan from the furnace and returns it to its position on the sample carousel.
4. The lower lid is returned to the furnace.
5. The upper lid is returned to the furnace.
6. The gripper returns to the home position.

Note: If the lids are not in place when you select **Unload Sample**, the error message: "Vertical step blocked" is displayed.

Align

The **Align** button accesses the align gripper wizard (see Chapter 7) for the AS 6. The wizard is a series of screens that walk you through aligning the gripper with respect to the furnace lid and the sample pans at locations 12 and 33. It also performs the furnace vertical alignment.

Reset

This button resets the system when an error occurs with the autosampler. Fix the cause of the problem and click on **Reset**. It clears the message from the LED display on the instrument; STANDBY should be redisplayed.



TGA 7 Control Panel

In addition to the common control panel buttons listed above, the TGA 7 control panel contains the following controls:

Clean Furnace Button 

Executes the Clean furnace procedure for the TGA 7. If a run is in progress when you select this button, the run ends immediately.

The clean furnace procedure for the TGA 7 involves lowering the furnace to expose it to the air and then heating it to approximately 900°C to burn off any materials coating the furnace surface.

WARNING: Since the furnace will be programmed to 900°C, make sure that the protective plastic visor is in the down position. DO NOT touch the furnace during this procedure.

With the control panel displayed, click on the **Clean Furnace** button. The furnace moves to the lowered position and the temperature is programmed to 900°C. When the cleaning procedure is complete, the furnace moves to the cool position and cools down to 30°C.

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Sample Weight Button 

Tells the analyzer to read the weight of the sample in the sample pan and display it in the Weight field in the Sample Info page in the Method Editor. This button is disabled during a run.

Zero Weight Button 

Tells the analyzer to read the weight of the empty sample pan; this is the offset weight which is subtracted from the total weight to obtain the weight of the sample in the pan. The weight is displayed in the Zero field in Sample Info page in the Method Editor. This button is disabled during a run.

Raise Furnace Button 

Moves the furnace to the Raised or Run position. If a run is in progress when you select this button, the run continues.

Lower Furnace Button 

Moves the furnace to the Lowered position. If a run is in progress when you select this button, the run ends immediately.

Cool Furnace Button 

Moves the furnace to the Cooling position. The fan is below the furnace. If a run is in progress when you select this button, the run ends immediately.

 Pyris 6 TGA Control Panel

In addition to the common control panel buttons listed above, the Pyris 6 TGA control panel contains the following controls:

Sample Weight Button 

Reads the weight of the sample in the sample pan and displays it in the Weight field in the Sample Info page in the Method Editor. This button is disabled during a run.

Zero Weight Button 

Reads the weight of the empty sample pan placed on the sample holder; this is the offset weight which is subtracted from the total weight to obtain the weight of the sample in the pan. It is

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displayed in the Zero field in Sample Info page in the Method Editor. This button is disabled during a run.

Cooling Air Button 

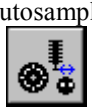
Enables/disables the small internal air pump which produces forced air that cools down the furnace. It can cool the furnace from 1000°C to 100°C in 6 minutes. If a run is in progress when you select this button, the run ends immediately. The button below indicates that the cooling air is disabled.

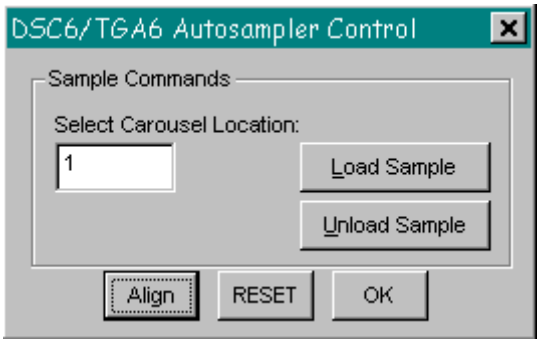


Autosampler Control Button 

Displays the Autosampler Control dialog box if an autosampler is installed on the Pyris 6 TGA. You can use the commands in the dialog box to control certain movements of the autosampler to handle an individual sample. You can also access the gripper alignment wizard which is used to realign the gripper after removing the and replacing the autosampler in order to install a new sample thermocouple. If no autosampler is present, the button is grayed out.

AS 6 Autosampler Control Dialog Box

The Autosampler Control dialog box is displayed when you click on the Autosampler Control button  on the Pyris 6 TGA control panel. Use this dialog box to control the autosampler when there is an error during a run and you need to unload a sample or reset the autosampler. Through this dialog box you can also perform gripper alignment, which should be done when you change the sample thermocouple in the Pyris 6 TGA. Close the dialog box by either clicking on the **OK** button or the **Close** button in the title bar.



Load Sample

When you click on **Load Sample,** note that the system does **NOT** check for the presence of a sample pan in the furnace:

1. The autosampler moves from the home position to the furnace and removes the upper lid. It puts the lid on the ring.

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2. The gripper goes to the specified carousel location and lifts the sample pan.
3. The sample pan is placed within the furnace.
4. The upper lid is returned to the furnace.
5. The gripper returns to the home position.

Unload Sample

When you click on **Unload Sample**:

1. The gripper moves from the home position to the furnace to remove the upper lid and place it on the ring.
2. Gripper retrieves the sample pan from the furnace and returns it to its position on the sample carousel.
3. The upper lid is returned to the furnace.
4. The gripper returns to the home position.

Note: If the lid is not in place when you select **Unload Sample**, the error message: "Vertical step blocked" is displayed.

Align

The **Align** button accesses the align gripper wizard (see Chapter 7) for the AS 6. The wizard is a series of screens that walk you through aligning the gripper with respect to the furnace lid and the sample pans at locations 12 and 33. It also performs the furnace vertical alignment.

Reset

This button resets the system when an error occurs with the autosampler. Fix the cause of the problem and click on **Reset**. It clears the message from the LED display on the instrument; STANDBY should be redisplayed.



DMA 7e Control Panel

In addition to the common control panel buttons listed above, the DMA 7e control panel contains the following controls:

Tare Probe 

The analyzer reads the weight of the probe when zero force is applied. This zeros the weight of the probe so it is not included in the run of the sample. You must tare the probe when you change the measuring system and before mounting a sample. The tare weight (Probe Weight) is displayed on the right-hand-side of the status bar at the bottom of the screen and in the dialog box.

Read Sample Height 

This button reads directly from the instrument the height of the sample on the sample platform. Press the **Probe Down** button on the analyzer to lower the probe to just before it touches the

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sample. Press the **Probe Hold** button. Click on the **Read Sample Height** button. The value is displayed in the Probe Position field in the Status Panel.

Sample Height Zero 

This button takes a height reading of the sample platform without a sample in place. This is the zero height. This is done so that the height of the platform is not included in the height of the sample. Press the **Probe Down** button to lower the probe so that it rests on the sample platform. Click on the **Zero Height** button. The value is displayed in the Probe Position field in the Status Panel. This is disabled during a run.

Apply Current Force 

The forces that are set up in the method are applied by the motor to the sample. If a run is in progress, this button has no effect since the forces are already applied.

Apply Zero Force 

Sets the static and dynamic forces applied by the motor to 0 mN; turns the Static and Dynamic Controls to Force, and sets the frequency to 1 Hz.

Static Control On/Off 

Toggles on and off the static force control set in the Set Controls dialog box in the Initial State page. When the control is on, the button is lit, i.e., has a white background.

Dynamic Control On/Off 

Toggles on and off the dynamic force control set in the Set Controls dialog box in the Initial State page. When the control is on, the button is lit, i.e., has a white background.

 TMA 7 Control Panel

In addition to the common control panel buttons listed above, the TMA 7 control panel contains the following controls:

Tare Probe 

Tells the analyzer to read the weight of the probe when zero force is applied. This zeros the weight of the probe so it is not included in the run of the sample. You must tare the probe when you change the measuring system and before mounting a sample. The tare weight (Probe Weight) is displayed on the right-hand side of the status bar at the bottom of the screen and in the dialog box.

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Read Sample Height 

Tells the analyzer to read the height of the sample. The value is displayed in the Height field of the Sample Info page in Method Editor. It also appears in the Probe Position box in the Status Panel. This is disabled during a run.

Sample Height Zero 

Tells the analyzer to read the zero offset (no sample in place). The value is displayed in the Zero field of the Sample Info page in Method Editor. It also appears in the Probe Position box in the Status Panel. This is disabled during a run.

Apply Current Force 

Tells the analyzer to apply the static force that is set up in the method. If a run is in progress, this button has no effect since the force is already applied.

Apply Zero Force 

Sets the static force applied by the force motor to 0 mN; turns the Static Control to Force, and sets the frequency to 1 Hz.

DTA 7 Control Panel

In addition to the common control panel buttons listed above, the DTA 7 control panel contains the following controls:

Read Zero Button 

Reads the difference in the temperatures of the sample and reference thermocouples and makes that the zero setting. The difference is displayed in the Zero field of the Sample Info page in Method Editor.

Enable Cover Control Button



Disable Cover Control Button



The furnace cover is used to control the heat convection generated by the furnace. When the cover is enabled, it will be closed when the furnace is heating or holding isothermally and during all run conditions. When the furnace is cooling and not in a run, the cover opens automatically to help

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dissipate heat more efficiently. When the cover is disabled, the cover will be open at all times. This setting is important if you are going to use an open sample tube.

An open sample tube can be used when performing decomposition experiments. The gases that escape can then be vented to a hood or the gases can be directed to another type of instrument for further analysis.

Furnace Lock Button 

Enables/disables the furnace lock. The furnace lock is a safety feature that prevents the furnace from being raised when the furnace is too hot. This keeps you from touching the sample area until the sample temperature falls below the furnace lock temperature. The default temperature is 55°C but can be changed in the Instrument page in Preferences.

Sample Info Page

The Sample Info page appears when you select the Sample Info tab of the Method Editor. This page contains the following sections:

Enter Sample Info Section

Sample ID

Enter a sample name of up to 40 characters. The entry field scrolls horizontally as you type.

Operator ID

Enter the name of the person who will run the experiment; the name can be up to 40 characters. The entry field scrolls horizontally as you type.

Comment

Enter any additional information about the sample or run; the entry can be a maximum of 160 characters. The entry field automatically wraps horizontally and scrolls vertically. Start typing on a new line by pressing **Enter**.

Enter Sample Weight Section

Weight

Enter the weight of the sample in milligrams. The default value is 1.000 mg.

For TGA methods, you can have the analyzer read the sample weight by using the **Sample Weight** button in the control panel.

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Zero (TGA only)

The zero weight value is read directly from the analyzer by selecting the **Zero Weight** button in the control panel; the value appears in the Zero field. The default value is 0.000 mg. This value is the weight of the empty sample pan either on the hangdown wire for the TGA 7 or Pyris 1 TGA or on the sample holder for the Pyris 6 TGA. The value is used to tare the weight of the sample pan before loading the sample.

Zero (DTA 7)

The difference in temperature between the sample and the reference thermocouple is displayed in this field. This value is read directly from the analyzer by selecting the **Read Zero** button in the control panel.

Save Data As Section

File Name

This field is for the name of the file in which the data collected from the run will be saved. You can enter the file name directly in this field or in the Browse dialog box. A Pyris software default file name (QSAVE.xxD) is used if you do not specify a default file name in the Save Preferences page. The validity of the drive\directory\file name is checked when the data are saved. You can also use incremental file names, e.g., if you enter <*user-defined file name*>### in the File Name field, the data file saved is <*user-defined file name*>xxx, where xxx starts at 000 and is incremented each time the method is used. This feature is useful when creating a Sample List in the Player Editor. If you do not specify the incremental file name and reuse the method without changing the file name entered, then Pyris Software will append a date – time stamp to the end of the user-defined file name in order to give the data file a unique file name and avoid overwriting an existing file. The format of the file name is <*user-defined name*>@YYMMDDHHMMSS.xxD, where YY is the year, MM is the month, DD is the day, HH is the hour (on a 240-hour clock), MM is the minutes, and SS is the seconds; xx in the extension represents the two-character code for the analyzer and D means data.

Browse

If you want to select a directory other than the default for storing the data file, click on the **Browse** button to display the Browse dialog box.

Directory

This field displays the drive and directory in which the data are saved after the run. This value reflects the disk\directory chosen in the Browse dialog box. If you do not select a specific directory, the default will be displayed.

Enter Sample Dimensions Section (DMA/TMA)

Measuring System/Geometry

From the drop-down list displayed when you click on the down arrow, select the measuring system that you are using in the analyzer and the associated geometry of the sample. The other items displayed in this section depend on this selection.

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Height

The sample height (in mm) is either entered manually or read from the instrument by clicking on the **Sample Height** button in the control panel. The value is displayed in the Height field of the Sample Info page. This value does not change when you load a new existing method.

Width

The sample width (in mm) is entered by the user. This value does not change when you load a new existing method.

Depth

The sample depth (in mm) is entered by the user. This value does not change when you load a new existing method.

Length

The sample length (in mm) (for an extension measuring system) is entered by the user. This value does not change when you load a new existing method.

Diameter

This diameter (in mm) (for cylindrical or disk-shaped samples) is entered by the user. This value does not change when you load a new existing method.

Thickness

This dimension is the thickness (in mm) of one of the two samples used with a shear measuring system. It is entered by the user. The maximum thickness is 3.000 mm. This value does not change when you load a new existing method.

Zero

The zero height value can be either entered manually or read from the instrument by clicking on the **Zero Height** button from the control panel. This is the position of the probe without a sample in place. The value is displayed in the Zero field. This value does not change when you load a new existing method.

Initial State Page

The Initial State page appears when you select the Initial State tab of the Method Editor. This page contains the following sections:

Set Initial Values Section

Temperature (all analyzers)

This field displays the initial temperature, i.e., the temperature of the thermocouple and the sample's environment when the run starts. The default value displayed is the Load Temperature

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value entered in the Instrument page in Preferences. Type a new value or use the spin buttons to increase or decrease the displayed temperature in 5°C steps. This value is used in the first step of the method program on the Program page.

Y Initial

Enter the initial ordinate (Y) value. Type the value or use the spin buttons to increase or decrease the displayed value in 5-mW steps.

Static Force or Static Stress (DMA/TMA)

The static force should be sufficient to keep the probe in continuous contact with the sample during the entire analysis. Static force is usually 15% greater than the dynamic force. The static force is held constant in a temperature scan, a time (isothermal) scan, and a frequency scan. In a dynamic stress scan, the static force changes proportionally to the increasing dynamic force to assure that the sample remains in tension throughout the analysis. Enter the initial value into the field or use the spin buttons to increase or decrease the displayed static force in 10-mN steps. If you choose to report stress (selected in Preferences), then the value is in kPa. This value is displayed in the first step of the method program on the Program page.

Dynamic Force or Dynamic Stress (DMA/TMA)

The dynamic force should be sufficient to produce an initial displacement amplitude of 5 – 50 μm. Dynamic force is held constant in a temperature scan, a time (isothermal) scan, and a frequency scan. It varies in a dynamic stress scan and is absent in a static force scan and a creep-recovery scan. Enter the initial value into the field or use the spin buttons to increase or decrease the displayed dynamic force in 10-mN steps. If you choose to report stress (selected in Preferences), then the value is in kPa.

Frequency (DMA/TMA)

Most applications use 1 Hz. This field displays the initial frequency at which the dynamic force is applied. The frequency is held constant in a temperature scan, a time (isothermal) scan, and a dynamic stress scan. There is no frequency in a static force scan or a creep-recovery scan. You cannot enter a value into the field; use the spin buttons to increase or decrease the displayed frequency in 0.2-Hz steps. This value is displayed in the first step of the method on the Program page.

DMA 7e Set Controls (DMA/TMA)

Click on the **Set Controls** button to display an additional box for entering initial static force control parameters (tension control and position control) and dynamic force control parameters (stress control, strain control, and amplitude control). You can also turn off dynamic force in this box so that only static force applies. The DMA 7e is like a TMA 7 in this state. When a Creep Recovery or a Static Stress Scan method is used, or when Dynamic Force is turned off, there are no controls available on this window. When a Dynamic Stress Scan method is used, there are no Dynamic Controls available.

Baseline File Section

Baseline subtraction performs a linear subtraction of the instrument baseline from a curve to improve the resolution of the transition of interest. Instrument baseline is established by placing empty sample pans in the sample and reference holders and performing a run under similar

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conditions that you run your samples. The data file saved is a baseline file. One should scan the analyzer under the proposed experimental conditions to check the curvature and noise level of the instrument baseline before analyzing samples. When you are going to run a sample, you can have the software perform a baseline subtraction using a baseline file that you collected. The Baseline File section of the Initial State page contains the following parameters:

Use Baseline Subtraction check box

Click on the check box to activate Baseline Subtraction. The baseline file selected here is subtracted from the data as it is collected to optimize the data. When you select Baseline Subtraction, the **Browse** button becomes selectable.

Directory

Displays the drive and directory in which the baseline file is stored. You cannot enter the drive/directory directly; you must use the **Browse** button to find and select the drive\directory.

File Name

Enter the name of the baseline file to be used for the run or select the file from the Browse box.

Browse

Activated when you click on the Baseline Subtraction check box. If you want to select a directory other than the default for storing the data file, click on the **Browse** button to display the Browse dialog box.

Instrument Baseline

In differential scanning calorimetry (DSC) and differential thermal analysis (DTA), it is recommended that one scan the analyzer before analyzing samples under the conditions you will be using for your samples to check the baseline curvature and noise level. This is done by placing empty sample pans in the sample and reference holders and performing a run using the method you will use for the samples.

The instrument's baseline may be curved or display noise for a number of reasons. There may be trace amounts of residue from a previous experiment attached to the sample holder. Decomposed or sublimated compounds may condense on the sample holder to distort the instrument baseline. Clean the sample holder with ethanol or acetone. If this does not solve the baseline problem, the cause may be the purge gas flow. Linearity of the baseline will be reduced if the purge gas flow rate is not constant or the purge gas contains a large amount of water vapor.

Another cause for an irregular instrument baseline may be the power supply. The mains supply is generally not sufficiently stable for sensitive instruments. Voltage spikes decrease the operating life of the instrument and produce a lot of noise on the baseline. The electrical characteristics of the instrument change over time. The instrument should be readjusted (balance and slope) to maintain a good instrument baseline.

Instrument baseline recorded for successive scans under the same conditions should be identical. If not, moisture may have condensed on the sample holder. Increase the flow of dry purge gas. In subambient mode, the baseline's shape strongly depends on the coolant. Its level should be maintained at a constant level.

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Set Purge Gas Section

The Set Purge Gas section of the Initial State page is selectable if your system is using a GSA 7 or a TAGS for changing purge gases. The gas and flow rate selected here are used during equilibration time. The purge gas and flow rate can be changed in a gas program entered on the Program page. It can also be changed in real time by selecting a different gas in the Purge Gas field in the control panel.

The values displayed here are the default gas and flow rate selected in Preferences. The section contains the following parameters:

Purge Gas

The default purge gas displayed (Gas A) is the gas selected in Purge Gas page of Preferences. Select the purge gas to be used during the equilibration time, before the first step begins, by clicking on the arrow to display the drop-down list of available gases. For a TAGS, the choices in the drop-down list are all available gases plus the option Gas Off. The gases selected in Preferences are marked with an asterisk and appear at the top of the list. For a GSA 7, the choices in the drop-down list are the two gases specified in Preferences (when editing a method in Data Analysis application, the choice of gases is the entire list). The gases selected in Preferences are marked with an asterisk and appear at the top of the list. If there is no gas accessory attached, then the name of the gas used at the start of the run is displayed in gray.

Flow Rate

The default purge gas flow rate displayed was entered in Preferences. This field is read only for a GSA 7 and no gas switching accessory. For a TAGS, increase or decrease the flow rate by entering a new value or using the spin buttons.

Equilibrate Within Section

The parameters in this section depend on the analyzer. Also, if you going to perform a frequency multiplex scan, remember that the values entered in this section apply to each scan within a multiplex run. In general, when you activate an Equilibrate check box and enter a threshold value, Pyris will monitor the addressed signal from the analyzer until it is within the threshold for 20 seconds (10 consecutive readings, each 2-s intervals).

NOTE: A run will begin when just one of the equilibrate conditions that you select is met.

NOTE: For Pyris Player, do not activate any options in the Equilibrate Within box for a DMA 7e method. If the method you select has any Equilibrate Within items selected, click on the **Edit Method** button in the Edit Step section and turn those selections off.

Temperature

This is the range within which the sensor temperature, set in Set Initial Values, can equilibrate. Enable the entry field by clicking on the check box. Enter a value directly or use the spin buttons to increase or decrease the value in 0.1°C steps. The run will not start until the change of the sensor temperature is plus or minus this value.

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Heat Flow (DSC analyzers)

Enable the entry field by clicking on the check box and then enter the value defining the amount by which the heat flow (Y) value can fluctuate before the run can start. The run will not start until the signal fluctuates no more or no less than this value. You can also use the spin buttons to increase or decrease the displayed value by 0.1 mW.

Weight (TGA analyzers)

For TGA analyzers, the Y signal is weight. The value in this field defines the amount by which the Y signal (in mg) can fluctuate before the run can start. Enable the entry field by clicking on the check box and then enter a value or use the spin buttons to increase or decrease the value in 0.1-mg steps. The run will not start until the Weight signal is within this value.

Delta T (DTA 7 only)

The Delta T signal is difference in temperature between the sample and the reference thermocouples. Enable the threshold entry field by clicking on the check box. Enter a value in the field or use the spin buttons to increase or decrease the value in 0.1°C steps. The run will not start until the Delta T signal is within this range for 20 s.

Turn off cover heater (Pyris 1 DSC only)

Click in the check box if you want the Pyris 1 DSC cover heater turned off when the run starts.

Height (TMA 7 only)

The value in this field defines the amount by which the height measured by the analyzer when using the **Read Height** button can fluctuate from the Height value measured and entered by the user before the run can start. Enable the entry field by clicking on the check box and then enter a value or use the spin buttons to increase or decrease the value in 0.01-mm steps. The run will not start until the height reading stays within this range.

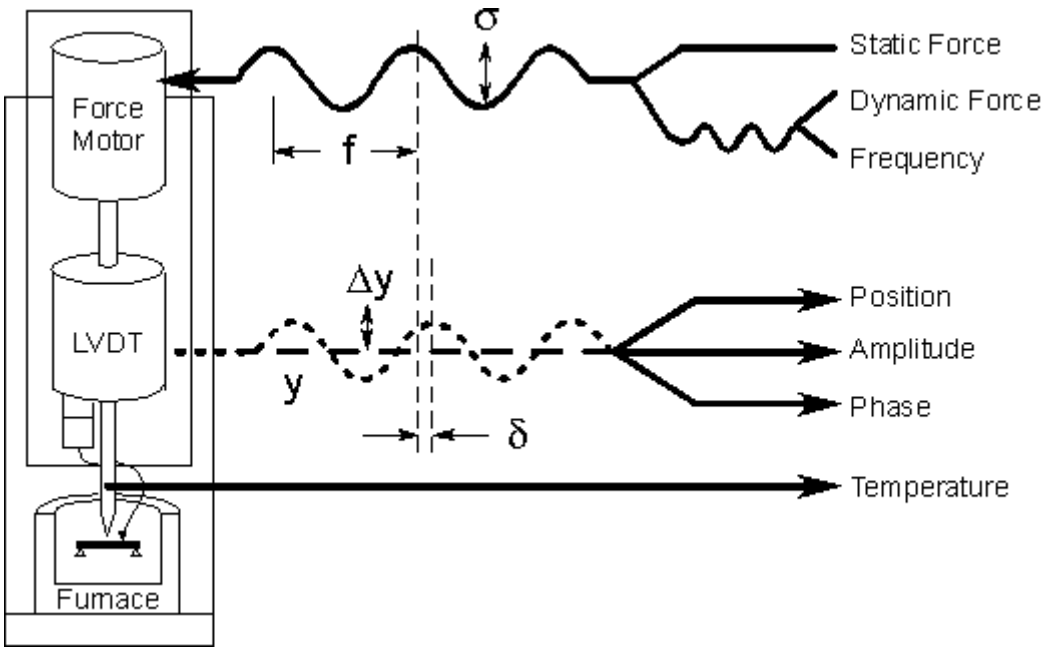
Amplitude (DMA/TMA)

The force motor applies a force to the sample which causes displacement. Displacement is separated into average position, average amplitude, and phase lag. The Equilibrate Within Amplitude value indicates the range in which the amplitude can fluctuate around the average amplitude before the run begins. Enter the value or use the spin buttons to increase or decrease the displayed value in 0.1-µm steps. The run will not start until the ordinate signal is within this threshold.

When a dynamic force is applied by the force motor of the DMA 7e at a certain frequency to the sample, the sample responds to the force with an oscillating displacement amplitude. The amplitude corresponds to the average energy recovered in one cycle of oscillation in an elastic deformation. The analyzer monitors the current force and displacement output values and calculates the amplitude which is saved in the data file. This signal is represented by the Amplitude curve. The diagram below shows the relationship among motor control variables, including amplitude. The amplitude is the most important output signal and is generated by the LVDT. Amplitude and phase angle are closely related; they are derived from the same measured vector.

Amplitude values between 5 and 500 µm can produce very high quality data even at very low phase angles (<1°). Amplitudes less than 10 µm can produce high-quality data if the phase angles are high (>1°). Amplitude signals can be increased or decreased by increasing and decreasing the dynamic stress. Data quality can be significantly improved by increasing the amplitude.

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Phase (DMA/TMA)

The force motor applies a force to the sample which causes displacement. Displacement is separated into average position, average amplitude, and phase lag. The Equilibrate Within Phase value indicates the range in which the phase lag value can fluctuate around the average phase before the run begins. Enter the value or use the spin buttons to increase or decrease the displayed value in 0.5° steps. The run will not start until the ordinate signal is within this threshold.

See the diagram above for the relationship among the motor control variables, including phase. Phase angle values between 1° and 45° can produce very high quality data even at much lower amplitudes. A phase angle can be changed, but only for a sample that has been mounted properly and is viscoelastic. In this case, changing the frequency of dynamic oscillation changes the phase angle. Reducing the phase angle generally increases data quality.

Wait no longer than

Enable the threshold entry field by clicking on the check box and then enter the maximum time to wait for the signal to equilibrate. If this time is reached and the signal has not finished equilibrating, the run will start anyway. You can also use the spin buttons to increase or decrease the displayed value in 1-min steps.

Data Collection Section

Sample Rate (DSC 7, Pyris 1 DSC, DTA 7)

Select the sample rate of Standard or Fast from the drop-down list box. This is the rate at which data are collected throughout the run. The standard data collection rate is 5 points per second; the fast rate is 20 points per second. Proper selection of a sample rate will increase the efficiency of the analysis. Generally, the slower scanning rate improves peak resolution while the fast scanning

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rate improves the usable sensitivity. The number of points saved in the date file also depends on the Data Sampling Options in the Step Details section of the Program page.

Set Purge Gas Section

The Set Purge Gas section of the Initial State page is selectable if your system is using a GSA 7 or a TAGS for changing purge gases. The gas and flow rate selected here are used during equilibration time. The purge gas and flow rate can be changed in a gas program entered on the Program page. It can also be changed in real time by selecting a different gas in the Purge Gas field in the control panel.

The values displayed here are the default gas and flow rate selected in Preferences. The section contains the following parameters:

Purge Gas

The default purge gas displayed (Gas A) is the gas selected in Purge Gas page of Preferences. Select the purge gas to be used during the equilibration time, before the first step begins, by clicking on the arrow to display the drop-down list of available gases. For a TAGS, the choices in the drop-down list are all available gases plus the option Gas Off. The gases selected in Preferences are marked with an asterisk and appear at the top of the list. For a GSA 7, the choices in the drop-down list are the two gases specified in Preferences (when editing a method in Data Analysis application, the choice of gases is the entire list). The gases selected in Preferences are marked with an asterisk and appear at the top of the list. If there is no gas accessory attached, then the name of the gas used at the start of the run is displayed in gray.

Flow Rate

The default purge gas flow rate displayed was entered in Preferences. This field is read only for a GSA 7 and no gas switching accessory. For a TAGS, increase or decrease the flow rate by entering a new value or using the spin buttons.

Set Controls Section (DMA/TMA)

The Set Controls section contains two boxes of parameters: Static Force Control and Dynamic Force Control. These are part of the initial values that you set before the start of a run.

NOTE: The total of static and dynamic forces must be less than the maximum force of the motor.

NOTE: All of the values in this section appear in the first method step on the Program page. If the first step cannot accommodate the control that is turned on, the control is turned off when you run the method.

If the method is a Dynamic Stress Scan, all Dynamic Force controls are disabled. Only Static Force controls are available. For Static Stress and Creep Recovery scans, or when Dynamic Force is turned off, there is no Set Controls section at all.

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Static Force Control

Click on the **Static Force** radio button if you want the static force (a steady force applied to the sample) to remain at the value entered in the Set Initial Values box throughout the run. There is no Tension Control or Position Control when you select Static Force Control.

- **Tension Control** is a system that controls the static force or stress applied to a sample in order to maintain a constant relationship between the dynamic and static controls. There is automatic adjustment of the static force to maintain constant sample contact. Tension Control applies a static force as a percentage of the dynamic force. For example, if Tension Control is 110%, then the analyzer will apply 10% more static force or stress than dynamic force or stress.

  Tension Control setpoint values depend on the measuring system used and the state of the sample. Other considerations may include how tacky the sample is or the compliance of the sample. Some typical values are given in the Setpoint Values table. The **Tension Control** button is a toggle; if this is turned on, then Position Control and Static Force Control are turned off. Click on the **Tension Control** radio button to activate it. When tension control is on, the applied static force necessary to maintain sample-to-probe contact is applied automatically. The analyzer will no longer apply the Static Force value entered in the Set Initial Values box but will apply a static force that is a percentage of the dynamic force. Type in the desired value or use the spin buttons to increase or decrease the value in 1% steps.

- The **Position Control** is a system that controls the static force or stress applied to a sample in order to maintain the sample at a constant position setpoint. The value entered is the setpoint to be applied when no static controls are on. Position Control varies the static force as the sample changes position as detected by the LVDT. For example, if a 5.000-mm fiber sample shrinks during a run and Position Control is on, the force will be automatically increased to maintain a 5.000-mm length.

  The **Position Control** button is a toggle; if this is turned on, then Tension and Static Force Control are off. Click on the **Position Control** radio button to activate it. When Position Control is on, the analyzer no longer applies the Static Force value entered in the Set Initial Values box but will apply a static force that will maintain the probe at a constant position. Type in the desired value or use the spin buttons to increase or decrease the value in 1-mm steps. Click in the Current box if you want the current Position Control value to be used when the method is run.

Tension Control Setpoint Values

Setpoint Min	Setpoint Max	Measuring System
105	110	3 Point Bending
0	5	Dual Cantilever
0	1	Single Cantilever
120	150	Extension
0	110	Parallel Plate
0	10	Parallel Plate (tacky sample)

Dynamic Force Control

Dynamic Force is the magnitude of the total cyclical force. Select this radio button if you want the dynamic force to remain at the value entered in the Set Initial Values box throughout the run.

- When **Stress Control** is selected, there is automatic adjustment of the applied dynamic force to achieve a constant stress value defined by the setpoint. When Stress Control is

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activated by clicking on the radio button, the DMA 7e operates as a constant stress analyzer throughout the run.

- When **Strain Control** is selected, there is automatic adjustment of the applied dynamic force to achieve a constant strain value defined by the setpoint. When Strain Control is activated by clicking on the radio button, the DMA 7e operates as a constant strain analyzer and the displacement amplitude is maintained. The strain is maintained throughout the run. Enter the setpoint value or use the spin buttons to increase or decrease the displayed value in 1% steps. Click on the Current box if you want the current Strain Control value to be used when the method is run.

- **Amplitude Control** is a system that controls the dynamic force or stress applied to a sample in order to maintain the sample at a constant amplitude setpoint. Click on the **Amplitude Control** radio button if you want the DMA 7e to operate as a constant amplitude analyzer. A constant dynamic displacement will be maintained throughout the experiment. Enter the setpoint value or use the spin buttons to increase or decrease the displayed value in 1-mm steps. Click on the Current box if you want the current Amplitude Control value to be used when the method is run.

Turn Off/On Dynamic Force

Click on this toggle button to activate or deactivate the Dynamic Force Control. If this radio button is selected, the entire Set Controls box is disabled. When selected, you are asked if you want to replace the current method with the default static force method.

Program Page

The Program page appears when you select the Program tab of the Method Editor. This page contains the following sections:

Method Steps Section

Initial Temperature

Displays the initial temperature entered in the Initial State page. Change the value by typing in the entry field or using the spin buttons. If you change the initial temperature here, the value will be updated on the Initial State page and in the first step in the method program. If you change the Temperature in the Initial State page, the method program is adjusted accordingly.

List of Method Steps

The steps of the method program are listed in the order in which they will occur. If you are creating a new method, a default step is listed initially. Information entered in the Initial State page is used in the first step. The first step is highlighted upon entering the Program page. Select a step in this list to highlight it for editing. Use the buttons to the right of the list to add, insert, or delete a step. The type of step you add or insert may affect the steps already listed.

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Add a Step

Displays the Method Step Options dialog box, in which you select the type of step you wish to append to the program. The types of steps available for selection vary with the type of analyzer you are using.

Insert a Step

Displays the Method Step Options dialog box in which you select the type of step to insert immediately before the highlighted step in the list of method steps.

Delete this Step

Deletes the highlighted step from the method. To delete a step, the remaining program must be a continuous temperature program.

End Condition

Displays the Set End Condition box in the Edit Step section of the Program page. Here you specify the end condition for your method.

Edit Step Section

The Edit Step section displays editable parameters for the highlighted step in the method. The types of steps available for selection depend on the type of analyzer you are using.

- Temperature Scan Step
- AutoStepwise Scan Step (TGA only)
- Isothermal Step
- Repeat Steps
- Heat-Cool Repeated Scan Step (DDSC only)
- Iso-Scan Repeated Scan Step (DDSC only)
- Frequency Scan Step
- Frequency MultiPlex Step
- Dynamic Force/Stress Scan Step
- Static Force/Stress Scan Step
- Creep/Recovery Step

Each step and its parameters are discussed later in this section.

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Set End Condition Section

The Set End Condition parameters are the conditions that the analyzer is to obtain at the end of a run. They appear when you select the **End Condition** button:

Temperature

Go To Load Temperature

Selects the load temperature entered in Instrument page in Preferences as the end condition for the current method. At the end of the run, the analyzer will be programmed to this temperature.

Hold

At the end of the run, the analyzer will hold at its current temperature.

Go To Temperature

Selecting Go To activates the Temperature entry field and indicates that a user-entered temperature is the end condition temperature. Enter the temperature to which the analyzer will return at the end of the run.

Frequency (DMA only)

Go To Load

Selects the load frequency entered in Preferences as the end condition for the frequency. At the end of the run, the analyzer will be programmed to this frequency.

Hold at Current

Selects the current frequency as the end condition frequency.

Go To

Selecting Go To activates the Frequency entry field and indicates that a user-entered frequency is the end condition frequency. Enter the frequency to which the analyzer will return at the end of the run.

Forces (DMA/TMA)

Go To Load

Selects the load forces entered in Preferences as the end condition for forces. At the end of the run, the analyzer will be programmed to these forces. For the TMA, only static force is involved.

Hold at Current

Selects the current static (DMA/TMA) and dynamic (DMA only) forces as the end condition forces.

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Go To

Selecting Go To deactivates the **Go To Load** and **Hold at Current** buttons and indicates that user-entered forces are the end condition forces. Enter the static force (DMA/TMA) and the dynamic force (DMA only) to which the analyzer will return at the end of the run.

Turn Off Cover Heater check box (Pyris 1 DSC only)

Programs the cover heater to turn off at the end of the current method.

Turn Off CryoFill (Pyris 1 DSC only)

Programs the Pyris 1 DSC CryoFill to turn off at the end of the run. When this feature is enabled, a sensor in the LN2 dewar in the Pyris 1 DSC detects the level of the LN2. If it drops below a certain level, the system will automatically bring in LN2 from the supply tank until it reaches the designated level.

Step Info Section

The Step Info parameters are displayed when the **Step Info** button on the Program page is selected. The Step Info section for most scan types contains the following controls (Creep/Recovery, Frequency, and Frequency Multiplex have different sections):

Detail

Enter a comment or identification for the highlighted step, up to 40 characters.

Data Sampling Options

For DSC analyzers, this field displays the data sampling option based on the sample rate selected in the Data Collection section of the Initial State page and enables the Select Value entry field. For the Fast sample rate, the only option is Data On and you cannot change the Select Value field. For the Standard sample rate, and for all other analyzers, the options are Seconds Between Points and Number of Points.

Select Value

Depending on the Data Sampling Option, this field displays the seconds between data points collected in the data file (in seconds) or the number of points in the data file. Use the spin buttons to increase or decrease the displayed value. The Total points in Run value, displayed for some analyzers, is adjusted accordingly. The smaller the number of seconds between points, the more data points collected. If the number of points collected is small, you may miss an important event in the run.

Total points in Run

The total number of points in the run, calculated from all steps in the method. This value is automatically updated if you change the data sampling options.

Sample Rate

Displays the sample rate (Standard or Fast) selected in the Data Collection section of the Initial State page.

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Total Time in Run

The total time of the run calculated from all the steps in the method. This value is automatically updated if you change the data sampling options.

Force Used (DMA/TMA)

The total force used for the step, the sum of static and dynamic forces as entered in the Initial State page.

Force Available (DMA/TMA)

Total amount of force available from the force motor.

AutoStepwise Scan Step Info Section (TGA only)

The Step Info section for an AutoStepwise Scan step contains the following fields that define how the TGA 7 or the Pyris 1 TGA method will run:

Detail

Enter an identifying or descriptive comment for this step; a maximum of 40 characters.

Weight

You can specify whether the weight of the sample will increase or decrease during the run by selecting **Gain** or **Loss** from the drop-down menu next to Weight. Indicate the amount by which the sample weight is to change in order to have the method switch to the new scan rate by entering a value in the "is more than" field. Select the weight change units to use: mg/min or %/min. The units used for the rate of weight loss in the remainder of this section are determined by this selection. The entry made in this line is called the entrance criterion for the autostepwise scan.

Scan Rate

Select either **Scan Rate** or **Isothermal**. If the rate of weight loss is greater than the value you entered in line 1 (the entrance criterion), the TGA 7 will continue to scan the temperature at Scan Rate you enter here or hold the temperature constant (by selecting Isotherm). The default value for Isotherm is 10.0 min and for Scan Rate is 2.0°C/min.

Return to Original Rate

If during this reduced rate segment of the run the rate of weight loss is less than the value entered here, then the analyzer returns to the original scan rate. The entry made in this line is called the exit criterion.

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There must be a difference between the entrance and exit criteria in order for the autostepwise scan to be valid:

	minimum value for entrance criterion	**maximum value for entrance criterion**	**minimum value for exit criterion**	**maximum value for exit criterion**
high balance range (<1300 mg)	0.05 mg/min	9999.9 mg/min	0.05 mg/min	9999.9 mg/min
ultrasensitive balance range (<25 mg)	0.0005 mg/min	99.999 mg/min	0.0005 mg/min	99.999 mg/min
Low balance range (<130 mg)	0.005 mg/min	999.99 mg/min	0.005 mg/min	999.9 mg/min

	minimum value for entrance criterion	**maximum value for entrance criterion**	**minimum value for exit criterion**	**maximum value for exit criterion**
high balance range (<1300 mg)	0.01 %/min	999.9 %/min	0.01 %/min	999.9 %/min
ultrasensitive balance range (<25 mg)	0.01 %/min	999.9 %/min	0.01 %/min	999.9 %/min
Low balance range (<130 mg)	0.01 %/min	999.9 %/min	0.01 %/min	999.9 %/min

For low balance range:	999.99 ≥ entrance criterion ≥ exit criterion ± 0.01 mg/min
	(entrance criterion – 0.01 mg/min) ≥ exit criterion ≥ 0.01 mg/min

For high balance range:	9999.9 ≥ entrance criterion ≥ exit criterion ± 0.01 mg/min
	(entrance criterion – 0.01 mg/min) ≥ exit criterion ≥ 0.01 mg/min

Data Sampling Options

The options are Seconds Between Points and Number of Points.

Select Value

Depending on the Data Sampling Option, this field displays the seconds between data points collected in the data file or the number of points in the data file. Use the spin buttons to increase or decrease the displayed value. The Total points in Run value, displayed for some analyzers, is adjusted accordingly. The smaller the number of seconds between points, the more data points collected. If the number of points collected is small, you may miss an important event in the run.

Gas Change Section

The Gas Change section is enabled only if a GSA 7 or a TAGS is being used and has been selected in the Configure Analyzer dialog box. This section is used to create a gas program to switch the purge gas at specified times or temperatures. The gas program is affected by the method program as follows:

1. When a method step is shortened in time, the remainder of the gas program is deleted.
2. When a method step is changed from heat to cool, the gas program is deleted.
3. When a DDSC method is changed from Iso-Scan to Heat-Cool or vice versa, no change is made in the gas program.

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4. When a DSC method is changed to a DDSC method or vice versa, the gas program is deleted.
5. Real-time gas program editing is limited to temperature program steps that have not yet started.
6. During a run, if a temperature program step is shortened to the point that the gas program steps cannot be performed, those gas program steps are deleted.
7. When a method is edited in Data Analysis application, the choice of gases is the entire available list, plus Gas Off.
8. The number of gas changes per method step is 10 for a TAGS and GSA 7. If there is no accessory, no gas changes are permitted.

The Gas Change section contains the following controls:

This step begins with

The purge gas and flow rate that is to be used at the beginning of the highlighted method step is displayed. If the highlighted step is the first step in the method, the gas specified on the Initial State page is displayed. Beneath this line are displayed the gas program entries that you create with the parameters below. When you have selected the purge gas, time or temperature at which to switch, and the flow rate, click on the **Add 1** button to add the line to the list. If you want to delete a line from gas program, highlight it and click on **Delete**.

Gas Step

The number of the current gas step. The initial default gas step is step 0.

Change to

The drop-down list displays all available gases plus the option Gas Off. The gases attached to the GSA 7 or the TAGS and selected in Preferences are indicated with an asterisk. Highlight the gas to which you want the system to change.

At

Enter the time or temperature at which the system is to switch purge gases.

Flow

The flow rate entered in Preferences for the selected gas is displayed. Enter a new flow rate for the purge gas if desired.

Method Step Options Dialog Box

The Method Step Options dialog box appears when you select the **Add a Step** or **Insert a Step** button on the Program page of the Method Editor. It lists all available step types for the current analyzer that can be added or inserted into the method. Highlight a step type in the list and double-click to select it and close the dialog box. The Edit Step section on the Program page adjusts accordingly.

Available step types are

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- Temperature Scan (all analyzers)
- AutoStepwise Scan (TGA only)
- Isothermal (all analyzers)
- Repeat Steps (all analyzers)
- DDSC Repeated Scan (DDSC only)
- Frequency Scan (DMA)
- Frequency Multiplex (DMA)
- Dynamic Force/Stress (DMA only)
- Static Force/Stress (DMA/TMA)
- Creep Recovery (DMA/TMA)

Edit Temperature Scan / Edit AutoStepwise Scan Step

For all analyzers, a Temperature Scan varies the sample temperature according to the entries in this section of the Program Page. The sample temperature can be heated, held, cooled, or programmed in combinations. The associated change in the sample's characteristics, depending on the analyzer, are measured.

The Temperature scan is recommended for most DMA thermal analyses. It is often used for determining glass transition (Tg), α transition (Tα), β transition (Tβ), annealing, brittleness/brittle point, deflection temperature under load, and softening point. In a DMA temperature scan, the sample temperature is varied while the static force, dynamic force (stress), and frequency are held constant. The sample position, amplitude (strain), and phase lag are measured versus temperature.

Typical applications for the use of the TMA Temperature scan are for determining glass transition (Tg), coefficient of thermal expansion, LCTE, heat deflection temperature, deflection temperature under load, softening point, and processing effects.

For a TGA 7 and Pyris 1 TGA, there is an additional selection from the Method Options dialog box: AutoStepwise Scan. You can select only one AutoStepwise step per method program. For the Pyris 1 TGA, your method can have multiple steps embedded among Temperature Scan and Isothermal Scan steps. Repeat steps are also available. AutoStepwise Scan is **not** available for the Pyris 6 TGA.

The Edit Step section for AutoStepwise Scan is the same as that for Temperature Scan. The AutoStepwise feature uses programmable criteria to automatically determine the start and end points of a weight loss. It is a technique where thermogravimetric reactions (e.g., vaporization causing heavy weight losses) may be studied more accurately by reducing the scan rate in a temperature program or holding the temperature constant when such a reaction is detected. This detection is done via monitoring the rate of weight loss during a temperature scan. If the rate of weight loss is greater than a preset value, the instrument will either continue to scan the temperature at the preset reduced rate (stepwise scan) or hold the temperature constant (stepwise isotherm). If, during this "reduced rate segment," the rate of weight loss is less than a preset value, then the normal scan is resumed. These parameters are entered in the AutoStepwise Step Info section.

The Edit Temperature Scan Step section contains the following controls depending on the analyzer:

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From (Starting Temperature)

Displays the starting temperature for the step based on the ending temperature of the preceding step. If a temperature scan is the first step in the method, the beginning temperature is the Initial Temperature that was entered in the Initial State page or in the Initial Temp field on this page.

To (Ending Temperature)

Displays the user-entered ending temperature for the current step. For the TGA 7 and Pyris 1 TGA AutoStepwise scan, the temperature range for a standard furnace is ±1000.0°C and for a high-temperature furnace it is ±1600.0°C. The spinner button increments to To field value in 5°C increments.

Rate

Displays the user-entered heating or cooling rate for the current step. This is the rate at which the analyzer heats or cools in °C/min. The default value is the preceding step's rate. For the TGA 7 and Pyris 1 TGA AutoStepwise scan, the rate range for the standard furnace is 0.1°C/min to 200.0°C/min and for a high-temperature furnace the rate range is 0.1°C/min to 40.0°C/min.

Static Force (Stress), Dynamic Force (Stress), and **Frequency** are the initial values entered in the Initial State page. They are for display only and cannot be changed in this box.

Edit Isothermal Step

An Isothermal Scan holds the temperature constant while the response of the sample is measured as a function of time.

Typical applications for the use of an Isothermal scan for the DMA 7e and the TMA 7 include cure characterization, fatigue tests, materials comparison, degradation characterization, cycles to failure analysis, fast mechanical characterization, and multiplexing (frequency). In a DMA/TMA isothermal scan, the sample temperature, static force, dynamic force (DMA only), and frequency (stress) (DMA only) are held constant. The sample position and displacement amplitude (strain) are monitored versus time.

The Edit Isothermal Step section contains the following controls depending on the analyzer:

For (Time)

Displays the user-entered time limit for the current step, i.e., the length of time the analyzer is held at the indicated temperature. The default value is the same time as the preceding isothermal step.

At (Temperature)

Displays the temperature for the current step based on the ending temperature of the preceding step in the method. If an isothermal step is the first step in the method, the temperature is the Initial Temperature entered in the Initial State page.

Static Force (Stress), Dynamic Force (Stress), and **Frequency** are the initial values entered in the Initial State page. They are for display only and cannot be changed in this box.

Edit Repeat Steps

Repeat Steps is a convenient way of duplicating steps in your method program. You can then edit the added steps individually. For example, the program may contain an isothermal – scan – isothermal series of steps. You can repeat these three steps however many times as needed and then change the temperatures on individual steps. The following steps that are affected by the changes are changed automatically. There must be at least two steps in the method step list in

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order to use Repeat Steps. The Edit Repeat Steps section for all analyzers contains the following controls:

Add Steps After/Insert Steps Before

Add Steps After is the number of the step after which the repeated steps will be added. This is always the last step currently displayed. Insert Steps Before is the number before which the repeated step(s) will be inserted. This number is that of the step highlighted when Repeat Steps is selected.

From Step

Displays the number of the first step in the group of steps to be repeated.

To Step

Displays the number of the last step in the group of steps to be repeated.

Repeat

Displays the number of times the specified group of steps should be repeated.

Edit Heat-Cool Repeated Scan Step

Selecting DDSC Repeated Scan replaces all current steps in the method. There are two types of DDSC Repeated Scans: Heat-Cool and Iso-Scan. You can select the one you want by clicking on the button displayed in the Edit section:

Heat-Cool **Iso Scan**




These buttons display the temperature and rate parameters for the Repeated Scan step.

The Heat-Cool Repeated Scan is used to extract the glass transition event from other events which may be obscuring it, such as a recrystallization on heating or enthalpy effects caused by prior thermal and mechanical history of the sample. If you are interested in the Storage, Loss, and Tan Delta curves, the Heat-Cool Repeated Scan is recommended because a better dynamic signal is obtained with this temperature program. Better resolution and sensitivity of the measurements may also be obtained by using this program.

Starting Temperature

The starting temperature of the heat-cool scan based on the Initial Temperature entered in the Initial State page or in the Initial Temp field on this page.

Rate 1

The heating rate of the heat scan. This affects the 2nd Temp value displayed and Rate 2. You determine the rate of the first segment and the system calculates the rate of the remaining segment along with the ending temperature and the length of the run.

The rate during a Heat-Cool should be selected so that there is a balance between obtaining an optimum equilibration time for the segment and the overall length of the run.

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2nd Temp

The second temperature which is the ending temperature of the heat scan and the starting temperature of the cool scan. If you change this value, then the Rate 1 field changes. There should not be too much of a difference between this value and the initial temperature.

Rate 2

The cooling rate of the cool scan is displayed automatically when you enter a 2nd Temp value. You cannot change this value.

3rd Temp

Enter the temperature at the end of the cool scan for the current step. If you change this value, the Rate 2 value is adjusted automatically. There should not be too much of a difference between this value and the second temperature value.

Edit Iso-Scan Repeated Scan Step

Selecting DDSC Repeated Scan replaces all current steps in the method program. There are two types of DDSC Repeated Scans: Heat-Cool and Iso-Scan. You can select the one you want by clicking on the buttons displayed in the Edit section:

Heat-Cool **Iso Scan**




These buttons display the temperature and rate parameters for the Repeated Scan step.

The Iso-Scan Repeated Scan is used for heating through a crystalline melt or when cooling through the recrystallization of a crystalline material. It does not force crystallization by a cooling segment during a melt or force simultaneous melting during the crystallization of a cooling experiment as a heat/cool program would. Use Iso-Scan for general survey analyses of unknown samples.

The Edit DDSC Iso-Scan Repeated Scan Step section contains the following controls:

Starting Temperature

Displays the starting temperature of the step.

Isothermal

Enter the length of time for the isothermal portion of the step. The length of the isothermal segment should be selected so that there is a balance between obtaining an optimum equilibration time for the segment and the overall length of the run. The shorter the isothermal segment, the shorter the overall experiment will be. When the isothermal segment is short, the Calibration Factor becomes more important in obtaining accurate specific heat results. If equilibrium has been by the end of each step, you can use a Calibration Factor, entered in the Step Info section, of 1. Otherwise, a Calibration Factor should be calculated for the experiment.

Rate

The heating or cooling rate of the scan. This value is adjusted automatically when you change the 2nd Temp value.

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2nd Temp

The second temperature for the current step which is the ending temperature of the scan.

DDSC Repeated Scan Step Info Section

When the **Step Info** button is selected for a DDSC Repeated Scan step, the following Step Info Section parameters are displayed:

Detail

Enter a comment or identification for the highlighted step, up to 40 characters.

Total points in Run

Displays the total number of points in the run, calculated from all steps in the method. This value is updated automatically when you select the **Fine Tune** button or when you leave the Program page.

Calibration Factor

The Calibration Factor is an instrument calibration factor that corrects for the case when the sample does not have enough time to reach equilibrium. If equilibrium has been reached at the end of each step in a DDSC Repeated Scan step, enter a value of 1. Otherwise, you will have to calculate the Calibration Factor.

Number Repetitions

Enter the number of times to repeat the Heat-Cool or Iso-Cool scan specified in the Edit Step section.

End Temperature

Displays the final temperature of the run as calculated by the software. This value is updated automatically when you select the **Fine Tune** button or when you leave the Program page.

Total Time

Displays the total time of the run as calculated by the software. This value is updated automatically when you select the **Fine Tune** button or when you leave the Program page.

Fine Tune

Adjusts the values specified in the Edit Step section and displayed in the Step Info section. If certain values, such as temperature, are not valid for the method, an error message is displayed. You can adjust the values and click on **Fine Tune** again. If there are no problems, the method steps will be displayed with the proper values.

Calculating the Calibration Factor for DDSC

The Calibration Factor parameter in the Step Info section of a DDSC method is a factor that corrects for the case when the sample does not have enough time to reach equilibrium. The Calibration Factor provides an additional calibration procedure for the dynamic curve types for the DDSC: Storage Cp, Loss Cp, Complex Cp. This calibration has no effect on the Total Cp curve, Total Heat Flow, or Tangent Delta.

Determine the Calibration Factor to use in Step Info by dividing the Total Cp by the Storage Cp at a single point. The result is a multiplier factor that compensates for the effect on Storage Cp caused by a short equilibrium time. It is sensitive to the size, shape, and encapsulation of the sample. For improved accuracy, the Calibration Factor can be calculated for a given sample if that material has a temperature region where the specific heat is changing slowly.

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The sample is run over a short temperature region under conditions that are optimal to obtain an accurate Total Cp and Storage Cp. Display the two curves in Data Analysis. Use Event Mark to mark the curves and obtain the Y value for each.

DMA 7e Edit Frequency Scan Step

The typical applications for use of a frequency scan include fingerprinting, molecular weight differences, molecular weight distribution differences, resonant and harmonic effects, multiplexing (frequency and temperature or stress), and trend analysis. Frequency scanning is for viscoelastic samples. Highly elastic samples will produce “linear” results. The viscosity curve (damping) will not exhibit any curvature. Highly liquid samples may not have sufficient modulus for analysis.

In a frequency scan the frequency of the dynamic force applied to the sample is varied automatically. The static force, dynamic force (stress), and temperature are held constant. The sample position and displacement amplitude (strain) are measured. Never vary two variables at the same time. Do not use motor controls when performing frequency scanning. Whenever a motor control makes a change, the rate of the change almost always interferes with the rate of oscillation (frequency) and produces unexpected results.

The Edit Frequency Scan Step section for the DMA 7e contains the following controls:

From (Starting Frequency)

The initial frequency at the start of the scan from the Initial State page.

To (Ending Frequency)

Enter the frequency at which to end the scan.

Temperature

The initial temperature from the Initial State page remains the same during the scan.

Frequency Scan and MultiPlex Scan Step Info Section

The Step Info fields for a Frequency Scan and a Frequency MultiPlex Scan are displayed when the **Step Info** button is selected:

Detail

Enter a comment or identification for the frequency scan, up to 40 characters.

Static Force

This is display-only. This is the static force value entered in the Initial State page.

Dynamic Force

This is display-only. This is the dynamic force value entered in the Initial State page.

Force Used

This is display-only. It the sum of the static and dynamic forces and remains constant throughout the Frequency scan. The frequency of the dynamic force applied to the sample is varied.

Force Available

This is display-only. This the value of the force motor in your DMA 7e. The Force Used can go up to the total force available.

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Edit Frequency MultiPlex Step

The Frequency MultiPlex Scan step is used for generating data for a Time – Temperature Superposition curve. The procedure for performing time – temperature superposition measurements involves generating a multiplex data set and then shifting the set to create a master curve. The master curve is then used to make predictions. The highest-quality multiplex data sets are generated by repeatedly performing a scan of one variable (frequency) over a number of variables (selected temperatures).

NOTE: In addition to the entries made on the Program page, the values entered in the Equilibrate Within section of the Initial State page are in effect after each scan of the Frequency MultiPlex run.

The Edit Frequency MultiPlex Step section for the DMA 7e contains the following controls:

From (Starting Frequency)

The initial frequency value entered in the Initial State page is displayed. This is the frequency at which the multiplex scan begins.

To (Ending Frequency)

Enter the frequency at which to end the scan.

Increment after each scan

Enter the increment to use to change the temperature. The frequency scan is performed at each increment. The increment determines the number of times the scan is performed. For example, if the initial temperature is 60.00°C and the ending temperature is 90.00°C and the increment is 5°C, the number of repetitions is 7.

End Temperature

Enter the temperature at which the last frequency scan is to run.

When the **Step Info** button is selected for the Frequency Multiplex scan, the Step Info Section is displayed.

Edit Dynamic Force/Stress Scan Step

The typical applications for use of a dynamic stress scan include determination of storage modulus (E'), complex modulus (E*), loss modulus (E''), linear viscoelastic region, proportional limit and elastic limit, specific modulus, and mechanical differences. The program amplitude of the dynamic force (sinusoidal oscillation) is varied in a dynamic stress scan. The static force can be programmed to change proportionally with the increasing dynamic force to assure that the sample remains in tension throughout the analysis. The frequency and temperature are held constant. The sample position and displacement amplitude (strain) are measured.

The Edit Dynamic Force/Stress Scan Step section for the DMA 7e contains the following controls:

From

The initial dynamic force, i.e., the dynamic force at which to start the scan. This value is from the Initial State page.

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To

Enter the ending dynamic force, i.e., the dynamic force at which to stop the scan. This value is incremented in 10-mN steps.

Rate

Enter the rate at which to increase the dynamic force from the initial to the ending value. The rate is increased in 5-mN/min intervals.

Temperature

The initial temperature, entered in the Initial State page, is the sample temperature throughout the scan since the temperature is held constant.

Frequency

The initial frequency, entered in the Initial State page, is the oscillation frequency throughout the scan since the frequency is held constant.

Edit Static Force/Stress Scan Step

Typical applications for the use of a static force/stress scan include determining the modulus (E), Young's modulus (Y), linear viscoelastic region, proportional limit, specific modulus, and mechanical differences. The program static force (stress) is varied using a static force scan (creep ramp). There is no dynamic force. The temperature is held constant. The sample position (strain) is measured.

The Edit Static Force/Stress Scan Step section for the DMA 7e and TMA 7 contains the following controls:

From (Starting Static Force)

The initial static force, i.e., the static force at which to start the scan. This value is from the Initial State page.

To (Ending Static Force)

Enter the ending static force, i.e., the static force at which to stop the scan. This value is incremented in 10-mN steps.

Rate

Enter the rate at which to increase the static force from the initial to the ending value. The rate is increased in 5-mN/min intervals.

Temperature

The initial temperature, entered in the Initial State page or in the Initial Temp field on this page, is the sample temperature throughout the scan since the temperature is held constant.

Edit Creep/Recovery Step

Typical applications for the use of creep/recovery scan include determining equilibrium modulus (E_e), equilibrium viscosity (η_e), relaxation map analysis, relaxation spectra, and mechanical differences. Equilibrium mechanical behavior of thermoplastics, thermosets, and elastomers is characterized for a specific stress at a specific temperature. The sample is taken from an equilibrium state with respect to static stress and temperature to a new static stress. (Shrinkage

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force measurements can be made with the Position Control on.) The time-dependent displacement response (strain) is measured. There is no dynamic force or frequency. The temperature is held constant. You can choose to vary the temperature, but in this case you must be sure that the isotherms are of equal length. The sample position (strain) is measured.

The Edit Creep/Recovery Scan Step section for the DMA 7e and TMA 7 contains the following controls:

Equilibrate

The scan begins at the initial static **Force** entered in the Initial State page. The system equilibrates for the amount of time entered in the **Time** field.

Creep

Enter the creep **Force** and the amount of **Time** the force is to be applied.

Recovery

Enter the recovery **Force** and the amount of **Time** the sample has to recover from the application of force.

Temperature

The initial temperature entered in the Initial State page or in the Initial Temp field on this page is displayed. You can have the temperature change by clicking in the check box to activate the temperature entry field. Enter a temperature to which to **Scan To.**

At

The rate at which the temperature is to increase or decrease during the creep/recovery scan if you elect to vary the temperature. It is derived from the change in temperature over the total amount of time of the scan (the equilibrate, creep, and recovery times).

Creep Recovery Step Info Section

When the **Step Info** button is selected, the Creep Recovery Step Info section containing the following parameters is displayed:

Detail

Enter a comment or identification for the highlighted step, up to 40 characters.

Data Sampling Options

Displays the data sampling option to be used for displaying of other values in this section. If you select Seconds Between Points, the Select Value field becomes effective. Change the seconds between data points collected and the Total Points in Run will be adjusted accordingly. The other choice is Number of Points. Select this option and the Select Value field displays the total number of points in the run as calculated from the creep time, recovery time, and number of repetitions.

Select Value

This field works in conjunction with the Data Sampling Options selection. It displays the seconds between data points collected (in seconds) or the total number of points to be collected in the run. You can change the seconds between points collected by using the spin buttons (you cannot type in an entry). If you change the Select Value entry, the total points in the run is adjusted accordingly.

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Repetitions

Enter the number of times to repeat the creep – recovery scan. This value affects the total number of points of the run, total time of the run, and the scanning rate used to get to the final scan temperature, if selected.

Total Points in Run

The total points collected during the creep – recovery run. This value is updated automatically if you change the creep time, recovery time, and the number of repetitions.

Total Time

The total time of the run as calculated by the software and dependent on the creep time, recovery time, and the number of repetitions of the creep – recovery scan.

Force Available

This is the value of the force motor in your DMA 7e. This is display only.

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Chapter 6
Menus, Dialog Boxes, and Toolbars

The items in the menu bar across the top of the Pyris window, underneath the title bar, vary depending on what Pyris window is displayed – Method Editor, Instrument Viewer, Data Analysis, Remote Monitor, or Pyris Player. The items in each drop-down menu also vary according to what type of analyzer you are using, what type of curve is displayed, and so on. The menu bar is inactive when a dialog box is displayed. Items displayed in gray lettering in the drop-down menus are not available with the current menu title selected.

- File Menu
- Edit Menu
- View Menu
- Display Menu
- Curves Menu
- Math Menu
- Calc Menu
- Restore Menu
- Tools Menu
- Window Menu
- Help Menu

In addition to menus, each window displays a toolbar. Most of the items on the toolbars appear in drop-down menus. The toolbars in Pyris are summarized at the end of the chapter.

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File Menus

The contents of a File menu varies depending on where you are in Pyris Software for Windows.

Instrument Viewer File Menu

The following items are on the File menu when the Instrument Viewer is displayed:

Print

This command is used to print the data file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box by typing **Ctrl + P** or clicking on the **Print** button on the toolbar: 

Print Dialog Box

The standard Windows Print dialog box appears when you select the **Print** command in the File menu throughout Pyris Software for Windows or the **Print** button  on the toolbar. It is the same dialog box you see when you print in other applications on your computer. The options in this dialog box allow you to specify how the data, method, calibration file, or play list should be printed.

Name

This is the name of the active printer. Select the drop-down arrow to display additional printers available to you. The four fields – Status, Type, Where, Comment – are informational only. They cannot be changed.

Print to file

Click in this checkbox to print the information to a file rather than directing it to the printer. You will be prompted to specify a file name and location.

Properties

Click on this button to set up options for the selected printer. The options available depend on the printer.

Print Range

Specify the pages you want to print:

- All prints the entire document.
- Pages prints the range of pages you specify in the from and to boxes.
- Selection prints the currently selected text.

Copies

Specify the number of copies you want to print for the specified page range.

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Collate

When more than one copy is to be printed, the Collate button is activated. Prints copies in page number order instead of separated multiple copies of each page.

Print Progress Dialog Box

The Printing dialog box is shown during the time that Pyris Software for Windows is sending output to the printer. The page number indicates the progress. To abort the print job, select the **Cancel** button.

Print Setup

This command allows you to first set up the page on which the curves will be printed, i.e., set headers and footers, and then set up the printer options. The Page Setup dialog box is displayed when the command is selected.

Page Setup Dialog Box

The Page Setup dialog box appears when you select **Print Setup** from the File menu in Instrument Viewer and Data Analysis. It comprises three tabbed pages: General, Header, and Footer. The fields in these pages are as follows:

General

Printer Name: The name assigned to the default printer of your system when configured in Windows. It is displayed for information only. You install printers and configure ports for your system using the Control Panel in Windows.

Title: Enter a title to be printed at the top of the printout. If you also include a header, the title appears below the header. The default is the title entered in the Graph page in Preferences. Click in the **Include Title** checkbox to include the title in the printout.

Margins: Enter or use the spin buttons to select the top, left, bottom, and right margins of your printout. The default values are 0.25 in. for each margin.

Setup: This button displays the Print Setup dialog box.

Header

Include Header: Click in the checkbox to have a header included in the printout.

Header Components: The components available for inclusion in the header of the printout are listed in this page. Click within the box to select/deselect the item. These items are from the Method Editor Sample Info page and the data file. Poisson's Ratio and Geometry apply to the DMA 7e and TMA 7 only.

Footer

Include Footer: Click in the checkbox to have a footer included in the printout.

Footer Components: Select whether you want the method steps of the method program and the date and time the data were collected printed in a footer at the bottom of the page.

Exit

Select this command to exit and close all parts of Pyris Software for Windows. You can also exit the Instrument Application by double clicking on the Control menu button at the upper-left-hand corner of the window or using the **Alt + F4** keys.

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Method Editor File Menu

The items on the File menu while in the Method Editor are as follows:

New Method

Use this command to create a new method. When you select this command, the default method is loaded into the Method Editor. Edit the default method and select **Save As** from the File menu to save the new method under a new file name; the default method remains unchanged. You can also execute the **New Method** command by typing **Ctrl + N** or by clicking on the **New** button on the toolbar: 

Open Method

Use this command to open an existing method. When you select this command, the Open Method dialog box appears. You can also access the Open Method dialog box (see below) by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Merge Method

Use this command to append the steps from an existing and saved method to the steps in the method currently loaded in the Method Editor. When you select this command, the Merge Method dialog box (see below), which is the same as the Open Method dialog box, appears. As soon as you select another method, its program lines are appended to those the current method's program on the Program page.

Save Method

Use this command to save the method currently loaded in the Method Editor. If the method is new and has not yet been saved, the Save As dialog box (see below) appears. You can save the method quickly by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Save Method As

Use this command to save the method currently loaded in the Method Editor under a different file name. When you select this command, the Save As dialog box (see beloe) appears.

Print

Use this command to print the current method file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box (see above) by typing **Ctrl + P** or by clicking on the **Print** button on the toolbar: 

Print Preview

Use this command to see what the current method file will look like when it is printed. The Print Preview window appears when you select this command. You can also access the Print Preview window by clicking on the **Print Preview** button on the toolbar: . The Print Preview window has its own toolbar.

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Exit

Select this command to exit and close all parts of Pyris Software for Windows. You can also exit the Instrument Application by double clicking on the Control menu button at the upper-left-hand corner of the window or using the Alt + F4 keys.

Calibration Window File Menu

The items in the File menu while in the Calibration window are as follows:

Open

Use this command to open an existing calibration file. When you select this command, the Open dialog box appears. You can also access the Open dialog box (see below) by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Save

Use this command to save the current calibration file. When you select this command, the file is saved automatically and will overwrite the existing file. If the calibration is new and has not yet been saved, the Save As dialog box (see below) appears. If you want to save the calibration file under a different file name, use **Save As**. You can also save the file by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Save As

Use this command to save the current calibration file under a new file name. When you select this command, the Save As dialog box (see below) appears.

Print

Use this command to print the current calibration file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box (see above) by typing the shortcut keys **Ctrl + P** or by clicking on the **Print** button on the toolbar: 

Print Preview

Use this command to see what the current calibration file will look like when it is printed. The Print Preview window appears when you select this command. You can also access the Print Preview window by clicking on the Print Preview button on the toolbar: . The Print Preview window has its own toolbar.

Print Setup

Use this command to select the standard Windows printing options before printing the calibration file. The Print Setup dialog box (see Pyris Player below) appears when you select this command.

Exit

Select this command to exit and close all parts of Pyris Software for Windows.

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Data Analysis File Menu

The items on the File menu while in the Data Analysis Application are as follows:

New Data

This command is used to select a new data file for display in the Data Analysis window. From the New Data File dialog box (see below), select the file you want to display. Any data that was displayed in the Data Analysis window is cleared and replaced by the new data file. You can also access the New Data File dialog box by typing **Ctrl + N** or by clicking on **New** button on the toolbar: 

Open Data

This command is used to open an existing data file in a **new** Data Analysis window. All other open Data Analysis windows remain open. From the Open Data File dialog box (see below), select the new data file you wish to display. You can also access the Open Data File dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the standard toolbar: 

Add Data

Select this command to add an existing data file to the active Data Analysis window. From the Add Data File dialog box select the data file you want to add to the display. That data file becomes the active curve. You can also access the Add Data File dialog box by typing **Ctrl + A** or by clicking on the **Add Curve** button in the toolbar: 

Close Data

This command closes the active Data Analysis window. Other open Data Analysis windows remain open. If any changes have been made to the active curve and have not yet been saved when you select **Close Data**, a dialog box listing the data files that have been modified is displayed, giving you a chance to save all or none of the changes.

Save Data

Select this command from the File menu to save the active curve and all derived curves, results, constructs, and annotations using the current file name. The data file is saved automatically without requesting confirmation, so be sure you want to save the file as is. If you want to save the data under a different file name, select **Save Data As**. You can also save the data file quickly by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Save Data As

Select this command to save the active curve and all derived curves, results, constructs, and annotations using a different file name. When you select this command, the Save Data As dialog box appears. You can also access the Save Data As dialog box (see below) by typing **Ctrl + S.**

Save All

Select this command to save all data files currently displayed.

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Print

This command is used to print the loaded data file. The standard Print dialog box appears when you select this command. You can also access the Print dialog box by typing **Ctrl + P** or clicking on the **Print** button on the toolbar: 

Print Preview

Select this command to see what the current data file will look like when it is printed. The Print Preview window appears when you select this command. You can also access the Print Preview window by clicking on the **Print Preview** button on the toolbar: . The Print Preview window has its own toolbar.

Print Setup

This command in Data Analysis allows you to first set up the page on which the curves will be printed, i.e., set headers and footers, and then set up the printer options. The Page Setup dialog box is displayed when the command is selected.

Exit

Select this command to exit and close all parts of Pyris Software for Windows.

Pyris Player File Menu

The following items are on the File menu when the Pyris Player window is open:

New Player

Use this command to create a new play list. When you select this command, the default Pyris Player file is loaded; the Edit Play List page is blank. Edit the default Player file and select **Save As** to save it under a new file name and retain the default Player file. You can also execute the **New Player** command by typing **Ctrl + N**.

Open Player

Use this command to open an existing play list file. When you select this command, the Open dialog box appears. You can also access the Open dialog box by typing **Ctrl + O** or by clicking on the **Open** button on the toolbar: 

Open Dialog Box

Depending on where you are in the software, the **Open** command on the File menu displays one of the following dialog boxes:

- Open Method
- Merge Method
- Open Data File
- New Data File

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- Open Calibration File
- Open Player

Look in

Lists the available folders and files. To see how the displayed folder fits into the hierarchy of drives and directories on your computer and network, click on the down arrow. To see what is inside a folder, click on it.

The box below the Look in field shows the folders and files in the selected location. You can click on a folder or a file in this box to open it. To open a folder one level higher, click on the Up One Level button .

File name

Instead of selecting the file from the list in the box above, you can type the name of the file you want to open into this field. You can use an asterisk as a wildcard. If necessary, you can enter the full path of the file.

Files of type

Lists the type of files to display. This is useful for narrowing the list of files displayed to only the files you are interested in. Use the down arrow to see a drop-down list of file extensions.

Save Player

Use this command to save the current play list file. When you select this command, the file is saved automatically and will overwrite the existing file. If the play list is new and has not yet been saved, the Save As dialog box (see below) appears. If you want to save the play list file under a different file name, use the **Save As** command. You can also save the file by typing **Ctrl + S** or by clicking on the **Save** button on the toolbar: 

Save Player As

Use this command to save the current play list under a new file name. When you select this command, the Save As dialog box appears.

Save Dialog Box

Depending on where you are in the software, the **Save** and **Save As** commands on the File menu display a dialog box with the fields described below. If you are creating a new method or play list or saving a new data file or calibration file and select **Save**, the Save As dialog box is displayed. This dialog box is also displayed when you click on the **Save** button  on the toolbar.

Save in

Lists the available folders and files. To see how the current folder fits into the hierarchy of drives and directories on your computer and network, click the down arrow. To see what is in a folder, click on it.

The box below the Save in field shows the folders and files in the selected location. You can also double click on a folder or file in that box to open it. To open the folder one level higher, click on the One Level Up button .

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File name

If you wish to save an existing file under a different file name, type the new file name in this field. Pyris Software for Windows supports long file names, i.e., up to 255 characters. Pyris adds the extension you specify in the Save as type box. If you wish to save an existing file under the existing file name, click on **OK** to accept the file name displayed.

Save as type

Specifies the type of file you are saving. The list includes all the available file types that this program can recognize. Each analyzer has its own associated extension for method files, calibration files, data files, and play list files.

Print

Selecting **Print** from the File menu or the **Print** button on the toolbar from any Pyris Player page initially displays the Print Type dialog box (see below) from which you choose the type of printout you want: summary or detailed. The summary printout includes the main-level items Prepare Sample, Data Analysis, and Sample Group which includes the Sample List and the Data Analysis List entries. The detailed printout includes the same information as a Summary printout but also includes additional information on the Sample List entries in a Sample Group.

When **Detail** is selected in the Print dialog box for View History or Sample History, the same information as given in **Summary** is given in addition to the date and time each event occurred, the method used, the data file name, and the sample weight.

History Summary yields a printout that includes just one-line entries for each time the play list was played back. The entry gives the date and time of the run.

In all cases, click on the **Print** button in the dialog box to display the standard Print dialog box.

You can also access the initial printing dialog box by typing the shortcut keys **Ctrl + P** or by clicking on the **Print** button on the toolbar with any of the six pages displayed: 

Pyris Player Print Type Dialog Box

When you select **Print** from the File menu in all pages in Pyris Player, an initial dialog box appears in which you select the type of printout you want: detailed or summarized. For the View History page and the Sample History page, an additional selection, History Summary, is available. Select the desired option by clicking on the radio button and then click on the **Print** button.

An example of a Summary printout is as follows:

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1: Sample Group:
1.1: Sample List: C:\Program Files\Pyris\Methods\short_iso.tg1m
1.1.1: Sample: @1: 1.811mg; C:\Program Files\Pyris\Data\samp1.tg1d
1.1.2: Sample: @2: 1.010mg; C:\Program Files\Pyris\Data\samp2.tg1d
1.1.3: Sample: @3: 1.790mg; C:\Program Files\Pyris\Data\samp3.tg1d
1.2: Data Analysis List:
1.2.1: Display Curve: Weight: Using Current Run
1.2.2: Peak Area: 1.2.1: Display Curve: 0.00 min to 3.40 min

A Detail printout of the same information may appear as follows:

1: Sample Group:
1.1: Sample List: C:\Program Files\Pyris\Methods\short_iso.tg1m
1.1.1: Sample: @1; 1.811mg; C:\Program Files\Pyris\Data\samp1.tg1d
Method: C:\Program Files\Pyris\Methods\short_iso.tg1m
Data: C:\Program Files\Pyris\Data\samp1.tg1d
Weight: 1.811 mg
1.1.2: Sample: @2; 1.010mg; C:\Program Files\Pyris\Data\samp2.tg1d
Method: C:\Program Files\Pyris\Methods\short_iso.tg1m
Data: C:\Program Files\Pyris\Data\samp2.tg1d
Weight: 1.010 mg
1.1.3: Sample: @3; 1.010mg; C:\Program Files\Pyris\Data\samp3.tg1d
Method: C:\Program Files\Pyris\Methods\short_iso.tg1m
Data: C:\Program Files\Pyris\Data\samp3.tg1d
Weight: 1.790 mg
1.2: Data Analysis List:
1.2.1: Display Curve: Weight: Using Current Run
Start at Step: 1; End at Step: 4; Endo Up: On; Start at Time Zero: Off
1.2.2: Peak Area: 1.2.1: Display Curve: 0.00 min to 3.40 min
Include: Height, Onset, End, Standard Baseline

When the View History page is displayed, the **History Summary** selection in the Print dialog box results in a printout of the history of a run that includes all the steps in the list. For a play list that includes a Sample Group, this means that the steps generated by the program as the play list is run are included. For example, for a Pyris 1 TGA with autosampler, when a Sample line in a Sample List is encountered, the program expands this line to include:

Prepare Sample
Load Sample
Raise Furnace
Start Method
Lower Furnace
Return Sample

If an error occurred, it is included in the summary printout.

Print Selected Item Only

Select this option to print the selected history information (summary or detail) of the focused history item. Otherwise, all history entries are printed.

Print

Displays the standard Print dialog box from which you initiate the printing process.

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Print Preview

Use this command to see what the current play list, sample history, or history list will look like when it is printed. Selecting **Print Preview** from the File menu initially displays Print Type dialog box from which you choose the type of preview you want: detailed or summary. Click on the **Print** button to display the Print Preview screen. Like the **Print** command, Summary will display just the main-level items in a play list. If a Sample Group is included, then the Sample List and Data Analysis List will also be displayed. For View History, Summary will display all the lines in the play list. Detail preview includes all lines in the play list and, for View History, preview includes date and time and error messages.

From the Print Preview screen, click on the **Print** button to display the standard Print dialog box. Click on the **Print** button in this box to print the display.

You can also access the Print Type dialog box for previewing by clicking on the **Print Preview** button  on the toolbar with any Pyris Player page displayed.

Print Setup

Use this command to select the standard Windows printing options before printing the calibration file or the play list. The Print Setup dialog box appears when you select this command.

Print Setup Dialog Box

The Print Setup dialog box appears when you select the **Print Setup** command in the File menu in Calibrate and Pyris Player. It also is displayed when you click on the **Setup** button in the Page Setup dialog box which is displayed first when you select **Print Setup** in the File menu in Instrument Viewer and Data Analysis. It is the standard Windows Setup dialog box you see in other applications on your computer. It contains the following fields:

Name

Select the printer you want to use from the list of printers available to you. The default printer of your system is the default. Click on the down arrow to see additional printers. You install printers and configure ports for your system using the Control Panel.

Properties

Click this button to access the dialog box in which you set up options for the printer. The options available depend on the features of the printer.

Size

Select the size of paper on which the document is to be printed.

Source

Some printers offer multiple trays for different paper sources. Specify the tray here.

Orientation

Choose Portrait or Landscape.

Network...

Choose this button to create a connection to a network printer. You must have the proper printer driver available.

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Exit

Select this command to exit and close all parts of Pyris Software for Windows.

Edit Menu

The contents of an Edit menu varies depending on where you are in the software but could contain the following items:

Undo

This command is not available from any window at this time.

Modify

This command appears in the Edit menu of Method Editor but is grayed out. It is unavailable at this time.

Delete

This command is available in Instrument Viewer, Data Analysis, and Remote Monitor. Select this command to delete the selected object such as the active curve or an annotation from a Data Analysis or Instrument Viewer window automatically. You can also delete the focused item quickly by typing **Shift** + **Delete** or by clicking on the **Delete Object** button on the toolbar: 

Copy

The function of this command depends on where in the software it is invoked.

In Pyris Player, use this command to copy a Sample Group or a Sample line in a play list to the clipboard. If the focused line is Sample Group when you select **Copy**, the entire Sample Group block is copied, i.e., Sample List and Data Analysis List. You can then select **Paste** from the Edit menu to paste the Sample Group immediately after the focused Sample Group. If the focused line is a Sample line when you select **Copy**, then **Paste** will insert another Sample line immediately after the focused line.

If you are in Instrument Viewer, the **Copy** command will copy method information such as file name, program steps, and so on, to the clipboard which can then be pasted into another document such as Word.

In Data Analysis, **Copy** places the X,Y data of the focused curve onto the clipboard. It can then be used in Excel or another software product.

An equivalent way to copy is to select the **Copy** button on the toolbar: 

Copy Image

This command is used to copy the Instrument Viewer or the Data Analysis window exactly as displayed. The image is copied to the clipboard from which it can be pasted into another application such as PowerPoint. The image does not include peripheral items such as the toolbar or control panel that you get when you perform a screen capture. The image contains the curves, axes, and axes labels. The pasted object can then be manipulated in another application, i.e.,

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resized, and its aspect ratio is maintained. This feature is the same as the Copy Image item on the menu seen in Data Analysis when you right click on the mouse.

Paste

Use this command to paste the items on the clipboard (by use of the **Copy** command) into the play list. The item or items are inserted immediately after the copied line or lines. You can then edit the line or lines as needed, e.g., the locations of the samples in a Sample Group. Another way to paste is to click on the **Paste** button on the Pyris Player standard toolbar: 

View Menus

The contents of a View menu varies depending on where you are in Pyris Software for Windows:

Instrument Viewer View Menu

The following items are on the View menu when the Instrument Viewer is displayed:

Calibrate

Select this command to access the Calibration window for the current analyzer. The Calibration window consists of tabbed pages, one for each type of calibration that you can perform on the analyzer.

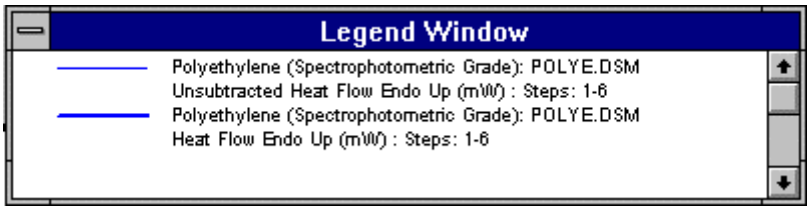
Monitor

Select this command to activate/deactivate the monitoring of the real-time status of an analyzer in the Instrument Viewer before the start of a run. You can also initiate the Monitor feature by clicking on the **Monitor** button on the toolbar: 

Legend

This command on the View menu in Instrument Viewer and Data Analysis displays the Legend window. The Legend window shows a description of each of the curves displayed. You can toggle the Legend window on and off by clicking on the **Legend** button  on the toolbar, selecting **Legend** from the View menu, or typing the **Ctrl + F1** key combination.

The Legend window is a small movable and sizable window containing information about the curves displayed. The curves are listed in the legend in the order in which they are opened. You can use the Legend window to quickly view important characteristics about a curve as well as make a curve active.



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The Legend window contains the following information about all curves displayed:

Line Type and Color displayed on the left side of the window. These are determined in the General Preferences page and Color Preferences page.

Sample ID and Data File Name displayed on the first line next to the line type.

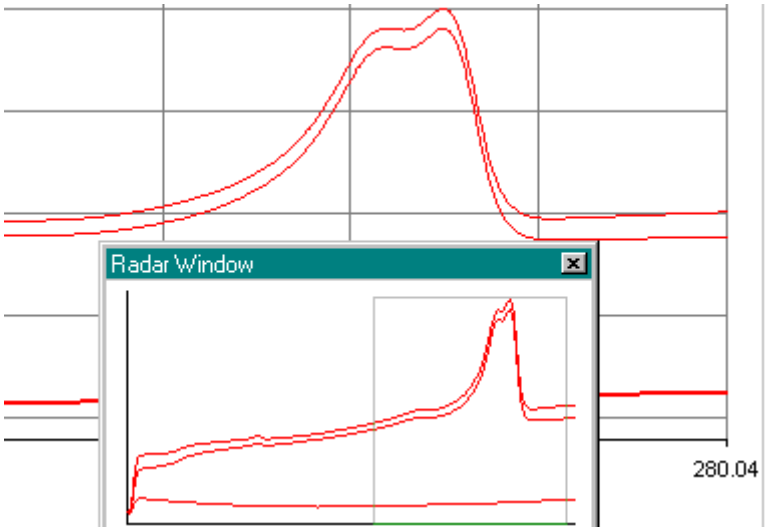
Step ID and Step Number displayed on the second line, with Total Number of Steps if there is more than one step in the method used to collect the data.

Units displayed next to Step ID in parentheses.

You can make a curve the active curve by positioning the dotted box around the curve description in the Legend window and clicking. The curve is then displayed as a thick solid line.

Radar

This command displays the Radar window:



This window shows the active curve in the Data Analysis window or the Instrument Viewer window at full scale while the curve in either of those windows is manipulated. In the Radar window the section of the full curve that is displayed in the Data Analysis or Instrument Viewer window is indicated by a rubberband box around it. This box can be manipulated by click-and-drag to change which area of the full curve is displayed in the Data Analysis or the Instrument Viewer window. The active curve is redrawn automatically to the new scale.

You can also access the Radar window by clicking on the **Radar** button on the Rescale Tools toolbar: 

You can toggle the Radar window display on and off by selecting Radar in the View menu or by clicking on the **Radar** button.

Grid

Select this command to toggle the X – Y grid on and off. You can also activate the grid by typing **Ctrl + F2** or by clicking on the **Grid** button on the toolbar: 

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Rescale Tools

The Rescale Tools command will display or remove from display the Rescale Tools toolbar.



The commands on the toolbar can be used to rescale a real-time curve in Instrument Viewer.

Toolbar

Select this command to display the Instrument Viewer standard toolbar. If the toolbar is already displayed, select this command to remove the toolbar.



Status Panel

Select this command to display the Status Panel of the current analyzer whether you are using the Method Editor, Instrument Viewer, or Data Analysis. In Remote Monitor, the status panel for the instrument on the monitored remote PC is displayed. It is the same status panel that you would see for an analyzer on your own PC.

This is a toggle item on the View menu. The Status Panel is a dockable panel and can be adjusted in size to display as many items as you need.

Control Panel

Select this command to display the instrument's control panel in an Instrument Application whether you are using the Method Editor, Instrument Viewer, or Data Analysis for the current analyzer. The item is grayed out in the View menu in Remote Monitor. You cannot display the control panel of a remotely monitored instrument.

Control Panel is a toggle item. The control panel is a dockable panel. The control panel contains buttons that control certain features of the analyzer, including starting and stopping a method, programming the temperature, cleaning the furnace, and changing the purge gas.

Method Editor View Menu

The items on the View menu while the Method Editor is displayed are as follows:

Calibrate

Select this command to access the Calibration window for the current analyzer. The Calibration window consists of tabbed pages, one for each type of calibration that you can perform on the analyzer.

Thermal Program

Select this command to toggle the Thermal Program window on and off. The Thermal Program window displays the method's steps in graphical form, temperature on the Y axis and time on the X axis. The graph is updated automatically as you change the program.

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Toolbar

Select this command to display the Method Editor standard toolbar. If the toolbar is displayed, select this command to remove the toolbar from display.



Status Panel

Select this command to display the Status Panel of the current analyzer whether you are using the Method Editor, Instrument Viewer, or Data Analysis. In Remote Monitor, the status panel for the instrument on the monitored remote PC is displayed. It is the same status panel that you would see for an analyzer on your own PC.

This is a toggle item on the View menu. The Status Panel is a dockable panel and can be adjusted in size to display as many items as you need.

Control Panel

Select this command to display the instrument's control panel in an Instrument Application whether you are using the Method Editor, Instrument Viewer, or Data Analysis for the current analyzer. The item is grayed out in the View menu in Remote Monitor. You cannot display the control panel of a remotely monitored instrument.

Control Panel is a toggle item. The control panel is a dockable panel. The control panel contains buttons that control certain features of the analyzer, including starting and stopping a method, programming the temperature, cleaning the furnace, and changing the purge gas.

Data Analysis View Menu

The items on the View menu while the Data Analysis window is displayed are as follows:

Method Used

Select this command to display the parameters of the method that was used to collect the active curve's data. The parameters are presented in the View Method Properties dialog box, which comprises seven pages, each of which you can print out or copy to an ASCII file. You can also display the method used by clicking on the **Method Used** button on the toolbar: 

View Method Properties Dialog Box

The View Method Properties dialog box, which comprises six pages, appears when you select the **Method Used** command in the View menu of a Data Analysis window or the **Method Used** button from the standard toolbar. The pages contain related parameters of the method used to collect the data that are displayed as the focused curve. The six pages are as follows:

- Sample Info Page
- Initial Conditions Page
- Equilibrate Within Page
- Calibration Page

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- Validation Page
- Method Step Information Page

Sample Info Page

The parameters on this page are the same as those on the Sample Info page of the Method Editor. They are saved in the data file. In addition, the date and time the data were collected and the total points in the run are displayed. The other items displayed depend on the analyzer. You can change the Sample ID, Operator ID, and Comment fields on this page.

Initial Conditions Page

The parameters displayed on this page are those entered in the Initial State page of the Method Editor. For the DMA 7e, this also includes the Set Controls values. They are saved in the data file. The items displayed depend on the analyzer. You cannot change any of the values displayed.

Equilibrate Within Page

The parameters displayed on this page are those selected in the Equilibrate Within section of the Initial State page of the Method Editor. If an equilibration item was selected, the check box next to that item is checked and the entered value is displayed. You cannot change any of the values displayed on this page.

Calibration Page

There are only two items displayed on this page: **Filename** — the name of the calibration file that was in effect when the data file was collected, and **Date/Time** — the date and time that the calibration file was created. You cannot change any of the values on this page.

Validation Page

This page contains only three items: **Validated** indicates whether the method was validated. **By** displays the name of the person who validated the method. **Date** is the date the method was validated. You cannot change any of the values on this page.

Method Step Information Page

This page displays the step information for each method step entered in the Program page of the Method Editor. When you highlight a step, the number of data points collected during that step is displayed as well as the gas steps associated with it. You cannot change any of the information on this page.

Buttons

Each page contains four buttons: **OK**, **Cancel**, **Print**, and **Create**. Click on **OK** to accept any changes made and close the View Method Properties window. Click on **Cancel** to ignore any changes made and to close the View Method Properties window. The **Print** button displays the Print Data File dialog box and the **Create** button displays the Create Data File dialog box.

Print Data File Dialog Box

This dialog box is displayed when you select **Print** in any of the View Method Properties pages. In addition to the method file, you can also include the data points of the data file with which this method file is associated and also the calibration information. The path of the method file is displayed as well.

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The **Print** button displays the standard Windows Print dialog box in which you set printing options and initiate the printing process. The printout lists all of the information displayed on the View Method Properties pages.

Create Data File Dialog Box

This dialog box is displayed when you select **Create** in any of the View Method Properties pages. Use this button to create an ASCII file of the method file. The path and file name under which the ASCII file will be saved is displayed. Click in the check box next to Include Data Points if you wish to include all data points in the ASCII file, and in the check box next to Include Calibration Information to include the calibration information in the ASCII file. When you click on **OK,** the method is saved to an ASCII file with the same name as the data file but with a .TXT extension. It is saved in the same directory as the data file.

Results

Select this command to display calculation results that were saved with the active curve. In the View Results dialog box you indicate which calculation results to display from the presented list. You can also delete results. Also shown in the dialog box are curves that cannot be displayed on the current axes.

View Results Dialog Box

The View Results dialog box appears when you select the Results command in the View menu of the Data Analysis window. It contains the following controls:

File

The directory path and file name of the active curve.

Select calculation results to display

Highlight one or more items on this list of calculation results that have been saved with the active curve. The results listed are viewable on the current axes.

Results that cannot be displayed on current axes

A list of results that cannot be shown in the Data Analysis window with the current axes.

View Results

Closes the dialog box and displays the highlighted results.

Cancel

Closes the dialog box without performing the operation.

Select All

Highlights all results in the list. All of the results will be displayed with the active curve.

Delete Results

Deletes the highlighted result(s) from the list. The results will be deleted from the data file **only** if you resave the data file.

Legend

See Instrument Viewer View Menu above.

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Radar

See Instrument Viewer View Menu above.

Grid

See Instrument Viewer View Menu above.

Rescale Tools

See Instrument Viewer View Menu above.

Toolbar

Select this command to toggle the display of the standard toolbar for a Data Analysis window on and off.



Status Panel

See Instrument Viewer View Menu above.

Control Panel

See Instrument Viewer View Menu above.

Pyris Player View Menu

The items on the View menu while in Pyris Player are as follows:

View Dependencies

When you select View Dependencies from the View menu in Pyris Player, the Dependencies List dialog box appears. The list includes all of the play list items that depend on the focused line in the list. This information is important when you want to delete the entry.

Dependencies List Dialog Box

The Dependencies List dialog box is displayed when you select **Show Dependencies** from the View menu in Pyris Player and if you select **Delete step** in the Player Steps area. It shows all of the play list entries that are dependent on the focused play list item. For example, you may have a play list containing the following lines:

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..

1.4: Start Method: . . .

.

.

.

2: Data Analysis: . . .

2.1: Display Curve:

2.2: Pause: Look at curve

2.3: Annotate Curve:

2.4: Derivative Curve:

The Dependencies List will contain the following lines if Start Method is the focused line:

2.1: Display Curve:
2.3: Annotate Curve:
2.4: Derivative Curve:

When trying to delete entries in the play list, this Dependencies List warns you that other entries in the play list depend on the presence of the line you want to delete.

Toolbar

Select this command from the View menu to display the standard toolbar for Pyris Player. If the toolbar is already displayed, select the **Toolbar** command to remove its display. Click on the buttons in the toolbar below to see the function of each:



Player Control Bar

Select this command from the View menu to display the control toolbar for the Pyris Player. If this toolbar is displayed, select **Control Bar** from the View menu to remove it from display. Click on each button on the toolbar below to see its function:



Curves Menus

The Curves menu is available when either the Data Analysis window or the Instrument Viewer is displayed. The contents of the Curves menu depends on the analyzer used to collect the data file displayed.

DMA/TMA Curves Menu

The following items appear in the Curves menu or in the Signals submenu for either the Data Analysis window or the Instrument Viewer for the DMA 7e and the TMA 7. The Signals submenu will appear only for a DMA 7e data file when dynamic force control is on.

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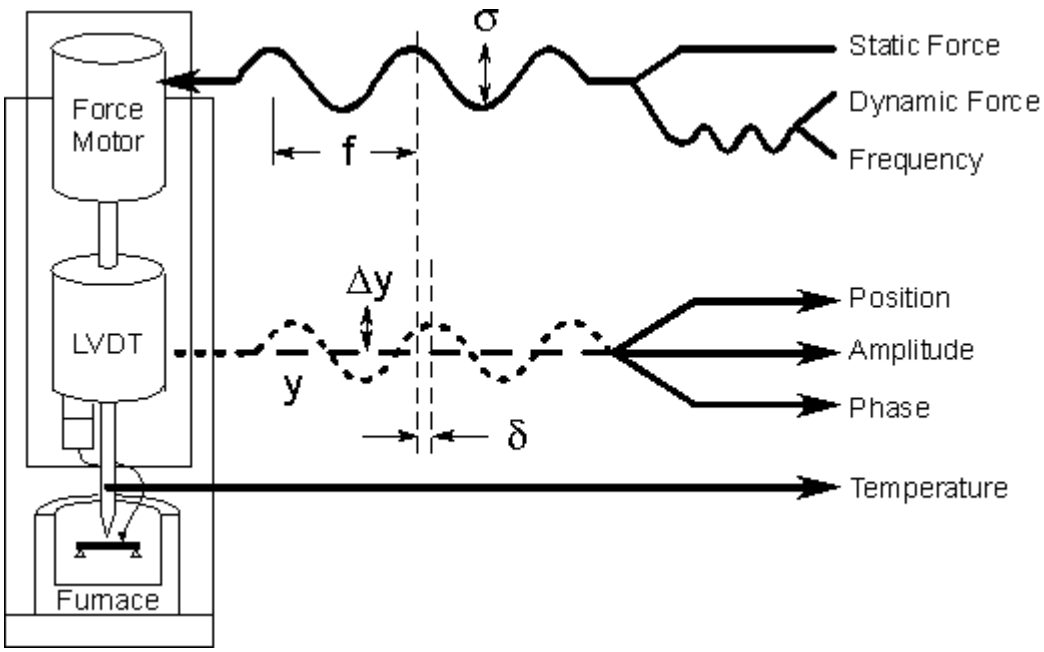
Probe Position

This command is available for DMA 7e and TMA 7 analyzers in Instrument Viewer and Data Analysis. In thermomechanical analysis (TMA) the deformation of a material under constant load (or constant strain) from a probe is recorded as a function of temperature or time. A sinusoidally varying stress is applied to the sample via a probe in dynamic mechanical analysis (DMA), producing an oscillating strain that lags behind the applied stress by a phase angle. During these analyses, the position of probe can be measured. the Probe Position curve shows the average position of the probe tip (in millimeters) during the run.

Static Force

This command is available for DMA 7e and TMA 7 analyzers only in Instrument Viewer and Data Analysis. In DMA experiments, the sample is stressed by applying a sinusoidally varying total force which comprises a static force and a dynamic force applied by the force motor. In TMA experiments, the force is static only. A static force (stress) is a continuous or steady force (pressure) or stress programmed to be applied by the force motor of the DMA or TMA to the sample. The **Static Force** command displays the curve of the static force during the run. The default static force may be sufficient is some scan modes (e.g., creep-recovery, frequency scan, isothermal scan, temperature scan).

DMA 7e Parameter Relationships



Dynamic Force

This command is available for a DMA 7e only in Instrument Viewer and Data Analysis. In DMA experiments, the sample is stressed by applying a sinusoidally varying total force which comprises a static force and a dynamic force applied by the force motor. The **Dynamic Force** command displays the curve of the dynamic force during the run. The default dynamic force is sufficient in temperature scans, isothermal scans, and frequency scans, and not needed in static force scans and creep-recovery.

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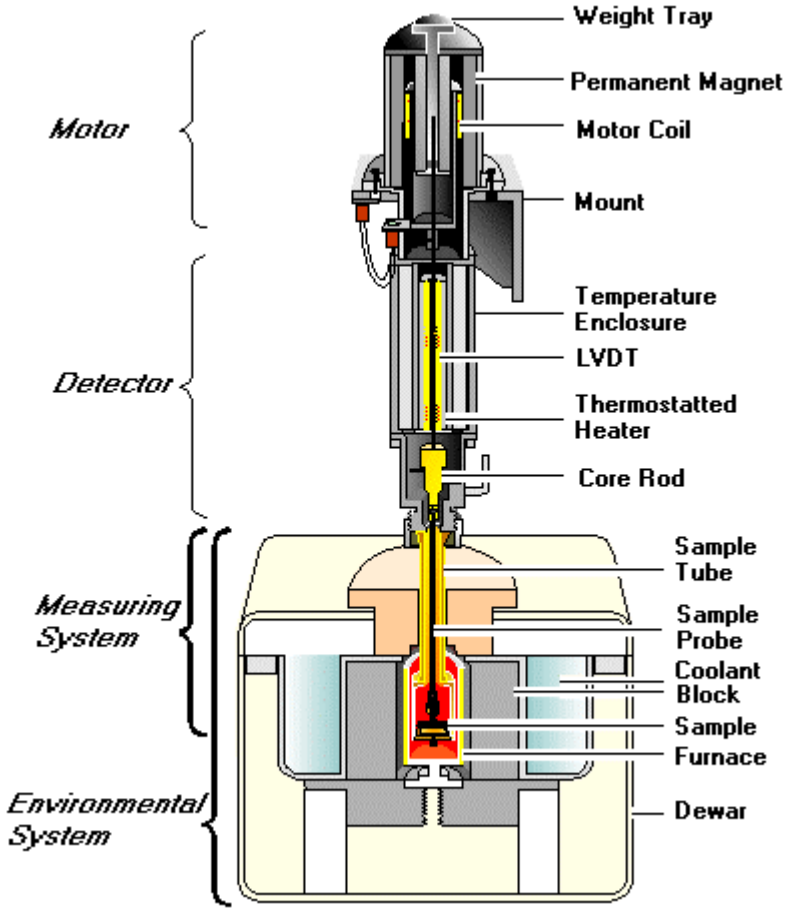
Frequency

This command is available for a DMA 7e only in the Instrument Viewer and Data Analysis. The frequency curve is the frequency of the dynamic force applied to the sample. The frequency is held constant in temperature scans, isothermal scans, and dynamic stress scans. It varies in frequency scans. There is no frequency in static force scans and creep-recovery.

Block Temperature

This command is available for a DMA 7e only in the Instrument Viewer and Data Analysis. The block is in the environmental system section of the DMA 7e (see figure below). It is the aluminum casing that surrounds the furnace and separates it from the coolant. Its temperature is recorded along with sample and program temperature during a run.

Cross Section of the DMA 7e



Sample Temperature

This command is available in Instrument Viewer and Data Analysis. Analyzers constantly monitor the sample temperature and output it as an analyzer signal that is collected with the raw data. Even in the absence of thermal gradients within the sample, the true sample temperature is unknown. Fluctuations in sample temperature due to heat evolution in the sample can remain undetected under these conditions. The nature of the atmosphere in the sample chamber can affect the measured value of the sample temperature because not all gases have the same thermal

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conductivity at a given temperature and pressure. In general, the sample temperature lags behind the program temperature.

Program Temperature

This command is available from the Curves menu in Data Analysis for the DMA 7e and the TMA 7. The program temperature is the temperature that the sample should be at according to the program created in the Program page of the Method Editor. If you are in Instrument Viewer, select Program Temperature to display the real-time program temperature signal. If you are not running an experiment, the curve should be flat since there is no temperature program in effect.

Amplitude

This command is available for a DMA 7e only in the Signals submenu of the Curves menu in Instrument Viewer and Data Analysis. When the force motor of the analyzer applies the specified force at a chosen frequency to the sample, it responds with an oscillating displacement amplitude. This amplitude corresponds to the average energy recovered in one cycle of oscillation in an elastic deformation. (See DMA 7e Parameter Relationships figure above.)

Phase Angle

This command is available for a DMA 7e only in Instrument Viewer and Data Analysis. When the force motor applies the specified force or stress at a chosen frequency to the sample, the sample responds with an oscillating displacement amplitude. It also responds to the applied force with a phase lag or angle which corresponds to the average energy lost in one cycle of oscillation in a viscous deformation. Phase angle is used to calculate the sample damping. (See DMA 7e Parameter Relationships figure above.)

Tangent Delta

This command is available for a DMA 7e only in Instrument Viewer and Data Analysis. Tangent delta is an indicator of the overall mechanical properties of a material, i.e., the relative amounts of energy stored and lost. It is often referred to as the mechanical loss factor. It is the ratio of damping to elasticity and is an indicator of the viscoelasticity of a sample. When a sample is dominated by elastic properties, tangent delta is low; when the sample is dominated by viscous properties, tangent delta is high. Tangent delta equals 1 when both elastic and viscous properties are present in equal proportions.

Modulus

The **Modulus** command is available for the DMA 7e in Instrument Viewer and Data Analysis. Modulus is the ratio of stress to strain and indicates how a sample reacts to mechanical energy. The **Modulus** command displays a submenu of the following items:

Storage Modulus

Storage modulus indicates the ability of a material to store mechanical energy, i.e., elasticity. It is the spring constant normalized for the sample size. Storage modulus is high when molecular mobility is restricted (e.g., as in crosslinked polymers and in polymers below the glass transition). When a polymer is heated above its glass transition temperature, there is an increase in free volume. When there is an increase in free volume, there is an increase in molecular mobility. Segments of the polymer align with the applied force. When this occurs in a sample, the storage modulus decreases.

Storage modulus is determined for each sample geometry (i.e., rectangle, cylinder, film, fiber, disk) using sample dimensions, amplitude, and the spring constant.

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This command calculates and displays the Storage Modulus curve of the focused data curve.

Loss Modulus

The loss modulus indicates the ability of a sample to dissipate mechanical energy. Loss modulus is the damping (dissipation of mechanical energy) normalized for sample dimensions. The loss modulus can be used to follow rheological changes that take place during processing. Loss modulus is determined for each sample geometry (i.e., rectangle, cylinder, film, fiber, disk) using sample dimensions, amplitude, and damping.

This command calculates and displays the Loss Modulus curve of the focused data curve. The shape of the Loss Modulus curve as a function of time or temperature can be correlated with viscosity curves on the same scale.

An additional option is available in the Modulus submenu in Data Analysis:

Complex Modulus

The complex modulus, also called the DMA modulus, is a representation of the bulk material behavior, i.e., the complete viscoelastic behavior of a material. The complex modulus contains both the elastic component (storage modulus) and the viscous component (loss modulus). This command calculates and displays the Complex Modulus curve of the focused data curve.

The Curves menu in Instrument Viewer and Data Analysis for the TMA 7 contains the following command:

Static Modulus

Static modulus is the ratio of static stress to static strain. Only the linear portion of the curve is of interest. This command calculates and displays the Static Modulus curve of the focused data curve.

Stress

The **Stress** command is available for the DMA 7e in Instrument Viewer and Data Analysis. Stress is a force applied to a cross-sectional area of a sample. In the DMA 7e, it is the force applied to the sample by the probe. The **Stress** command displays a submenu of the following items:

Static Stress

Static stress is a steady pressure (force per unit area) (in Pa) applied to the sample. Static stress must provide sufficient stress to prevent lifting of the probe as a result of the dynamic stress and to insure proper sample-to-measuring system contact. If the static stress is too low, bouncing could occur. If it is too high, misleading results could result. This command calculates and displays the Static Stress curve of the focused data curve.

Static Stress is also on the Curves menu for the TMA 7 in Instrument Viewer and Data Analysis.

Dynamic Stress

The dynamic stress is the oscillatory stress applied to the sample. It is superimposed upon the static stress. The dynamic stress selected should produce an amplitude between 5 and 500 µm throughout the entire run. The dynamic stress (in Pa) is calculated using the sample dimensions (form factors), amplitude, and the spring constant. Each measuring system (3-point bending, extension, parallel plate, and single and dual cantilever) and geometry (rectangle, cylinder, disk, fiber, film) combination has a unique form factor containing sample

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dimensions. This command calculates and displays the Dynamic Stress curve of the focused data curve.

Strain

The **Strain** command is available for the DMA 7e on the Curves menu in Instrument Viewer and Data Analysis. Strain is the ratio of the change in length of an extension sample to the original length of that sample. The **Strain** command displays a submenu of the following items:

Static Strain

Static Strain is also on the Curves menu for the TMA 7 in Instrument Viewer and Data Analysis.

Static strain is the measured displacement of a sample due to an applied static stress relative to the sample size. Static stress is a continuous and steady pressure (force) or stress programmed to be applied to the sample. Select this command to calculate and display the Static Strain curve of the focused curve.

Dynamic Strain

Dynamic strain is the measured displacement of a sample due to an applied dynamic stress relative to the sample size. Dynamic stress is a continuous and oscillatory pressure (force) or stress programmed to be applied to the sample. Select this command to calculate and display the Dynamic Strain curve of the focused curve.

Viscosity

The **Viscosity** command is available for the DMA 7e on the Curves menu in Instrument Viewer and Data Analysis. Viscosity is the resistance of a material to flow under stress. It is expressed in terms of the ratio of shear stress to rate of shear. The **Viscosity** command displays a submenu of the following items:

Storage Viscosity

Storage viscosity (in Pa s) is the observed storage modulus divided by the frequency (of the dynamic force applied to the sample). Select this command to calculate and display the Storage Viscosity curve of the focused curve.

Loss Viscosity

The loss viscosity (in Pa s) is the observed loss modulus divided by the frequency (of the dynamic force applied to the sample). Select this command to calculate and display the Loss Viscosity curve of the focused curve.

The Viscosity submenu in Data Analysis contains the following additional item:

Complex Viscosity

Complex viscosity (in Pa s) is the observed complex modulus divided by the frequency (of the dynamic force applied to the sample). Select this command to calculate and display the Complex Viscosity curve of the focused curve.

Expansion Coefficient

This command is available for a DMA 7e Probe Position curve only. The coefficient of expansion is the change in volume of the sample per degree temperature increase from the initial temperature. It is derived using the volumetric expansion values which are calculated from the

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probe position. Select this command to calculate and display the Expansion Coefficient curve of the focused curve.

Compliance

The **Compliance** command is available for the DMA 7e on the Curves menu in Data Analysis. Compliance is the ability of a material to yield under stress; the ratio of change in strain to the change in stress which produces it. It is the reciprocal of the modulus. The Compliance command displays a submenu of the following items:

Storage Compliance

Storage compliance (in 1/Pa) is the reciprocal of the storage modulus. Storage modulus indicates the ability of a material to store mechanical energy, i.e., elasticity. It is the spring constant normalized for the sample size. Storage modulus is high (storage compliance is low) when molecular mobility is restricted (e.g., as in crosslinked polymers and in polymers below the glass transition). Select this command to calculate and display the Storage Compliance curve of the focused curve.

Loss Compliance

Loss compliance (in 1/Pa) is the reciprocal of the loss modulus. Loss modulus indicates the ability of a sample to dissipate mechanical energy. Loss modulus is the damping (dissipation of mechanical energy) normalized for sample dimensions. Select this command to calculate and display the Loss Compliance curve of the focused curve.

An additional option is available in the Compliance submenu in Data Analysis:

Complex Compliance

Complex compliance (in 1/Pa) is the reciprocal of the complex modulus. Complex modulus is a representation of the bulk material behavior, i.e., the complete viscoelastic behavior of a material. The complex modulus contains both the elastic component (storage modulus) and the viscous component (loss modulus). Select this command to calculate and display the Complex Compliance curve of the focused curve.

The Curves menu in Instrument Viewer and Data Analysis for the TMA 7 contains the following command:

Static Compliance

Static compliance (in 1/Pa) is the reciprocal of static modulus or the ratio of static strain to static stress. Only the linear portion of the curve is of interest. Select this command to calculate and display the Static Compliance curve of the focused curve.

Time - Temperature Superposition

This command is available on the Curves menu in Data Analysis for DMA 7e Frequency or Frequency MultiPlex scans. Select this command to access the TTS dialog box in which you select the curves to be used to create a Master curve needed for time–temperature superposition on the Reference curve. Other parameters are also selected in this dialog box. After completing the TTS dialog boxes, the TTS curve is displayed.

Time–temperature superposition (TTS) is a purely empirical method for predicting material properties beyond the range of practical laboratory analysis. It allows for the prediction of very long time behavior (e.g., shelf life, warpage) and very short time behavior (process speeds, failure modes) of a material or the material behavior at higher or lower temperatures than original analysis. The dynamic range of laboratory analysis from short times to long times (50 to 0.01 Hz)

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is not sufficient. It can be increased by using TTS. Results collected using "standard" DMA 7e test conditions can be used to predict performance of plastics and composites over very long times using TTS. TTS is used to generate master curves that give material properties at times or temperatures that exceed typical laboratory experimental times and temperatures.

The procedure for performing TTS involves first generating a multiplex data set and then shifting the data with respect to a reference curve to generate a master curve. **Multiplexing** is the generation of a family of curves where two parameters are varied and plotted together. For example, a frequency scan performed at several different temperatures and plotted will produce a multiple data set. The **master curve** is a composite curve generated from frequency multiplex data. It may be used to predict material behavior over a very wide range of times and temperature. Time–temperature superposition is performed for curves whose X axis is frequency. Predictions can be made directly from the master curve at the reference temperature over very long or short times. The **reference temperature** is the temperature of the reference curve from which all other multiplexed data sets will be shifted relative to this temperature. It is typically 298 K. A reference temperature can be chosen to give the best fit for the WLF equation using typical values for C1 or 8.86 and C2 of 101.6. This will typically give a reference temperature of about 5 K above the glass transition temperature.

The master curve can also be shifted to a higher or a lower temperature, and from that position predictions can be made over very long or short times at the new temperature.

The highest quality multiplex data sets are generated by repeatedly performing a scan of one variable (frequency) over a selected variable (temperature). It is a simple matter to shift the multiplex data to generate a master curve. The quality of a TTS shift and, therefore, the quality of the predictions can then be evaluated using an equation that fits your sample.

A Time – Temperature Superposition curve is available for frequency or multiplex frequency data collected by a DMA 7e. The Time – Temperature Superposition dialog box contains the following fields:

Reference Curve

The curve you highlighted in the Data Analysis window is used as the Reference curve for TTS. The available curves are those curves displayed in the active Data Analysis window. The default curve is the active curve. If you want to select another curve for the Reference curve, click on **Cancel** to return to the Data Analysis window and click on another curve to highlight it.

Select the curves to be added to be used to create the Master Curve

The curves listed here are all the curves displayed in the active Data Analysis window except the Reference curve. Highlight the curves to be used to create the Master curve. Click on each one while pressing down the **Ctrl** key, or, while pressing down the left mouse button, drag the cursor over the curves to highlight them.

Select the material and constants

Select the material whose constants C1 and C2 you wish to use in the WLF equation which is used to calculate the amount of shift. This list is created and maintained by the user. Enter the name of a material that is close in molecular structure to the material used in the frequency scan; then enter the two constants for the material from a materials table. Any material and constant values entered will be saved for future use.

Next

Click on this button to display the TTS-2 dialog box and continue with TTS.

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TTS-2 Dialog Box

Reference Curve

The legend information for the focused curve selected as the Reference curve in the preceding dialog box is displayed.

Reference Temperature

The Program Temperature of the Reference curve.

Are the Shift Factors acceptable?

The shift factor for each curve that the Master curve comprises is displayed. The shift factor is the amount a data set must be moved in order to align a portion of it with the Reference curve. If you are not satisfied with these shift factors, click on the **Back** button to return to the first dialog box and select a new set of curves.

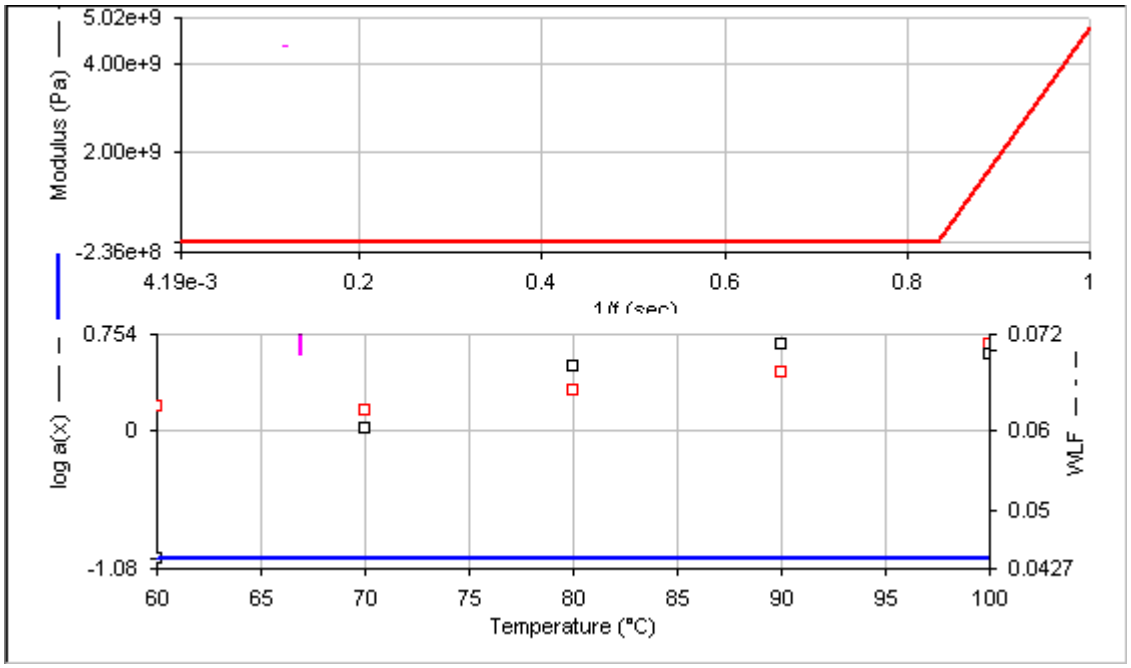
Back

Returns you to the preceding dialog box for selection of new curves to create a new Master curve.

Finish

Completes the TTS calculation and displays the TTS curve.

An example of a TTS curve is shown below:



Two additional items are on the Curves menu when in Data Analysis:

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Step Select

This command is available while in Data Analysis. Select this toggle command to activate/deactivate the Step Select feature. When you select a Curves menu command after activating Step Select, the Step Select dialog box appears in which you choose the steps from the method program for which you want to display the data curve.

Step Select Dialog Box

The Step Select dialog box appears when you select one of the commands in the Curves menu and the **Step Select** command is active. This is a toggle item. Use this function to display one or more method steps in a data file rather than the entire data file. The Step Select dialog box contains the following fields:

Data File

The active curve's data file name and the directory in which it resides.

Curve Type

The curve type you selected in the Curves menu before this dialog box was displayed.

List of Method Steps

A list of all the steps in the method associated with the active curve. Select one or more steps in the list by highlighting.

OK closes the dialog box and adds the curve(s) representing the highlighted method step(s) to the Data Analysis window. **Cancel** closes the dialog box without performing the operation. **Select All** highlights all method steps in the list.

Start Time at Zero

This command is available while in Data Analysis. Select this toggle command to activate/deactivate the Start Time at Zero feature. When activated, data from each step in the method program can be displayed as starting at zero minutes so that data from different steps can be compared.

The following items are on the Curves menu in Instrument Viewer and Data Analysis for the TMA 7 only:

Static Modulus

Static modulus is the ratio of static stress to static strain. Only the linear portion of the curve is of interest. This command calculates and displays the Static Modulus curve of the focused data curve.

Static Compliance

Static compliance (in 1/Pa) is the reciprocal of static modulus or the ratio of static strain to static stress. Only the linear portion of the curve is of interest. Select this command to calculate and display the Static Compliance curve of the focused curve.

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Static Stress

Static stress is a steady pressure (force per unit area) (in Pa) applied to the sample. Static stress must provide sufficient stress to prevent lifting of the probe as a result of the dynamic stress and to insure proper sample-to-measuring system contact. If the static stress is too low, bouncing could occur. If it is too high, misleading results could result. This command calculates and displays the Static Stress curve of the focused data curve.

Static Strain

Static strain is the measured displacement of a sample due to an applied static stress relative to the sample size. Static stress is a continuous and steady pressure (force) or stress programmed to be applied to the sample. Select this command to calculate and display the Static Strain curve of the focused curve.

DSC Curves Menu

The following items are in the Curves menu for the Pyris 1 DSC, DSC 7, and Pyris 6 DSC. What the Curves menu contains will depend on whether you are in the Instrument Viewer or the Data Analysis window.

Heat Flow

The heat flow is the amount of energy applied to or removed from either the sample furnace alone or both the sample and the reference furnaces to compensate for the energy change occurring in the sample. The **Heat Flow** command for a DSC analyzer displays the heat flow curve of the focused curve. It is the heat flow signal **after** baseline subtraction is performed, if indicated in the Initial State page in the Method Editor.

Derivative Heat Flow

This command is available in Instrument Viewer for DSC 7, Pyris 1 DSC, and DSC 6 heat flow curves. This command displays the first derivative of a real-time heat flow curve. The first derivative is used to find an inflection point in a curve, i.e., the point where a peak begins. The derivative is the instantaneous slope or rate of change of the data curve as a function of time.

Baseline Heat Flow

Select this command to display the heat flow curve of the baseline file used in baseline subtraction for the active curve of a DSC analyzer. The baseline file is the result of running empty sample pans in the sample and reference holders under the same conditions your samples are to be run. This establishes the noise level and curvature of the instrument baseline before analyzing samples. If you did not indicate Use Baseline Subtraction in the method, the baseline curve is a flat line at zero.

Unsubtracted Heat Flow

This command is available from the Curves menu for DSC 7, Pyris 1 DSC, and Pyris 6 DSC. Select this command to display the heat flow signal of the analyzer before baseline subtraction is performed. The heat flow is the amount of energy applied to or removed from either the sample furnace alone or both the sample and reference furnaces to compensate for the energy change occurring in the sample.

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Sample Temperature

This command is available for Instrument Viewer and Data Analysis. Analyzers constantly monitor the sample temperature and output it as an analyzer signal that is collected with the raw data. Even in the absence of thermal gradients within the sample, the true sample temperature is unknown. Fluctuations in sample temperature due to heat evolution in the sample can remain undetected under these conditions. The nature of the atmosphere in the sample chamber can affect the measured value of the sample temperature because not all gases have the same thermal conductivity at a given temperature and pressure. In general, the sample temperature lags behind the program temperature.

Program Temperature

The program temperature is the temperature that the sample should be at according to the program created in the Program page of the Method Editor. If you are in Instrument Viewer, select Program Temperature to display the real-time program temperature signal. If you are not running an experiment, the curve should be flat since there is no temperature program in effect.

Endotherms Up

Heat flow curves use the endotherm convention currently set. Select this toggle command to activate/deactivate the Endotherms Up feature. With endotherms up, the Y axis increases from bottom to top; with endotherms down, it decreases from bottom to top. Curves already on screen are not affected by a change in the endotherm setting. The endotherm convention in effect for curves displayed is saved when the data file is saved.

Data generated by a power-compensated DSC is correctly displayed with the endotherms up.

Two additional items are on the DSC Curves menu when in Data Analysis:

Step Select

Select this toggle command to activate/deactivate the Step Select feature. When you select a Curves menu command after activating Step Select, the Step Select dialog box appears in which you choose the steps from the method program for which you want to display the data curve.

Start Time at Zero

Select this toggle command to activate/deactivate the Start Time at Zero feature. When activated, data from each step in the method program can be displayed as starting at zero minutes so that data from different steps can be compared.

DDSC Curves

If you are running the Pyris 1 DSC in DDSC mode or are using the DDSC accessory with the DSC 7, then you will see additional items on the Curves menu. The data collected with DDSC is raw heat flow data. The data curve can be smoothed and analyzed like any DSC heat flow curve. From this data, dynamic and static curve types can be calculated. These curve types are on the Curves menu:

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Dynamic Curves

Storage Cp

Select this command to display the storage specific heat data for the active curve. The storage specific heat is the simple linear specific heat that results from the uptake of energy into the various atomic and molecular mechanisms of energy storage as the sample is heated.

Loss Cp

Select this command to display the loss specific heat data for the active curve. The loss specific heat is the component of the dynamic specific heat that is out of phase with the temperature change because the heat flow has resulted in a structural change in the sample material rather than merely raising the temperature.

Complex Cp

Select this command to display the complex specific heat data for the active curve. The complex specific heat is the vector sum of the storage specific heat and the loss specific heat curves.

Tangent Delta

Select this command to display the tangent delta data for the active curve. The tangent delta curve is the ratio of the loss specific heat to the storage specific heat.

Enthalpy

This command is available for DDSC and only if the active curve is a specific heat curve. Select this command to display the enthalpy data for the active curve. Selecting the **Enthalpy** command calculates the enthalpy change of the material, which is the integral of the specific heat over the specified temperature range. Enthalpy curves are generally calculated from the total specific heat curve but can be generated from any other specific heat curve generated by Pyris.

Static Curves

Total Cp

Select this command to display the total specific heat data for the active curve. Total specific heat is the heat flow data expressed in specific heat units. The raw or smoothed heat flow data must be the first curve displayed in order to generate a total specific heat curve.

Heat Flow

The heat flow is the amount of energy applied to or removed from either the sample furnace alone or both the sample and the reference furnaces to compensate for the energy change occurring in the sample. The **Heat Flow** command displays the heat flow curve of the focused curve. It is the heat flow signal **after** baseline subtraction is performed, if indicated in the Initial State page in the Method Editor.

Enthalpy

See Dynamic Curves above.

Enthalpy can be static or dynamic depending on which type of specific heat curve is used to calculate the enthalpy.

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DTA 7 Curves Menu

The following items appear in the Curves menu for the DTA 7 in Data Analysis and Instrument Viewer:

Heat Flow

This command converts a DTA curve in °C to a heat flow curve in mW. The heat flow curve represents the heat flow signal **after** baseline subtraction is performed, if indicated in the Initial State page in the Method Editor. The heat flow is the amount of energy applied to or removed from either the sample furnace alone or both the sample and reference furnaces to compensate for the energy change occurring in the sample.

The DTA 7 can display the raw data as from a differential thermal analyzer where the curve is ΔT vs. time or temperature. ΔT is the difference in temperature between the sample and the inert reference material. The raw data can also be displayed as from a differential scanning calorimeter where the curve is heat flow vs. temperature or time. The DTA's signal is optimized and conditioned by the software so that the output is calibrated in units of mW, facilitating peak area measurements. This mode is used for those applications where quantitative analysis based on peak area is desired.

Baseline Heat Flow

Select this command to display the heat flow curve of the baseline file used in baseline subtraction. The baseline file is the result of running empty sample pans in the sample and reference holders under the same conditions your samples are to be run. This establishes the noise level and curvature of the instrument baseline before analyzing samples. If you did not indicate Use Baseline Subtraction in the method, the baseline curve is a flat line at zero.

Unsubtracted Heat Flow

Select this command to display the heat flow signal **before** the baseline subtraction is performed. The heat flow is the amount of energy applied to or removed from either the sample furnace alone or both the sample and reference furnaces to compensate for the energy change occurring in the sample. The DTA heat flow curve is the normal DTA 7 temperature differential signal (Delta T curve in °C) converted to a heat flow signal (in mW).

Delta T

Delta T is the difference in temperature between the sample and an inert reference material. The signal is displayed as Delta T (in °C) vs. temperature or time. Note that high-temperature peaks are weaker when displayed as Delta T vs. temperature or time since conventional DTA sensitivity will decrease as a function of increasing temperature. These peaks should be displayed as a Heat Flow curve.

Baseline Delta T

Delta T is the difference in temperature between the sample and an inert reference material in a DTA 7. The signal is displayed as Delta T (in °C) vs. temperature or time. Baseline Delta T displays the Delta T curve of the baseline file used in baseline subtraction for the active curve. If you did not select Use Baseline Subtraction in the method, the baseline curve is a flat line at zero.

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Unsubtracted Delta T

This command displays the Delta T signal of the DTA 7 **before** baseline subtraction is performed. Delta T is the difference in temperature between the sample and an inert reference material.

Sample Temperature

This command is available in Instrument Viewer and Data Analysis. Analyzers constantly monitor the sample temperature and output it as an analyzer signal that is collected with the raw data. Even in the absence of thermal gradients within the sample, the true sample temperature is unknown. Fluctuations in sample temperature due to heat evolution in the sample can remain undetected under these conditions. The nature of the atmosphere in the sample chamber can affect the measured value of the sample temperature because not all gases have the same thermal conductivity at a given temperature and pressure. In general, the sample temperature lags behind the program temperature.

Program Temperature

The program temperature is the temperature that the sample should be at according to the program created in the Program page of the Method Editor. If you are in Instrument Viewer, select Program Temperature to display the real-time program temperature signal. If you are not running an experiment, the curve should be flat since there is no temperature program in effect.

Furnace Temperature

The DTA 7 high-temperature furnace provides a uniform temperature environment for the sample and the reference materials. The furnace thermocouple maintains this uniform environment. If a problem should occur with the furnace, the Furnace Temperature curve can be used to display the signal of the temperature of the furnace.

Heat Flow Calibration

When DTA 7 data is converted to heat flow data for display of a Heat Flow curve, a k *vs.* T (where T is temperature) curve is used to convert Y values from °C to mW. k is the rate of reaction of a material undergoing a transition. After the Heat Flow curve is obtained, it may be fit to the theoretical shape of the curve obtained by another equation that takes into account the linear relationship of temperature with time in differential thermal analysis and the scanning rate. The Heat Flow Calibration curve is the theoretical heat flow curve. Display it along with a Heat Flow curve as a diagnostic tool.

Endotherms Up

Heat flow curves use the endotherm convention currently set. Select this toggle command to activate/deactivate the Endotherms Up feature. With endotherms up, the Y axis increases from bottom to top; with endotherms down, it decreases from bottom to top. Curves already on screen are not affected by a change in the endotherm setting. The endotherm convention in effect for curves displayed is saved when the data file is saved.

These two additional items are on the Curves menu when in Data Analysis:

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Step Select

Select this toggle command to activate/deactivate the Step Select feature. When you select a Curves menu command after activating Step Select, the Step Select dialog box appears in which you choose the steps from the method program for which you want to display the data curve.

Start Time at Zero

Select this toggle command to activate/deactivate the Start Time at Zero feature. When activated, data from each step in the method program can be displayed as starting at zero minutes so that data from different steps can be compared.

TGA Curves Menu

The following items are in the Curves menu for the Pyris 6 TGA, TGA 7, and Pyris 1 TGA for either the Instrument Viewer or the Data Analysis window:

Weight

Thermogravimetric analysis (TGA) examines the mass change of a sample as a function of temperature in the scanning mode or as a function of time in the isothermal mode. The TGA analyzer monitors the sample weight and outputs it as an analyzer signal that is stored in the data file. Select this command in Instrument Viewer and in Data Analysis to display the sample weight signal from the analyzer **after** baseline subtraction is performed, if indicated in the Initial State page of the Method Editor.

Derivative Weight

Select this command to display the first derivative of a real-time weight curve. The first derivative is used to find an inflection point in a curve, i.e., the point where a peak begins. The derivative is the instantaneous slope or rate of change of the data curve as a function of time.

Baseline Weight

Select this command to display the weight curve of the baseline file used in baseline subtraction. The baseline file is the result of running an empty sample pan under the same conditions under which your samples are to be run. This establishes the noise level and curvature of the instrument baseline before analyzing samples. If you did not indicate Use Baseline Subtraction in the method, the baseline curve is a flat line at zero.

Unsubtracted Weight

Thermogravimetric analysis (TGA) examines the mass change of a sample as a function of temperature in the scanning mode or as a function of time in the isothermal mode. The TGA analyzer monitors the sample weight and outputs it as an analyzer signal. Select this command to display the sample weight signal from the analyzer **before** baseline subtraction is performed.

Sample Temperature

This command is available in Instrument Viewer and Data Analysis. Analyzers constantly monitor the sample temperature and output it as an analyzer signal that is collected with the raw data. In TGA analyzers, the nature of the microbalance precludes the thermocouple from being in direct contact with the sample. The thermocouple instead measures the temperature of the atmosphere in

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close proximity to the sample. Even in the absence of thermal gradients within the sample, the true sample temperature is unknown. Fluctuations in sample temperature due to heat evolution in the sample can remain undetected under these conditions. The nature of the atmosphere in the sample chamber can affect the measured value of the sample temperature because not all gases have the same thermal conductivity at a given temperature and pressure. In general, the sample temperature lags behind the program temperature.

Program Temperature

The program temperature is the temperature that the sample should be at according to the program created in the Program page of the Method Editor. If you are in Instrument Viewer, select Program Temperature to display the real-time program temperature signal. If you are not running an experiment, the curve should be flat since there is no temperature program in effect.

These two additional items are on the Curves menu when in Data Analysis:

Step Select

Select this toggle command to activate/deactivate the Step Select feature. When you select a Curves menu command after activating Step Select, the Step Select dialog box appears in which you choose the steps from the method program for which you want to display the data curve.

Start Time at Zero

Select this toggle command to activate/deactivate the Start Time at Zero feature. When activated, data from each step in the method program can be displayed as starting at zero minutes so that data from different steps can be compared.

Math Menu

The Math menu is available only for Data Analysis windows. The Math menu for all analyzers contains the following commands:

Derivative

This command on the Math menu calculates and displays the first derivative of the active curve. When you select this command, the first derivative curve is displayed and becomes the active curve.

Add

This command adds one or more curves to the active curve. When you select this command, the Add dialog box appears from which you select the curves to add to the focused or active curve. This command is available only when two or more curves are displayed in a Data Analysis window. The new curve is added to the display and becomes the active curve.

Add Dialog Box

The **Add** command in the Math menu creates a new curve by adding the ordinate values of one or more curves to the ordinate values of the active curve. To use the **Add** command, there must be at least two curves of the same type displayed in the active Data Analysis window. The Add dialog box lists all of the curves that can be added to the active curve. Select the curve or curves you

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want to add, then select the **Calculate** button. The new curve is added to the display and becomes the active curve. When you save the new curve, it will be attached to the data used in the original active curve.

List of available curves

All curves whose ordinate values can be added to the ordinate values of the active curve. Select one or more curves in the list to highlight them for addition.

Calculate

Closes the dialog box, adds the ordinate values of the highlighted curve or curves to those of the active curve, and displays the new curve in the current Data Analysis window.

Subtract

This command subtracts a displayed curve from the active curve. When you select this command, the Subtract dialog box appears from which you select the curve to subtract. This command is available only when two or more curves are displayed in a Data Analysis window. The new curve is added to the display and becomes the active curve.

Subtract Dialog Box

The **Subtract** command in the Math menu creates a new curve by subtracting the ordinate values of a selected curve from the ordinate values of the focused curve. To use the **Subtract** command there must be at least two curves of the same type displayed in the Data Analysis window. The Subtract dialog box contains the following fields:

List of available curves

All curves whose ordinate values can be subtracted from the ordinate values of the active curve. Select one curve in the list to highlight it for subtraction.

Calculate

Closes the dialog box, subtracts the ordinate values of the highlighted curve from those of the active curve, and displays the new curve in the active Data Analysis window.

Average

This command in the Math menu displays the average curve calculated from two or more displayed curves. In the Average dialog box you select the curves to average with the active curve. The curves available for selection are those already displayed in the Data Analysis window so two or more curves have to be displayed when this command is selected. The resulting average curve is added to the display and becomes the active curve.

Average Dialog Box

You can average the ordinate values of one or more curves with those of the focused curve to create an average curve. There must be at least two curves of the same type displayed in the Data Analysis window in order to use the **Average** command. The Average dialog box appears when you select the Average command in the Math menu.

List of available curves

All displayed curves whose ordinate values can be averaged with the ordinate values of the active curve are listed. Highlight one or more curves in the list to be averaged with the focused curve.

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Calculate

Adds the ordinate values of the highlighted curve(s) to those of the active curve and computes the average. The average curve is displayed in the current Data Analysis window.

Smooth

This command smoothes the active curve. When you select this command, the Smooth dialog box appears in which you enter the left and right limits, the type of smoothing, and the window size. The smoothed curve replaces the active curve.

Smooth Dialog Box

The **Smooth** command in the Math menu removes noise from the focused curve. You select the section to smooth and the smoothing algorithm to use. Two **X**'s appear in the Data Analysis window when you select the Smooth command, along with the dialog box:
Left Limit

Enter the left limit for the smooth calculation. When the left limit is selected directly on the curve by clicking on and dragging an **X** to the desired location, the value in the entry field automatically changes to reflect the new position.

Right Limit

Enter the right limit for the smooth calculation. When the right limit is selected directly on the curve by clicking on and dragging the other **X** to the desired location, the value in the entry field automatically changes to reflect the new position.

Algorithm

Select an algorithm from the list of algorithms that can be used in the smooth calculation:

- Standard: A simple sliding average algorithm. Each point is smoothed based on its current value and a group of points that make up the window centered on it. This type of smoothing results in moderate smoothing of noise.
- Median: A sliding average, like Standard smooth, but each point in the window is weighted differently, with the point in the center of the smooth window weighted heaviest and points on the ends weighted least. This results in moderate smoothing with better peak height preservation.
- Average: Another sliding average algorithm that results in better baseline smoothing than Standard or Median, but there may be some degradation of peak height. Use this algorithm for smoothing DDSC curves, using the size of the repeat step as the window size.
- Savitzky-Golay: A weighted smoothing algorithm that is very good for smoothing baselines and maintaining peaks. However, there may be slight shifting in the peak value.

Window Size

Enter a window size (in points) to be used in the smooth calculation. The default window size is 5 points in all cases except DDSC Heat Flow curves. In this case, the default window size is the number of points in one repeat unit.

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Calc Menu

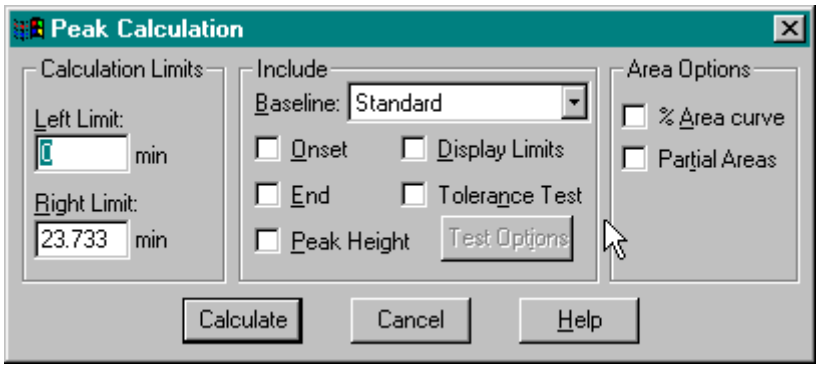
The Calc menu is available on the menu bar in Data Analysis. It contains the following commands, some of which are analyzer-dependent:

Peak Area

This command is used to perform an area calculation based on the limits you set on the active curve. In the Peak Calculation dialog box you can also select the type of baseline to use, whether to include onset, offset, or peak height features, and the type of curve to calculate — percent area or partial area.

Peak Calculation Dialog Box

The peak area calculation determines the area, starting point, midpoint, and end point of a peak transition. This calculation is available for all curves. In heat flow curves, peak transitions are associated with melting, crystallization, and curing. The peak area calculation can be used with derivative weight loss curves to determine degrees of weight loss.



When you select **Peak Area** from the Calc menu, the Peak Calculation dialog box appears along with two **X**'s. You can use the mouse to drag the **X**'s to the desired location on the peak to set the limits of the calculation or you can use the keyboard to type in the limits. The limits should completely encompass the peak transition. Use the dialog box as follows:

Calculation Limits

Left Limit

Enter the left limit for the calculation. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired position, the value in the entry field automatically updates.

Right Limit

Enter the right limit for the calculation. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired position, the value in the entry field automatically updates.

Include

The items in this section of the dialog box can be selected for display with the curve and on the printout.

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Baseline

After setting the limits, select the type of baseline to be used in the calculation and displayed:

- **Standard** is a straight, limit to limit baseline.
- **Sigmoidal** baseline can be used when the curve before the transition is at a different level than it is after the transition. This most commonly occurs in heat flow data. When sigmoidal baseline is chosen, tangents are drawn from the beginning of each specified limit. You can adjust these before the calculation is performed.
- **Horizontal from Right** can be used when the curve before the transition is at a different level than it is after the transition. It draws a line from the curve at the right limit to the left limit's X value. A vertical line is drawn from the horizontal line at the left limit X value (either up or down) to the curve.
- **Horizontal from Left** can be used when the curve before the transition is at a different level than it is after the transition. It draws a line from the curve at the left limit to the right limit's X value. A vertical line is drawn from the horizontal line at the right limit X value (either up or down) to the curve.
- **Horizontal at Zero** can be used when the curve before the transition is at a different level than it is after the transition. It draws a horizontal line at Y = 0.0 from the left limit to the right limit. A vertical line is drawn from the horizontal line at the left limit (either up or down) to the curve and a vertical line is drawn from the horizontal line at the right limit (either up or down) to the curve.

The **Onset** value is calculated by finding the intersection of the baseline and the extrapolated tangent at the inflection point of the leading edge of the peak. The **End** value is calculated by finding the intersection of the baseline and the extrapolated tangent at the inflection point of the trailing edge of the peak. The **Peak Height** is the distance from the baseline to the peak. You can select one, two, or all three items. Select **Display Limits** to have the X,Y values of the left and right limits also displayed on the curve.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you click on Tolerance Test, the **Test Options** button becomes active. Click on the button to display the dialog box.

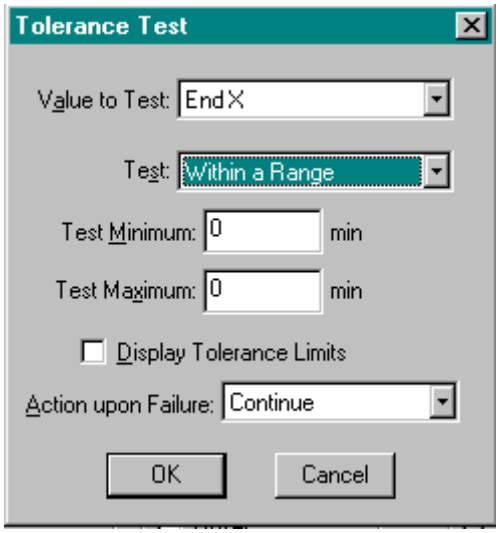
Area Options

Select **% Area Curve** to calculate and display a percent area curve based on the peak area calculation. If you select **Partial Areas**, the Partial Areas dialog box will appear after the Peak Area is calculated and displayed. You can select both items.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:

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Values to Test are Peak X, Onset X, End X, Peak Height, Peak Area, and Delta H.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Partial Areas Dialog Box

A partial areas calculation is a convenient way to mark the amount of completion of the peak transition at various points. It is available for any curve for which a peak area calculation can be performed.

A partial areas calculation begins immediately after a peak area calculation is completed. In the case of a heat flow curve, this intermediate dialog box is displayed in which you establish the standard ΔH value to be used in calculating the percent area values. This is useful, for example, when a sample is known to be less than 100% crystalline and the percent areas should be reported relative to 100% crystallinity. In such a case, the ΔH reported by the peak area calculation can be

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adjusted by the known percent crystallinity of the sample. The default value displayed is the ΔH value calculated during peak calculation and displayed on the plot. You can also enter another value which is a percent of the ΔH based on the condition of the sample (i.e., percent cured, percent solid for melting of a completely crystalline material; percent solid in a melting experiment). Click on **OK** and the next Partial Areas dialog box is displayed.

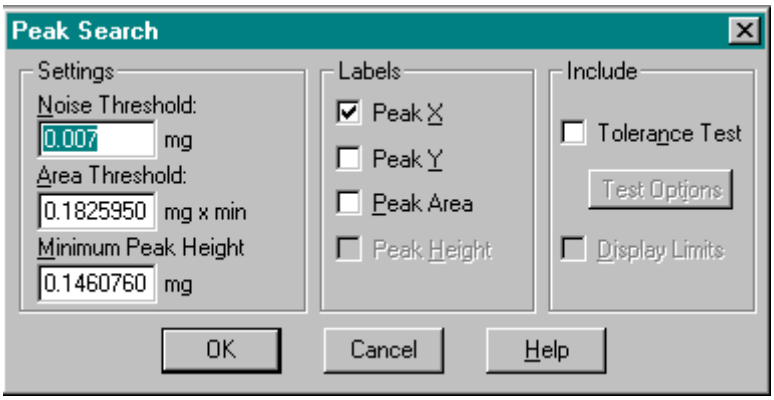
Partial area values are calculated either by entering the desired X-axis point in the **Enter Limit** field or by entering a specific value in the **Enter Percent Area** field in the Partial Areas dialog box. You can also select the position on the curve by directly clicking on the curve; a vertical line will be drawn and the value in the dialog box will be displayed. Click on **Calculate** and the limit, percent area, and area are displayed in a table. The dialog box remains open so you can continue to perform this calculation for other points.

Peak Search

This command is used to perform a peak search of the focused curve based on minimum height threshold or area threshold. When you select this command, the Peak Search dialog box appears in which you enter the minimum peak height and whether to include labels.

Peak Search Dialog Box

The Peak Search calculation is used to determine peak maxima and minima on a curve. Peak search is available for all curves. It is useful for quickly identifying all of the significant peaks in a curve.



Settings

Noise Threshold

Enter a noise threshold in Y-axis units for which the Pyris software will search. This parameter enables Pyris to discriminate between baseline noise and peaks. If the vertical difference between the inflection point on the leading and trailing edges of the peak and the top of the peak exceeds the Noise Threshold, then Pyris recognizes the potential start of a peak. The Noise Threshold must be met on only one side of the peak. All peaks in the active curve whose heights are greater than this value will be identified. The lower the Noise Threshold value, the more sensitive peak detection is.

Area Threshold

Area Threshold is used to discriminate between noise spikes and peaks. This parameter is used after the Noise Threshold to confirm the potential start of peaks that pass the Noise Threshold test. After passing that test, the data points must continue to pass the test and the

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cumulative sum of the data points on the leading edge must eventually exceed the area threshold for the peak to be confirmed. The higher the area threshold value, the more difficult it is to confirm a peak.

Minimum Peak Height

Minimum Peak Height is used to avoid finding a false peak top because of noise. To find the top of a peak, Pyris tries to identify a local maximum bunched point value. When a bunched point is lower than the previous one, the previous point is considered to be the potential peak top. To avoid finding a false peak top because of noise, Pyris performs a confirmation test by summing the differences between the potential top and subsequent bunched points. If the sum exceeds two-thirds of the Area Threshold value, the potential peak top is confirmed. If a higher bunched point is found before the area test is passed, a new potential top is identified and the area test is restarted. Because of this top-of-the-peak test, the choice of an area threshold value affects both peak confirmation and how shoulders are detected on the leading edge of a larger peak.

Labels

Select Peak X, Peak Y, or Peak Area or any combination of the three to be displayed on each peak found. When you select Peak Area, Peak Height and Display Limits become available for selection.

Include

Tolerance Test

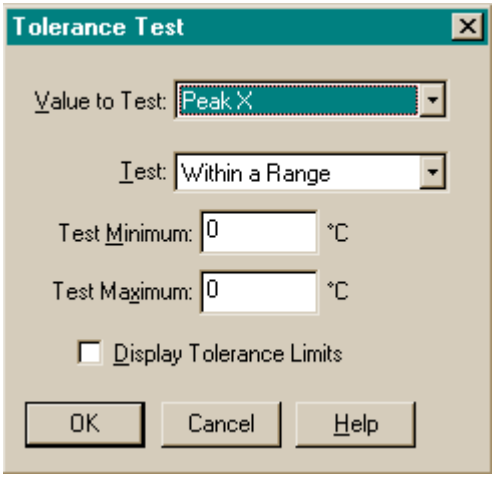
You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you click on the Tolerance Test box, the **Test Options** button is available. Click on the button to display the Tolerance Test dialog box.

Display Limits

If you want the limits (e.g., Area, X values) that were calculated to be displayed in the Data Analysis window, click on this box.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



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Value to Test is Peak X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Tg (DSC only)

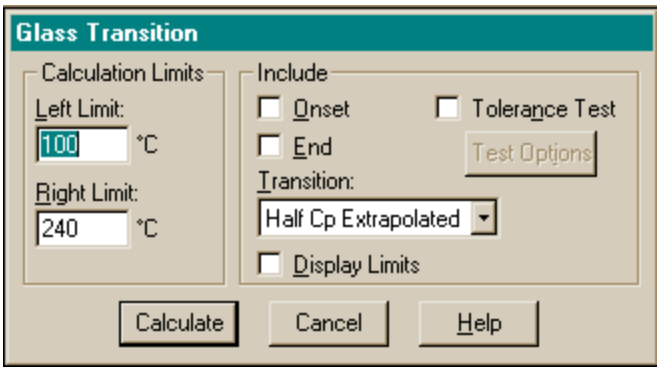
Select this command to perform a glass transition calculation based on the active curve. The Tg calculation is used to determine the starting point, midpoint, and end point of a glass transition. When you select this command, the Glass Transition dialog box appears in which you enter the calculation limits and whether to include the onset and end points and the transition point.

Glass Transition Dialog Box

The Glass Transition (Tg) calculation is used to determine the starting point, midpoint, and end point of a glass transition. It is available for heat flow curves (the Step transition is used for other curves). When a material is heated through the glass transition region, it changes from a rigid to a flexible state and its heat capacity changes. This is reflected as a change in the heat flow.

The Glass Transition dialog box appears when you select the Tg command in the Calc menu. When it is displayed, two red **X**'s appear on the active curve (they may overlap each other). The dialog box contains the following fields:

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Calculation Limits

Left Limit

Enter the left limit for the glass transition calculation. When the left limit is selected directly on the curve by clicking on and dragging an **X** to the left, the value in the entry field automatically updates. The left limit should precede the beginning of the glass transition.

Right Limit

Enter the right limit for the glass transition calculation. When the right limit is selected directly on the curve by clicking on and dragging an **X** to the right, the value in the entry field automatically updates. The right limit should follow the end of the glass transition.

Include

You can include the onset and end points in the display of the calculation results. The onset value is calculated by finding the intersection of the extrapolated tangent at the first limit and the extrapolated tangent at the inflection point. The end value is calculated by finding the intersection of the extrapolated tangent at the second limit and the extrapolated tangent at the inflection point. You can also indicate whether you want the X,Y ordinates of the limits displayed in the Data Analysis window.

Transition

Select from the drop-down list the glass transition type you want to use in the calculation:

- Inflection Point reports the point between the limits at which the slope of the curve changes from increasing to decreasing or vice versa.
- Half Cp Extrapolated reports the point on the curve where the specific heat change is half of the change in the completed transition.
- Half-Width reports the point on the curve that is halfway between the onset and end points.
- Fictive Temperature reports the point on the enthalpy curve where the change of slope occurs.

Tolerance Test

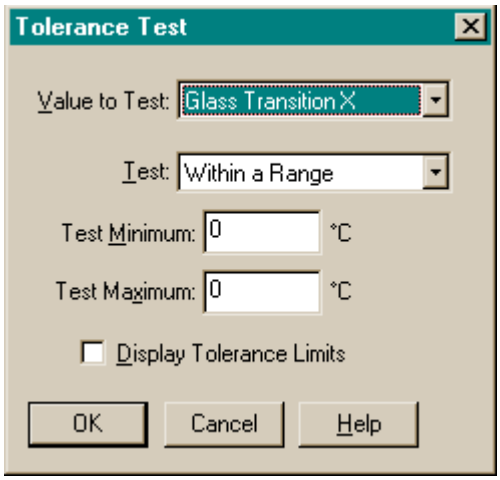
You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

Calculate closes the dialog box, displays tangent lines on the curve, and displays the Adjust Tangents dialog box. **Cancel** closes the dialog box without performing the calculation.

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Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Glass Transition X, Delta Cp, Onset X, and End X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Adjust Tangents Dialog Box

The Adjust Tangents dialog box appears automatically when you click on the **Calculate** button in the Onset, Oxidative Induction, Glass Transition, and Step Transition dialog boxes.

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Left Tangent radio button

Activates the left tangent for adjustment. In general, the left tangent corresponds to the point where the curve begins to deviate from the baseline. Use the Up and Down buttons to adjust the left tangent line or move the line on the graph.

Right Tangent radio button

Activates the right tangent for adjustment. For an onset calculation for a typical heating curve, the right tangent corresponds to the point of maximum slope of the leading edge of the peak. For a glass transition calculation, the right tangent should be placed at a position on the curve where there is not much activity since glass transition measures the change in specific heat. For a step transition calculation, the right tangent should be where the curve starts to go to back to baseline. For an oxidative induction calculation for DSC, the right tangent should be placed at the point of maximum slope of the leading edge of the peak; for TGA OIT, it should be placed where the curve starts to go back to baseline. Use the Up and Down buttons to adjust the right tangent line or move the line on the graph.

Increment

Enter the increment value to use when adjusting the tangents. The default value is 1.

Calculate

After adjusting the tangents, click on Calculate to close the dialog box, complete the calculation, and display the results on the curve.

Restore

If you do not like where you have positioned the tangent line on the curve, you can restore the line to its original position by clicking on the **Restore** button.

Glass Transition of Polymers

The glass transition (Tg) value for a polymer indicates a transition from a rigid to a flexible structure. It causes a change in heat capacity and a shift in baseline. Below Tg the polymer loses its flexible working behavior. Glass transition effects are relatively small in magnitude compared with melting point.

For amorphous polymers or the amorphous regions of partially crystalline polymers, the glass transition is exhibited when a viscous or rubbery state is transformed into a glass-like state. Relaxation phenomena occur above and below the glass transition temperature since neither the viscous nor the glassy state is an equilibrium state. Differential scanning calorimetry measures the glass transition of polymers as a stepped increase in the heat capacity of the sample during heating. This is caused by a increased molecular motion in the polymer.

Glass transition can be observed in amorphous polymers, e.g., poly(vinyl chloride) and poly(vinyl acetate). A small amount of organic solvent in the sample shifts Tg to lower temperatures. Samples containing low molecular mass compounds measured in open sample pans may display a large endothermic peak immediately after the glass transition. The peak is the result of the vaporization of the low molecular mass component. If the sample baseline changes sharply after the glass transition, the Tg value is not reliable. The sample should be dried until no more vaporization is seen and then it should be reheated to determine Tg.

In some cases, glass transition may be difficult to measure. The Tg of a partially crystalline polymer is difficult to detect by DSC because enhancement of molecular motion in the amorphous regions is restricted by the crystalline regions. (Restricted motion of amorphous regions can be observed by using DMA techniques.) In densely crosslinked polymers it is difficult to observe Tg because of restriction of the main chain motion and the sample baseline step occurs over a broad temperature interval.

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Reference

Hatakeyama, T., Quinn, F.X.: *Thermal Analysis: Fundamentals and Applications to Polymer Science*, Wiley, New York, 1994.

Step

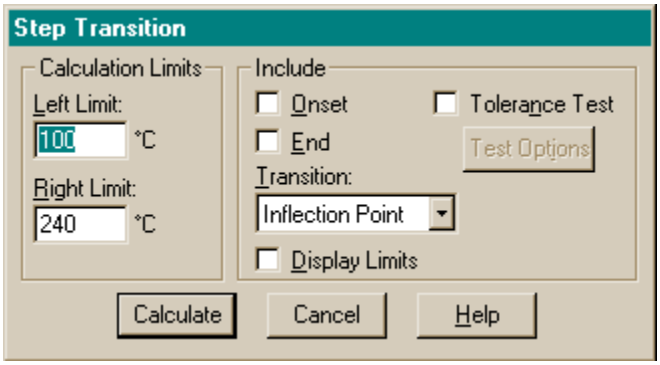
This command is available for all TGA analyzers and DMA/TMA analyzers. Select this command to perform a Step transition calculation based on the active curve. It determines the starting point, midpoint, and end point of a step transition. When you select this command, the Step Transition dialog box appears in which you enter calculation limits, whether to include onset or end points, and the type of transition to use: inflection point, half width, or half height.

Step Transition Dialog Box

The Step calculation is used for all data. It determines the starting point, midpoint, and end point of a step transition. It is similar to the Glass Transition (Tg) calculation used for heat flow curves of DSC and DTA analyzers. An example of the use of a Step calculation is the determination of the onset, end, and inflection points of water loss on a TGA 7 weight loss curve. DMA is a sensitive way to measure the glass transition of polymers. Side chain or main chain motion in specific regions of the polymer and local mode relaxation which cannot be monitored by DSC can be observed with DMA. From the variation in the temperature of the tan δ peak of a DMA curve as a function of frequency, a transition map can be compiled.

The results of a Step calculation are the X and Y values at the limits, onset, and end points, if you choose to display them, the transition point, either the inflection point, half-height, or half-width point, and the delta Y between the first tangent and the transition point.

The Step Transition dialog box appears when you select the Step command in the Calc menu and two **X**'s appear on the active curve. The dialog box contains the following fields:



Calculation Limits

Left Limit

Enter the left limit for the step transition calculation. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the value in the entry field automatically changes to reflect the position.

Right Limit

Enter the right limit for the step transition calculation. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired location, the value in the entry field automatically changes to reflect the position.

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Include

You can include the onset and the end points of the transition in the display of the result of the calculation. The onset value is calculated by finding the intersection of the extrapolated tangent at the first limit and the extrapolated tangent at the inflection point. The end value is calculated by finding the intersection of the extrapolated tangent at the second limit and the extrapolated tangent at the inflection point. You can also include the display of the X,Y ordinates of the limits.

Transition

Select the transition type to use in the step transition calculation:

- Inflection Point reports the point between the limits at which the slope of the curve changes from increasing to decreasing or vice versa.
- Half-Height reports the point on the curve that is halfway between the extrapolated tangent lines.
- Half-Width reports the point on the curve that is halfway between the onset and end points.

Tolerance Test

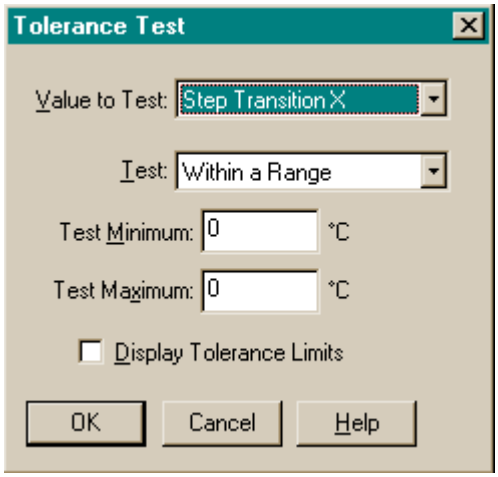
You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

Calculate

Closes the dialog box, displays tangents lines on the curve, and displays the Adjust Tangents dialog box (see above).

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Step Transition X, Delta Y, Onset X, and End X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values,

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depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Crystallinity (DSC only)

The Crystallinity option is available for heat flow curves. When you select Crystallinity, the program automatically begins and loads the active data file. The Crystallinity software calculates the crystallinity of the sample from the data file and displays the crystallinity curve. Use the online Help file associated with the Crystallinity software for additional help.

The Crystallinity calculation applies to heat flow versus temperature data from DSC analyzers. When **Crystallinity** is selected from the Calc menu, the active curve is copied to the Windows Clipboard, the third-party Crystallinity software loads, and curve on the clipboard is placed and used in the software. **The Crystallinity software has its own online help**.

Crystallization of polymer systems usually gives rise to a semicrystalline morphology consisting of crystalline and amorphous phases. This is called a two-phase model and a characteristic quantity in such a model is crystallinity. Crystallinity represents the amount of crystalline material present in the system. To find this value, a measured quantity (e.g., volume or enthalpy) is compared with the extreme values of that quantity. These extremes are the reference states. In the two-phase model, the reference states are (100%) amorphous and (100%) crystalline. The model assumes additivity of extensive quantities such as volume and enthalpy for the two phases.

DSC curves can be used to calculate the mass crystallinity as a function of the temperature for any desired thermal history because the form in which the sample is supplied is not very critical and because DSC enables dynamic measurements (with cooling and heating at certain rates and isothermal periods). DSC is the only technique that enables rapid and quantitative measurement of crystallinities in a freely selectable manner.

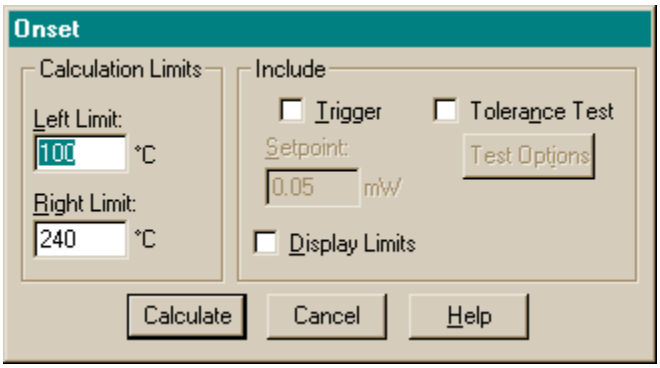
Onset

This command is used to perform an Onset calculation on the active curve. The Onset calculation determines the beginning of any transition that is distinguished by a significant change from the baseline. When you select this command, the Onset dialog box appears in which you enter the left and right limits for the onset calculation.

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Onset Dialog Box

The onset calculation determines the beginning of any transition that is distinguished by a significant change from the baseline. It is available for any curve type generated by any analyzer. An example of its use is the determination of the beginning of a melt on a DSC heat flow curve. When you select Onset from the Calc menu, two **X**'s are displayed on the active curve and the dialog box appears:



Calculation Limits

Left Limit

Enter the left limit for the onset temperature calculation. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired position, the value in the entry field automatically updates.

Right Limit

Enter the right limit for the onset temperature calculation. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired position, the value in the entry field automatically updates.

Include Trigger check box

The trigger indicates the point at which the curve deviates from the pretransition baseline by a specified amount, called the setpoint. To include the Onset trigger point in the display of the calculation results, click in the Trigger box. The Setpoint entry field is then activated in which you enter the trigger value. The trigger value is calculated by locating the first time or temperature for which the vertical distance between the curve and initial tangent line is greater than the value of the trigger. The initial tangent line is defined as the left-hand limit tangent line for a heating curve on temperature X axis or the right-hand limit tangent line for a cooling curve on a temperature X axis.

Setpoint

Enter the setpoint for the trigger. The default value is 1% of the currently displayed Y scale and the range is 0.1% to 20% of the same scale. The amount of deviation in the trigger can be set by changing the setpoint value.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; clicking on it displays the Tolerance Test dialog box.

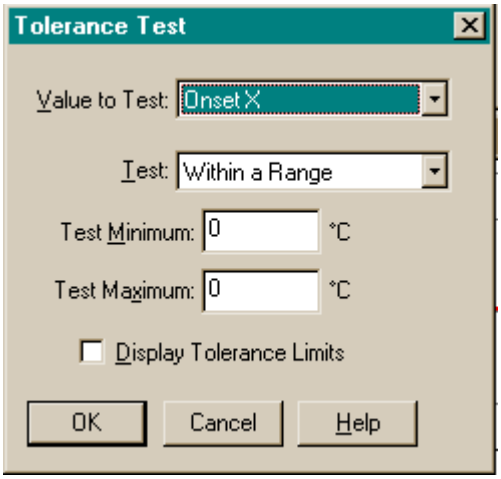
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Calculate

Closes the dialog box, displays tangents lines on the curve, and displays the Adjust Tangents dialog box (see above).

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Onset X and Trigger X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

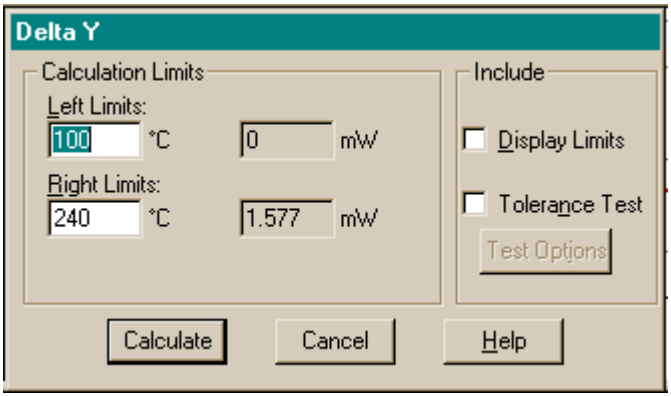
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Delta Y

Select this command to perform a Delta Y calculation based on the active curve. The Delta Y calculation determines the change in the ordinate axis after you select two points on the X axis for the active curve. When you select this command, the Delta Y dialog box appears in which you enter the X-axis values for the two points.

Delta Y Dialog Box

The Delta Y calculation determines the difference in ordinate units between two selected points on a curve. It is available for any curve type generated from any analyzer. An example of its use is the determination of the percent weight loss that occurs between two temperatures on a TGA curve. Two **X**'s appear on the active curve when you select the **Delta Y** command in the Calc menu. The dialog box contains the following fields:



Calculation Limits

Left Limits

Enter the X value of the left limit for the Delta Y calculation. The Y value for that point is displayed but cannot be changed manually. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the values in **both** entry fields change automatically.

Right Limits

Enter the X value of the right limit for the Delta Y calculation. The Y value for that point is displayed but cannot be changed manually. When the right limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the values in **both** entry fields change automatically.

Include

Display Limits

You can have the X,Y ordinates of the left and right limits used in the Delta Y calculation displayed on the curve along with the Delta Y value.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

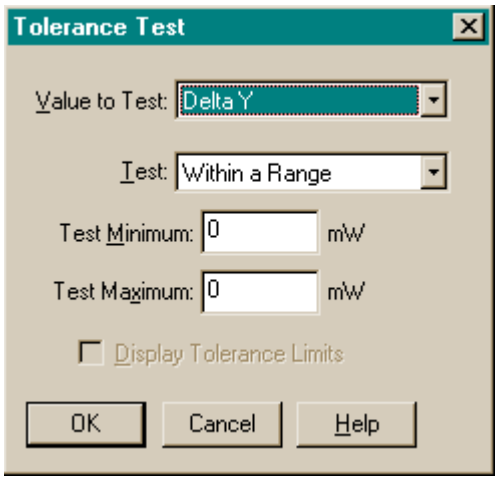
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Calculate

Closes the dialog box, performs the calculation, and displays the results on the curve.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test isDelta Y.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

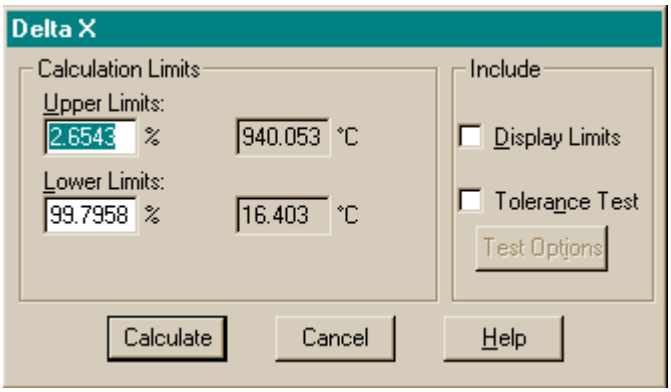
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Delta X

This command is available for TGA Weight and Weight % curves and all DMA/TMA curves. Select this command to perform a Delta X calculation based on the active curve. The Delta X calculation determines the change in the abscissa axis after you select two points on the Y axis for active curve. When you select this command, the Delta X dialog box appears in which you enter the Y-axis values for the two points.

Delta X Dialog Box

The Delta X calculation determines the difference in abscissa units between two selected points on a curve. It is available for TGA and DMA/TMA curves. An example of its use is the determination of a temperature corresponding to a specific heat loss on a TGA curve. Two **X**'s appear on the active curve when you select the **Delta X** command in the Calc menu. The dialog box contains the following fields:



Calculation Limits

Upper Limits

Enter the Y value of the upper limit for the Delta X calculation. The X value for that point is displayed but cannot be changed manually. When the upper limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the value in **both** entry fields automatically update.

Lower Limits

Enter the Y value of the lower limit for the Delta X calculation. The X value for that point is displayed but cannot be changed manually. When the lower limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired location, the values in **both** entry fields automatically update.

Display Limits

You can have the X,Y ordinates of the left and right limits used in the Delta X calculation displayed on the curve along with the Delta X value.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

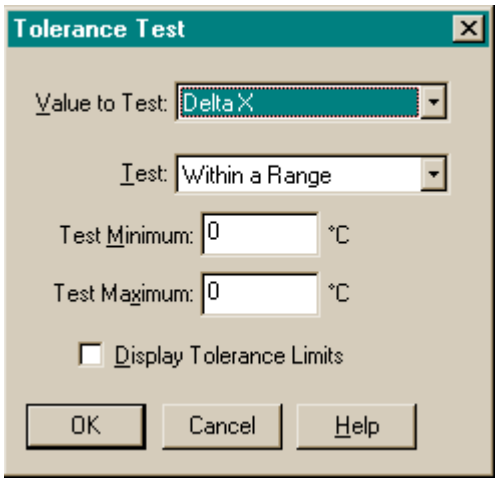
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Calculate

Closes the dialog box, performs the calculation, and displays the results on the curve.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Delta X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

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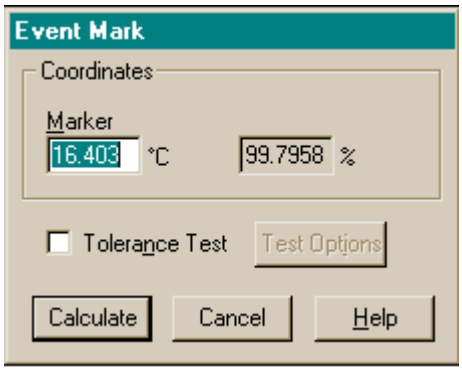
Event Mark

Select this command to perform an Event Mark calculation based on the active curve. The Event Mark calculation lets you mark or label the X and Y coordinates of events on the active curve. When you select this command, the Event Mark dialog box appears in which you enter the X-axis value of the event you want to mark, click on **Calculate**, and the marker is automatically displayed along with annotation.

Event Mark Dialog Box

Event Mark lets you label the X and Y coordinates of an event on a curve. A tic mark appears at the user-selected position and the mark is annotated with the X value and unit and the Y value and unit. You can mark an unlimited number of events on the data curve before performing a calculation. You can move the event mark annotation around on the screen by clicking and dragging it to a new position.

The Event Mark dialog box appears when you select the **Event Mark** command in the Calc menu and an **X** appears on the active curve at the minimum X value.



Marker

Enter the X value of the event you wish to mark. The Y value for that X value is automatically displayed in the grayed-out field to the right. This is for information purposes only. You may also drag the **X** to the desired position on the curve. Its value is displayed in the Marker field.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

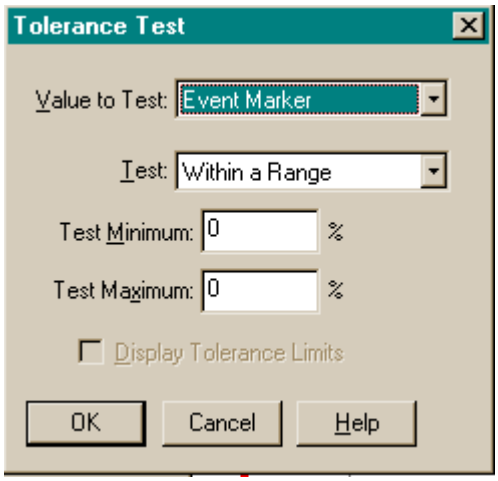
Calculate

Closes the dialog box, performs the calculation, and displays the result on the curve.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:

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Value to Test is Event Marker.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Trigger

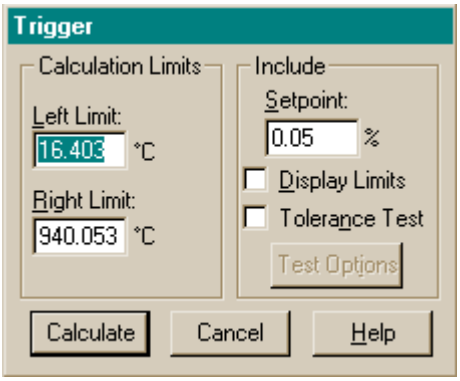
Select this command to perform a Trigger calculation based on the active curve. The Trigger calculation determines the beginning of a transition without a clearly defined onset in the curve. When you select this command, the Trigger dialog box appears in which you enter calculation limits and the set point of the trigger.

Trigger Dialog Box

The Trigger calculation determines the beginning of a transition without a clearly defined onset in the curve. This transition is the point at which the difference between the curve and the tangent from the first limit is equal to the Setpoint value. This calculation is available for any curve type

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generated from any analyzer. It is useful whenever the onset of a transition is very gradual. This Trigger calculation is identical to that included in the Onset calculation. Two **X**'s appear on the active curve when you select the **Trigger** command in the Calc menu. The dialog box contains the following fields:



Calculation Limits

Left Limit

Enter the left limit for the Trigger calculation. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the value in the entry field automatically updates.

Right Limit

Enter the right limit for the Trigger calculation. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired location, the value in the entry field automatically updates.

Include

Setpoint

Enter the trigger setpoint value. The default value is 0 in the same units as the Y axis of the active curve.

Display Limits

You can have the X,Y ordinates of the left and right limits used in the Trigger calculation displayed on the curve along with the Trigger value.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. when you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

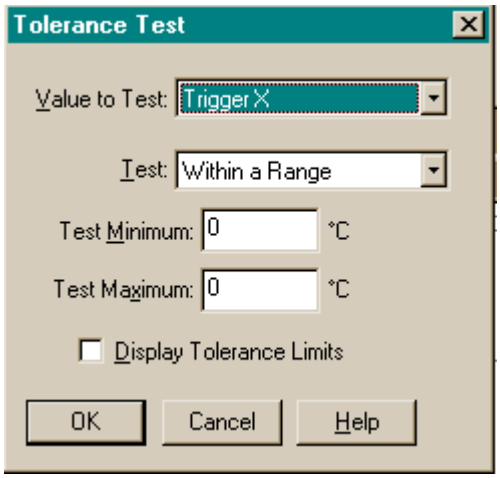
Calculate

Closes the dialog box, performs the calculation, and displays the results on the curve.

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Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Trigger X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

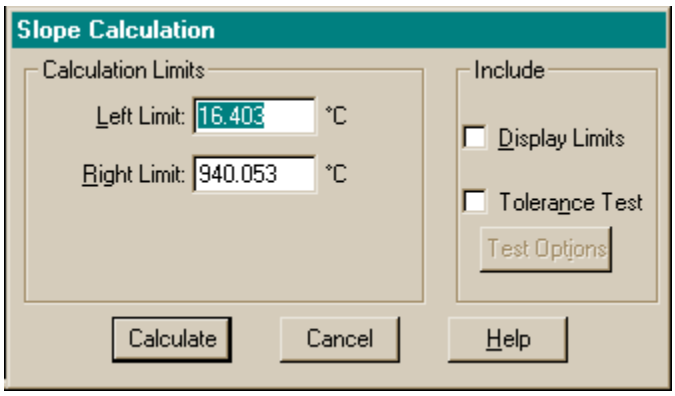
Slope

Select this command to calculate the slope of a user-defined section of the active data curve. When you select this command, the Slope dialog box appears in which you enter the limits used to calculate the slope.

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Slope Dialog Box

The Slope calculation determines the slope and the inverse slope of a user-defined section of the active curve. It is available for any curve type generated by any analyzer. The Slope dialog box appears when you select the **Slope** command from the Calc menu and two **X**'s appear on the active curve.



Calculation Limits

Left Limit

Enter the left limit for the slope calculation; this is X1. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the value in the entry field automatically updates.

Right Limit

Enter the right limit for the slope calculation; this is X2. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired location, the value in the entry field automatically updates.

Include

Display Limits

You can include the X,Y ordinates of the left and right limits used in the slope calculation on the display of the result, along with the Slope and Inverse Slope values.

Tolerance Test

You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

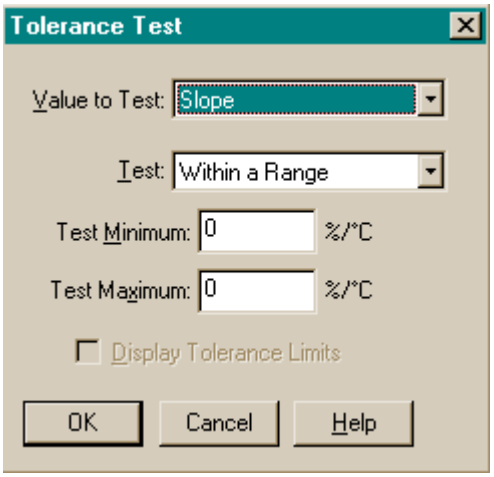
Calculate

Closes the dialog box, performs the slope calculation, and displays the results on the curve.

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:

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Value to Test is Slope and Inverse Slope.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Oxidative Induction (DSC only)

The Oxidative Induction calculation is a special case of the onset calculation. It must be used on a normalized heat flow curve displayed on a time scale. This calculation determines the beginning of any transition that is distinguished by a significant change from the baseline. When you select this command, the Oxidative Induction dialog box appears in which you enter the limits of the calculation and indicate other parameters to include in the displayed results.

Oxidative Induction Dialog Box

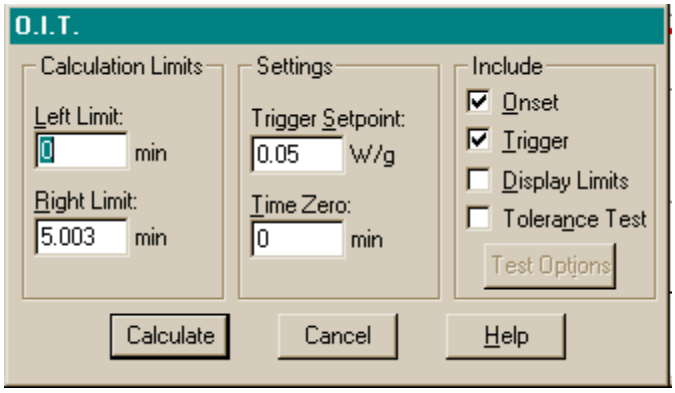
The oxidative induction time calculation is a special case of the onset calculation. The OIT calculation, like onset, determines the beginning of any transition that is distinguished by a significant change from the baseline. However, it must be used on a normalized heat flow curve

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displayed on a time scale. The calculated results are based on a reference time called Time Zero. By default, this is the time that the gas change occurred during the run. If no gas change was programmed in the method, the default for Time Zero is zero minutes.

The OIT calculation determines the time at which antioxidant in a sample is used and the sample begins to degrade. Generally, the sample is heated past the melt in an inert atmosphere, then held isothermally while the atmosphere is changed to oxygen. The amount of time in the oxygen atmosphere before degradation occurs is the reported calculation result.

The Oxidative Induction dialog box appears when you select the **Oxidative Induction** command in the Calc menu. You may first see a message box stating that the curve needs to be normalized and the X axis needs to be scaled to time. Click on **OK** and the display is fixed automatically. When the OIT dialog box is displayed, two **X**'s appear on the active curve.



Calculation Limits

Left Limit

Enter the left limit for the OIT calculation. When the left limit is selected directly on the curve by clicking on and dragging the leftmost **X** to the desired location, the value in the entry field automatically updates.

Right Limit

Enter the right limit for the OIT calculation. When the right limit is selected directly on the curve by clicking on and dragging the rightmost **X** to the desired location, the value in the entry field automatically updates.

Settings

Trigger Setpoint

This field is available if the Trigger check box under Include is selected. Enter the setpoint for the trigger value. The default value is 1% of the currently displayed Y scale.

Time Zero

The OIT calculation is based on the reference time called Time Zero. Time Zero is the time that the sample atmosphere changes to oxygen during the run. If no gas change was programmed, the default for Time Zero is 0 minutes. Enter the time at which there is a gas change.

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Include

You can include the onset and trigger points in the display of the calculation results. The onset value is calculated by finding the intersection of the tangents to the left and right limits selected, relative to Time Zero. Select Trigger to enable the Setpoint entry field and to calculate and include the trigger point with the results. The trigger indicates the point, relative to Time Zero, at which the curve deviates from the pretransition baseline by the amount equal to the Trigger Setpoint. You can also indicate if you want the X,Y coordinates of the limits displayed in the Data Analysis window.

Tolerance Test

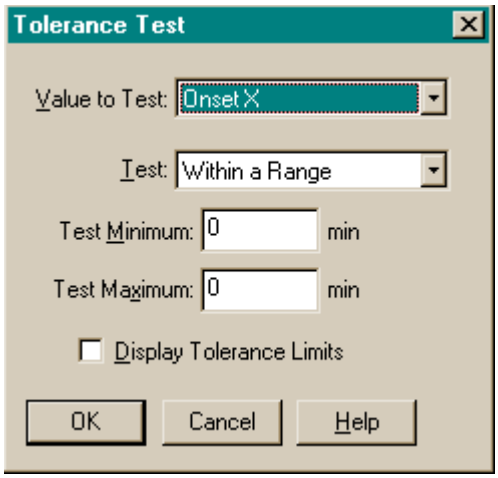
You can have the results of the calculation tested to see if they meet tolerance criteria that you set in the Tolerance Test dialog box. When you select Tolerance Test, the **Test Options** button becomes active; click on it to display the Tolerance Test dialog box.

Calculate

Closes the dialog box, performs the calculation, and displays the Adjust Tangents dialog box (see above).

Tolerance Test Dialog Box

When Tolerance Test is selected, the **Test Options** button becomes selectable. Click on it to display the Tolerance Test dialog box:



Value to Test is Onset X and Trigger X.

Test types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

You can choose not to **Display Tolerance Limits** on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue

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- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Specific Heat - Single Curve Command

When a material is subjected to a linear temperature program, the heat flow rate into the sample is proportional to its instantaneous specific heat. Since the scanning rate of the DSC analyzer is linear and the instrument measures heat flow directly, the specific heat of a sample material is easily calculated from any DSC scan.

Select this command to display automatically the specific heat curve for the focused curve, which must be a heat flow curve generated from a DSC 7, Pyris 6 DSC, or a Pyris 1 DSC. The heat flow curve must contain a scanning step. If it contains more than one scanning step and there are no isothermal steps, one specific heat curve is generated. If the focused curve contains both isothermal and scanning steps, a separate Specific Heat curve is created for each group of scanning steps. If the focused curve contains isothermal steps only, no Specific Heat curve is created.

Specific Heat - Multiple Curves Command

Select this command to generate a specific heat curve from two or more curves. The focused curve must be a heat flow curve generated by a DSC 7, Pyris 6 DSC, or a Pyris 1 DSC. Each curve must be the result of an Iso-Scan-Iso temperature program, where the scanning step could have either heating or cooling. When you select this command, the Specific Heat window is displayed in which you enter the Baseline and Reference curves for use in the calculation in addition to other details.

Specific Heat Window

The specific heat is the amount of energy required to increase the temperature of a unit quantity of material (e.g., 1 g or 1 mole) 1°C. When a sample is subjected to a linear temperature program, the heat flow rate into the sample is proportional to its instantaneous specific heat. Because a DSC analyzer measures the heat flow rate as a function of temperature, specific heat values are quickly determined. Since the scanning rate of the DSC is linear and the instrument measures heat flow directly, the specific heat of a sample material is easily calculated from the scan.

The Specific Heat calculation can be performed on a heat flow, unsubtracted heat flow, or baseline heat flow curve obtained by a DSC 7, Pyris 6 DSC, or Pyris 1 DSC. The curve must contain a scanning step. If it contains more than one scanning step and no isothermal steps, one Specific Heat curve is generated and you will see only the option **Single Curve** displayed on the Specific Heat submenu. When that option is selected, the Specific Heat curve is displayed automatically and becomes the active curve.

If the curve contains a minimum of three steps – isothermal, scan, isothermal – a separate Specific Heat curve is created for each group of scanning steps and **Multiple Curves** is available for selection on the Specific Heat submenu. When Multiple Curves is selected, the Specific Heat window appears. Entries made here are used to create the Specific Heat curve.

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The window contains two tabbed pages: Method and Details. The Method page contains the following fields:

Select the Baseline Curve

The Legend text for each displayed curve that meets the following criteria is displayed in this box. The curve must (1) be a Heat Flow curve, (2) have the form of Iso–Scan–Iso, (3) have the leading and trailing isothermal steps and the scanning step be at least 1 minute long, and (4) have the starting and ending temperatures in the scan be the same in all curves. The Baseline curve results from performing a run using an empty sample pan (baseline pan) of the same type used for the sample run and using the same method. Select the curve you want for the baseline curve by clicking on it. Baseline curve selection is available if you have two or more curves displayed.

Reference Curve check box

If you want to use a reference curve in the calculation of specific heat, click in the box.

Select the Reference Curve

The Legend text for each displayed curve that meets the following criteria is displayed in this box which appears when you click in the Reference Curve check box. The curve must (1) be a Heat Flow curve, (2) have the form of Iso–Scan–Iso, (3) have the leading and trailing isothermal steps and the scanning step be at least 1 minute long, and (4) have the starting and ending temperatures in the scan be the same in all curves. The Reference curve results from performing a run with a reference material, e.g., sapphire, contained in a reference pan of the same type used for the sample run and using the same method. Select the curve you want for the reference curve. Reference curve selection is available if you have three or more curves displayed.

Details tab

To add details to the specific heat calculation, click on this tab to display the Specific Heat Details page.

Specific Heat Details Page

In performing a Specific Heat — Multiple Curves calculation, this page is used to enter details on the sample pans and the reference material that were used in collecting the data curves to be used in the Specific Heat calculation.

Reference Material

Enter the name of the reference material that you used to create the Reference curve, if applicable. You can also select the name from the drop-down menu.

Pan Material

Enter the name of the pan material used in the sample run, the baseline run, and the reference run. You can also select the name from the drop-down menu.

Sample Pan Weight

Enter the weight (mg) of the sample pan used for the sample run. This is used if the heat flow curve is not normalized.

Baseline Pan Weight

Enter the weight (mg) of the sample pan used for the baseline run.

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Reference Pan Weight

Enter the weight (mg) of the sample pan used for the reference run, if applicable.

Include these Details in Displayed Results check box

Click in the check box to have these details annotated to the display in the Data Analysis window.

Purity (DSC only)

To perform a Purity calculation, the curve must be a heat flow curve and the X axis must be temperature. The data may be subtracted, smoothed, or optimized. The Purity Analysis program determines the purity of a sample by fitting a portion of the DSC data to the Van't Hoff relationship. The fit of the data determines the reliability of the purity measurement for the sample. A succession of dialog boxes helps define the parameters used to calculate the purity of the data curve.

Purity Analysis

The determination of the purity of an organic compound by DSC is based on the fact that the presence of even minute amounts of impurity in a sample broadens its melting range and lowers the final melting point from T_0, the melting temperature of an infinitely pure material, to a lesser temperature, T_m. As the impurity contents increases, the melting point decreases and the range of melting broadens. Even minute differences in the impurity content of the sample result in distinct differences in the melting peak shape and the final melting temperature.

The fit of a portion of the DSC data to the Van't Hoff relationship determines the reliability of the purity measurement for the sample. The three purity parameters determined by the Purity Analysis program are (1) mole fraction of impurity, (2) x-correction value, and (3) theoretical melting point (T0) of pure sample.

There are two methods for calculating the purity parameters: the standard method and the multiple linear regression (MLR) method. Most samples can be analyzed using either method. However, samples that begin to decompose during the melt should be analyzed using the MLR method.

To perform a purity analysis on DSC data, you may use data that is subtracted, smoothed, or optimized. You may also modify the data by the values entered in the dialog boxes that make up the Purity Analysis program. These are

1. Peak area calculation limits
2. Calculation method used, thermal resistance constant, molecular weight of the sample, and heat capacity of the sample pan
3. Baseline adjustment for MLR method
4. Peak area percent

   You can also choose to display a curve of the temperature versus the reciprocal of the fraction of sample melted by clicking in the check box for

5. View T vs 1/F

Calculate Peak Area Dialog Box

Calculate Peak Area is part of the Purity calculation. Purity is calculated using the van't Hoff equation. The result of this equation is the inverse of F_s, which is the fraction of the sample melted at a specified temperature (T_s). F_s is equal to A_s/A_T, where A_T is the total area of the curve and T_s is the area of the curve up to T_s. In the Calculate Peak Area dialog box you specify the limits to use in the calculation of A_T.

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Left Limit

Enter the left limit or the peak start temperature. You may also click on and drag the left **X** to the desired location; the value in the Left Limit entry field changes automatically to reflect the position of the **X**.

Right Limit

Enter the right limit of the peak end temperature. You may also click on and drag the right **X** to the desired location; the value in the Right Limit entry field changes automatically to reflect the position of the **X**.

Next

Accepts the values entered here and displays the next dialog box, Select Calculation Method.

Select Calculation Method Dialog Box

In performing a purity analysis calculation, you must select the type of method to use for calculating purity parameters. This is done in the Select Calculation Method dialog box:

Select Method

Purity analysis provides two methods of calculating the purity parameters: Standard and Multiple Linear Regression (MLR). The calculation method selection should depend on the baseline of the data curve that you are analyzing for purity.

If your DSC data is characterized by a conventional melting curve, in which the melting peak is complete, the total area under the peak is calculated. Therefore, it is not necessary to create a new baseline. In this case, you can use either the standard or the MLR method of calculation for the purity analysis. The standard method involves an iterative calculation of the x-correction, followed by a multiple linear regression on the two remaining purity parameters.

In some DSC analyses, the sample may decompose during the melting phase. This decomposition may produce a melting curve in which the melting peak is incomplete and the total area under the peak cannot be obtained. For these analyses you must use the multiple linear regression method of calculating the purity of the sample. This requires creating a new baseline for the data curve. The MLR method uses a 3 x 3 multiple linear solution which determines the three purity parameters simultaneously. This method does not require obtaining the total area under the peak.

R0 (Thermal Resistance Constant)

Enter the thermal resistance constant for your sample pan. The value depends on the type of pan you are using, the way the pan has been crimped, and the temperature of the reaction. For most experiments, use the default value of 72.00°C/W. However, under certain conditions it may be necessary to calculate R0 for the sample pan. These conditions are

- use of a nonstandard sample pan such as graphite, alumina, or high pressure capsule
- the reaction takes place over a high temperature range, e.g., 400°C

If you want to calculate R0, see Determining the Thermal Resistance Constant for the Sample Pan below.

Molecular Weight

Enter the molecular weight in g/mol of your sample material. Purity calculations cannot be performed until the molecular weight has been entered.

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Cp (pan)

Enter the heat capacity for the sample pan. This value depends on the type of pan used. The value is used to perform a minor correction in the purity calculation. Use the default 0.023 J/°C for most applications. However, if you are using high pressure capsules and want to calculate the heat capacity of your sample pan material, see Determining the Heat Capacity of Sample Pans below.

Thermal Resistance Constant of a Sample Pan

The value for the thermal resistance constant (*R*0) for the sample pan depends on the type of sample pan you are using, the way the pan has been crimped, and the temperature of the reaction. For most experiments, the default value may be used. However, under certain conditions it may be necessary to calculate the *R*0 for the sample pan. These conditions are

a. if the sample pan is a nonstandard pan, e.g., graphite, alumina, or high pressure capsule

b. if the reaction takes place over a very high or very low temperature range, e.g., above 400°C or below –50°C.

If you want to calculate *R*0 instead of using the default value, follow the procedure below:

1. Place a moderate amount (10 – 20 mg) of a pure melting point standard material in the type of sample pan that you are using for your experiment. The melting point should be within 200°C of the region being investigated.
2. Place the sample pan in the sample holder.
3. Place an empty sample pan in the reference holder of the analyzer.
4. Set up and run an experiment. Use a slow scanning rate (typically the scan rate you will use for your purity experiments) and start the scan at a low temperature so that at least three minutes of data is taken before the onset of the melting endotherm.
5. When the experiment is finished and the data curve is displayed, select Delta Y from the Calc menu. Enter the Left and Right Limits for calculating Delta Y, choosing two points on the leading edge of the curve. Alternatively, you can drag the **X**'s to the desired locations on the curve.
6. Click on the **Calculate** button; the results are displayed in the window.
7. Note the Delta Y value (in mW) and the temperatures at X1 and X2.
8. Using the equation

$$R0 = \Delta T / \Delta Y \times 1000$$

calculate the thermal resistance of your sample pan. The value is multiplied by 1000 to convert mW to W which is the unit used in purity analysis.

For example, (0.797°C / 10.0967 mW) x 1000 = 0.797°C / 0.0100967 W = 71.943°C/W.

This *R*0 value can also be found using the Slope calculation.

Determining the Heat Capacity of Sample Pans

The heat capacity of a sample pan is determined with the following equation:

$$Cp(pan) = T_c \times W$$

where Cp is the heat capacity (in J/°C) of the sample pan at constant pressure, T_c is the thermal capacity (in J/g °C) of the sample pan, and W is the weight (in g) of the empty sample pan and lid.

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For example, a typical weight of an empty aluminum pan with a lid is 0.026 g. Therefore,

Cp(pan) = (0.9026)(0.026)
= 0.023 J/°C at 25°C

Thermal Capacity of a Sample Pan

Thermal capacity is the quantity of heat necessary to produce unit change of temperature in unit mass. Thermal capacity is expressed in J/g °C. It is numerically equivalent to the specific heat, the ratio of the thermal capacity of a substance to the thermal capacity of water at 15°C. The table below provides the thermal capacity T_c of four sample pan materials.

Sample Pan Material	–23°C	25°C	227°C	427°C
Aluminum	0.8622	0.9026	0.9956	1.0871
Graphite	0.5679	0.7107	1.2193	1.5468
Gold	0.1266	0.1289	0.1334	0.1385
Platinum	0.1298	0.1328	0.1383	0.1437
Stainless Steel		0.48		

Adjust Baseline Dialog Box

For purity analysis, the Adjust Baseline dialog box appears if you select the MLR Calculation method and you click on the **Next** button in the Select Calculation Method dialog box. A baseline is displayed on the curve with **X**'s indicating the left and right limits. If the data show no evidence of sample decomposition, accept the baseline by clicking on the **Next** button. If your data show an incomplete melting curve, generate a new baseline by changing the limits.

Left Limit

Enter the value for the left baseline limit. This value should be before the melting curve begins. You may also click on and drag the **X** to the desired location. The value in the entry field changes automatically.

Right Limit

Enter the value for the right baseline limit. This value should be lower than the temperature of the peak. If you position the right baseline limit close to the left baseline limit, the baseline that is drawn by the system will appear as an extension of the data before the melting curve begins. You may also click on and drag the **X** to desired location. The value in the entry field changes automatically.

Adjust % Area Limits Dialog Box

In performing a purity analysis calculation, the Adjust % Area Limits dialog box is displayed. If using an MLR method, the baseline calculated in using the baseline limits entered in Adjust Baseline is displayed in the dialog box. If you are not satisfied with the baseline, click on the **Back** button to return to the Adjust Baseline dialog box. Also appearing on the curve are two vertical lines indicating the left and right limits for the partial area under the curve that is to be used in the purity calculation.

The Van't Hoff plot is generated from a series of sample temperatures and fraction melted results, in the range of approximately 5% – 60% melted. The range used is adjusted using the fields in this dialog box:

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Left Limit

Enter a new left limit for the percent area. The default value places the left limit at the temperature where approximately 5% of the sample has melted. You may also click on and drag the left vertical red line to the desired position. The value in the entry field, as well as the %, is adjusted automatically.

Right Limit

Enter a new right limit for the percent area. The default value places the right limit at the temperature where approximately 60% of the sample has melted. You may also click on and drag the left vertical red line to the desired position. The value in the entry field, as well as the %, is adjusted automatically.

View T vs 1/F

In performing a purity analysis calculation, the View Curves dialog box appears when you click on the **Next** button in the Adjust % Area Limits dialog box. To see how the data fits the Van't Hoff relationship, select **T vs 1/F** by clicking in the check box. In a **new** window, entitled Van't Hoff Plot, the corrected data points are shown as red boxes and the line drawn through them shows how the multiple linear regression fits the data points. The blue boxes represent the uncorrected values calculated for $1/F$ (F is the fraction of sample melted) at given temperatures.

Back

Returns you to the preceding dialog box, Adjust % Area Limits, without accepting any change made in this dialog box.

Finish

Accepts the entry made in this dialog box and proceeds to calculate the purity of the displayed data using the input values entered in all of the preceding dialog boxes. The results are displayed automatically.

NOTE: To close the **T vs 1/F** window, do one of the following: Click on the **Close** button on the upper-right-hand corner of the Data Analysis window; this closes both windows. Make the Data Analysis window the focused window, select **View Results** from the View menu, select the Purity entry in the "Select calculation results to display" list, and then click on the **Delete Results** button. This closes the Van't Hoff Plot window only.

Kinetics

This command is available for TGA and DSC analyzers. The Kinetics software is an optional package that must be installed on your computer.

The kinetics calculation applies to heat flow vs. temperature for DSC data and to weight vs. temperature for TGA data. In order to use the third-party Kinetics software, (1) you must select three or more curves that were collected by the same type of analyzer (DSC or TGA), (2) the X axis must be temperature, (3) each curve must be a single-step scan, and (4) the temperature range must be the same for all curves and the scan rate must be different.

The Kinetics software is used to obtain the kinetic parameters that characterize a thermally initiated reaction. This program uses a multilinear regression method to fit a single data curve (at a constant heating rate) to the Arrhenius relationship and thereby determine the pre-exponential factor, activation energy, and order of reaction.

The rate of reaction is expressed by

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$d\alpha/dt = k(1 - \alpha)\exp(n)$

where $d\alpha/dt$ is the rate of reaction, α is the degree of conversion or the fraction of the sample that reacted, t is time, n is the reaction order, and k is the Arrhenius rate constant. The software calculates the kinetics parameters – activation energy and degree of conversion – that characterize a thermally initiated reaction. The software fits the data to the Arrhenius relationship $k = Z \exp(-E_a/RT)$, where Z is the preexponential constant, E_a is the activation energy of the reaction, R is the universal gas constant, and T is the absolute temperature.

Activation energy of a thermal event can be directly determined from a series of thermogravimetric or differential scanning calorimetric runs performed at different scanning rates. If a series of runs is made at different scanning rates, each curve is shifted up on the temperature scale with increasing scanning rate so that a plot of the logarithm of the scanning rate versus the inverse of the absolute temperature (at the same conversion or weight loss %) is linear, with a slope directly proportional to the activation energy and known constants.

Various calculation inputs can be adjusted in order to make the data more meaningful. For each calculation the data fit is indicated by the confidence limits for the kinetic parameters and by the fit of the partial area data to the Arrhenius relationship (the plot of ln k vs. $1/T$). If satisfactory, the parameters and key inputs can be saved with a comment.

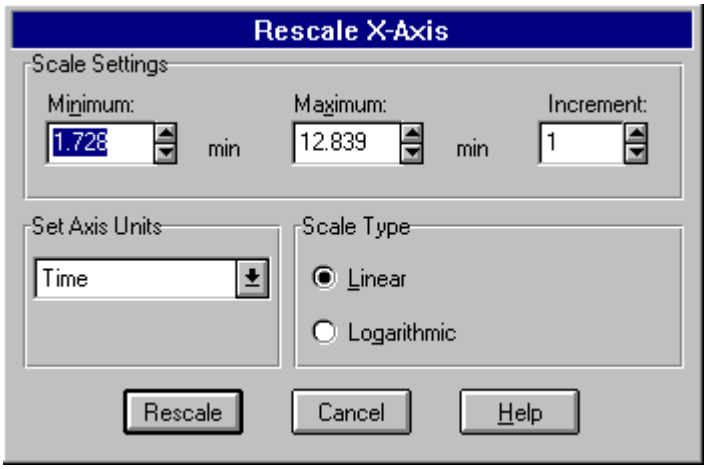
Display Menu

The items in the Display menu for both Data Analysis and Instrument Viewer are listed below. Some are analyzer-dependent.

Rescale X

The **Rescale X** command in the Display menu or the **Rescale X** button  on the Rescale Tools toolbar is used to rescale the X axis of the active curve in the Data Analysis window or the Instrument Viewer. In the Rescale X-Axis dialog box you change the minimum and maximum X values, units used for display, and the scale type.

Rescale X-Axis Dialog Box



Scale Settings

Minimum

Displays the minimum value for the X axis. Enter a new value or use the spin buttons to change the displayed value. The spin buttons will increase or decrease the value by the increment set in the Increment entry field.

Maximum

Displays the maximum value of the X axis. Enter a new value or use the spin buttons to change the displayed value. The spin buttons will increase or decrease the value by the increment set in the Increment entry field.

Increment

Displays the value used to increase or decrease the Minimum and Maximum entry fields when using the spin buttons. Type a value or use the spin buttons to change the displayed value in multiples of 10.

Set Axis Units

Displays a list of available axis units, e.g., time, temperature, dynamic strain, dynamic stress, dynamic force, frequency. The default selection is the current axis unit.

Scale Type

Linear

Changes the X axis to a linear scale.

Logarithmic

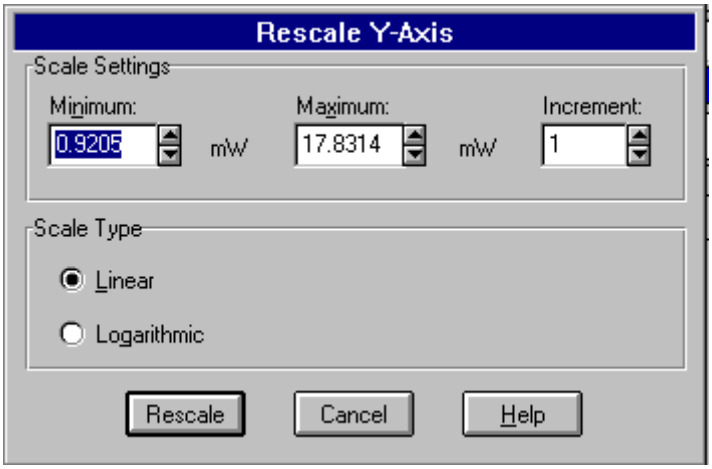
Changes the X axis to a logarithmic scale.

Rescale Y

The **Rescale Y** command in the Display menu or the **Rescale Y** button  on the Rescale Tools toolbar is used to rescale the Y axis of the active curve in the Data Analysis window or the Instrument Viewer. In the Rescale Y dialog box you can change the minimum and maximum Y values and the scale type.

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Rescale Y-Axis Dialog Box



Scale Settings

Minimum

Displays the minimum value for the Y axis. Enter a new minimum value or use the spin buttons to change the displayed value. The spin buttons will increase or decrease the value by the value set in the Increment field.

Maximum

Displays the maximum value for the Y axis. Enter a new maximum value or use the spin buttons to change the displayed value. The spin buttons will increase or decrease the value by the value set in the Increment field.

Increment

Displays the value used to increase or decrease the Minimum and Maximum values when using the spin buttons. Type a value or use the spin buttons to change the displayed value in multiples of 10.

Scale Type

Linear

Changes the Y axis to a linear scale.

Logarithmic

Changes the Y axis to a logarithmic scale.

Auto-Rescale

Auto-Rescale is available for the Instrument Viewer only. When this command is toggled on, the Instrument Viewer display is automatically rescaled when the data reaches the edge of the display. For example, if the X axis is 0 to 3 minutes and the run is 5 minutes, when the run goes past 3 minutes, the X axis rescales to be from 0 to 3.5 minutes. It continues to rescale until 5 minutes is reached. All data in the curves displayed remain displayed regardless of the original scaling. When

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Auto-Rescale is turned off, the current scale is maintained and data collected past the displayed range is not seen. A checkmark is displayed next to the menu command when Auto-Rescale is activated.

Normalize Y

This command is available for heat flow curves of DSC and DTA data and for Probe Position data for the DMA 7e. Select this command to change the Y axis of the active curve to a normalized scale. A curve is normalized by dividing the Y value by the sample weight. All curves associated with the active Y axis are also normalized. This is a toggle item on the Display menu. The normalized active curve is displayed as a dashed line rather than a solid line.

Weight % (TGA only)

Select this command to change the Y axis to Weight %; the label changes accordingly.

Log X

Select this command to toggle the X axis between linear and logarithmic scale. The Log X plot requires that the curve and the X-axis minimum values be greater than 0. When the X-axis scale is logarithmic, a checkmark appears next to the menu command. When a log scale is changed to linear, the new display uses the same minimum and maximum that were used for the log scale. You can also toggle the axis scaling by clicking on the **Log X** button on the Rescale Tools toolbar: 

Log Y

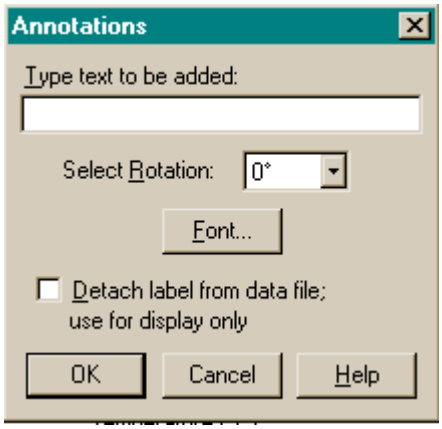
Select this command to toggle the Y axis between linear and logarithmic scale. The Log Y plot requires that the curve and the Y-axis minimum values be greater than 0. When the Y-axis scale is logarithmic, a checkmark appears next to the menu command. When a log scale is changed to linear, the new display uses the same minimum and maximum that were used for the log scale. You can also toggle the axis scaling by clicking on the **Log Y** button on the Rescale Tools toolbar: 

Annotate

Select this command from the Display menu or click on the **Annotate** button  on the Rescale Tools toolbar to add annotations to the Instrument Viewer or Data Analysis display. In the Annotations dialog box you enter text for a label to be displayed with the active curve. The label can be "attached" to the curve, i.e., it is saved in the data file, or it can be used for display only. You can select the font and orientation of the text.

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Annotations Dialog Box



Type text to be added

Enter the text (up to 40 characters) you want to display with the focused curve.

Select Rotation

From the drop-down list, select the angle at which to display the label: 0°, 90°, 180°, or 270°.

Font

This button displays the standard Windows Font dialog box in which you can select a typeface, type style, and type size for your text label.

Detach label from data file; use for display only

When you click in this check box, the annotation will not become part of the data file but will appear when the curve is displayed. The label will be rescaled with the curve. If the label is attached to the data file, it will not be rescaled with the curve.

Font Dialog Box

The Font dialog box appears when you select the **Font** button in the Graph page of Preferences or in the Annotatations dialog box. All annotations and results are subsequently displayed in the font selected. The Font dialog box contains the following fields:

Font

Displays a list of available fonts on your system. Use the scroll bar to display additional fonts.

Font style

Displays a list of available font styles for the selected font: regular, italic, bold, and bold italic.

Size

Displays a list of available font sizes for the selected font. Use the scroll bar to display additional sizes.

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Effects

When Strikeout is selected, all annotations and results are displayed with a strikeout through them. When Underline is selected, all annotations and results are displayed underlined.

Color

Displays a list of available colors in which the selected font can be displayed.

Sample

Displays a sample of the currently selected font, style, size, color, and effect.

Script

Lists the available language scripts for the specified font. Pick the one appropriate for the language for which your computer is set up.

Help Menu

The Help menu contains the following commands:

Contents & Index

Select this command to open Pyris Help. The Contents, Index, and Search tabs are in the left pane, and the contents of topics are displayed in the right pane or panes.

Quick Help

Select this command to display the Quick Help main menu. Quick Help can also be accessed from the Pyris Help Contents.

Multimedia Presentations

This option provides access to the Help containing multimedia, i.e., audio and video. These help topics hardware related. They show how to install a hangdown wire in a TGA 7, how to prepare samples for the DSC, and how to install a measuring system for a DMA 7e, to name a few.

About

This command displays the About Pyris box which contains the Pyris Software for Windows copyright information and software version number. Click on the **OK** button to clear the box from the screen.

Restore Menu

The Restore menu is available when the Calibration window is displayed. The commands displayed will depend on the analyzer used. They are as follows:

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Temperature

This command restores the default temperature calibration value. Temperature calibration is initially performed by the Service Engineer by running high-purity standard and reference materials with known temperature transitions in the temperature ranges of interest. You should restore the default temperature calibration value before performing a temperature calibration.

Heat Flow

This command is available for DSC and DTA 7 analyzers. It restores the default energy calibration value. Energy or heat flow calibration is initially performed by the Service Engineer by running high-purity standard and reference materials with known transition energy values in the ranges of interest. You should restore the default heat flow calibration value before performing a heat flow calibration.

Weight

This command is available for TGA analyzers. It restores the default weight calibration value. Weight calibration is initially performed by the Service Engineer by using a 100-mg Class M calibration standard. You should restore the default weight calibration value before performing a weight calibration.

DMA Calibration

This command is available for the DMA 7e analyzer ONLY. It restores the default DMA calibration values. DMA calibration is initially performed by the Service Engineer upon installation and must be performed before any other calibration. You should restore the default DMA calibration values before performing a DMA calibration.

Height

This command is available for the DMA 7e and TMA 7 analyzers. It restores the default height calibration value. Height calibration is initially performed by the Service Engineer upon installation and calibrates the displacement transducer that is used to measure the position and amplitude axis. You should restore the default height calibration value before performing a height calibration.

Force

This command is available for the DMA 7e and TMA 7 analyzers. It restores the default force calibration value. Force calibration is initially performed by the Service Engineer upon installation and calibrates the force motor that is used to apply the static and dynamic forces to the sample. You should restore the default force calibration value before performing a force calibration.

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Eigendeformation

This command is available for the DMA 7e and TMA 7 analyzers. It restores the default eigendeformation calibration value. Eigendeformation calibration is initially performed by the Service Engineer upon installation and calibrates the very small movement of the analyzer when large forces are applied. Eigendeformation calibration is performed after height and force calibrations. You should restore the default eigendeformation calibration value before performing eigendeformation calibration.

All

Select this command to restore all default calibration values for the current analyzer.

Tools Menu

The Tools menu contains the following commands:

Preferences

Select this command to display the Preferences window for your particular analyzer. It contains 6 tabbed pages that you use to set conditions for your analyzer. In Data Analysis, the Preferences window contains 4 tabbed pages. See Chapter 9 for more detail on Preferences.

Validate Method

Select this command to validate the current method. You must have Administrator permissions to access this command. When you select this command, the method is marked as validated in the Method Editor and when it appears in the Method Used dialog box.

Convert ANF File

This command is available in Data Analysis only. Select this command to convert an ANF file to a data file that Pyris can use. ANF files are the result of converting data files from Perkin Elmer Series 7 UNIX instruments as well as from other analyzers by using the data file conversion software provided with Pyris Software for Windows. When you select this command, the Convert ANF Data Files dialog box is displayed. Select the file to be converted. Click on **Open** and the file is converted automatically. Pyris gives it the extension associated with the analyzer that collected the data and it is saved in the same directory as the ANF file. Retrieve the file using Add Data or Open Data in the File menu.

Convert PC Series File

This command is available in Data Analysis only. Select this command to convert a PC Series data file to a data file that Pyris can use. PC Series files are in ASCII format. When you select this command, the Convert PC Series Data Files dialog box is displayed. Select the file to be converted. [Available file types are DSC 7 Temp (.D7), DSC 7 Iso (.I7), DSC 7 SpHeat (.C7),

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TGA 7 (.G7), TMA 7 (.T7).] Click on **Open** and conversion is performed automatically. The converted file has the Pyris extension associated with the instrument that collected the data, e.g., dsd for a DSC 7 data file, and it is saved on the same directory as the PC Series file. Retrieve the file using Open Data or Add Data from the File menu.

Import X-Y Data

Select this command to import into Pyris a data file that is in ASCII format. The Import Data dialog box is displayed from which you select the .txt file you want to import. This command can be used to import a data file from another instrument whose file format is incompatible with Perkin-Elmer Thermal Analysis analyzers' data file formats. If you can save the data file in .txt format then you can import it into Pyris Software for Windows.

NOTE: (1) The text file must consist of two tab-delimited columns. (2) The first item in each column is the data description for that column (e.g., Heat Flow Endo Up, Specific Heat). This text will appear in the axis label. (3) The first column is the X-axis data and the second column is the Y-axis data.

Tables

This command is available for all analyzers in Data Analysis only. Select this command to display the Curve Tables dialog box which is used to create a table for the data of the active curve. The table contains the X and Y values of data points of the curve. You enter the initial X value and an increment value for X. The table can be written to a file in ASCII format or sent to the clipboard for pasting into another application.

Curve Tables Dialog Box

The Curve Tables dialog box appears when you select the **Tables** option in the Tools menu in Data Analysis. Upon selection, the data points for the active curve are automatically displayed in two columns with the X axis in the left column and the Y axis in the right column. The dialog box contains the following items with which you can adjust the table for your use:

Starting X

The default value for the initial X value in the table is the X value of the first data point in the data file. Use the spin buttons to increase or decrease the value by 1 or type in the desired value.

Increment

You can modify the number of data points in the table by choosing to use X values at a specified increment. Use the spin buttons to increase or decrease the Increment value or type it in. When you change the increment, the Number of Data Points field is adjusted automatically.

Number of Data Points

This is display only. The default value is the total number of data points in the data file. If you adjust the Starting X and the Increment, the Number of Data Points is adjusted accordingly. The value displayed here is the number of data points that will be saved to the clipboard or written to a file.

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Fill

Fills the table area with those data points selected by using the parameters in Starting X and Increment.

Write to File

Displays the Write Table to File dialog box. In the File name field enter the file name for the table. The default file name is OUTPUT.TXT and the default directory is the same directory as the data file. Click on **Save**. The file is in ASCII format.

Copy to Clipboard

Saves the ASCII table to the clipboard. You can then paste it into another application where it can be manipulated as needed.

Reset

Restores the table to its default listing that includes all data points. The Number of Data Points is adjusted to display the original number of data points. Starting X and Increment return to their original values.

Print

Displays the standards Windows from which you can print out the Curves table.

Remote Monitor

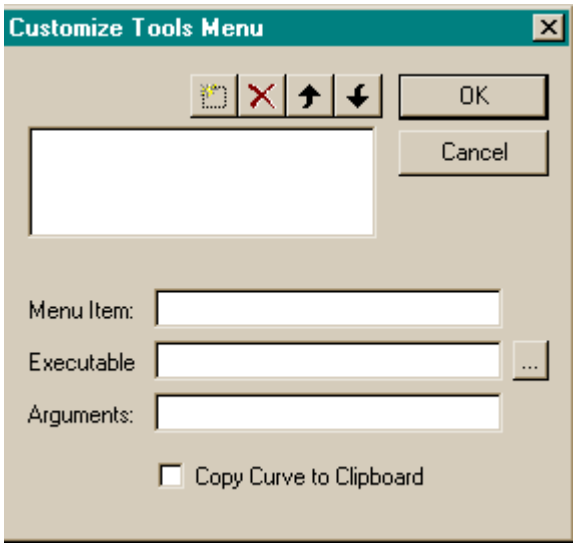
Select this command when in the Data Analysis application not associated with a particular analyzer to access the Remote Monitor feature on your system. You must have the Remote Monitor application already installed and the security button must be in place. Remote Monitor allows you to see real-time data from analyzers attached to remote PCs via the network.

Customize

Select this command if you want to add an application to the Tools menu, e.g., Microsoft Access or PowerPoint, to make access easier. When you select the custom item from the Tools menu, the executable file is run using the arguments that you specified in the Arguments field in the dialog box and application opens. For example, you can add Microsoft Word to your Tools menu and use a particular .doc file as an argument. When Word is selected from the Tools menu, it is opened with the .doc file displayed. If you indicate that you want the active curve at the time of selection placed on the clipboard, you can paste the data, in ASCII form, into the application.Upon selection, the Customize Tools Menu dialog box is displayed.

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Customize Tools Menu Dialog Box



Add Button

Adds a blank box to the menu item list box. Click on the button to display "Empty Item" in the Menu Item field and in the tools list box. Type in the name of the menu item you want to add.

Delete Button

Deletes the highlighted menu item in the menu item list box.

Move Item Buttons

The up and down arrow buttons move the highlighted item in the list box up or down, thus moving its position in the Tools menu up or down. Use these buttons to adjust your Tools menu display.

Menu Item

Enter the name for the custom item to be used for display in the Tools menu. This entry appears in the list box above.

Executable

Enter the executable file name for the item being added to the Tools menu. Use the Menu button (. . .) to display the Open dialog box from which you can find and select the .exe file.

Arguments

Enter any arguments needed in order for the executable file to be "hooked" to the Tools menu.

Copy Curve to Clipboard

Copies to the clipboard the curve active at the time you select the menu item from the Tools menu.

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Window Menu

The Window menu contains the following commands:

Cascade

Select this command to arrange all open windows so that they overlap.

Tile Horizontal

Select this command to arrange all open windows as nonoverlapping horizontal tiles.

Tile Vertical

Select this command to arrange all open windows as nonoverlapping vertical tiles.

Arrange Icons

This command arranges all icons along the bottom of the window.

Window 1, 2, 3, 4

The four most recently opened windows are listed at the bottom of the Window menu. Choose the number that corresponds to the window you want displayed. A checkmark appears next to the name of the active window.

Control Menu

The Control menu of a standard window is displayed by clicking on the upper-leftmost box. It contains the following Windows commands:

Restore

Select this command to return the active window to its size and position before you chose the **Maximize** or **Minimize** command.

Move

When you select this command, the cursor changes to a four-headed arrow:



You can move the window by either using the arrow keys to position the window and then clicking on the mouse to anchor it or by pressing the mouse button and dragging. This command is unavailable if you maximize the window. A shortcut for this command is to type **CTRL + F7.**

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Size

When you select this Windows Control menu command, the cursor changes to a four-headed arrow:



You can size the active window with the arrow keys. This command is unavailable if the window is maximized. After the pointer changes to the four-headed arrow:

1. Press one of the cursor arrow keys to move the pointer to the border you want to reposition.
2. Press a cursor arrow key to move the border.
3. Press **Enter** when the window is the size you want.

A shortcut to sizing a window is to use the mouse to drag the size bars at the corners or edges of the window.

Minimize

Select this command to reduce the application window to an icon. A shortcut is to click on the **Minimize** button on the title bar.

Maximize

Select this command to enlarge the active window to fill the maximum space it can occupy. A shortcut is to click on the **Maximize** button on the title bar or double click on the title bar.

Close

Select this command to close the active window or dialog box. Double-clicking on a Control menu box or on the Pyris Manager logo in the upper-left-hand corner of a window is the same as choosing the **Close** command. The Method Editor and Instrument Viewer windows do not have **Close** as a selectable option in the Control menu. You can close all windows at once with the **Close** command on the application's Control menu. Choosing Close from the Pyris Manager logo Control menu closes Pyris Software for Windows for that instrument. The Pyris Manager remains open.

Rescale Tools Toolbar

When a Data Analysis window or an Instrument Viewer is displayed, you can use the buttons on the Rescale Tools toolbar to rescale the display, shift the curve, or change the slope. For data analysis, these tools can be used to optimize the display of the data, compare data, and annotate curves. If the Rescale Tools toolbar is not already displayed, display it by selecting **Rescale Tools** from the View menu. Some of the items on the toolbar are also options in some of the menus.

Radar

This is the same as the Radar item on the View menu for Instrument Viewer and Data Analysis.

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Swap Y Axes 

Use this button to swap the position of the focused curve's Y-axis label. The focused curve is indicated by a thick solid line. The Swap Y Axes dialog box appears.

Swap Y Axes Dialog Box

At the top of the Swap Y Axes dialog box, the file name, sample ID, and the method step used to collect the data are given. Select the Y axis to use for the focused curve from the drop-down list which lists all available Y axes labels currently displayed in the Data Analysis window or the Instrument Viewer. They are listed as they are displayed from left to right and named Y1, Y2, etc. After making your selection, click on **OK** to perform the swap and close the dialog box.

Rescale X 

Use this function to rescale the X axis of the active curve in the Data Analysis window or the Instrument Viewer. In the Rescale X-Axis dialog box you change the minimum and maximum X values, units used for display, and the scale type.

Rescale Y 

Use this button to rescale the Y axis of the active curve in the Data Analysis window or the Instrument Viewer. In the Rescale Y dialog box you can change the minimum and maximum Y values and the scale type.

Full X Scale 

The **Full X Scale** button changes the X axis for the active curve to the full scale, i.e., the full contents of the data file with respect to the X axis are displayed. If you have rescaled the X axis or used the rubberband box in the Radar window to zoom in on a particular section of the curve, Full X Scale returns you to the full X-axis range of the data file. In Instrument Viewer, Full X Scale returns the default X scale.

Full Y Scale 

The **Full Y Scale** button changes the Y axis for the active curve to the full scale, i.e., the full contents of the data file with respect to the Y axis are displayed. If you have rescaled the Y axis or used the rubberband box in the Radar window to zoom in on a particular section of the curve, Full Y Scale rescales the Y axis of the active curve to display the full Y range of all curves using the active Y axis. In Instrument Viewer, the Y scale returns to the default scale.

Full Scale 

The **Full Scale** button displays the full contents of the data file, i.e., at the full X- and Y-axes scales of the data file. If you have changed the X or the Y scale and want to return to the full display of the curve, use this button. In Instrument Viewer, both axes return to the default scales.

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Previous Scale 

If you rescaled the curves in the Data Analysis window or the Instrument Viewer by creating a rubberband box with the click and drag method or by using the Rescale Tools toolbar or options in the View menu, select the **Previous Scale** button to display the curve using the display that was used immediately preceding the current display. Each time you rescale the X and Y axes, the system "remembers" those scale settings. (Changes between linear and log scales do not apply.)

Log X 

Clicking on this button toggles the X axis between linear and logarithmic scale. The Log X plot requires that the curve and the X-axis minimum values be greater than 0. When the X-axis scale is logarithmic, a checkmark appears next to the menu command. When a log scale is changed to linear, the new display uses the same minimum and maximum that were used for the log scale.

Log Y 

Clicking on this button toggles the Y axis between linear and logarithmic scale. The Log Y plot requires that the curve and the Y-axis minimum values be greater than 0. When the Y-axis scale is logarithmic, a checkmark appears next to the menu command. When a log scale is changed to linear, the new display uses the same minimum and maximum that were used for the log scale.

Shift Curve 

The **Shift Curve** button is used to shift or offset any heat flow, baseline heat flow, or unsubtracted heat flow curve vertically along the Y axis according the entries made in the Shift Curve dialog box. This is helpful if there are two or more curves displayed and they are overlapping at some point. Move the focused curve up and away from the other curve to compare the curves.

Shift Curve Dialog Box

When you click on the **Shift Curve** button, the Shift Curve dialog box appears. It contains the following fields:

Shift From

Displays the X- and Y-axes coordinates from which the active curve will shift. Only the X coordinate is changed. When selecting the Shift From coordinate directly on the curve by dragging the **X** to the desired position, both values in the Shift From fields are updated automatically.

Shift To

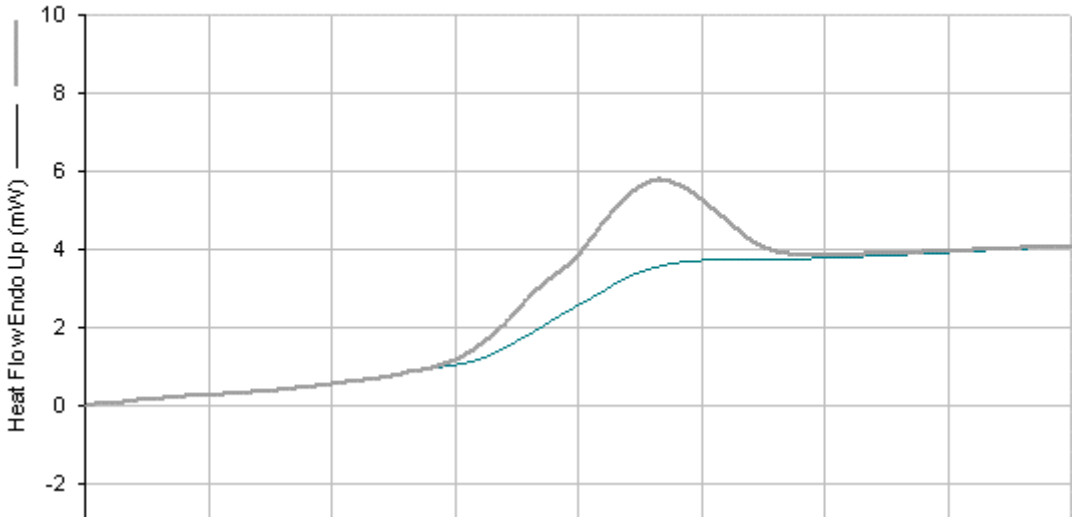
Enter the Y coordinate to which to shift the active curve. When selecting the Shift To coordinate directly on the curve, the value in the Shift To entry field is updated automatically.

Align All check box

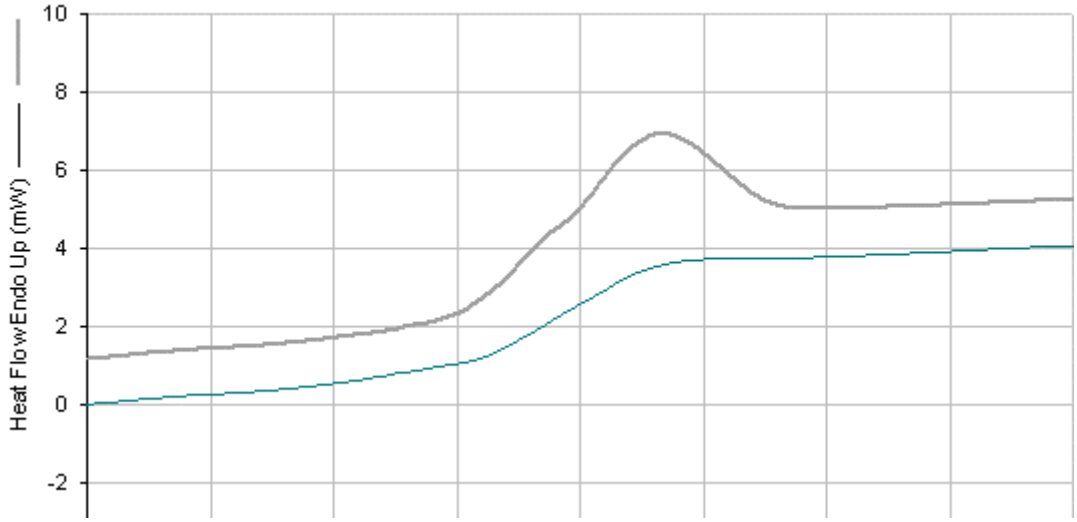
Selecting this box shifts all curves that use the same Y axis as the active curve to the Shift To coordinate.

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Curves before Shift Curve was used



Curves after Shift Curve was used



Change Slope 

You can change the slope of any heat flow, baseline heat flow, or unsubtracted heat flow curve in Data Analysis using the **Change Slope** button  on the Rescale Tools toolbar. Only the active curve is affected; the axes and scales do not change. If the button is grayed out, you cannot shift the active curve.

When you click on the **Change Slope** button, the Change Slope dialog box appears and the current slope is drawn with a thick red line.

Change Slope Dialog Box

The dialog box contains the following fields:

Select Pivot Point

The entry field displays the current X value of the pivot point for the slope calculations. The Y value is display only; it cannot be changed. The pivot point is indicated by a small box around it. Enter a new X value for the pivot point. You cannot move the box.

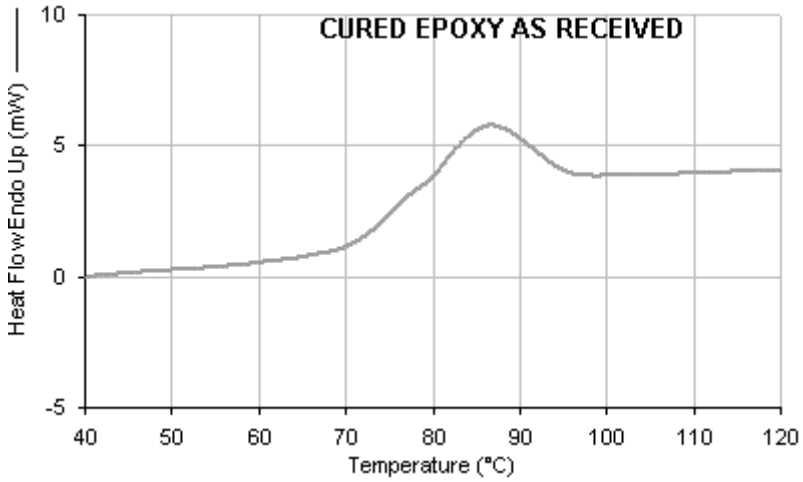
Select Slope Point

Enter the new Y value for the slope point; the X value is changed automatically. You can also select the slope point directly on the curve by moving the red **X**. The point on the curve at the same X-axis coordinate will be moved to the slope point, and the entire curve will be sloped accordingly.

Slope Setting

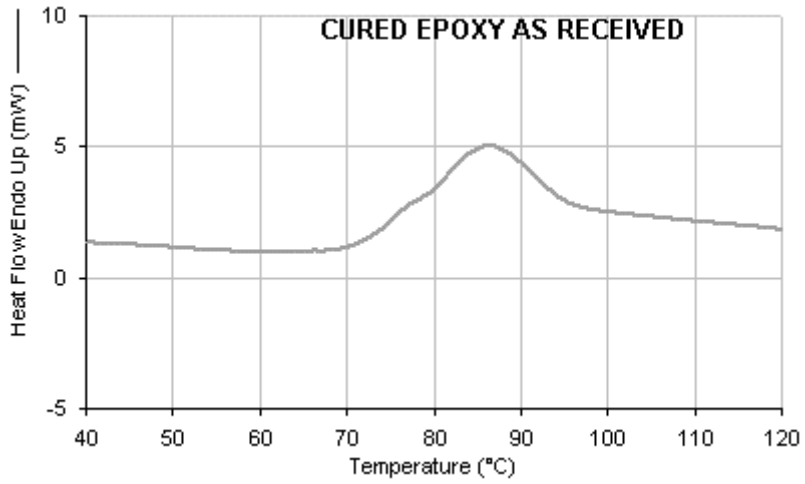
Align Endpoints: When selected, the slope point and the pivot point align on the same Y-axis point.

Curve before changing the slope:



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Curve after changing the slope:



Legend

This is the same as the option on the View menu in Instrument Viewer and Data Analysis.

Annotate

Use this button to add annotations to the Instrument Viewer or Data Analysis display. In the Annotations dialog box (see Annotate under Display Menu) you enter text for a label to be displayed with the active curve. The label can be "attached" to the curve, i.e., it is saved in the data file, or it can be used for display only. You can select the font and orientation of the text.

Temp/Time

The **Temp/Time** button is used to toggle the X axis between temperature and time. The curve is redisplayed automatically with the new X axis.

Standard Toolbars

The standard toolbar is displayed across the top of the Pyris window, beneath the title bar. The buttons available for selection depend on where you are in the software. If you position the cursor in the upper-right-hand corner of a button, a tool tip will be displayed explaining what the button's function is. Each toolbar was presented in Chapter 2 in "Navigating in Pyris Software for Windows."

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Instrument Viewer Button 

Select this button from the standard toolbar to display the Instrument Viewer window. This button is available from all windows.

Method Editor Button 

Select the Method Editor button to display the Method Editor window. This button is available from all windows.

Data Analysis Button 

The Data Analysis button displays the Data Analysis window. First the Open Data File dialog box is displayed from which you select a data file. This button is available from all windows.

Pyris Player Button 

Select the **Pyris Player** in the Method Editor, Instrument Viewer, and Data Analysis windows to access the Pyris Player Editor window. The Pyris Player window contains six pages that are used to create a new play list or edit an existing list, view the play list, view the history of the run of a play list, view a sample list (which is used in a sample group), and view the history of the running of a sample list, i.e., what occurred for each sample. Pyris Player has its own toolbar.

New Button 

In Method Editor, use this button to create a new method. When you select this button, the default method is loaded into the Method Editor. Edit the default method and select **Save As** from the File menu to save the new method under a new file name; the default method remains unchanged. This button is equivalent to the New Method command on the File menu.

In Data Analysis, this button is used to select a new data file for display. From the New Data File dialog box, select the file you want to display. Any data that was displayed in the Data Analysis window is cleared and replaced by the new data file. This button is equivalent to the New Data command in the File menu.

In Pyris Player, use this command to create a new play list. When you select this command, the default Pyris Player file is loaded; the Edit Play List page is blank. Edit the default Player file and select **Save As** to save it under a new file name and retain the default Player file. This button is equivalent to the New Player command on the File menu.

Open Button 

In Method Editor, use this button to open an existing method. When you select this button, the Open Method dialog box appears. This is equivalent to the Open Method command on the File menu.

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In Data Analysis, this button is used to open an existing data file in a **new** Data Analysis window. All other open Data Analysis windows remain open. From the Open Data File dialog box, select the new data file you wish to display. This is the same as Open Data on the File menu.

In Calibration, use this button to open an existing calibration file. When you select this button, the Open dialog box appears. The Open command on the File menu is the same as selecting this button.

In Pyris Player, use this button to open an existing play list file. When you select this button, the Open dialog box appears. The Open Player command on the File is the same as selecting this button.

Add Data Button 

Select this button to add an existing data file to the active Data Analysis window. From the Add Data File dialog box select the data file you want to add to the display. That data file becomes the active curve. This is the same as the Add Data option on the File menu.

Save Button 

In Method Editor, use this button to save the method currently loaded in the Method Editor. If the method is new and has not yet been saved, the Save As dialog box appears. This is equivalent to Save on the File menu.

In Data Analysis, select this button to save the active curve and all derived curves, results, constructs, and annotations using the current file name. The data file is saved automatically without requesting confirmation, so be sure you want to save the file as is. If you want to save the data under a different file name, select **Save Data As** from the File menu.

In Calibration, use this button to save the current calibration file. When you select this button, the file is saved automatically and will overwrite the existing file. If the calibration is new and has not yet been saved, the Save As dialog box appears. If you want to save the calibration file under a different file name, use **Save As** in the File menu.

In Pyris Player, use this button to save the current play list file. When you select this button, the file is saved automatically and will overwrite the existing file. If the play list is new and has not yet been saved, the Save As dialog box appears. If you want to save the play list file under a different file name, use the **Save As** command in the File menu.

Print Button 

In Method Editor, use this button to print the current method file. The standard Print dialog box appears when you select this button. This is the same as selecting **Print** from the File menu.

In Data Analysis, this button is used to print the data file. The standard Print dialog box appears when you select this button. This is the same as selecting **Print** from the File menu.

In Instrument Viewer, this button is used to print the displayed data file. The standard Print dialog box appears when you select this button. **Print** on the File menu is equivalent to selecting this button.

In Calibration, use this button to print the current calibration file. The standard Print dialog box appears when you select this button. Selecting **Print** in the File menu will also display the Print dialog box.

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In Pyris Player, selecting the **Print** button on the toolbar from any Pyris Player page initially displays a dialog box from which you choose the type of printout you want: summary or detailed. The summary printout includes the main-level items Prepare Sample, Data Analysis, and Sample Group which includes the Sample List and the Data Analysis List entries. The detailed printout includes the same information as a Summary printout but also includes additional information on the Sample List entries in a Sample Group.

When **Detail** is selected in the Print dialog box for View History, the same information as given in Summary is given in addition to the date and time each event occurred.

History Summary yields a printout that includes just one-line entries for each time the play list was played back. The entry gives the date and time of the run.

In all cases, click on the **Print** button in the dialog box to display the standard Print dialog box.

Print Preview Button 

In Method Editor, use this button to see what the current method file will look like when it is printed. The Print Preview window, which has its own toolbar, appears when you select this button.

In Data Analysis, select this button to see what the current data file will look like when it is printed. The Print Preview window, which has its own toolbar, appears when you select this command.

In Calibration, use this button to see what the current calibration file will look like when it is printed. The Print Preview window, which has its own toolbar, appears when you select this command.

In Pyris Player, use this button to see what the current play list, sample history, or history list will look like when it is printed. Selecting this button initially displays the Print Type dialog box from which you choose the type of preview you want: detailed or summary. Click on the **Print** button in the dialog box to display the Print Preview screen. Like the **Print** command, Summary will display just the main-level items in a play list. If a Sample Group is included, then the Sample List and Data Analysis List will also be displayed. For View History, Summary will display all the lines in the play list. Detail preview includes all lines in the play list and, for View History, preview includes date and time and error messages.

From the Print Preview screen, click on the **Print** button to display the standard Print dialog box. Click on the **Print** button in this box to print the display.

Delete Button 

This button is available in Instrument Viewer, Data Analysis, and Remote Monitor. Select this button to delete the selected object such as the active curve or an annotation from a Data Analysis or Instrument Viewer window automatically.

Copy Button 

The function of this button depends on where in the software it is invoked.

In Pyris Player, use this button to copy a Sample Group or a Sample line in a play list to the clipboard. If the focused line is Sample Group when you select **Copy**, the entire Sample Group block is copied, i.e., Sample List and Data Analysis List. You can then select **Paste** from the Edit

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menu or the Paste button from the toolbar to paste the Sample Group immediately after the focused Sample Group. If the focused line is a Sample line when you select **Copy**, then **Paste** will insert another Sample line immediately after the focused line.

If you are in Instrument Viewer, the **Copy** button will copy method information such as file name, program steps, and so on, to the clipboard which can then be pasted into another document such as Word.

In Data Analysis, **Copy** places the X,Y data of the focused curve onto the clipboard. It can then be used in Excel or another software product.

Paste Button 

In Pyris Player, use this button to paste the items on the Clipboard (placed there by use of the **Copy** command) into the play list. The item or items are inserted immediately after the copied line or lines. You can then edit the line or lines as needed, e.g., the locations of the samples in a Sample Group.

Method Used Button 

Select this button to display the parameters of the method that was used to collect the active curve's data. The parameters are presented in the View Method Properties dialog box, which comprises seven pages, each of which you can print out or copy to an ASCII file.

Monitor Button 

In Instrument Viewer, select this button to activate/deactivate the monitoring of the real-time status of an analyzer before the start of a run. This is same as the Monitor command in the View menu.

Grid Button 

In Instrument Viewer and Data Analysis, select this button to toggle the X – Y grid on and off. This button is the same as the Grid command on the View menu.

Print Preview Toolbar

The Print Preview toolbar comprises the following options:

Print

Displays the Print dialog box to initiate a print job.

Next Page

Previews the next page.

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Prev Page

Previews the previous page.

One Page / Two Page

Previews one or two pages at a time.

Zoom In

Takes a closer look at the page.

Zoom Out

Takes a larger look at the page.

Close

Returns from Print Preview to the editing window.

Pyris Player Toolbar

The toolbar used to run a play list, the Pyris Player toolbar, is discussed in Chapter 9, *Pyris Player*.

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Chapter 7
Calibration and Alignment

This chapter discusses the calibration routines for each instrument. A general discussion of calibration is presented, followed by a description of each type of calibration, e.g., Temperature Calibration, Furnace Calibration, etc. Some calibrations apply to more than one instrument. Following calibration, the alignment procedures for the AS 6 and Pyris 1 TGA autosamplers are presented.

Calibration

An analyzer is calibrated when it is first installed by a service engineer. An analyzer remains calibrated, even when the system is turned off, as long as there are no major changes to operating conditions. The **Calibration** option is available when you are in Method Editor or Instrument Viewer. When selected, a Calibration window opens up, displaying tabbed pages, one for each type of calibration available for that particular analyzer. Click on the desired tab and that calibration routine's page is displayed.

When the Calibration window is opened, the values of the calibration file in effect are displayed. Select **Open** from the File menu to open another existing calibration file. Click on **Save and Apply** to apply the calibration values from that file to the analyzer. Select **Close** to exit calibration and continue with your work.

You can create a calibration file and save it without applying its values to the analyzer by selecting **Save** or **Save As** from the File menu.

If you select **Close** from a calibration window and the current values have not been saved, you will be prompted to save them at this time. If you do not save the calibration values, they will not be applied to the instrument.

You can restore the default calibration values at any time by using the commands in the Restore menu. The Restore menu appears when the Calibration window is displayed. The options in the menu are instrument-specific.

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Calibration Reference Material

To perform a Temperature calibration of any analyzer or the Heat Flow calibration of a DSC analyzer, you should use a standard calibration reference material. If your lab must comply with ISO 9000, Perkin Elmer provides ISO 9000 compatible reference materials. Reference materials comply with ISO instrumentation calibration. Certified reference materials are traceable to national or international standards through an unbroken chain of custody. Not every calibration material is a CRM.

For calibrating a DSC 7, Pyris 6 DSC, and a Pyris 1 DSC, the reference materials usually used are indium and zinc, which are supplied with the instrument for calibration purposes. Other reference materials are also available from Perkin Elmer:

Reference Material	Part No.	Transition Temp. (°C)	Transition Energy
Indium	0319-0033	156.60	28.45
Tin	0319-0034	231.88	60.46
Lead	0319-0035	327.47	23.01
Zinc	0319-0036	419.47	108.37
Potassium sulfate	not applicable	585.0 + 0.5	33.26
Potassium chromate	not applicable	670.5 + 0.5	35.56

Recommended reference materials for calibrating a DSC when operating in the subambient temperature range are listed below:

Reference Material	Transition	Transition Temp. (°C)	Transition Energy
Cyclopentane	Crystal	–151.16	69.45
Cyclopentane	Crystal	–135.06	4.94
Cyclohexane	Crystal	–87.06	79.58
Cyclohexane	Melt	6.54	31.25
Water	Melt	0.00	333.88
n-Hexane	Melt	–90.56	140.16
n-Octane	Melt	–56.76	182.0
n-Decane	Melt	–29.66	202.09
N-Dodecane	Melt	–9.65	216.73
n-Octadecane	Melt	28.24	241.42
Hexatriacontane	Crystal	72.14	18.74
Hexatriacontane	Melt	75.94	175.31
P-Nitrotoluene	Melt	51.64	

To calibrate a TGA, magnetic calibration reference materials are used to perform the Curie point temperature calibration on the standard furnace:

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Reference Material	Part No.	Magnetic Transition Temp. (°C)
kit of 5 reference materials: alumel, nickel, Perkalloy, iron, Hisat-50	0219-0071	
one 2-in. piece of 99.99% alumel wire	0998-8015	154.2
three 2-in. pieces of 99.99% nickel wire	N519-0869	354
three 2-in. pieces of Perkalloy wire	N519-0616	596
three 2-in. pieces of 99.99% iron wire	0998-8017	780

Pyris 1 DSC Calibration

There are three calibration routines for a Pyris 1 DSC:

- Temperature
- Heat Flow
- Furnace

Once your Pyris 1 DSC is calibrated, it should remain so for a long time, provided there are no major changes in operating conditions. Conditions that could affect the current calibration are

1. If the operating range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is still valid.
2. If you change the purge gas type or flow rate, verify that the temperature is still calibrated.
3. If you change the coolant or coolant accessory, you may need to recalibrate.
4. If the analyzer has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. If so, condition the analyzer by performing several heating and cooling runs with an empty sample holder, then check the calibration by running standard materials.

Before calibrating the Pyris 1 DSC, it is sometimes necessary to restore default calibration values. Typically, it is better to restore defaults when you are changing the temperature range that you are using. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, restoring defaults may be necessary. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

For Temperature and Heat Flow calibrations, run high-purity reference materials (typically indium and zinc) with known temperature and energy transitions. The data obtained from these runs are used in the Pyris software calibration routines to calibrate the Pyris 1 DSC. Once the analyzer is calibrated, it will remain calibrated even when the system is turned off, as long as there are no changes in the operating conditions.

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Temperature Calibration

Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so before completing the reference material sample runs.

To perform the Pyris 1 DSC Temperature calibration, complete a scan for each reference material under the same conditions that you run your samples. After each run is completed, perform a Peak Area calculation and include the Onset temperature. Record the ΔH (J/g) and Onset results; you will need the Onset result for Temperature calibration and you can use the ΔH result for Heat Flow calibration.

When all of the reference materials have been run and calculations performed, enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the table on the Temperature calibration page. Click on the check box in the "Use" column for each reference that is to be used in the calibration.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

If you are performing a Furnace calibration (for DDSC mode), it is essential to **Save and Apply** the new Temperature calibration first.

Heat Flow Calibration

Select the Heat Flow tab to display the Heat Flow page of the Calibration window. A Heat Flow calibration uses a single standard. The Heat Flow calibration value can be slightly modified by running a new reference sample while the existing calibration is applied.

To perform the Pyris 1 DSC Heat Flow calibration, complete a scan for the reference material under the same conditions that you run your samples or use one of the scans completed for the Temperature calibration. After the run is completed, perform a Peak Area calculation and record the ΔH (J/g) result.

Enter the reference material name, expected ΔH, the measured ΔH, weight of the reference material, and calibration method name in the Calibration table.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

Furnace Calibration

The Pyris 1 DSC Furnace calibration routine is used when your analyzer has the DDSC accessory installed. The calibration linearizes the Pyris 1 DSC furnace by matching the program temperature to the sensor temperature over the range that you enter. If you have just completed a Temperature calibration procedure, you must **Save and Apply** the new Temperature calibration before starting the Furnace calibration.

Select the Furnace tab to display the Furnace page of the Calibration window. Enter the minimum and maximum temperature limits, then select the **Begin Calibration** button and follow the instructions in the dialog box. To accept the Furnace calibration values, click on **Save and Apply**.

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DSC 7 Calibration

There are three calibration routines for a DSC 7:

- Temperature
- Heat Flow
- Furnace (DDSC only)

Once your DSC 7 is calibrated, it should remain so for a long time, provided there are no major changes in operating conditions. Conditions that could affect the current calibration and require you to recalibrate the DSC 7 are

1. If the operating range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is still valid.
2. If you adjust the Balance control on the analyzer, you may need to recalibrate. Always optimize the DSC 7 baseline before calibrating the analyzer.
3. If you change the purge gas type or flow rate, verify that the temperature is still calibrated.
4. If you change the coolant or coolant accessory, you may need to recalibrate.
5. If the analyzer has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. In this case, condition the DSC 7 by performing several heating and cooling runs with an empty sample holder, then check the calibration by running standard materials.

Before calibrating the DSC 7, it is sometimes necessary to restore default calibration values. Typically, it is better to restore defaults when you are changing the temperature range that you are using. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, restoring defaults may be necessary. The Temperature calibration can be slightly modified by running a new reference sample while the existing calibration is applied. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

For Temperature and Heat Flow calibrations, run high-purity reference materials (typically indium and zinc) with known temperature and energy transitions. The data obtained from these runs are used in the Pyris software calibration routines to calibrate the DSC 7. Once the analyzer is calibrated, it will remain calibrated even when the system is turned off, as long as there are no changes in the operating conditions.

Temperature Calibration

Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so **before** completing the reference material sample runs.

To perform the DSC 7 Temperature calibration, complete a scan for each reference material under the same conditions that you run your samples. After each run is completed, perform a Peak Area calculation and include the Onset temperature. Record the ΔH (J/g) and Onset results; you will need the Onset result for Temperature calibration and you can use the ΔH result for Heat Flow calibration.

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When all of the reference materials have been run and calculations performed, enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the table on the Temperature page. Click on the check box in the "Use" column for each reference that is to be used in the calibration.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

If you are performing a Furnace calibration next (if your DSC 7 has the DDSC installed), it is essential to **Save and Apply** the new Temperature calibration first.

Heat Flow Calibration

Select the Heat Flow tab to display the Heat Flow page of the Calibration window. A Heat Flow calibration uses a single standard. The Heat Flow calibration value can be slightly modified by running a new reference sample while the existing calibration is applied.

To perform the DSC 7 Heat Flow calibration, complete a scan for the reference material under the same conditions that you run your samples or use one of the scans completed for the Temperature calibration. After the run is completed, perform a Peak Area calculation and record the ΔH (J/g) result.

Enter the reference material name, expected ΔH, measured ΔH, weight of the reference material, and calibration method name in the Calibration table.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

Furnace Calibration

The Furnace Calibration is available for a DSC 7 if the DDSC accessory is installed. This calibration linearizes the DSC 7 furnace by matching the program temperature to the sensor temperature over the range that you enter. If you have just completed a Temperature calibration procedure, you must **Save and Apply** the new Temperature calibration before starting the Furnace calibration.

Select the Furnace tab to display the Furnace page of the Calibration window. Enter the minimum and maximum temperature limits, then select the **Begin Calibration** button and follow the instructions in the dialog box. To accept the Furnace calibration values, click on **Save and Apply**.

Pyris 6 DSC Calibration

There are two calibration routines for a Pyris 6 DSC:

- Temperature
- Heat Flow

It is recommended that a baseline correction be performed before temperature calibration.

The Pyris 6 DSC analyzer has been calibrated at the factory for both temperature and heat flow. Under normal conditions, the Pyris 6 DSC does not need temperature recalibration. Temperature calibration and heat flow calibration should be checked using the precrimped samples of indium

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and zinc before obtaining data. Some conditions that could affect the current calibration of the Pyris 6 DSC:

1. If the operating range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is valid.
2. If you change the purge gas type or flow type, verify that the temperature is still calibrated.
3. If you change the coolant, you may need to recalibrate.
4. If the Pyris 6 DSC has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. If so, condition the analyzer by performing several heating and cooling runs with an empty sample holder, then check the calibration by running standard materials.

Before calibrating the Pyris 6 DSC, it is sometimes necessary to restore default calibration values. Typically, it is better to restore defaults when you are changing the temperature range that you are using. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, restoring defaults may be necessary. The Temperature calibration can be slightly modified by running a new reference sample while the existing calibration is applied. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

For Temperature and Heat Flow calibrations, run high-purity reference materials (typically indium and zinc) with known temperature and energy transitions. The data obtained from these runs are used in the Pyris software calibration routines to calibrate the Pyris 6 DSC. Once the analyzer is calibrated, it will remain calibrated even when the system is turned off, as long as there are no changes in the operating conditions.

Temperature Calibration

Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so before completing the reference material sample runs.

To perform the Pyris 6 DSC temperature calibration, complete a scan for each reference material under the same conditions that you use to run your samples. After each run is completed, perform a Peak Area calculation and include the Onset temperature. Record the ΔH (J/g) and Onset results; you will need the Onset result for Temperature calibration and you can use the ΔH result for Heat Flow calibration.

When all of the reference materials have been run and calculations performed, enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name used in the table on the Temperature page. Click on the check box in the “Use” column for each reference that is to be used in the calibration.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

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Heat Flow Calibration

Select the Heat Flow tab to display the Heat Flow page of the Calibration window. A Heat Flow calibration uses a single standard. The Heat Flow calibration value can be slightly modified by running a new reference sample while the existing calibration is applied.

To perform the Pyris 6 DSC Heat Flow calibration, complete a scan for the reference material under the same conditions that you run your samples or use one of the scans completed for the Temperature calibration. After the run is completed, perform a Peak Area calculation and record the ΔH (J/g) result.

Enter the reference material name, the expected ΔH, the measured ΔH, the weight of the reference material, and the calibration method name in the Calibration table.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Select **Close** to close the Calibration window and begin using the new calibration values.

Baseline Correction of the Pyris 6 DSC

Generally, it is not necessary to perform a baseline correction on the analyzer but if the baselines have too much slope (>3 mW at a scan rate of 20°C/min with no sample pans in the furnace), you should correct the baseline. Baseline correction eliminates a static offset and baseline drift during a temperature scan. The correction is a linear correction between two points.

To correct the baseline you must determine the temperature range over which it is to be optimized. Then you must select the coolant or cooling accessory to use and set the purge gas rate. Use the **baseline.d6m** method to obtain routine baselines. When the run is finished, save the data file. This file is used to correct the baseline of subsequent runs.

DDSC Calibration

The Dynamic Differential Scanning Calorimetry (DDSC) accessory for the DSC 7 and the Pyris 1 DSC allows you to operate the analyzer as either a standard DSC 7 or Pyris 1 DSC or in DDSC mode. Upon installation of the DDSC, the analyzer is calibrated for Tp, the program temperature, and Ts, the sensor temperature.

NOTE: The DSC 7 or the Pyris 1 DSC with the DDSC accessory is calibrated only from the standard mode. Once you calibrate the sensor temperature, the DDSC mode will be calibrated automatically.

The calibration routines for an analyzer with a DDSC accessory are

- Temperature Calibration
- Heat Flow Calibration
- Furnace Calibration

Once the calibration routines are performed, the temperature and heat flow calibrations should remain unchanged for a long time, provided there are no changes in the operating conditions of the instrument. Conditions that could affect the current calibration of the DSC 7 or Pyris 1 DSC with the DDSC accessory and require recalibration are

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1. If the operating temperature range of your experiments changes, recalibration may be necessary. Run a reference material in the new range of interest to determine if the current calibration is valid.
2. If the Balance control is adjusted, recalibration may be necessary. Therefore, always optimize the baseline before the instrument is calibrated.
3. If the purge gas type or purge gas flow rate is changed, the calibration should be checked for highest accuracy. Switching between helium and another gas will require recalibration as well.
4. If you change the coolant or cooling accessory, recalibration may be necessary.
5. Since the temperature calibration is slightly dependent on scan rate for DDSC analyzers, final calibration should be performed at the scan rate that you will use for your experiments. As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating rates, or heating and cooling rates, it is better to calibrate at the slowest rate to be used. When operating in DDSC mode, it is best to calibrate at a slow rate of $\leq$ 5°C/min.
6. If the instrument has been turned off for a long period of time (i.e., weeks or months), the instrument may appear to require recalibration. Condition the instrument by performing several heating and cooling runs with the sample holder empty and then check the calibration by running reference materials.

TGA 7 Calibration

There are two calibration routines for a standard furnace and a high temperature furnace TGA 7:

- Weight
- Furnace

For a standard furnace TGA 7, there is an additional calibration routine:

- Temperature

Once your TGA 7 is calibrated, it should remain so for a long time, provided there are no major changes in operating conditions. The conditions that could affect the current calibration are

1. If the operating range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is still valid.
2. If you install a new furnace, verify that the temperature is still calibrated.
3. If you change the purge gas type or flow rate, verify that the temperature is still calibrated.
4. If you install a new thermocouple or change the position of the thermocouple, verify that the temperature is still calibrated.
5. If you change the hangdown wire or sample pan, verify that the weight is still calibrated.

If the analyzer has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. In this case, condition the TGA 7 by performing several heating and cooling runs with an empty sample holder, then check the calibration by running standard materials.

Before calibrating the TGA 7, it is sometimes necessary to restore default calibration values. Typically, it is better to restore defaults when changing the temperature range that you are using. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, you may need to restore defaults. The Temperature calibration can be slightly modified by running

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a new reference sample while the existing calibration is applied. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

Temperature Calibration

For the TGA 7 Temperature calibration, run Curie point calibrations for high-purity reference materials. (For the high temperature furnace TGA 7 Temperature calibration, refer to ASTM Method E1582 for another procedure.) The data obtained from these runs are used in the Pyris software calibration routines to calibrate the TGA 7 temperature. Once the analyzer is calibrated, it will remain calibrated even when the system is turned off, as long as there are no changes in the operating conditions.

Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so before completing the reference material sample runs.

To perform the TGA 7 Temperature calibration, complete a scan for each reference material under the same conditions that you run your samples. After each run is completed, perform an Onset calculation at the end of the Curie point transition. Record the Onset temperature.

When all of the reference materials have been run and calculations performed, enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the table on the Temperature Calibration page. Click on the check box in the “Use” column for each reference that is to be used in the calibration.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

If you are performing a Furnace calibration next, it is essential to **Save and Apply** the new Temperature calibration first.

Weight Calibration

In a Weight calibration, you compare the known weight of a reference material with the actual weight read from the TGA 7. The Weight calibration value can be slightly modified by recalibrating while the existing calibration is applied.

Enter the known weight of the reference material in the Weight page of the Calibration window, then select the **Begin Calibration** button and follow the instructions in the dialog boxes. You can cancel the calibration at any time by selecting the **Cancel** button in any dialog box.

Furnace Calibration

The TGA 7 Furnace calibration is a nine-point calibration between two specified temperature limits. It linearizes the TGA 7 furnace by matching the program temperature to the thermocouple temperature over the range that you enter.

If you have just completed a Temperature calibration procedure, you must **Save and Apply** the new Temperature calibration before starting the Furnace calibration.

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Select the Furnace tab to display the Furnace page of the Calibration window. Enter the Minimum and Maximum temperature limits, then select the **Begin Calibration** button and follow the instructions in the dialog box. You can minimize the Instrument Application window while the TGA 7 furnace is being calibrated. To accept the Furnace calibration values, click on **Save and Apply**.

Pyris 6 TGA Calibration

There are three calibration routines for a Pyris 6 TGA:

- Furnace
- Temperature
- Weight

The Pyris 6 TGA analyzer has been calibrated by the service engineer that installed it. Under normal conditions, the Pyris 6 TGA does not need temperature recalibration. If it is necessary to calibrate the Pyris 6 TGA, Furnace calibration **must** be performed before Temperature calibration. Temperature calibration should be checked using the two or three of the four reference materials provided and the weight calibration should be checked using the reference weight provided before performing any runs.

Once your Pyris 6 TGA is calibrated, it should remain so for a long time, provided there are no major changes in operating conditions. The conditions that could affect the current calibration are

1. If the operating temperature range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is still valid.
2. If you change the purge gas type or flow rate, verify that the temperature is still calibrated.
3. If you install a new sample thermocouple, or if the existing one has been disturbed, verify that the temperature is still calibrated.
4. If you change the sample pan, verify that the weight is still calibrated.
5. If the analyzer has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. In this case, condition the Pyris 6 TGA by performing several heating and cooling runs with an empty sample holder, then check the calibration by running standard materials.

Before calibrating the Pyris 6 TGA, it is sometimes necessary to restore default calibration values. Typically, it is better to restore defaults when changing the temperature range used. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, you may need to restore defaults. The Temperature calibration can be slightly modified by running a new reference sample while the existing calibration is applied. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

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Furnace Calibration

The Pyris 6 TGA Furnace calibration is a nine-point calibration between two specified temperature limits. It linearizes the Pyris 6 TGA furnace by matching the program temperature to the thermocouple temperature over the range that you enter.

Unlike other analyzers, do **not** do a Temperature calibration before the Furnace calibration. The Furnace calibration is to be done first.

Select the Furnace tab to display the Furnace page of the Calibration window. Enter the Minimum and Maximum temperature limits, then select the **Begin Calibration** button and follow the instructions in the dialog box. You can minimize the Instrument Application window while the Pyris 6 TGA furnace is being calibrated. To accept the Furnace calibration values, click on **Save and Apply**.

Temperature Calibration

For the Pyris 6 TGA Temperature calibration, run Curie point calibrations for the high-purity reference materials provided with the instrument. The data obtained from these runs are used in the Pyris software calibration routines to calibrate the Pyris 6 TGA temperature. Once the analyzer is calibrated, it will remain calibrated even when the system is turned off, as long as there are no changes in the operating conditions.

Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so before completing the reference material sample runs.

To perform the Pyris 6 TGA Temperature calibration, you complete two scans for each reference material under the same conditions that you run your samples. The first run should use the lower scanning rate (e.g., 5°C/min) and the second run the higher scanning rate (e.g., 50°C/min). You must run at least two reference materials. After each run is completed, perform an Onset calculation at the end of the Curie point transition. Record the Onset temperature.

When all of the reference materials have been run and calculations performed, in the Temperature calibration page enter the reference material name, expected onset temperature, measured onset temperature, and the scanning rates used.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them.

Weight Calibration

In a Weight calibration, you compare the known weight of a reference material with the actual weight read from the Pyris 6 TGA. The Weight calibration value can be slightly modified by recalibrating while the existing calibration is applied.

Enter the known weight of the reference material in the Weight page of the Calibration window, then select the **Begin Calibration** button and follow the instructions in the dialog boxes. You can cancel the calibration at any time by selecting the **Cancel** button in any dialog box.

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Pyris 1 TGA Calibration

There are three calibration routines for the Pyris 1 TGA:

- Temperature
- Weight
- Furnace

The high temperature furnace Pyris 1 TGA Calibration window contains two calibration routine pages:

- Weight
- Furnace

Once your Pyris 1 TGA is calibrated, it should remain so for a long time, provided there are no major changes in operating conditions. The conditions that could affect the current calibration are

1. If the operating range of your experiments changes, you may need to recalibrate the temperature. Run a standard in the new range of interest to determine if the current calibration is still valid.
2. If you install a new furnace, verify that the temperature is calibrated.
3. If you change the purge gas type or flow rate, verify that the temperature is still calibrated.
4. If you install a new thermocouple or change the position of the thermocouple, verify that the temperature is still calibrated.
5. If you change the hangdown wire or sample pan, verify that the weight is still calibrated.
6. If the analyzer has been turned off for a long time (i.e., weeks or months), it may appear to require recalibration. In this case, condition the Pyris 1 TGA by performing several heating and cooling runs with an empty sample pan, then check the calibration by running standard materials.

Before calibrating the Pyris 1 TGA, it is sometimes necessary to restore the default calibration values. Typically, it is better to restore defaults when changing the temperature range that you are using. If you are changing the purge gas or flow rate, or if the analyzer has not been used for some time, you may need to restore defaults. The Temperature calibration can be slightly modified by running a new reference sample while the existing calibration is applied. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button, click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

Temperature Calibration

For the Pyris 1 TGA Temperature calibration, run Curie point calibrations for high-purity reference materials. (For the high-temperature furnace Pyris 1 TGA Temperature calibration, refer to ASTM Method E1582 for another procedure.) The data obtained from these runs are used in the Pyris software calibration routines to calibrate the Pyris 1 TGA temperature.

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Select the Temperature tab to display the Temperature page of the Calibration window. If you are restoring default calibration values, do so before completing the reference material sample runs.

To perform the Pyris 1 TGA Temperature calibration, complete a scan for each reference material under the same conditions that you will run your samples. After each run is completed, perform an Onset calculation at the end of the Curie point transition. Record the Onset temperature.

When all of the reference materials have been run and calculations performed, enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the table on the Temperature page. Click on the check box in the "Use" column for each reference that is to be used in the calibration.

When all of the information has been entered, click on the **Save and Apply** button to save the calibration values and apply them. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

If you are performing a Furnace calibration next, it is essential to **Save and Apply** the new temperature calibration first.

Weight Calibration

In a Weight calibration, you compare the known weight of a reference material with the actual weight read from the Pyris 1 TGA. The Weight calibration value can be slightly modified by recalibrating while the existing calibration is applied. Enter the known weight of the reference material in the Weight page of the Calibration window, then select the **Begin Calibration** button and follow the instructions in the dialog boxes. The instructions are slightly different for a Pyris 1 TGA with an autosampler.

Furnace Calibration

The Pyris 1 TGA Furnace calibration is a nine-point calibration between two specified temperature limits. It linearizes the Pyris 1 TGA furnace by matching the program temperature to the thermocouple temperature over the range that you enter.

NOTE: If you have just completed a Temperature calibration procedure, you must Save and Apply the new Temperature calibration before starting the Furnace calibration.

Select the Furnace tab to display the Furnace page of the Calibration window. Enter the Minimum and Maximum temperature limits, then select the **Begin Calibration** button and follow the instructions in the dialog box. You can minimize the Instrument Application window while the Pyris 1 TGA furnace is being calibrated so you can use other applications on the computer. To accept the Furnace calibration values, click on **Save and Apply**.

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DMA 7e Calibration

There are six calibration routines for a DMA 7e:

- DMA Calibration
- Height
- Force
- Eigendeformation
- Temperature
- Furnace

All calibrations are performed by a Service Engineer upon installation of your DMA 7e so it is not necessary for you to perform a complete calibration when you begin to use your analyzer. Periodic checking of the calibration is all that is needed to verify accurate calibration. Once the DMA 7e is calibrated, it should remain so for a long time, provided there are no changes in the instrument's operating conditions. The analyzer remains calibrated even when it is turned off. Conditions that could affect the current calibration are

1. If the operating temperature range of your experiments changes, you may need to recalibrate the temperature axis. Check the temperature calibration in the range of interest (to determine if the current calibration is still valid) by measuring the melting point of a reference material.
2. If the purge gas or purge gas flow rate is changed, the temperature calibration should be checked.
3. If a new furnace is installed, the temperature calibration should be checked.
4. If a new thermocouple is installed or if the position of the thermocouple is changed, the temperature calibration should be checked.
5. The eigendeformation calibration should be checked and performed if the sample tube and/or probe is changed and the samples being analyzed are very stiff (that is, a high modulus of >10 GPa).
6. If a different measuring system is installed, the temperature and other calibrations should be checked.

Before calibrating the DMA 7e, it is sometimes necessary to restore default calibration values. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting **All**. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

DMA Calibration

DMA Calibration performs necessary operations in the firmware and generates lookup tables stored in memory. Run the DMA Calibration with a 3-point bending measuring system installed.

Select the DMA tab in the Calibration window to display the DMA page. Click on **Begin Calibration**, remove any samples, lower the probe, raise the furnace assembly, and click on **OK** in the dialog box and calibration begins. Progress of the calibration is indicated by the elapsed

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time displayed. When complete, click on **OK.** To perform the next calibration, Height, you must select **Save and Apply** to save the calibration values and apply them.

Height Calibration

This procedure calibrates the displacement transducer that is used to measure the position and amplitude (ordinate) axis of the DMA 7e. To perform a Height calibration, you need the sapphire height displacement standard shipped with the instrument. Measure the height of the standard.

Select the Height tab in the Calibration window to display the Height page. Click on **Begin Calibration**. Prepare the analyzer for zero reading by removing any samples on the sample platform. Press the Probe Up button on the analyzer and check for any samples on the sample platform. Press Probe Down to lower the probe until it rests on the sample platform. Click on **OK**. After the Y signal has stabilized, select **OK** if you want the current Y value entered as the new zero value. Calibration sets the location of the empty sample platform as 0 mm.

You then place the displacement standard on the sample platform and lower the probe to rest on the standard. Wait for the Y signal to stabilize and then select **OK** to save the height calibration value. To accept the new height factor, **Save and Apply**.

Force Calibration

Select this tab to calibrate the force motor that is used to apply the static and dynamic forces to the sample. To perform the Force calibration, you need to 50-g-force calibration reference material and weight platform shipped with the instrument.

Select the Force tab in the Calibration window to display the Force page. Click on **Begin Calibration**, remove any sample from the platform, install the weight tray, and place the 50-g weight on the tray. When calibration is complete, remove the weight and the weight tray and the new force value is saved automatically.

Eigendeformation Calibration

This procedure calibrates the very small movement of the analyzer itself when large forces are applied. After performing the calibration, the compliance of the system is subtracted from the probe position signal. Increasing force with no sample present will yield zero displacement. This calibration should be performed after Height and Force calibration.

Select the Eigendeformation tab in the Calibration window to display the Eigendeformation page. Click on **Begin Calibration,** remove any sample from the platform, lower the probe, and wait for the calibration. You will then insert the steel reference material, lower the probe, and wait for the calibration to complete. Click on **Save and Apply** to accept the eigendeformation calibration values.

Temperature Calibration

This one-point temperature calibration allows you to run one reference material to match the thermocouple temperature and the sample temperature. In this calibration, the onset melting temperature for indium is determined using the 3-point bending measuring system.

Select the Temperature tab in the Calibration window to display the Temperature page. To perform the DMA 7e Temperature calibration, complete a scan for indium reference material

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under the same conditions that you run your samples. After the run is completed, perform an Onset calculation and record the Onset temperature. Enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the Calibration table. Click on the check box in the Use column. Select **Save and Apply** to send the new calibration value to the analyzer and save the calibration file.

Furnace Calibration

The DMA 7e Furnace calibration is a nine-point calibration between two specified temperature limits. It linearizes the DMA 7e furnace by matching the program temperature to the thermocouple temperature over the range that you enter.

Perform a Furnace calibration **after** a Temperature calibration. Select the Furnace tab in the Calibration window to display the Furnace page. Enter the Minimum and Maximum temperature limits, then select **Begin Calibration** and follow the instructions in the dialog box. To accept the Furnace calibration values, click on **Save and Apply**.

TMA 7 Calibration

There are five calibration routines for the TMA 7:

- Height
- Force
- Eigendeformation
- Temperature
- Furnace

All calibrations are performed by a Service Engineer upon installation of your TMA 7 so it is not necessary for you to perform a complete calibration when you begin to use your analyzer. Periodic checking of the calibration is all that is needed to verify accurate calibration. Once the TMA 7 is calibrated, it should remain so for a long time, provided there are no changes in the instrument's operating conditions. The analyzer remains calibrated even when it is turned off. Conditions that could affect the current calibration are

1. Operating temperature range of your experiments changes. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
2. Purge gas or purge gas flow rate is changed.
3. New furnace is installed.
4. New thermocouple is installed or the position of the thermocouple is changed.
5. The Eigendeformation calibration should be checked and performed if the sample tube and/or probe is changed and the samples being analyzed are very stiff.

Before calibrating the TMA 7, it is sometimes necessary to restore default calibration values. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting All. This activates the **Save and Apply** button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

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Height Calibration

This procedure calibrates the displacement transducer that is used to measure the position and amplitude (ordinate) axis of the TMA 7. To perform a Height calibration, you need the sapphire height displacement standard shipped with the instrument. Measure the height of the standard.

Select the Height tab in the Calibration window to display the Height page. Click on **Begin Calibration**, prepare the analyzer for zero reading by removing any samples on the sample platform. Click on **OK**. Lower the probe until it rests on the sample platform. After the Y signal has stabilized, select **Apply** if you want the current Y value entered as the new zero value. Calibration sets the location of the empty sample platform as 0 mm.

You then place the displacement standard on the sample platform and lower the probe to rest on the standard. Wait for the Y signal to stabilize and then select **Save and Apply** to save the height calibration values and apply them.

Force Calibration

Select this tab to calibrate the force motor that is used to apply the static force to the sample. To perform the Force calibration, you need to 50-g-force calibration reference material and weight platform shipped with the instrument.

Select the Force tab in the Calibration window to display the Force page. Click on **Begin Calibration**, remove any sample from the platform, install the weight tray, and place the 50-g weight on the tray. When calibration is complete, remove the weight and the weight tray and the new force value is saved automatically.

Eigendeformation Calibration

This procedure calibrates the very small movement of the analyzer itself when large forces are applied. After performing the calibration, the compliance of the system is subtracted from the probe position signal. Increasing force with no sample present will yield zero displacement. This calibration should be performed after Height and Force calibration.

Select the Eigendeformation tab in the Calibration window to display the Eigendeformation page. Click on **Begin Calibration,** remove any sample from the platform, lower the probe, and wait for the calibration. You will then insert the steel reference material, lower the probe, and wait for the calibration to complete. Click on **Save and Apply** to accept the eigendeformation calibration values.

Temperature Calibration

This one-point temperature calibration allows you to run one reference material to match the thermocouple temperature and the sample temperature. In this calibration, the onset melting temperature for indium is determined using the penetration probe.

Select the Temperature tab in the Calibration window to display the Temperature page. To perform the TMA 7 Temperature calibration, complete a scan for indium reference material under the same conditions that you run your samples. After the run is completed, perform an Onset calculation and record the Onset temperature. Enter the reference material name, expected onset temperature, measured onset temperature, and calibration method name in the Calibration table. Click on the check box in the Use column. Select **Save and Apply** to send the new calibration value to the analyzer and save the calibration file.

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Furnace Calibration

The TMA 7 Furnace calibration is a nine-point calibration between two specified temperature limits. It linearizes the TMA 7 furnace by matching the program temperature to the thermocouple temperature over the range that you enter.

Perform a Furnace calibration after a Temperature calibration. Select the Furnace tab in the Calibration window to display the Furnace page. Enter the Minimum and Maximum temperature limits, then select **Begin Calibration** and follow the instructions in the dialog box. To accept the Furnace calibration values, click on **Save and Apply**.

DTA 7 Calibration

There are three calibration routines used to calibrate the DTA 7:

- Temperature
- Heat Flow
- Furnace

All calibrations are performed by a Service Engineer upon installation of your DTA 7 so it not necessary for you to perform a complete calibration when you begin to use your analyzer. Once the calibration is performed, the analyzer will be continuously calibrated, even when the system is turned off. Unless major changes to the analyzer's condition are made, the DTA 7 should remain calibrated.

All three calibrations can be performed separately or together. The Furnace calibration must be performed **after** the Temperature calibration. The recommended calibration procedure is to perform a 2-standard Temperature calibration when the instrument is first installed or when the DTA 7 sample and reference thermocouples are changed. Temperature calibration should be performed if either the furnace thermocouple or the furnace is changed. Conditions that could affect the furnace, temperature, or energy calibration of the DTA 7 are

1. Operating temperature range of your experiments changes. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
2. Slope control is adjusted. Always optimize the DTA 7 baseline before the instrument is calibrated.
3. Purge gas type or flow rate changes. Calibration should be checked for highest accuracy.
4. New furnace or new furnace thermocouple is installed. The Temperature and Furnace calibrations should be performed again.
5. New pair of sample and reference thermocouples are installed. Temperature calibration should be performed again.
6. Instrument has been turned off for a long time (i.e., weeks or months). It may appear to need recalibration. Condition the DTA 7 by performing several heating and cooling runs with the sample and reference cups empty and then check the calibration by running standard reference materials.

NOTE: The Temperature calibration is dependent on scan rate. Therefore, the final calibration should be performed at the scan rate that you will use for your experiments.

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As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating and cooling rates, it is better to calibrate at the slowest rate you will be using.

Before calibrating the DTA 7, it is sometimes necessary to restore default calibration values. You can restore default calibration values by selecting the appropriate calibration routine from the Restore menu while in the Calibration window. You can even restore all calibration values by selecting All. This activates the Save and Apply button; click on it and then, in the Save As dialog box, select the calibration file to be used or enter a new file name under which to save the values displayed in the calibration window.

Temperature Calibration

This two-point calibration consists of running two high-purity standards (shipped with the instrument) and measuring the melt onset for each material. The measured onset temperature is compared with the expected onset temperature to calibrate the temperature axis over a very broad temperature range.

To perform the DTA 7 Temperature calibration, complete a scan for aluminum under the same conditions that you run your samples. After the run is completed, perform a Peak Area with Onset calculation and record the Onset temperature. Do the same for the gold reference material. Select the Temperature tab in the Calibration window to display the Temperature page. Enter the information in the Temperature page and select **Save and Apply** to send the new calibration values to the analyzer and save the calibration file.

Heat Flow Calibration

Two high-purity standards are used to calibrate ΔH. This calibration is used to run in the DSC mode or when data collected in the DTA mode needs to be converted to Sample Heat Flow data.

To perform the Heat Flow calibration, accurately weigh the reference material. The accuracy and precision to which you weigh the reference material relates directly to the accuracy and precision of the energy measurements made on the DTA 7. Complete a scan of the reference material and perform a Peak Area calculation and note the ΔH result. Select the Heat Flow tab in the Calibration window to display the Heat Flow page. Enter the information and select **Save and Apply** to send the new calibration values to the analyzer and the save the calibration file.

Furnace Calibration

This calibration performs a nine-point temperature calibration between user-selected limits. It must be performed **after** the Temperature calibration. Remove the cups from the sample and reference holders. Select the Furnace tab in the Calibration window to display the Furnace page and enter Minimum and Maximum temperatures. Click on **Begin Calibration** and the Furnace calibration will start; it takes up to 2.5 hours. Select **Save and Apply** to send the new calibration values to the analyzer and save the calibration file.

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Temperature Calibration Page

Prior to performing a temperature calibration, you must perform runs using standard reference materials. Perform a peak area with onset calculation for each data file. The onset temperature obtained is used in the temperature calibration.

The Temperature Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Reference Material

Enter the names of the reference materials used in the calibration runs.

Expected Onset

Enter the expected onset temperatures for each calibration reference material. That information is provided with the reference material.

Measured Onset

Enter the measured onset temperatures calculated from the peak area with onset calculations performed on the data collected for each calibration reference material.

Method

Enter the name of the calibration method used for each calibration run.

Use

When selected, the calibration values on that line will be used in calculating the calibration factors.

Save and Apply

Applies the values entered in the table to the analyzer and saves the values in the file specified in the Save As dialog box. You can overwrite the current calibration file.

Pyris 6 TGA Temperature Calibration Page

Temperature calibration for the Pyris 6 TGA is different than that for the other Pyris analyzers. Temperature calibration must be performed **after** a Furnace calibration. If you try to make entries or changes in the Pyris 6 TGA Temperature Calibration page before performing a Furnace

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calibration, a message is displayed. For a Pyris 6 TGA, you must perform two runs for either two or three reference materials at two different scanning rates.

The items on the Pyris 6 TGA Temperature Calibration page are as follows:

Operator

Enter a operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Reference Material

The names of the three supplied reference materials are displayed by default. Change the names as needed.

Expected Onset

Enter the expected onset temperatures for each calibration reference material. That information is provided with the reference material.

Measured Onset at Rate 1 or Rate 2

Two runs are performed for each of the two or three reference materials you choose to use for calibration: alumel, iron, and perkalloy. Enter the measured onset results at the two scanning rates for each reference material.

Rate 1 and Rate 2

Enter the scanning rates used for the two runs per reference material. The recommended rates are 5°C and 50°C.

Select References

Click on the radio button for the references you want to use for the calibration: References 1 and 2 or all three.

Save and Apply

Applies the values entered in the table to the analyzer and saves the values in the file specified in the Save As dialog box. You can overwrite the current calibration file.

Heat Flow Calibration Page

The Heat Flow Calibration page appears when you select the Heat Flow tab in the DSC 7, Pyris 1 DSC, Pyris 6 DSC, or DDSC Calibration window. It is recommended that indium be used when performing a heat flow calibration. You can use the same data file that was used for temperature calibration. Perform a peak area with onset calculation and note the heat of fusion (ΔH). Then you are ready to perform a heat flow calibration.

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The Heat Flow Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Range

This is display-only. The Range is selected in the Instrument page in Preferences. It is an indication of the amount of dynamic energy that the analyzer can detect. You should have a good sense of the transition energy of the materials that you will be running. The Low selection will work with most experiments.

Reference Material

Enter the name of the reference material used in the calibration run.

Expected (J/g)

Enter the expected heat of fusion (ΔH) for the calibration material. This is provided with the standard reference material.

Measured (J/g)

Enter the measured heat of fusion (ΔH) from the peak area with onset calculation performed on the data collected for each calibration material.

Weight

Enter the weight (in mg) of the sample used in the calibration run.

Method

Enter the name of the calibration method used in the calibration run.

Save and Apply

Applies the values entered in the table to the analyzer and saves the values in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

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Furnace Calibration Page

The Furnace Calibration tab appears on the Calibration window if you have the DDSC accessory installed in a DSC 7 or have the DDSC software installed for the Pyris 1 DSC. Furnace calibration is a two-point calibration that matches the program temperature scale to the sensor temperature scale at two isothermal temperatures. It is available when the defaults are set to the sensor temperature.

The Furnace Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and automatically updated when a calibration routine is completed successfully and the values are calculated.

Minimum Setpoint

Enter the minimum temperature for the calibration. The minimum temperature is based on the ability of the cooling device you are using for the experiment. Therefore, be sure that this entry is one that the analyzer can reach and be in control. For example, if you are using an ice bath and you select 20°C for the minimum setpoint, the analyzer may not be able to be in control at that temperature. Therefore, 25°C or 30°C may be a better entry.

Maximum Setpoint

Enter the maximum temperature for the calibration.

Begin Calibration

Click on this button to begin calibration immediately and to display the Furnace Calibration dialog box.

Save and Apply

Applies the values just calculated to the analyzer and saves them in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

Furnace Calibration Dialog Box

The Furnace Calibration dialog box appears when you click on the **Begin Calibration** button on the Furnace Calibration page. While the furnace calibration is proceeding, the approximate time remaining in the calibration routine is displayed. You may stop the calibration by clicking on the **Stop Calibration** button. This cancels the calibration routine and closes the dialog box. The **Minimize App** button minimizes the Pyris application to an icon while continuing to display the Furnace Calibration dialog box. This button changes to **Restore App** with which you can restore the Pyris application to your screen.

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Weight Calibration Page

Weight calibration allows you to calibrate the weight (ordinate) axis of a TGA analyzer. A calibration standard is provided with the instrument to perform the weight calibration.

The Weight Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Range

This is display-only. The Range is selected in the Instrument Page in Preferences. It is an indication of the amount of dynamic energy that the analyzer can detect. You should have a good sense of the transition energy of the materials that you will be running. The Low selection will work with most experiments.

Reference Weight

Enter the weight of the reference material used in the calibration routine. This is provided with the reference material.

Measured Weight

The weight of the reference material as measured by the analyzer during calibration is displayed.

Begin Calibration

For a TGA 7 and a Pyris 1 TGA without autosampler, clicking on this button displays the first weight calibration dialog box which instructs you to prepare the analyzer for a zero reading, i.e., read the weight of the empty sample pan.

For a Pyris 6 TGA, the first weight calibration dialog box instructs you to place an empty sample pan on the balance and replace the furnace cover.

For a Pyris 1 TGA with autosampler, the first weight calibration dialog box instructs you to place an empty crucible at position 1 of the autosampler tray.

After following the instructions, clicking on **OK**, and performing the Read Zero calibration, the second weight calibration dialog box is displayed which instructs you to place the reference weight in the sample pan for a weight reading. For a Pyris 6 TGA, the second weight calibration dialog box instructs you to place the reference weight in the sample pan, place the pan in the balance, and replace the furnace cover. For a Pyris 1 TGA with autosampler, the second weight calibration dialog box instructs you to place the reference weight in the empty crucible in position 1. After you click on **OK**, the furnace will be raised and the weight will be read.

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Save and Apply

Applies the calibration value to the analyzer and saves the values in the calibration file you designate in the Save As dialog box.

First Weight Calibration Dialog Box

This dialog box appears when you select the **Begin Calibration** button on the TGA 7 and Pyris 1 TGA without autosampler Weight Calibration page. It gives instructions on preparing the analyzer for the zero reading, i.e., the weight of the sample pan without any sample in it. Click on **OK** and the reading is made and the second dialog box is displayed. The **Cancel** button cancels the calibration routine and closes the dialog box.

Pyris 6 TGA Weight Calibration Dialog Box 1

This dialog box appears when you select the **Begin Calibration** button on the Pyris 6 TGA Weight Calibration page. It gives instructions on preparing the analyzer for the zero reading, i.e., the weight of the sample pan without any sample in it. Place the empty sample pan (crucible) on the balance and replace the cover on the furnace. Click on **OK** and the reading is made. The second dialog box is displayed.

Pyris 1 TGA Weight Calibration Dialog Box 1

This dialog box appears with you select the **Begin Calibration** button on the Pyris 1 TGA with autosampler Weight Calibration page. It gives instructions on preparing the analyzer for the zero reading, i.e., the weight of an empty crucible. Place the crucible in position 1 of the autosampler. Click on **OK** and the autosampler will go to the load position. The analyzer will load the crucible and its weight will be read. The crucible will then be unloaded and the second dialog box is displayed.

Second Weight Calibration Dialog Box

This dialog box appears after you have performed the zero reading for a TGA 7 and Pyris 1 TGA without autosampler. It instructs you to prepare the analyzer for the reference material's weight reading. Click on **OK** to have the weight measured and close the dialog box. The measured weight is displayed in the Measured field on the Weight page. **Cancel** aborts the calibration routine and closes the dialog box.

Pyris 6 TGA Weight Calibration Dialog Box 2

This dialog box appears after you have performed the zero reading for a Pyris 6 TGA. It instructs you to prepare the analyzer for the weight reading. Place the reference weight in the sample pan that is still on the balance. Replace the furnace cover. Click on **OK** and the weight is measured and displayed in the Measured field on the Weight page.

Pyris 1 TGA Weight Calibration Dialog Box 2

This dialog box appears after you have performed the zero reading for the Pyris 1 TGA with autosampler. It instructs you to prepare the analyzer for the reference material's weight reading.

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Place the reference weight in the crucible at location 1 of the autosampler. After you click **OK**, the crucible will be loaded, the furnace will be raised, the weight will be read. The crucible will then be unloaded. The measured weight is displayed in the Measured field on the Weight Calibration page.

Furnace Calibration Page

The furnace calibration for a DTA 7, Pyris 6 TGA, TGA 7, Pyris 1 TGA, DMA 7e, and a TMA 7 is a 9-point temperature calibration between user-selected upper and lower temperature limits. The thermocouple temperature will be matched to the programmed furnace temperature when this calibration is complete.

For a DTA 7, TGA 7, and Pyris 1 TGA, furnace calibration must be performed **after** temperature calibration. For a Pyris 6 TGA, it must be performed **before** temperature calibration.

For the DTA 7, Pyris 6 TGA, TGA 7, and Pyris 1 TGA, when selecting minimum and maximum temperatures for the furnace calibration, select the limits so that they encompass the temperature range where you plan to operate. If you normally run from 100°C to 900°C, select 100°C and 900°C as the Minimum and Maximum Setpoints. For the DMA 7e or the TMA 7, select 50°C and 250°C for the minimum and maximum temperatures.

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Minimum Setpoint

Enter the minimum temperature at which the calibration begins.

Maximum Setpoint

Enter the maximum temperature at which the calibration ends. The software calculates the other seven points.

Begin Calibration

Displays the furnace calibration dialog box and begins the calibration routine.

Save and Apply

Applies the calibration values to the analyzer and saves them in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

Furnace Calibration Dialog Box

The Furnace Calibration dialog box appears when you select the **Begin Calibration** button on the Furnace Calibration page. While the furnace calibration is proceeding, the approximate time

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remaining in the calibration routine is displayed. You may stop the calibration by clicking on the **Stop Calibration** button. This cancels the calibration routine and closes the dialog box. The **Minimize App** button minimizes the Pyris application to an icon while continuing to display the Furnace Calibration dialog box. This button changes to **Restore App** with which you can restore the Pyris application to your screen.

DMA Calibration Page

The DMA Calibration routine must be completed before any other calibration is performed. It performs necessary operations in the firmware and generates lookup tables that are stored in memory.

The DMA Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Begin Calibration

Click on this button to display the DMA Calibration dialog box. Follow the instructions and the calibration is performed.

Save and Apply

Applies the calibration values to the analyzer and saves them in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

DMA Calibration Dialog Box

The DMA Calibration dialog box appears when you select the **Begin Calibration** button on the DMA Calibration page. It instructs you to remove the sample from the sample platform, lower the probe, and raise the furnace of the analyzer. Click on the **OK** button to initiate the calibration and display the next dialog box.

Second DMA Calibration Dialog Box

As the DMA Calibration routine is in progress, the dialog box displays the approximate time remaining in the calibration. Click on the **Cancel** button if you wish to cancel the calibration and return to the Calibration window. At the end of the calibration the last dialog box appears with the message that the calibration is complete.

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Last DMA Calibration Dialog Box

When the DMA Calibration routine is complete, you see the message “DMA 7 Calibration is Complete” displayed.

Height Calibration Page

Height calibration is used to calibrate the displacement transducer that is used to measure the position and amplitude (ordinate) axis of the DMA 7e or the TMA 7.

The DMA/TMA Height Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Reference Height

Enter the height of the reference material. A sapphire height displacement standard, suitable for height calibration over the full range of the system, is provided with the instrument.

Measured Height

The height of the reference material as measured by the analyzer is displayed automatically after the calibration procedure.

Begin Calibration

Click on this button to display the first Height Calibration dialog box. Follow the instructions and the calibration is performed.

Save and Apply

Applies the calibration values to the analyzer and saves them in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

Height Calibration Dialog Box

The Height Calibration dialog box appears when you select the **Begin Calibration** button on the Height Calibration page. It instructs you to prepare the analyzer for the zero reading. Before clicking on **OK**, remove any sample from the sample platform, lower the probe, and raise the furnace of the analyzer. Click on the **OK** button to initiate the zero height calibration and display the Read Zero dialog box. Clicking on **Cancel** will cancel the calibration routine and return you to the calibration window.

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Read Zero Dialog Box

This dialog box appears after you have initiated the zero height reading. The Y signal is displayed in the Read Zero field. Once you are satisfied with the reading, click on **OK** to accept the value and display the next dialog box. Click **Cancel** to abort the calibration routine and return to the calibration window.

Second Height Calibration Dialog Box

This dialog box appears after accepting the Read Zero value during a Height calibration. It instructs you to raise the analyzer's probe, position the sapphire displacement standard on the platform, and then lower the probe. Click on **OK** to initiate the height calibration. The Read Height dialog box is displayed. Click **Cancel** to abort the calibration routine and return to the calibration window.

Read Height Dialog Box

This dialog box appears after initiating the height reading. The Y signal is displayed in the Read Value field. Once you are satisfied with the stability of the reading, click on **OK** to accept the value and enter it as the height calibration value in the Measured field and clear the dialog box. Click **Cancel** to abort the calibration routine and return to the calibration window.

Force Calibration Page

Force calibration is used to calibrate the force motor of the DMA 7e or the TMA 7 that is used to apply the static and dynamic forces to the sample. Force calibration takes two readings at different forces and linearizes the results.

The DMA/TMA Force Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Begin Calibration

There are no reference and measured entry fields in this calibration routine. Just click on this button to display the Force Calibration dialog box, follow the instructions, and the calibration is performed.

Save and Apply

Applies the calibration value to the analyzer and saves it in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

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Force Calibration Dialog Box

The Force Calibration dialog box appears when you click on **Begin Calibration** button on the Force Calibration page. It instructs you to prepare the analyzer for force calibration by removing the sample from the sample platform and placing the weight tray in the analyzer. Click on the **OK** button to initiate the force calibration and display the next dialog box. Clicking on **Cancel** will cancel the calibration routine and return you to the Force Calibration page.

Second Force Calibration Dialog Box

This dialog box instructs you to place the standard 50-g weight onto the weight tray. Calibration proceeds automatically. After a short time, the calibration is finished, the dialog box clears, and you return to the Force Calibration page. Click **Cancel** to abort the calibration routine and return to the Force Calibration page.

Eigendeformation Calibration Page

Eigendeformation calibration is used to calibrate the very small movement of the analyzer itself when large forces are applied. After performing the calibration, the compliance of the system is subtracted from the probe position signal. Therefore, increasing force with no sample present will yield zero displacement. Perform the Eigendeformation calibration after height and force calibration.

The DMA/TMA Eigendeformation Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

Begin Calibration

Click on this button to display the Eigendeformation Calibration dialog box. Follow the instructions and the calibration is performed.

Save and Apply

Applies the calibration value to the analyzer and saves it in the calibration file that you designate in the Save As dialog box. You can overwrite the current calibration file.

Eigendeformation Calibration Dialog Box

The Eigendeformation Calibration dialog box appears when you click on the **Begin Calibration** button on the Eigendeformation Calibration page. It instructs you to prepare the analyzer for eigendeformation calibration by removing the sample from the sample platform and lowering the

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probe. When you are ready, click on **OK**. The next dialog box is displayed. Click **Cancel** to abort the calibration routine and return to the Eigendeformation Calibration page.

Eigendeformation Calibration Step One

While the calibration is in progress, the time remaining in the calibration is counted down in the dialog box for Eigendeformation Calibration - No Sample. When time is up, the next dialog box is displayed. Click **Cancel** to abort the calibration routine and return to the Eigendeformation Calibration page.

Second Eigendeformation Calibration Dialog Box

This dialog box instructs you to raise the probe and insert the steel calibration reference material that is provided with the instrument. Lower the probe and click on **OK** to proceed to the next dialog box. Click **Cancel** to abort the calibration routine and return to the Eigendeformation Calibration page.

Eigendeformation Calibration Step Two

While the calibration is in progress, the time remaining is counted down in the dialog box for Eigendeformation Calibration - Steel Sample. When complete the last dialog box is seen. Click **Cancel** to abort the calibration routine and return to the Eigendeformation Calibration page.

Last Eigendeformation Calibration Dialog Box

The last dialog box in Eigendeformation Calibration states that the calibration has been completed successfully. Raise the probe and remove the steel sample from the platform. Click on **OK** to clear the dialog box and return to the Eigendeformation Calibration window.

Heat Flow Calibration Page

It is recommended that aluminum and gold be used when performing a heat flow calibration for the DTA 7. You can use the same data files that were used for temperature calibration. Perform a peak area calculation and note the heat of fusion (ΔH). Then you are ready to perform a heat flow calibration.

The Heat Flow Calibration page contains the following fields:

Operator

Enter an operator name up to 40 characters; it will be saved with the calibration file.

Date and Time

This is display-only and is automatically updated when a calibration routine is completed successfully and the values are calculated.

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Reference Material

Enter the names of the reference materials used in the calibration runs. The defaults are Aluminum and Gold.

Expected (J/g)

Enter the expected heat of fusion for each calibration material. This is provided with the standard reference material.

Measured (°C s)

Enter the measured heat of fusion from the peak area calculation performed on the data collected for each calibration material.

Expected Temperature

Enter the expected melting point for the reference material which is provided with the standard reference material.

Weight

Enter the weight (in mg) of the sample used in the calibration run.

Save and Apply

Applies the values entered in the table to the analyzer and saves the values in the file specified in the Save As dialog box. You can overwrite the current calibration file.

AS 6 Align Gripper Wizard

Align AS 6 Gripper - Start

You should perform an align gripper procedure if you installed a reference pan into the Pyris 6 DSC or you changed the sample thermocouple of the Pyris 6 TGA. Occasionally, the gripper may start to pick up a lid or pan in a faulty manner. Realigning the gripper may solve the problem.

You access the Gripper Alignment wizard when you click on the **Align** button in the DSC 6/TGA 6 Autosampler Control dialog box. This first screen reminds you that the furnace cover should be in place; you do **not** need to place pans at locations 12 and 33 on the sample tray. If you have a Pyris 6 DSC, make sure that the lower furnace lid is on the ring and not in the furnace. Click on **Next** to go to the next screen.

AS 6 Upper Cover Alignment

If you are running the Align Gripper wizard from the Diagnostic Panel, when you click on **Next** in the first wizard screen, the gripper arm swings around to find the home position and then it goes to the furnace cover. The second screen appears. You can use the **Move Up, Move Down, Move Counterclockwise**, and **Move Clockwise** buttons, or use the arrow keys on the keyboard (the

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right arrow is Clockwise), the adjust the position of the fingers with respect to the knob on the lid. The indentation on the fingers should be just blow the lip on the knob. Make sure that the distance from each finger to the lid is the same. The **Move Up** and **Move Down** buttons move the gripper in 0.1-mm steps. When you are satisfied, click on **Next** to display the next wizard screen. The gripper removes the upper lid and places it on the ring. The gripper remains above the upper lid. For a Pyris 6 DSC, the next wizard deals with the lower lid. For the Pyris 6 TGA, you see the AS6 Location 12 Alignment wizard.

AS 6 Lower Lid Vertical Position Calibration

Place the lower lid calibration tool into the Pyris 6 DSC furnace with the small end down; make sure that the tool is all the way down. Place the lower lid on top of the tool. Click on the **Next** button. The gripper positions itself over the lower lid.

AS 6 Lower Lid Alignment

The gripper is now over the lower lid on the calibration tool. Use the buttons on the wizard or the arrow keys on the keyboard to position the gripper so that the indentation of each finger is just below the lid's knob. Use the **Move Counterclockwise** and **Move Clockwise** buttons (or the left and right arrow keys, respectively) to position the fingers around the knob so that each finger is equidistant from the knob. Click on the **Next** button. The gripper removes the lower lid and places it on the ring.

AS 6 Remove Lower Lid

This screen just tells you to remove the Lower Lid Calibration tool from the furnace carefully. Click on **Next**. The gripper swings over to position 12.

AS 6 Location 12 Alignment

The gripper swings over to location 12 on the front sample tray. Use the four buttons on the screen or the arrow keys to move the gripper lower. The fingers should be equidistant from the dimple in the tray and about 0.1 mm above the tray. You do not need to have a sample pan in position for the procedure. Click on **Next** to proceed to the next wizard screen. The gripper swings over to position 33.

AS 6 Location 33 Alignment

After aligning the gripper at location 12, the gripper swings around to location 33 at which you perform the same steps in order to make sure that the gripper's fingers are equidistant from the dimple in the sample tray and that they are 0.1 mm from the tray. Click on **Next** to proceed to the next wizard screen. For the Pyris 6 DSC, the gripper goes over the lower furnace lid; for the Pyris 6 TGA, the gripper goes over the upper lid.

AS 6 Lower Cover on Ring Alignment

Service engineers will see this wizard screen after the Location 33 Alignment for a Pyris 6 DSC. (The Pyris 6 TGA does not have a lower lid so this alignment does not exist for that instrument.) After adjusting the gripper at location 33 and clicking on **Next**, the gripper positions itself over the lower lid on the ring. Use the buttons on the wizard screen and/or the arrow keys on the keyboard

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to adjust the gripper fingers so that they are equidistant from the lower lid's knob and that the indentations of the fingers are just below the lip of the knob. Click on **Next**; the gripper lifts the lower lid and places it in the furnace. It then moves to the upper furnace lid position.

AS 6 Upper Cover on Ring Alignment

After aligning the lower lid for the Pyris 6 DSC, or performing the Location 33 alignment for the Pyris 6 TGA, the next step is to align the gripper with respect to the upper furnace lid while it is on the ring. The gripper should be positioned over the lid. Use the buttons on the wizard and/or the arrow keys to manipulate the gripper so that the fingers are equidistant around the lid's knob and that the indents of the fingers are just below the knob so that they can pick it up. Click on **Next**. The gripper lifts the lid and places it on the furnace; then it returns to the home position and the fingers close. The next wizard screen is displayed.

AS 6 Furnace Position Alignment

Make sure that the alignment crucible (an upside down TGA sample pan) is in position 0. Click on the **Begin** button. The furnace alignment procedure takes about 5 minutes. The gripper swings around to the furnace. It removes the upper lid and places it on the ring. For the Pyris 6 DSC, it removes the lower lid and places it on the ring. The gripper then retrieves the alignment crucible and places it in the furnace, places the lower lid within the furnace (for the Pyris 6 DSC), places the upper lid on the furnace, and goes to the home position. If these steps are accomplished without error, FINISHED is displayed in the status box. Click on **Next**.

AS 6 Alignment Finished

Service engineers performing the Align Gripper procedure have access to additional wizard screens prior to this one.

The last screen just informs you that you are finished with the alignment. Click on **Finish** to exit the wizard and return to the DSC6/TGA6 Autosampler Control dialog box.

Pyris 1 TGA Align Gripper Wizard

Start Align Gripper

You should go through the alignment procedure for the gripper on the Pyris 1 TGA autosampler if you have changed the hangdown wire. The **Align Gripper** button on the Autosampler Control dialog box accesses the Align Gripper wizard. This first wizard screen tells you to make sure that there is no crucible on the hangdown wire. When you click on the **Next** button, the next wizard screen appears.

Align Gripper - Move

This wizard screen informs you that when you click on the **Next** button, the furnace will go to the Lowered position, if not there already, and the autosampler will go to the Load position. The next wizard screen is then displayed.

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Align Gripper - Direction

After the furnace and autosampler have moved to the Lowered and Load positions, respectively, you can now move the gripper with respect to the hangdown wire. Look at the groove or "V" in the gripper arms with respect to the hangdown wire from the left side of the autosampler. The hangdown wire should be directly in the middle of the grooves. If not, use the Move In and the Move Out features in the wizard screen. Click on a step size button to fine tune the movement of the gripper. Move In moves the gripper to the left with respect to the hangdown wire (i.e., the gripper moves in toward the analyzer). Move Out moves the gripper to the right with respect to the hangdown wire (i.e., the gripper moves out away from the analyzer). Click on the **Next** button to proceed to the next wizard once the hangdown wire is positioned correctly inside the gripper.

Align Gripper - Close and Open

After the hangdown wire is aligned within the grooves of the gripper arms, close the gripper around the hangdown wire. Make sure that the wire is not pulled excessively or rotated. Open the gripper. Close and open the gripper a few times to check your work. If the gripper needs more fine tuning, click on the **Back** button to return to the Direction wizard. If the hangdown wire sits within the gripper correctly, click on **Next** for the next wizard screen.

Align Gripper - All Done

You are all done with aligning the gripper. This wizard suggests that you should check your work by performing a Tare All. Do this by opening a new Pyris Player playlist. Click on **Add a step** and choose Sample Group. Click on the **Tare All** button. In the Advanced Tare All dialog box, select Use Only One Tray and select the second option: This Group Only; Populate from Tray. Click on **OK** and the system will tare each crucible found in the sample tray. It is a convenient way to see that the hangdown wire and gripper are aligned correctly.

NOTE: We recommend that you align the tray before performing a Tare All. Click on **Align Tray** in the Autosampler Control dialog box once you exit this wizard.

Once you exit this wizard by clicking on the **All Done** button, the furnace goes to the Cooling position and the autosampler goes to the Safe position.

Pyris 1 TGA Align Tray Wizard

Start Align Tray

If you have changed the hangdown wire or have just aligned the gripper, you should perform the Tray Alignment procedure. Access the Align Tray wizard for the Pyris 1 TGA autosampler by clicking on the **Align Tray** button on the Autosampler Control dialog box. This first wizard screen tells you to make sure that there is no crucible on the hangdown wire. When you click on **Next**, the second wizard screen appears.

NOTE: Before aligning the tray, be sure to align the gripper by using the Align Gripper wizard. This is also accessed from the Autosampler Control dialog box.

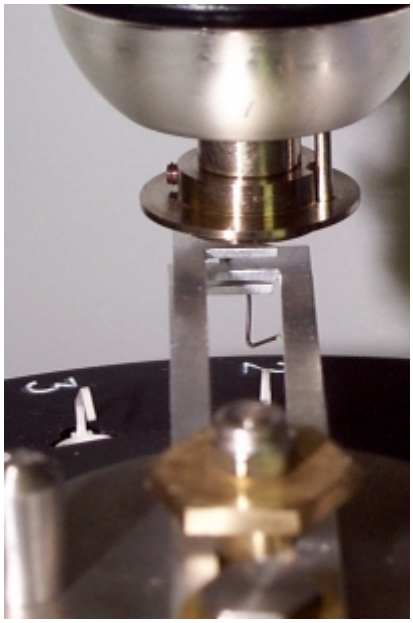
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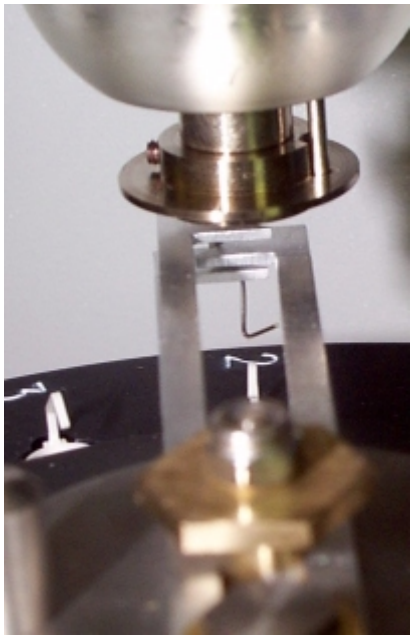
Align Tray - Move

This wizard screen informs you that when you click on the Next button, the furnace will go to the Lowered position, the autosampler will go to the Load position, and the gripper will close around the hangdown wire. Click on **Next** to display the next wizard screen.

Align Tray - Direction

After the furnace and autosampler have moved to the Lowered and Load positions, respectively, and the gripper has closed, check the position of the crucible handle with respect to the hangdown wire. In the left-hand photo below, note the position of the right side of the hangdown wire with respect to the left side of the crucible handle. The crucible is too far to the right. Using Move Right and a Medium step will move the tray counterclockwise and actually move the crucible to the left. The result is shown in the right-hand photo. The inner side of the hangdown wire should align with the left side of the crucible handle.





Align Tray - All Locations

After aligning the crucible in position 1 of the sample tray with the hangdown wire, proceed with aligning the rest of the crucibles. Click on **Next Position** to rotate the tray to the next position. When are done aligning all 20 locations, click on **Next** for the next wizard screen.

When the hangdown wire and crucible handle are aligned properly, click on **Next** for the next wizard screen.

Align Tray - All Done

To make sure that all 20 positions of the sample tray align correctly with the hangdown wire, and therefore all crucibles will be picked up and returned without error, perform a Tare All. After leaving this wizard, open Pyris Player, select New from the File menu to use an empty play list. Click on Add a step and select Sample Group. Click on the Tare All button. In the Advanced Tare

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All dialog box, select Use Only One Tray and select the second option: This Group Only; Populate from Tray. Click on **OK** and the system will tare each crucible found in the sample tray. It is a convenient way to see that the hangdown wire and crucibles are aligned correctly. If they are not, the hangdown wire will either miss the crucible handle, or will put the crucible back into the tray unevenly.

Press **All Done** to leave this wizard. The furnace goes to the Cooling position, the gripper opens, and the autosampler returns to the Safe position.

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Chapter 8
Preferences

You can change the default values for many functions in Pyris Software for Windows by using the **Preferences** item on the Tools menu in Instrument Application and Data Analysis Application. Each Preferences window contains nine tabbed pages when opened in Instrument Viewer and four tabbed pages when opened in Data Analysis. Each page is described below.

General Preferences Page

The General page appears when you select the General tab in the Preferences window. The General page contains the following fields:

Line Types

You can select the line type to be used for displaying up to four curves and then select a line type for each of those curves whose axes are not shown. Display the selection of line types by clicking on the down arrow for the drop-down list. The order of display for the Y axes is as follows:

1. Y1 axis left inside axis
2. Y2 axis right inside axis
3. Y3 axis left outside axis
4. Y4 axis right outside axis

Tooltips

Tooltips are the familiar text displays that popup when your cursor moves over a specific area of the screen such as a toolbar button or field in a screen. The tooltip's text can be short or a more descriptive long version. You may also turn tooltips off altogether.

DMA Reports

Indicates how you want the DMA data displayed and reported: as force (mN) or stress (Pa).

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Color Preferences Page

The Colors page appears when you select the Colors tab in the Preferences window. The Colors page contains the following fields:

Set Color For

Displays the colors for graphical window components, including the background, grid, and the first 12 curves displayed. Select an item in the list and then select a color in the Colors box.

Colors

Displays all the possible colors that can be assigned to the graphical window components listed in the Set Color For list box. The color assigned to the currently selected item in the list is displayed in a double box.

Graph Preferences Page

The Graph page appears when you select the Graph tab in the Preferences window. The Graph page contains the following fields:

Title

Enter the title that will appear on your printouts if you check in the Include Title box in the Page Setup dialog box. The default title is "Perkin Elmer Thermal Analysis."

Logo

Enter the file name of the graphics file of your logo. This will appear on your printouts.

Auto-Rescale

When Full Scale functions are selected in the Rescale Tools toolbar, the X-axis and Y-axis scales change so that all of the data can be displayed. White space can be placed automatically between the curves and the sides of the graphical window by using the Auto-Rescale settings. They are set in this page. Enter the top, bottom, left, and right percentages to be used for displaying data at full scale. Type a value in each entry field or use the spin buttons to change the displayed values in 1% increments. The higher the percentage, the more space displayed between the curve and the edge of the window.

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Font

Displays the Font dialog box. Below the button are the name of the selected font, the font size, and an example.

Save Preferences Page

The Save page appears when you select the Save tab in the Preferences window. The Save page contains the following fields:

Automatic Save Every

Click in this box to enable the Automatic Save feature in the software. Real-time data files will automatically be saved in the time interval specified in the entry field. Type in a value or use the spin buttons to change the displayed value in one-minute increments. The default value is 30 minutes. This feature is useful for particularly long runs.

Use file name

Enter a default data file name to be used for automatic saves or if you do not enter a file name in the Save Data As field in the Method Editor. A date/time stamp in the format of YYMMDDHHMMSS (year, month, day, hour, minute, second) is appended to the file name every time it is used so data files are not overwritten. The default file name is QSAVE.XXX where XXX is the standard extension associated with the analyzer.

Directory Paths

Path to Data Files

Enter the complete directory path in which to store data files.

Path to Method Files

Enter the complete directory path in which to store method files.

Path to Player Lists

Enter the complete directory path in which to store play list files.

Browse

Use this button to select the drive and the directory in which to save the data, method, or play list file.

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Real-Time Curves Preferences Page

The Real-Time Curves page appears when you select the Real-Time Curves tab in the Preferences window. The Real-Time Curves page contains the following fields:

Real-Time Curve Selection

Curve #1

Select the default curve type for the first real-time curve displayed. The available curve types are the same as listed on the Curves menu for each analyzer.

Curve #2

Select the default curve type for the second real-time curve displayed. The available curve types are the same as listed on the Curves menu, minus the curve selected for Curve #1.

Curve #3

Select the default curve type for the third real-time curve displayed.

Curve #4

Select the default curve type for the fourth real-time curve displayed.

X-Axis Displayed at Start of Run

Select Sample Temperature or Time as your X axis display in the Instrument Viewer as the run starts. You can change the X axis using Rescale X during the run.

At Start of Each Run

If you change the types of curves displayed while in Instrument Viewer and/or the X axis, you can have the software use those settings for subsequent runs instead of changing them back to those set in this Preferences page. Click in the radio button next to **Use Current Settings**. If you always want the Preferences page settings used at the start of each run, select **Use Settings Shown Here**.

Remote Access Preferences Page

The Remote Access page appears when you select the Remote Access tab in the Preferences window. You must have the optional Remote Monitor software installed on your computer in order to see this tab.

The Remote Access page is used to set permission for others on a network to access your computer and analyzers. There are three permission levels:

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No Access

Forbids any other PC from accessing the analyzers running on your PC.

Status Panel and Curve Display only

Allows other PCs on the network to monitor the status of the analyzers running on your PC and displays the Instrument Viewer screen for the remote analyzer.

Stop Run, Status Panel, and Curve Display

Allows other PCs on the network to monitor the status of and stop a run on the analyzers running on your PC and displays the Instrument Viewer screen for the remote analyzer.

Purge Gas Preferences Page

The Purge Gas page appears when you select the Purge Gas tab in the Preferences window. Use this page to select the purge gas connected to each available port on the analyzer or gas switching accessory attached to the analyzer. The gas switching accessory must be configured into the system during Configuration in order for the correct number of gas ports to be displayed on this page. If no accessory is configured, only Gas A will be displayed. If a GSA 7 is being used, then there are two purge gas ports: Gas A and Gas B. A TAGS attached to your thermal system provides four purge gas ports. The entries made on this page are displayed in the Initial State page of the Method Editor and at the bottom of the control panel.

Purge Gas Settings

Select the purge gas attached to the Gas A port of your analyzer, GSA 7, or TAGS from the drop-down list. If you change the purge gas attached to the port, you must update the entry here. It is important that these settings reflect the actual system. Data may be affected by inaccurate information.

Initial Flow Rate

Enter the initial flow rate by typing in the value or using the spin buttons. This value is displayed on the Initial State page of the Method Editor and at the bottom of the control panel.

Autosampler Preferences Page

The Autosampler page appears when you select the Autosampler tab in the Preferences window. This page is included in the Preferences window if the analyzer has an autosampler.

Autosampler Load Range

This section defines the temperature at which samples will be allowed to load or unload. If the current sample temperature is outside of the range defined by the **Minimum Temperature** and

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Maximum Temperature values, for any autosampler operation, whether it is in a playlist or not, the analyzer will be programmed to the **Temperature Setpoint** before the operation is allowed to proceed. The Go To Temp rate is used to go to the Temperature Setpoint. The program temperature must be equal to the Temperature Setpoint and the sample temperature must be within 5°C of the program temperature in order to proceed.

The minimum and maximum temperatures for the DSC 7 autosampler are limited to 0°C and 70°C, respectively, because of the tip of the robot arm. All other autosamplers are allowed to operate at the analyzer temperature limits. The Temperature Setpoint must fall between those two temperatures.

Pyris 1 DSC Autosampler Preference Page

The Autosampler page appears when you select the Autosampler tab in the Preferences window for the Pyris 1 DSC. This page is included in the Preferences window if the analyzer has an autosampler.

Autosampler Load Range

This section defines the temperature at which samples will be allowed to load or unload. If the current sample temperature is outside of the range defined by the **Minimum Temperature** and **Maximum Temperature** values, for any autosampler operation, whether it is in a playlist or not, the analyzer will be programmed to the **Temperature Setpoint** before the operation is allowed to proceed. The Go To Temp rate is used to go to the Temperature Setpoint. The program temperature must be equal to the Temperature Setpoint and the sample temperature must be within 5°C of the program temperature in order to proceed.

The minimum and maximum temperatures for the Pyris 1 DSC autosampler are limited to 0°C and 70°C, respectively, because of the tip of the robot arm.

Number of Retries

The Pyris 1 DSC autosampler may be set to try to remove a sample or reference from the furnace area more than once. The default is 0 retries, i.e., it will attempt to remove the sample or reference pan only once. The maximum is 5 retries.

Use Initial Check

This feature is available for the playlist operations Load Sample, Load Reference, Return Sample, and Return Reference for the Pyris 1 DSC autosampler only. If you clicked in the Use Initial Check box to activate the feature, when the playlist starts and any one of these four commands is attempted, the autosampler will perform an initial check once and **once only,** i.e., once per playback. If you stop and then restart a playlist, the check will be done once again. The initial check will look for the presence of a sample in the furnace, the presence of a reference in the furnace, and the presence of at least one platinum cover on the receptacle. Playback will proceed with the internal state of the playback set accordingly.

If the first playlist operation involving a sample is Return Sample, with initial check, the presence or absence of a sample pan in the sample holder will be detected. If a sample pan is detected, it will be removed. If there is no pan present, no error will be generated and playback will continue.

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PID Controls Preferences Page

The PID Controls page appears in the Preferences window for a DMA 7e or a TMA 7. When using Position Control with the DMA 7e or the TMA 7, the probe is maintained at a constant position using a “Proportional + Integral + Derivative” motor control system. These values are valuable in fine tuning the probe position. The motor control system compares the measured sample position to the program position and generates a motor command that is related to the difference between these positions. The motor command is then multiplied by the values you enter in this page.

The DMA 7e and the TMA 7 also use a “Proportional + Integral + Derivative” furnace control system to control the sample temperature. The amount of heat applied to the furnace, as it is heated or cooled, is determined by the temperature of the sample thermocouple and the furnace control system. The furnace control system compares the sample temperature to the program temperature and generates a furnace command that is related to the difference between these temperatures. The command is multiplied by the furnace gain value. After multiplication, the furnace command is used to program the amount of heat applied by the furnace. Furnace control is used when the instrument furnace is in the raised position, either before or during the analysis. When the furnace is lowered, the furnace temperature alone is used to control the furnace.

Position Control

Proportional

The motor control system produces a correction that is proportional to the difference between the user-selected setpoint and the current probe position. A control equation that uses only proportional control will typically maintain a probe position within a close range of the setpoint. It typically does not converge to the setpoint and there is often some constant offset. The higher the proportional gain the greater the response of the control system and it is likely to overshoot the setpoint.

Integral

The motor control system produces a correction based on the integral of the difference between the user-selected setpoint and the current probe position. The area accumulated by the offset, as swept through time, is added to the control instructions in order to increase or decrease the static force until the probe position moves to the setpoint, gradually zeroing out any remaining offset. This term is usually not used in controlling the probe position.

Derivative

The motor control system produces a correction based on the time derivative of the difference between the setpoint and the probe position. This control minimizes overshoot and undershoot of the setpoint by controlling the slope of the probe position versus time curve. This component of the motor control typically responses more slowly and can eliminate any constant offset between the probe position and the probe setpoint.

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Temperature Control

Proportional

The furnace control system produces a correction that is proportional to the difference between the program temperature and the sample temperature. A control equation that uses only proportional control will typically maintain a sample temperature within a close range of the program temperature. It typically does not converge to the program temperature and there is often some constant offset. The higher the proportional gain the greater the response of the furnace control system and it is likely to overshoot the program temperature.

Integral

The furnace control system produces a correction based on the integral of the difference between the program temperature and the sample temperature. The area accumulated by the offset, as swept through time, is added to the control instructions until the sample temperature moves toward the program temperature, gradually zeroing out any remaining offset. This term is usually not used in controlling the furnace temperature.

Derivative

The furnace control system produces a correction based on the time derivative of the difference between the program temperature and the sample temperature. This control minimizes overshoot and undershoot of the program temperature by controlling the slope of the sample temperature versus time curve. This component of the furnace control typically responses more slowly and can eliminate any constant offset between the sample temperature and the program temperature.

DSC 7 and Pyris 1 DSC Instrument Page

The DSC 7 Instrument page and the Pyris 1 DSC Instrument page are exactly the same. They each appear when you select the instrument's tab in the Preferences window. The DSC 7 Instrument page is available only when in DSC 7 Instrument Application and the Pyris 1 DSC Instrument page is available only when in Pyris 1 DSC Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used in all methods as the default End Condition. When the **Go To Load** button in the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature, the temperature setpoint, or the user-specified temperature entered in the control panel. Scanning rates greater than 100°C/min are normally used only for rapidly heating or cooling the analyzer to starting temperatures or to selected isothermal temperatures. Typical experimental scanning rates range from 5°C/min to 40°C/min.

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Maximum Temperature

Enter the default maximum temperature that the analyzer will be allowed reach. This parameter helps prevent the melting of sample pans. For example, since aluminum melts at 660°C, enter a maximum temperature of 600°C.

Data

Range

Select the default data range sensitivity for the ordinate scale. The data range is indicative of the amount of dynamic energy the analyzer can detect. High range for the DSC 7 and the Pyris 1 DSC is 720 mW full scale and low range is 320 mW full scale.

Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (heat flow) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

Lag Compensation

This value compensates for the lag of the analyzer in maintaining the scanning rate when heating up to the designated temperature. It helps the analyzer get up to the designated scanning rate faster. Although the thermal lag is small for power-compensated DSC analyzers (i.e., the peak shifts up by about 1.3°C as the scan rate in increased from 5°C/min to 20°C/min), it may be significant for some applications. In cases where temperature must be independent of scan rate, correction of measured temperature for differences in scan rate can be performed. See Determine the Lag or Rate Compensation for an explanation on how to determine the value to enter.

Equilibration Constant

This field is for DDSC analyzers only. Enter the number of equilibration heat – cool cycles that are to be run at the beginning and the end of a run. It determines how long the DDSC equilibrates.

Environment

Cooling Device

Select the cooling device attached to your analyzer from the drop-down list. Available cooling devices are Intracoolers 1 and 2 for the DSC 7, Intracoolers 1P and 2P for the Pyris 1 DSC, CCA 7, water bath, ice bath, and CryoFill for the Pyris 1 DSC. This parameter has no effect on the software; it is for information only.

Helium Purge in Use

If you are using the Liquid Nitrogen Subambient Accessory on the DSC 7 or the CryoFill Liquid Nitrogen Cooling System with the Pyris 1 DSC for cooling, you must use helium for the purge gas. In this case, select this check box. The thermoconductivity of helium is high so when you click in the Helium Purge in Use check box the system will limit the maximum Go To Load temperature to 300°C.

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Liquid Nitrogen in Use

When using the Liquid Nitrogen Subambient Accessory or the CryoFill LN2 Cooling System for cooling, select this box.

Pyris 6 DSC Instrument Page

The Pyris 6 DSC Instrument page appears when you select the DSC 6 tab in the Preferences window. The Pyris 6 DSC Instrument page is available only when in DSC 6 Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used in all methods as the default End Condition. When the **Go To Load** button in the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel. Proper selection of the heating rate increases the efficiency of your analysis at the desired sensitivity. Slower heating rates improve peak resolution while faster heating rates improve the usable sensitivity. Heating rates greater than 40°C/min are usually used for rapidly heating or cooling the Pyris 6 DSC to the beginning temperature or the next program step or to selected isothermal temperatures in the temperature program.

Data

Display Language

Select the language in which the LCD display on the analyzer is to display its information: English, French, German, Italian, Japanese, or Spanish.

Ordinate Filter

The ordinate filter filters out noise from the Y signal (heat flow). Click on the box to allow data smoothing.

Furnace Constant

The furnace constant is calculated at the factory and is supplied on the Pyris 6 DSC Data Sheet shipped with the instrument. Enter the value from the sheet here.

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Heat Flow Conversion

The heat flow conversion (or calorimetric sensitivity) is calculated at the factory and is supplied on the Pyris 6 DSC Data Sheet shipped with the instrument. Enter the value from the sheet here.

Environment

Cooling Device

Select the cooling device attached to your Pyris 6 DSC from the drop-down list. Available cooling devices are circulating water or chilled gas. This parameter has no effect on the software; it is for information only.

TGA 7 Instrument Page

The TGA 7 Instrument page appears when you select the TGA 7 tab in the Preferences window. The TGA 7 Instrument page is available only when in TGA 7 Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used as the default End Condition in all methods. When the **Go To Load** button on the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel. Scanning rates from 0.1°C/min to 200°C/min for a standard furnace or 0.1°C/min to 100°C/min for high temperature furnace can be used. The exact scanning rate used depends on the experiment you are performing and the end result you are trying to achieve.

Most typical TGA experiments use a rate ranging from 5°C/min to 50°C/min. However, you may want to heat or cool very rapidly to a selected temperature and then hold there isothermally or scan at a controlled rate. In such cases, very fast heating or cooling rates (e.g., 100°C/min to 200°C/min) are used to raise or lower the sample temperature very quickly.

Y Data

Low Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 130 mg.

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High Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 1300 mg.

Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (weight) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

Pyris 6 TGA Instrument Page

The Pyris 6 TGA Instrument page appears when you select the Pyris 6 TGA tab in the Preferences window. The Pyris 6 TGA Instrument page is available only when in Pyris 6 TGA Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used as the default End Condition in all methods. When the **Go To Load** button on the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel. Proper selection of the heating rate increases the efficiency of your analysis at the desired sensitivity. Slower heating rates improve peak resolution while faster heating rates improve the usable sensitivity. Heating rates greater than 40°C/min are usually used for rapidly heating or cooling the Pyris 6 TGA to the beginning temperature or the next program step or to selected isothermal temperatures in the temperature program. The maximum heating rate is 100°C/min.

Data

Display Language

Select the language in which the LCD display on the analyzer is to display its information: English, French, German, Italian, Japanese, or Spanish.

Ordinate Filter

The ordinate filter filters out noise from the Y signal (weight). Click on the box to allow data smoothing. There is a 12.5-second delay from the time an event occurs until the time it is observed on the screen when the Ordinate Filter is used.

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Furnace Constant

The furnace constant is calculated at the factory and is supplied on the Pyris 6 TGA Data Sheet shipped with the instrument. Enter the value from the sheet here.

Environment

Cooling Device

Select the cooling device attached to your Pyris 6 TGA from the drop-down list. The only available cooling device is circulating water. This parameter has no effect on the software; it is for information only.

Cooling Air Operating Temperatures

Cooling air to cool the furnace is provided by a small air pump mounted inside the instrument and is enabled and disabled by clicking on the **Cooling Air** button on the control panel. The Lower and Upper entries define the temperatures at which the pump will turn off and turn on. If the Cooling Air feature is enabled, if the furnace temperature is above the value entered in the Upper field, the pump starts. Once the furnace has cooled down to the temperature in the Lower field, the pump stops. The minimum for Lower is 0°C and the maximum for Upper is 1000°C.

DMA 7e Instrument Page

The DMA 7e Instrument page appears when you select the DMA 7e tab in the Preferences window. The DMA 7e Instrument page is available only when you are in DMA 7e Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used as the default End Condition in all methods. When the **Go To Load** button on the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel.

Load Static Force

Enter the default static force of the DMA 7e. When the **Go To Load** button is selected, not only does the analyzer heat or cool to the load temperature, it also applies the load conditions specified for force (or stress) and frequency. When entering a load static force value, take into consideration that the static force during a run should be low enough to prevent excessive bending or drift of the sample. This is measuring-system dependent. Too high a value can cause error in the phase angle. Static force should be maintained within analyzer limits throughout the entire analysis.

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Load Dynamic Force

Enter the default dynamic force of the DMA 7e. When the **Go To Load** button is selected, not only does the analyzer heat or cool to the load temperature, it also applies the load conditions specified for force (or stress) and frequency. When entering a load dynamic force take into consideration that the dynamic force during a run should be sufficient to produce a dynamic amplitude between 5 and 500 µm throughout the analysis. Dynamic force should be maintained within analyzer limits throughout the entire analysis.

Load Frequency

Enter the default frequency at which the forces are applied to the sample by the DMA 7e force motor. When the **Go To Load** button is selected, not only does the analyzer heat or cool to the load temperature, it also applies the load conditions specified for frequency and force. A frequency should be selected to produce a reasonable phase angle while avoiding resonant frequencies. Typical industry standards are between 1 and 11 Hz.

Data

Poisson's Ratio

This value in indicative of the volumetric changes exhibited by a material as it is stressed or strained by the DMA 7e. It is used to generate the shear moduli curves from data taken on a flexure measuring system.

System Damping Factor

This factor compensates for the loss modulus of the analyzer itself during a run. The default is 0.01 mN-s/mm.

Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (probe position) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

Phase Bias

This value is an offset to the phase signal.

Environment

Cooling Device

Select the cooling device attached to your DMA 7e from the drop-down list. Available cooling devices include Intracoolers 1 and 2, CCA 7, water bath, ice bath, and liquid nitrogen.

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TMA 7 Instrument Page

The TMA 7 Instrument page appears when you select the TMA 7 tab in the Preferences window. The TMA 7 Instrument page is available only when you are in TMA 7 Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used as the default End Condition in all methods. When the **Go To Load** button on the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel.

Load Static Force

Enter the default static force of the TMA 7. When the **Go To Load** button is selected, not only does the analyzer heat or cool to the load temperature, it also applies the load static force. When entering a load static force value, take into consideration that the static force during a run should be low enough to prevent excessive bending or drift of the sample. This is measuring-system dependent. Too high a value can cause error in the phase angle. Static force should be maintained within analyzer limits throughout the entire analysis.

Data

Poisson's Ratio

This value in indicative of the volumetric changes exhibited by a material as it is stressed or strained by the TMA 7. It is used to generate the shear moduli curves from data taken on a flexure measuring system.

Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (probe position) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

Environment

Cooling Device

Select the cooling device attached to your TMA 7 from the drop-down list. Available cooling devices include Intracoolers 1 and 2, CCA 7, water bath, ice bath, and liquid nitrogen.

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DTA 7 Instrument Page

The DTA 7 Instrument page appears when you select the DTA 7 tab in the Preferences window. The DTA 7 Instrument page is available only when you are in DTA 7 Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used as the default End Condition in all methods. When the **Go To Load** button in the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature or the user-specified temperature entered in the control panel.

Furnace Lock Temp

Enter the temperature at which the Furnace Lock engages. The furnace interlock mechanism prevents the furnace from being raised while at elevated temperatures. It remains locked until the Furnace Lock Temperature is reached. This minimizes exposure to any hot surface. The Furnace Lock is enabled and disabled by clicking on the **Furnace Lock** button on the control panel. The default Furnace Lock Temp is 55°C.

Data

Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (heat flow) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

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Pyris 1 TGA Instrument Page

The Pyris 1 TGA Instrument page appears when you select the Pyris 1 TGA tab in the Preferences window. The Pyris 1 TGA Instrument page is available only when in the Pyris 1 TGA Instrument Application.

Analyzer Constants

Load Temperature

Enter the default Load Temperature which is used in all methods as the default End Condition. When the **Go To Load** button on the control panel is selected, the analyzer automatically heats or cools to the load temperature.

Go To Temp Rate

Enter the rate at which the analyzer is to heat or cool in order to go to the load temperature, the temperature setpoint, or the user-specified temperature entered in the control panel. Scanning rates from 0.1°C/min to 200°C/min for a standard furnace or 0.1°C/min to 100°C/min for a high temperature furnace can be used. The exact scanning rate used depends on the experiment you are performing and the end result you are trying to achieve.

Most typical TGA experiments use a rate ranging from 5°C/min to 50°C/min. However, you may want to heat or cool very rapidly to a selected temperature and then hold there isothermally or scan at a controlled rate. In such cases, very fast heating or cooling rates (e.g., 100°C/min to 200°C/min) are used to raise or lower the sample temperature very quickly.

Y Data

The Pyris 1 TGA has three balance mechanism ranges. The Pyris software stores a calibration value for each of these ranges. When you change the selection here, a new set of values is sent to the analyzer.

High Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 1300 mg.

Low Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 130 mg.

Ultrasensitive Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 25 mg.

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Ordinate Filter Factor

This value is a measure of how much of the ordinate signal (weight) is filtered by the analyzer; 0 = no data smoothing, 1 = minimal data smoothing, 3 = normal data smoothing, and 6 = maximum data smoothing.

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Chapter 9
Pyris Player

The Pyris Player is used to create a play list of steps that control an analyzer with an autosampler. In general, a play list's steps tell the analyzer/autosampler to load a specific sample from the sample tray, start a method, unload the sample, and perform postrun analyses and display specified curves. Pyris Player can also be used with an instrument that does not have an autosampler. Play lists can be created that automate postrun activities.

The Pyris Player features are

- ability to perform other tasks while a play list is being run
- ability to edit the currently running play list
- complete history files generated to record success or failure of each item in a list
- ability to create a list of samples that are to be analyzed with the same method and then have the data files analyzed using the same data analysis list

The Pyris Player is accessed in Instrument Application or Data Analysis by clicking on the **Pyris Player** button  on the standard Pyris toolbar. The Pyris Player consists of six tabbed pages: Setup, Edit Play List, View List, View Sample List, View History, and View Sample History. In addition to the standard toolbar (see Chapter 6), Pyris Player also has its own toolbar.

Pyris Player Toolbars

When you enter Pyris Player, the standard toolbar seen in Instrument Viewer, Method Editor, and Data Analysis is replaced by a smaller toolbar and an additional toolbar whose buttons control the playback of the play list. These toolbars can be moved to other positions at the top of the screen. It is convenient to have them next to each other directly beneath the menu bar.

Pyris Player Standard Toolbar

The Pyris Player standard toolbar is as follows:



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Each button is described in Chapter 6, "Standard Toolbars" section.

Pyris Player Control Bar

The Pyris Player control bar appears below the standard toolbar when you open Pyris Player. This bar controls the playback of the play list.



Pyris Player Start Button 

Select this button to start the playback of the play list at the top of the list, i.e., from the first entry. When this button is selected, other buttons become available for selection and this button turns gray. Playback of the play list will start at the top of the list even if the focused line is not the first line of the play list.

Start at Current Step Button 

This button is selectable when the highlighted or current step is Prepare Sample, Data Analysis, Sample Group, or a Sample line in a Sample List of a Sample Group. If the highlighted step is any other type of entry, the button is grayed out.

Resume at Current Step Button 

Select this button to resume playback of the play list at the current step. The button becomes activated when you click on the **Pause Playback** button on the control bar.

Pause Button 

Select this button to pause the playback of the play list. You may want to change a sample, a magazine in a DSC autosampler, or the samples in the sample tray of the Pyris 1 TGA autosampler. The Pause command stops the playlist so you can give the appropriate commands from the Autosampler Control dialog box in order to change a sample, or you can edit a method that is below the step that is paused. Edit the method, select **Save** from the File menu, then click on the Resume at Current Step button.

Skip Ahead Button 

Select this button to skip to the highlighted step after the current step is complete. The highlighted step must be a main-level step, i.e., Prepare Sample or Data Analysis. If the highlighted step is not a main-level item, the play list will skip to the main-level step immediately preceding the highlighted step.

If your play list contains a Sample Group, the skipping rules are different: (1) You can skip into a Sample Group from another part of a play list; (2) you can skip out of one Sample Group into another Sample Group; (3) you can skip samples within a Sample List. The skipped samples are indicated in the View History page.

If a run is in progress when you click on **Skip Ahead**, you have to click on the **Stop Method** button to end the run. The playback of the play list will then continue at the highlighted item.

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Rules of Skip Ahead

- Items that remain in the current block when **Skip Ahead** is executed are not run and are marked as skipped in the View History page by a blue arrow .
- Items in a Sample List that follow the line that was executing with **Skip Ahead** is selected are skipped and are marked as such in the View History page.
- Items in a Sample List that are ahead of the highlighted step when **Skip Ahead** is selected are not run and are marked as skipped in the View History page.
- In order to use the **Skip Ahead** button, the Edit Play List page must be displayed when you click on the button.

Stop Now Button 

Select this button to stop playback of the play list. Data from all preceding runs is saved. If a method is running when you click on the **Stop Now** button, you will have to click on the **Stop Method** button on the control panel to stop the run.

Stop at End of Block Button 

Select this button to end the playback of the play list at the end of the current block. All of the remaining steps in the current block are played. A block is defined as all of the items under a main-level item: Prepare Sample, Data Analysis, and Sample Group. A Sample Group block encompasses all of the Sample List items and the generated items used to run them, i.e., Load Sample, Start Method, and Return Sample, and the Data Analysis List.

Pyris Player Setup Page

If you select Pyris Player while in an Instrument Application, the Player opened is for that instrument alone. You can open Pyris Player from another Instrument Application for another instrument on your system. For example, if you have a Pyris 1 DSC with an autosampler and a DSC 7 Robotic System as your Thermal Analysis system, you can have a play list for each instrument operating simultaneously.

If you are in Data Analysis Application when you select Pyris Player, use the Setup page to select the instrument with which you would like Player to be associated.

Analyzer Type

If you are in the Data Analysis Application when you click on the Pyris Player button on the toolbar, then the drop-down list for Analyzer Type will display all available instruments from which to select the desired instrument. When you go to the Edit Play List page, the last play list displayed for that instrument is seen. If you are in an Instrument Application, the only instrument listed in the drop-down list is that particular instrument.

Lot ID

If your laboratory groups samples into lots, this field is where you can record the lot number of the samples you are going to run with the play list. This is particularly useful for runs on an autosampler.

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Comment

You can enter free-form text describing what the playlist does in the Comment field. There is no limit to how long the comment can be.

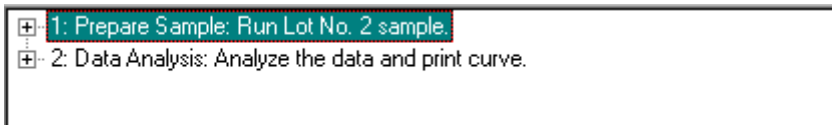
Pyris Player Edit Play List Page

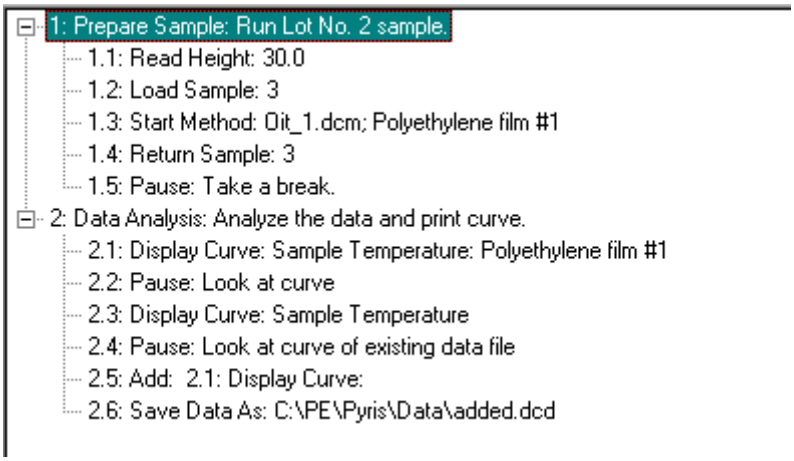
The Edit Play List page is where you can create and save play lists or open existing lists to edit or run. The Player Steps area shows the steps of the list as you add, insert, or delete steps using the buttons to the right. The area underneath the Player Steps area — the Edit Step area — contains fields in which you enter values to be used in the play list. Enter the values and then click on the step in the list in order for the new values to be displayed in the Player Steps area. There are three main levels in a play list: Prepare Sample, Sample Group, and Data Analysis. These main levels contain second-level entries. Your play list does not have to have all three. A typical play list may contain a Prepare Sample section and a Data Analysis section. Another list may be just Data Analysis procedures. A Sample Group comprises a Sample List and a Data Analysis list. The samples in a Sample List are similar and are run using the same method. The data are then analyzed using the same data analysis commands. Sample Groups are particularly useful with an autosampler.

The Edit Play List page contains the following items:

Player Steps Box

This area displays the steps of the play list as you add, insert, or delete them using the buttons to the right. The play list is initially displayed in expanded mode as it is created. You can compress parts of the play list by clicking on the minus signs next to the main-level items Prepare Sample, Sample Group, and Data Analysis. The second-level steps beneath will disappear and the minus sign will be replaced by a plus sign. You can see the play list alone and in its own window by clicking on the View Play List tab.





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Add a Step Button

Use this button to start creating a play list or to add a step to the end of an existing play list. The Player Step Options dialog box is displayed when you click on **Add a step**. The items available for adding to an existing play list will depend on the type of analyzer and what is already in the play list. A play list always starts out with either Prepare Sample, Sample Group, or Data Analysis.

Insert a Step Button

As soon as one step is in the Player Steps area, the **Insert a step** button becomes selectable. When you insert a step, it appears above the step that is highlighted when you select **Insert a step**. The items that appear in the Player Step Options dialog box for insertion depend on the main-level item immediately above the insertion point.

Delete this Step Button

Highlight the step you want to remove from the play list and click on **Delete this step**. If other steps in the play list are dependent on this step, a Dependencies List dialog box is displayed. For example, if you try to delete a Display Curve step, you may be reminded that an Add Curve step below it depends on that step.

Dependencies List Dialog Box

The Dependencies List dialog box is displayed when you select **Show Dependencies** from the View menu in Pyris Player and if you select **Delete step** in the Player Steps area. It shows all of the play list entries that are dependent on the focused play list item. For example, you may have a play list containing the following lines:

.
.
1.4: Start Method: . . .
.
.
.
2: Data Analysis: . . .
2.1: Display Curve:
2.2: Pause: Look at curve
2.3: Annotate Curve:
2.4: Derivative Curve:

The Dependencies List will contain the following lines if Start Method is the focused line:

2.1: Display Curve:
2.3: Annotate Curve:
2.4: Derivative Curve:

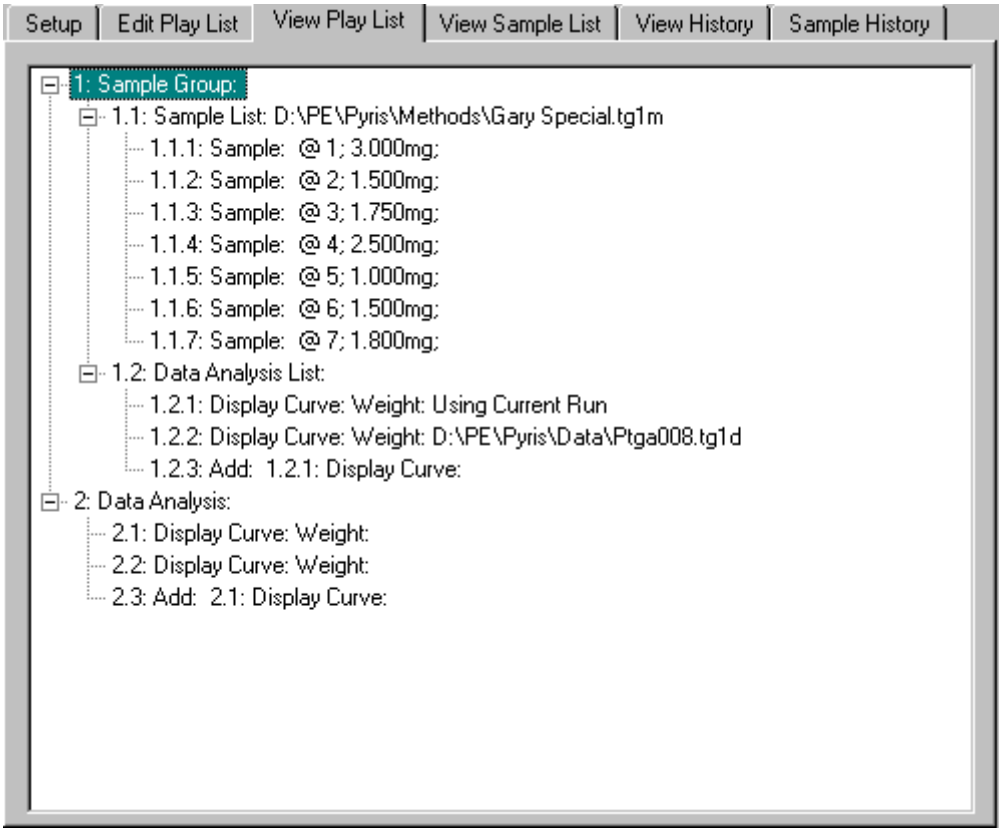
When trying to delete entries in the play list, this Dependencies List warns you that other entries in the play list depend on the presence of the line you want to delete.

Edit Step Section

Whenever an item is added to or inserted into the play list, an item-sensitive Edit Step section appears below the Player Steps section. The step number and item name are shown. Parameters specific to the play list item are displayed for editing. Each Edit Step section is discussed in detail below.

Pyris Player View Play List Page

The Pyris Player View Play List page shows the current play list only. This view is useful for displaying the play list in compressed or expanded form. Since there are no buttons or an Edit Step area, the play list has more room for display. This makes it easier to read and to get a better overview of what the play list is doing. Use the "+" and the "–" symbols to the left of the first-level items (numbered 1, 2, 3, etc.) to expand or compress the list beneath that item. Prepare Sample, Sample Group, and Data Analysis are like folders that contain additional folders and/or steps.



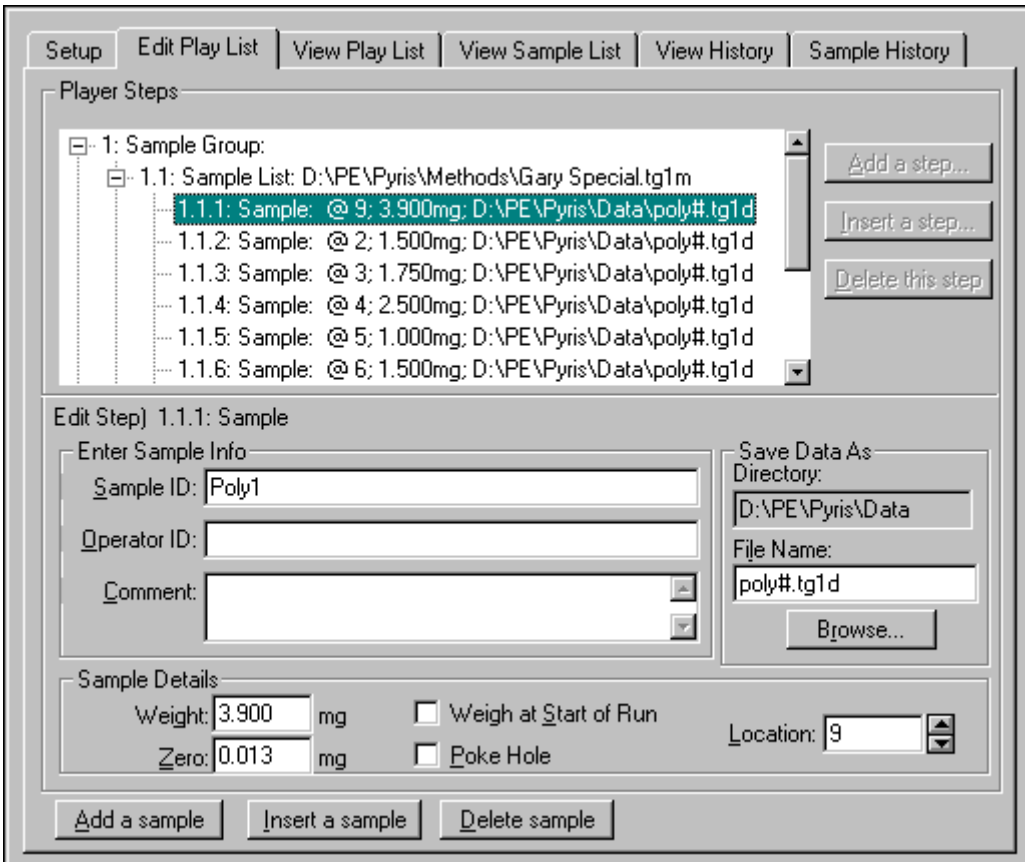
Pyris Player View Sample List Page

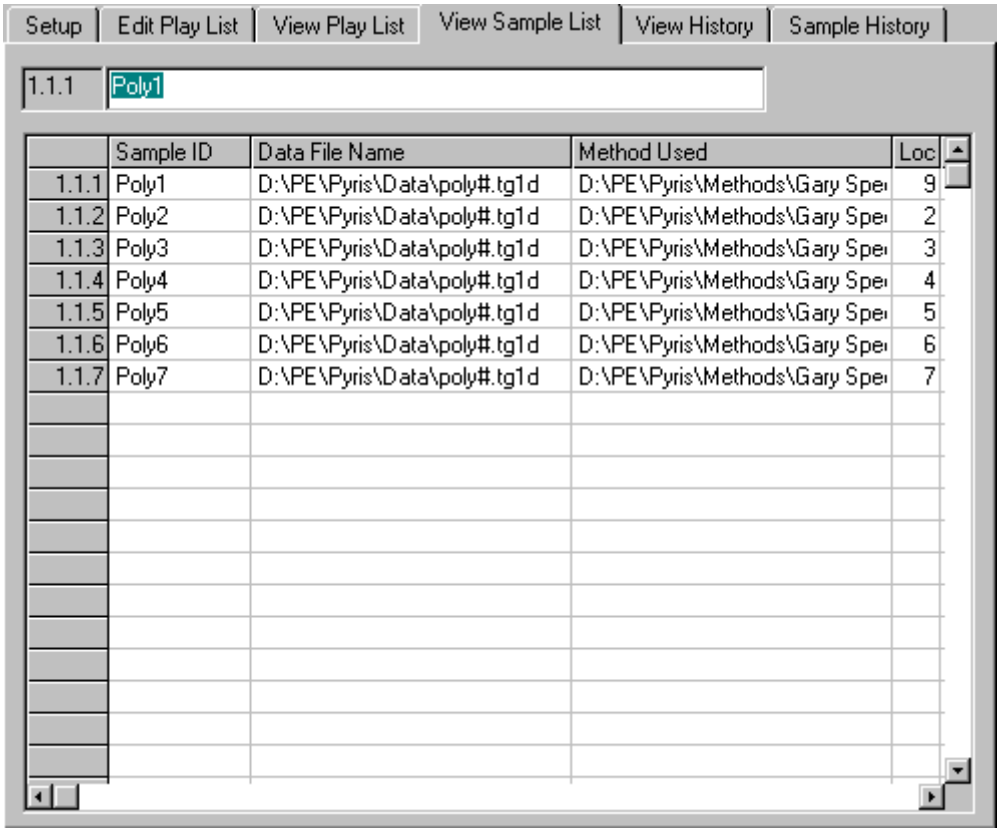
The View Sample List page is a more compact display of the samples to be run in your play list. You can edit the information about a sample or samples in this list rather than on the Edit Play List page. Data in every column can be edited except for the Method Used column for samples in a

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Sample List as part of a Sample Group. Since the method used applies to all the samples in the Sample List, you cannot change the method for a particular sample. You would need to change the method for the Sample List in the Edit Play List page.

A play list in the Edit Play List page and its associated View Sample List page are shown below:



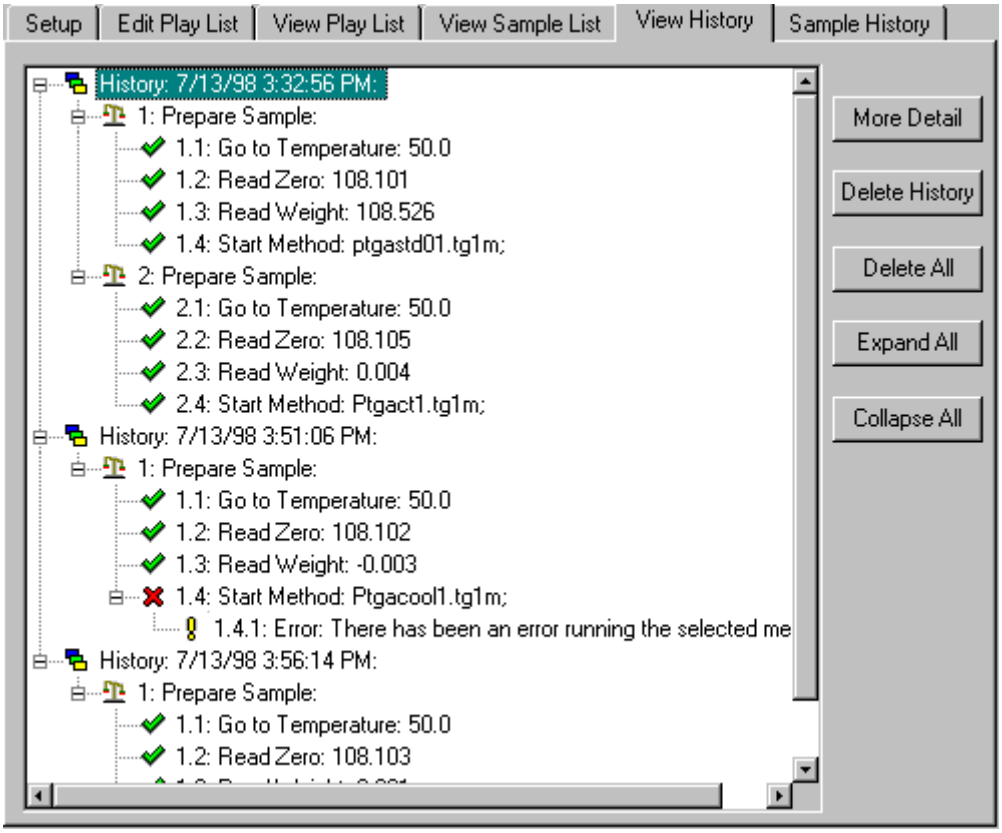


Place the cursor in a field on any sample line and that value is displayed in the display field above the sample list. There you can change the value. When in the Sample File Name or Method Used field, a **Browse** button appears next to the display field. If you want to change the directory in which the data file will be saved, click on the **Browse** button to display the Save in dialog box to change the path. For a method, when you click on the **Browse** button, the Look in dialog box is displayed in which you can look for and select another method to use.

Pyris Player View History Page

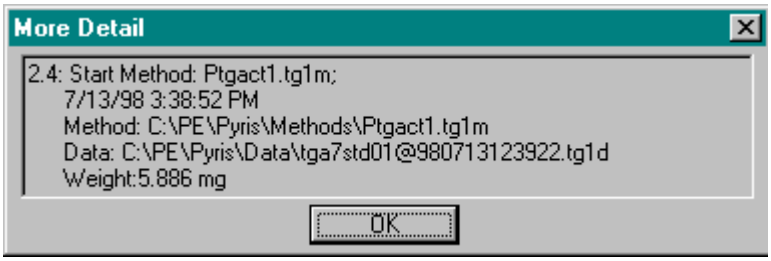
The View History page of Pyris Player lists the history of the playback of the play list. It is also a real-time update list so as a play list is in playback mode, the history list is dynamically revised to reflect the status of a play list step. There is a history entry, indicated by the symbol , for each time the play list is run, with the date and time of the run displayed. Click on the "+" to the left of History entry to "open up" the history folder; this displays the main-level entries of the play list: Prepare Sample, Sample Group, and Data Analysis. Click on the "+" next to those entries to open the folders and display the second-level steps in the folders. If there is a green checkmark before the step, the operation was performed successfully. If appears in front of a step, the operation failed. Double click on the "+" to the left of the to display an additional Error line that explains why the playback of the play list entry failed. A blue down arrow indicates that the step was skipped. A Pause is indicated by a blue flag .

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More Detail

Click on this button with a history entry highlighted to display a dialog box showing the date and the time that the entry was executed. Some entries' More Detail dialog boxes will contain additional information. For example, for a Start Method entry, the following dialog box is seen:



Delete History

You can delete a history entry by clicking on this button. The focused line must be a History line when you select this feature. You are warned that the entire block, i.e., all the entries associated with that run, will be deleted. You can cancel the deletion request by clicking on the **Cancel** button.

Delete All

This button will delete all of the history entries in the View History page. You are warned that the entire History List will be deleted and are given the chance to cancel the request.

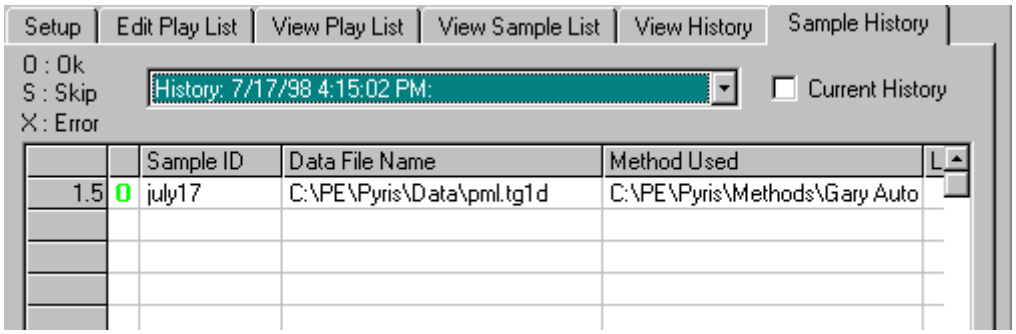
Expand All/Collapse All

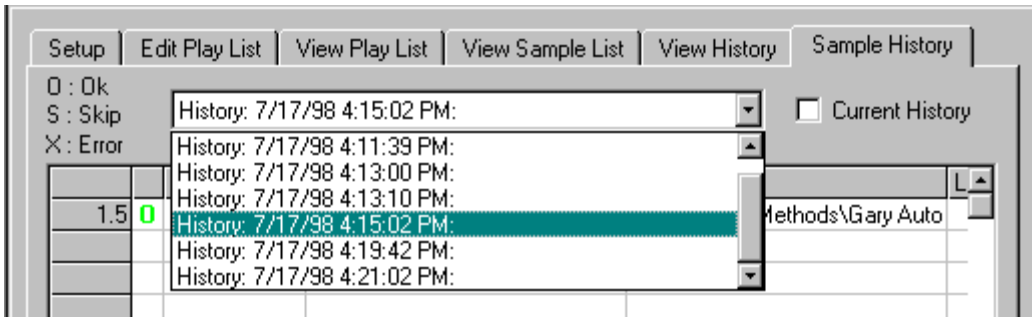
These buttons toggle the display of the History List between full view and collapsed view. The sample History List shown above is expanded, i.e., all entries for all History files are displayed. The same History List collapsed is as follows:



Pyris Player Sample History Page

The history of the samples run by the current play list can be displayed in the Sample History page. This page can also be used to dynamically display the current run's history. To select which History file to display, click on the drop-down arrow in the display field above the sample listing to show all of the History files for the play list.





To show the current run's history, click in the **Current History** check box.

The first column in the Sample History page displays the item number in the play list that generated the data file. The second column displays the status code for the sample in the History file. If the sample ran with no errors, an **O** is displayed. If the sample was skipped for any reason, e.g., there was no sample pan in the designated autosampler location, an **S** is displayed. If there was an error running the sample, e.g., there was a problem with the selected method, an **X** is displayed.

The remaining columns are similar to those in the View Sample List page: the data file name, method used, location (relevant for autosamplers), and Detail Description. If there was an error, this field displays the error message generated. If the run was OK, this field displays the sample weight. These fields cannot be edited.

Player Step Options Dialog Box

This dialog box is displayed when you select **Add a step** or **Insert a step** in the Edit Play List page. When you begin to create a play list and the Play Steps area is blank, the only items in the Player Step Options dialog box are (1) Prepare Sample, (2) Data Analysis, and (3) Sample Group. The options listed subsequently depend on the main-level item immediately above where the line is to be added or inserted. They also depend on the analyzer. When you add a step from anywhere in the play list, it is appended to the end of the list. When you insert a step, it is inserted above the highlighted step. In this case, the items in the Player Step Options dialog box depend on the main-level item immediately above the insertion point.

When you select an item, the Edit Step area below the Player Steps area changes accordingly.

The following items appear in the Player Step Options dialog box for all analyzers:

- Prepare Sample
- Data Analysis
- Sample Group
- Pause
- Load Sample
- Start Method
- Return Sample
- Change Calibration
- Go To Temperature
- Display Curve

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The following additional items are available for the play list for the analyzers indicated:

- Read Height (DMA 7e and TMA 7)
- Read Zero (DMA 7e, TMA 7, all TGAs, DTA 7)
- Read Weight (all TGAs)
- Raise Furnace (TGA 7 and Pyris 1 TGA)
- Lower Furnace (TGA 7 and Pyris 1 TGA)
- Cool Furnace (TGA 7 and Pyris 1 TGA)
- Load Reference (Pyris 1 DSC)
- Return Reference (Pyris 1 DSC)

If Data Analysis is the last main-level item in the list when you select **Add a step** or it is above the highlighted item when you select **Insert a step**, then **Display Curve** is an option in the Player Step Options dialog box.

If Display Curve is the last item in the list when you select **Add a step** or it is above the highlighted item when you select **Insert a step**, then the following items are listed in the Player Step Options dialog box:

- Math Options
- Calculation Options
- Rescale Options
- Delete Curve
- Copy to Clipboard
- Save Data As
- Save All
- Print
- Pause
- Run Program

Each of these play list items is described in detail below, including its Edit Step area.

Prepare Sample

Prepare Sample is a main-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button. Typically, you would select this entry to start a new play list. Once this item is selected, the choices in the Player Edit Options list expand to include analyzer-specific steps such as Read Zero, Read Weight, Raise Furnace, or Close Cover. You can enter a Prepare Sample command anywhere in an existing play list. If you are in a Sample Group and you want to insert Prepare Sample, the entry will appear above the Sample Group entry.

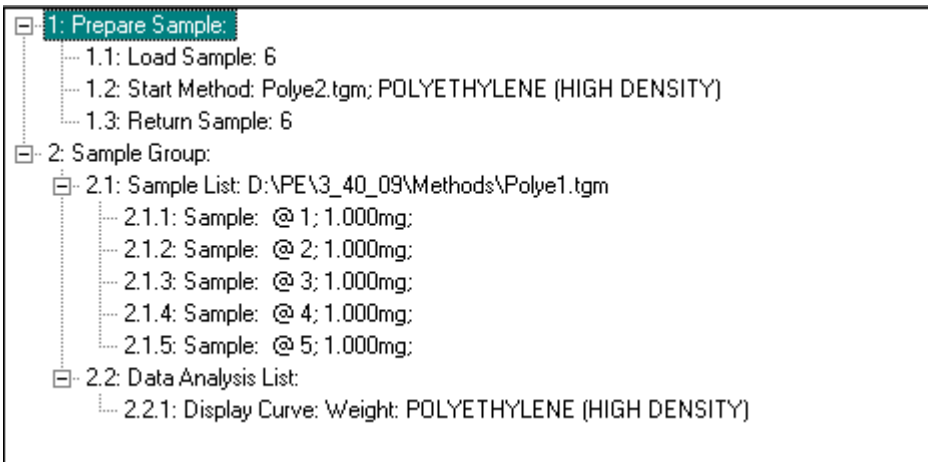
When Prepare Sample is added to the play list, the Edit Step area displays a Comment field in which you enter any free-form text that may describe what the play list will do or the types of samples will be run. The comment can be a maximum of 80 characters.

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Data Analysis

Data Analysis is a main-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button. This entry is very useful for a play list for an instrument that does not have an autosampler but where the play list will be used to perform postrun analyses. This entry would typically be followed by Display Curve and then additional items that perform operations on a curve or curves. The curves can be those created by playing back the play list or existing data files. Data Analysis includes the same options that appear on the Math and Calc menus and Rescale toolbar in the non-Player Data Analysis, e.g., Add, Subtract, Peak Area, Delta Y, etc.

A Sample Group has its own Data Analysis List section. The only difference between Data Analysis in a regular play list and the Data Analysis List is the data files available for selection. For a Data Analysis List, you can select a play list item, an existing file, or the data from the current run. For a regular Data Analysis, the curves available for analysis are existing files or data files collected by the play list above the current location in the play list. However, you cannot select a curve from a preceding line in a Sample List. For example, in the play list below, when Display Curve is selected for the Data Analysis List and you click on the Select Existing File radio button, the only file displayed in the file drop-down list is the file created in step 1.2:



When Data Analysis is added to the play list, the Edit Step area displays a Comment field in which you can enter any free-form text that may describe the Data Analysis that you are going to set up. The comment can be a maximum of 80 characters.

Edit Step: Comment

For Prepare Sample, Data Analysis, or Data Analysis List in Sample Group, the Edit Step area displays a Comment box in which you can enter free-form text describing the play list step. You can enter multiple lines of text. When you reach the end of a line, simply keep typing; the text will wrap to the next line automatically. You also can press **Enter** to start a new line. The Comment field is a convenient area in which to describe the group of lines that make up Prepare Sample or Data Analysis, e.g., the type of samples to be run and the results you expect.

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Sample Group

Sample Group is a first-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button. When Sample Group is added to a play list, second-level entries Sample List and Data Analysis List are automatically added. The Edit Step area contains a Comment field, an **Add a sample** button and an **Advanced** button. These are discussed below in the Edit Step sections relevant to Sample Group, Sample List, and Sample.

Edit Step: Sample Group

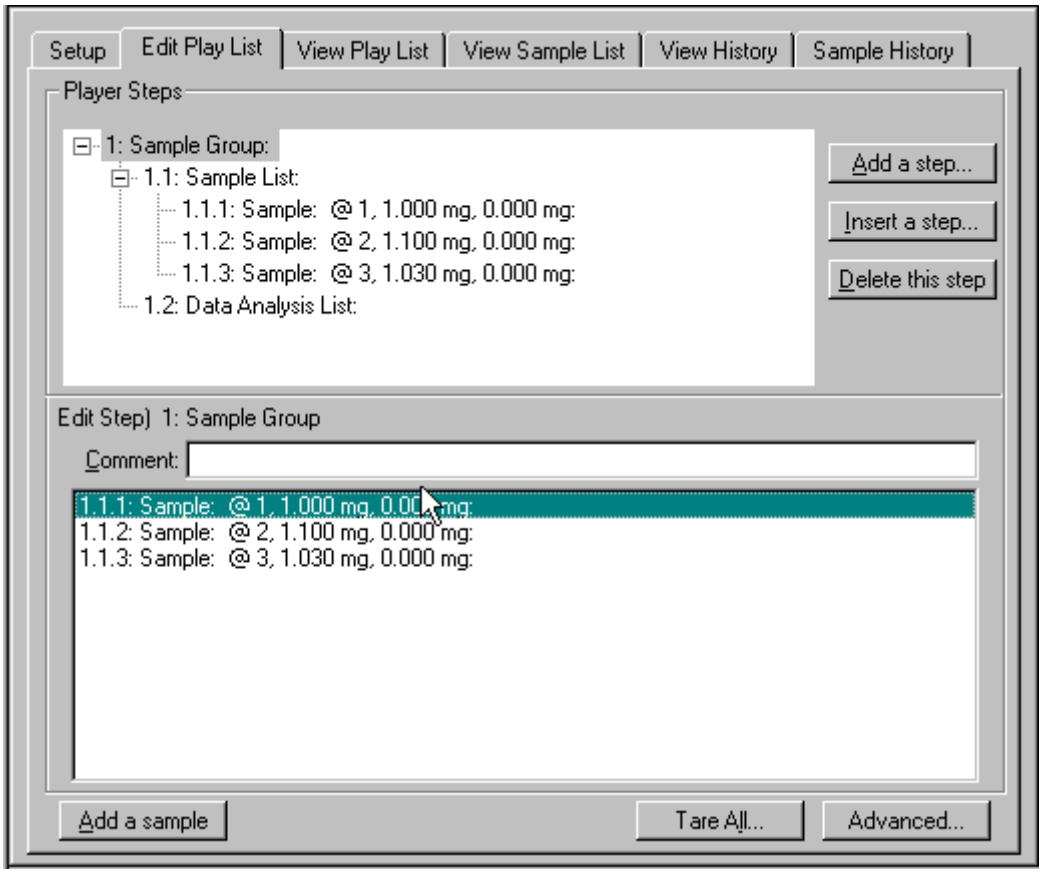
When you select Sample Group from the Player Step Options dialog box, the Player Steps area displays the default items of a Sample Group:

1: Sample Group:

1.1: Sample List:

1.2: Data Analysis List:

The Edit Step area, which consists of a Comment field and a large blank area, is empty. As soon as you begin to add samples to the Sample List, the samples are listed in the blank area:



Comment

Enter free-form text describing the sample group about to be created. The entry appears next to Sample Group in the Player Steps area. Note that the text does not wrap when it reaches the end of the Comment box but keeps scrolling to the left.

Add a sample

Nothing is displayed in the blank area until you add a sample to the list. When you click on **Add a sample**, the Edit Step: Sample area is automatically displayed and a Sample line with default values is added to the Sample List. To return to the Edit Step: Sample Group display, click on the Sample Group line in the Play Steps area. The Sample line is displayed in the Sample List and also in the list of samples below the Comment box.

Tare All button

With the Sample Group line highlighted, the **Tare All** button is displayed for Pyris 1 TGA and Pyris 6 TGA autosampler systems. If you click on this button before entering any samples in the Sample List, the Advanced Tare Options dialog box (see below) appears. From that box you can select how you want crucibles or sample pans in the autosampler to be tared with respect to the Sample List and how the tray is filled. From that dialog box the TGA Tare/Weigh System dialog box (see below) or the Pyris 6 AS Tare/Weigh dialog box (see below) is displayed as the system performs the steps to tare each crucible.

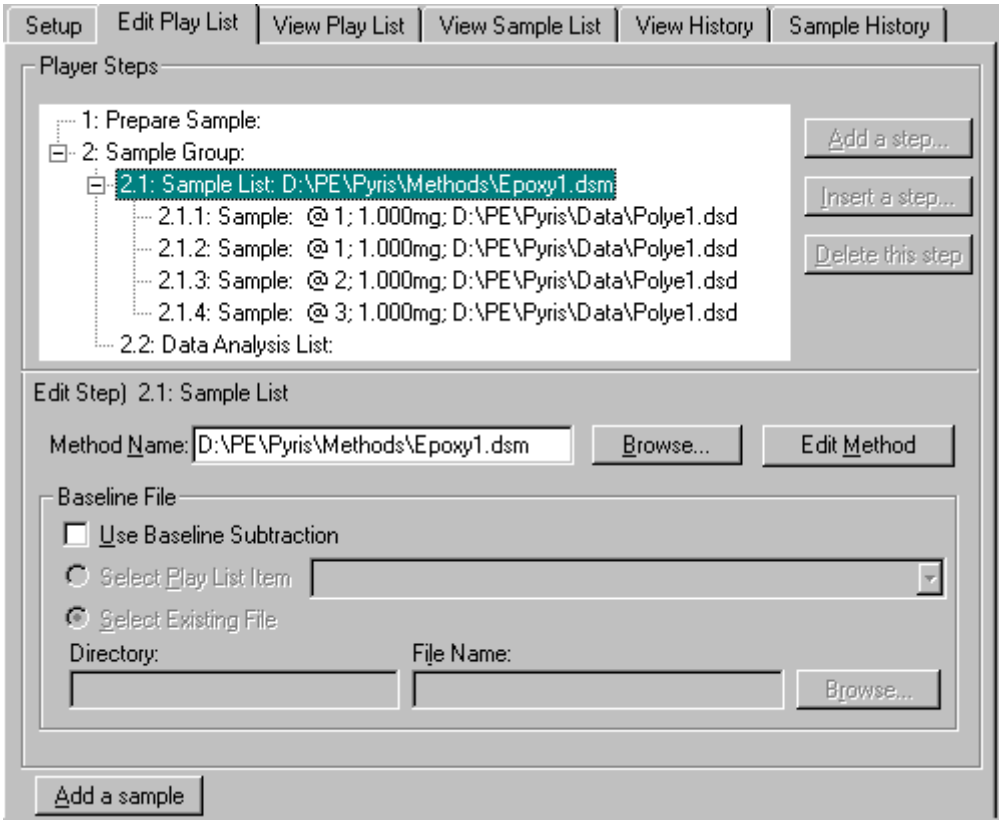
Advanced button

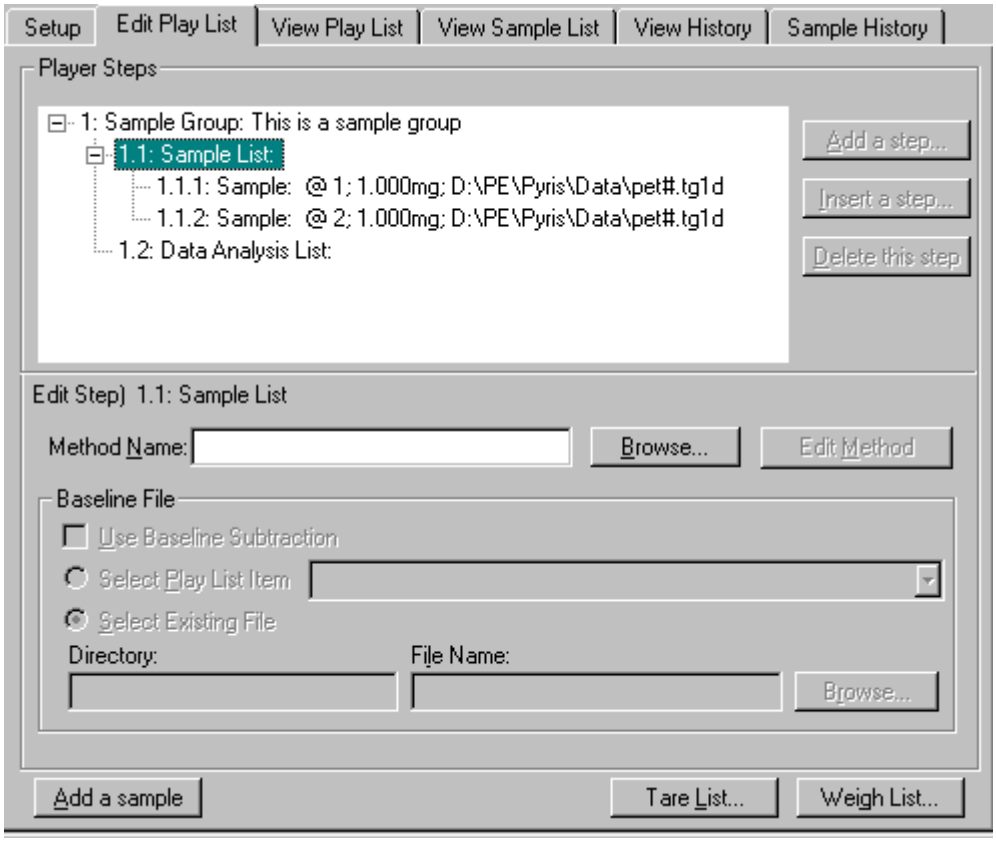
With the Sample Group line highlighted, the **Advanced** button is displayed for Pyris 1 TGA and Pyris 6 TGA autosampler systems. Click on this button to access the Furnace Burnout dialog box. Furnace burnout is used to burn off volatile residue in the furnace after a specified number of samples have been run.

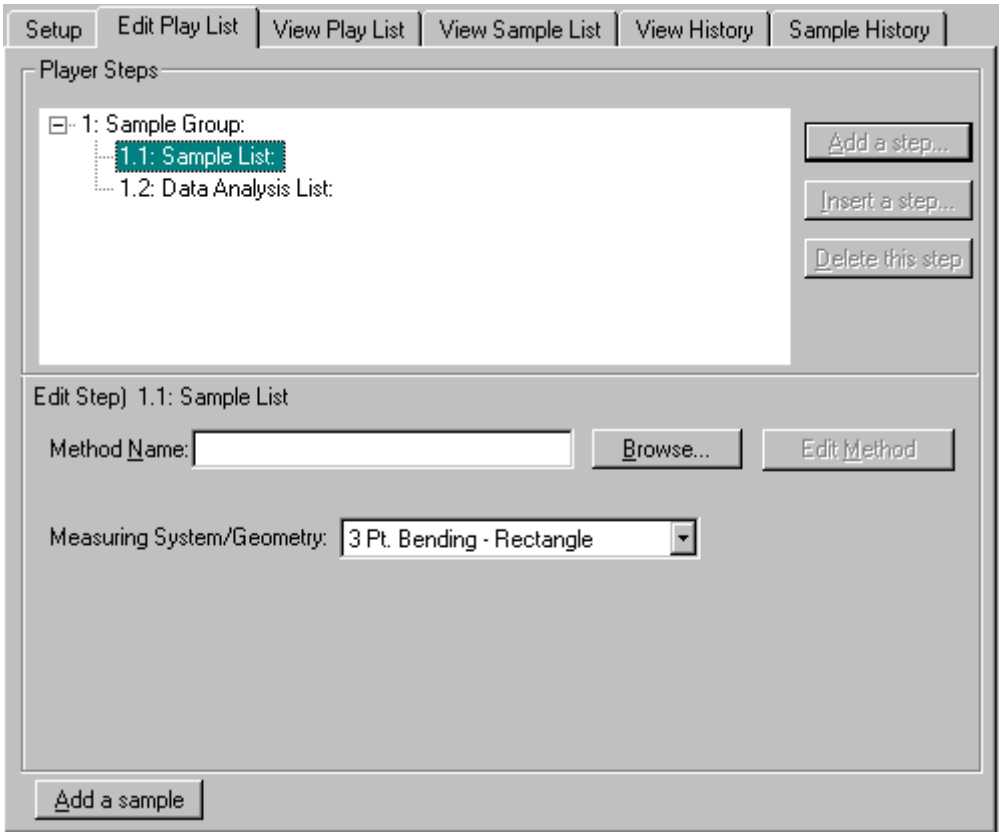
Edit Step: Sample List

When you click on the Sample List line in the Sample Group, the Edit Step: Sample List area is displayed. The areas for a DSC, Pyris 1 TGA, and DMA 7e analyzer are shown below:

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Setup
Edit Play List
View Play List
View Sample List
View History
Sample History
Player Steps
1: Prepare Sample:
2: Sample Group:
2.1: Sample List: D:\PE\Pyris\Methods\Epoxy1.dsm
2.1.1: Sample: @ 1; 1.000mg; D:\PE\Pyris\Data\Polye1.dsd
2.1.2: Sample: @ 1; 1.000mg; D:\PE\Pyris\Data\Polye1.dsd
2.1.3: Sample: @ 2; 1.000mg; D:\PE\Pyris\Data\Polye1.dsd
2.1.4: Sample: @ 3; 1.000mg; D:\PE\Pyris\Data\Polye1.dsd
2.2: Data Analysis List:
Add a step...
Insert a step...
Delete this step
Edit Step) 2.1: Sample List
Method Name: D:\PE\Pyris\Methods\Epoxy1.dsm
Browse...
Edit Method
Baseline File
Use Baseline Subtraction
Select Play List Item
Select Existing File
Directory:
File Name:
Browse...
Add a sample


Setup
Edit Play List
View Play List
View Sample List
View History
Sample History
Player Steps
1: Sample Group: This is a sample group
1.1: Sample List:
1.1.1: Sample: @ 1; 1.000mg; D:\PE\Pyris\Data\pet#.tg1d
1.1.2: Sample: @ 2; 1.000mg; D:\PE\Pyris\Data\pet#.tg1d
1.2: Data Analysis List:
Add a step...
Insert a step...
Delete this step
Edit Step) 1.1: Sample List
Method Name:
Browse...
Edit Method
Baseline File
Use Baseline Subtraction
Select Play List Item
Select Existing File
Directory:
File Name:
Browse...
Add a sample
Tare List...
Weigh List...



Method Name

Displays the full path, i.e., drive and directory, of the method selected. If you have added a sample to the list by clicking on Add a sample in the Sample Group area and then click on Sample List, the following message is displayed: "The method for running the sample list was not specified." Click **OK** to clear the message. Select your method by using the Browse button; in the dialog box, find the file on your system. The file name is displayed following the drive and directory.

Edit Method button

Click on this button to access the Initial State and the Program pages. The fields in these pages are exactly the same as for a method in Method Editor.

Baseline File

If you want to use baseline subtraction, i.e., the program will subtract the baseline file from the data file before saving it, click on the Use Baseline Subtraction box. Once you activate the feature, the Directory and File Name fields become available. You must select an existing baseline file by using the Browse button. You cannot use a playlist item for a baseline file (the selection is grayed out).

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Measuring System/Geometry (DMA/TMA only)

From the drop-down list displayed when you click on the down arrow, select the measuring system that you are using in the analyzer and the associated geometry of the sample.

Add a sample button

Use this button to add a sample to the end of an existing sample list. A line is added to the Sample List, with the line number incremented. The location in the Edit Step area is incremented also. The Edit Step: Sample area is displayed.

Tare List button (Pyris 1 TGA and Pyris 6 TGA autosampler systems only)

Use this button to have the system tare all of the empty crucibles or sample pans in the locations used in the sample list. The TGA Tare/Weigh System dialog box or the Pyris 6 AS Tare/Weigh System dialog box is displayed and the steps necessary to tare each crucible or pan are performed automatically. The Zero value for each location is filled in automatically in the Sample List.

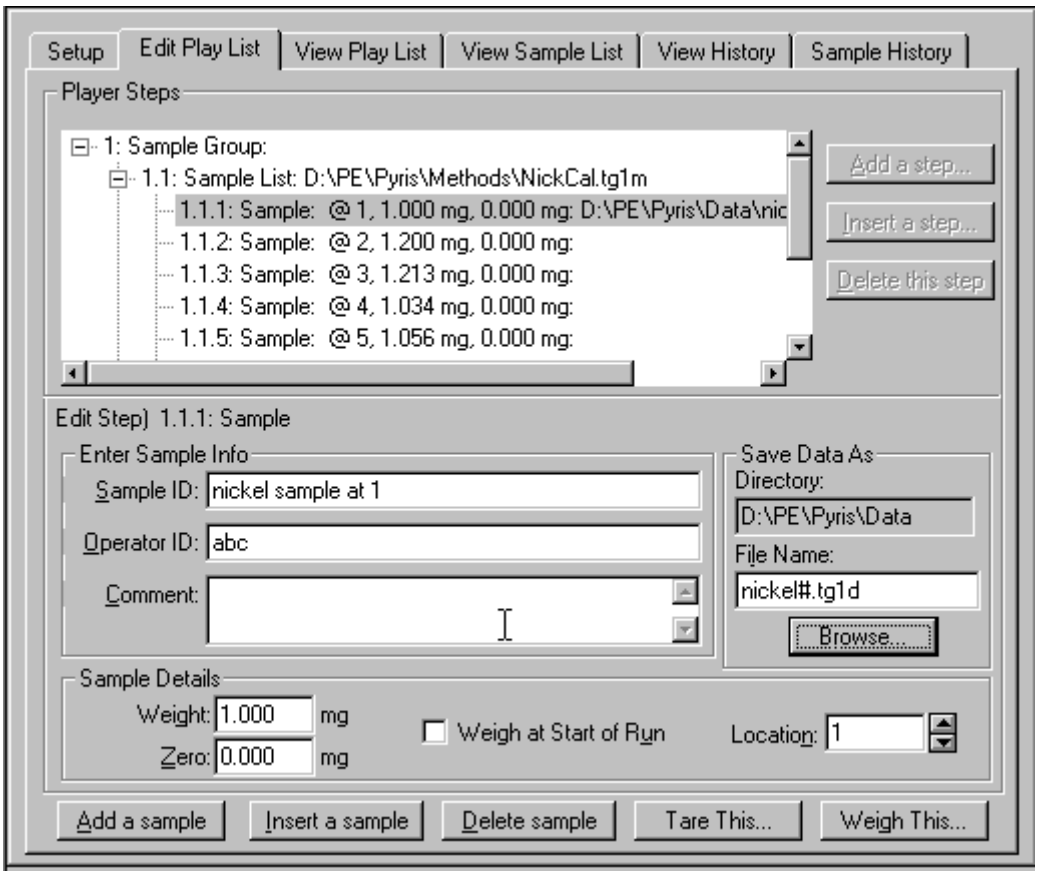
Weigh List button (Pyris 1 TGA and Pyris 6 TGA autosampler systems only)

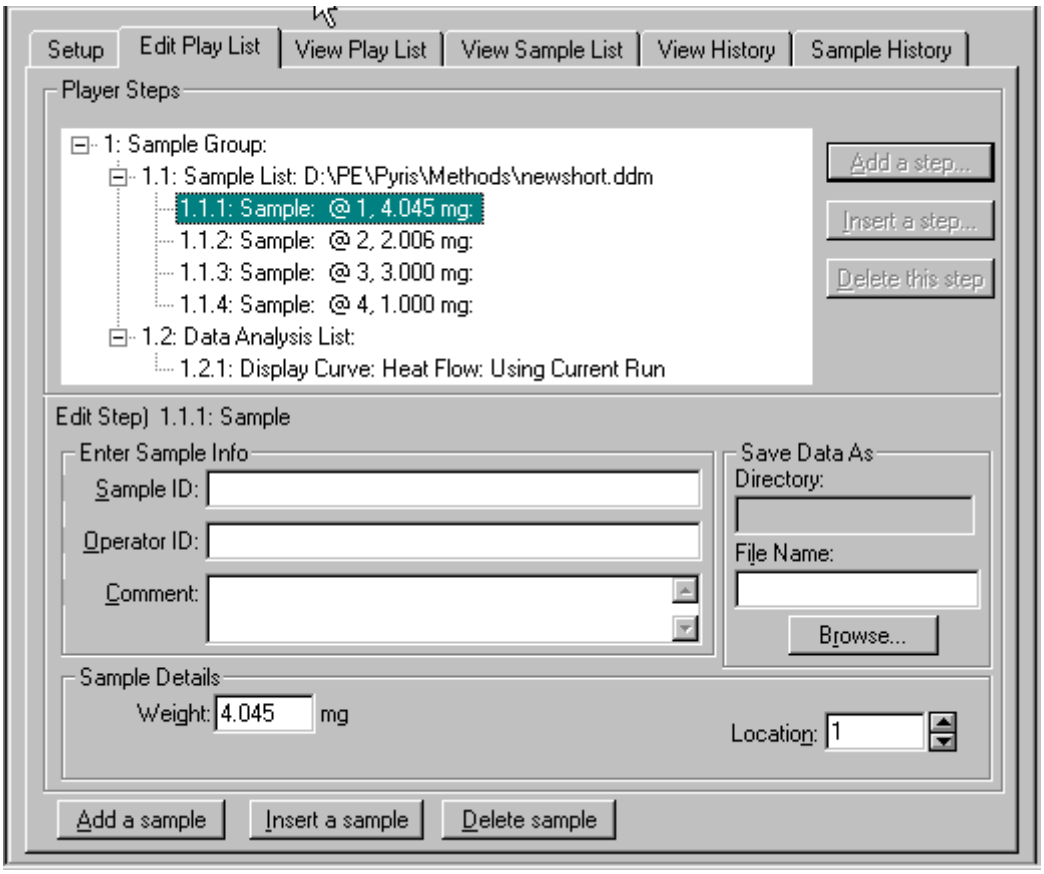
Use this button to have the system weigh the samples in the crucibles at the locations specified in the sample list. The TGA Tare/Weigh System dialog box or the Pyris 6 AS Tare/Weigh System dialog box is displayed and the steps necessary to weigh each sample are performed automatically. The Weight value for each sample in the sample list is filled in automatically.

Edit Step: Sample

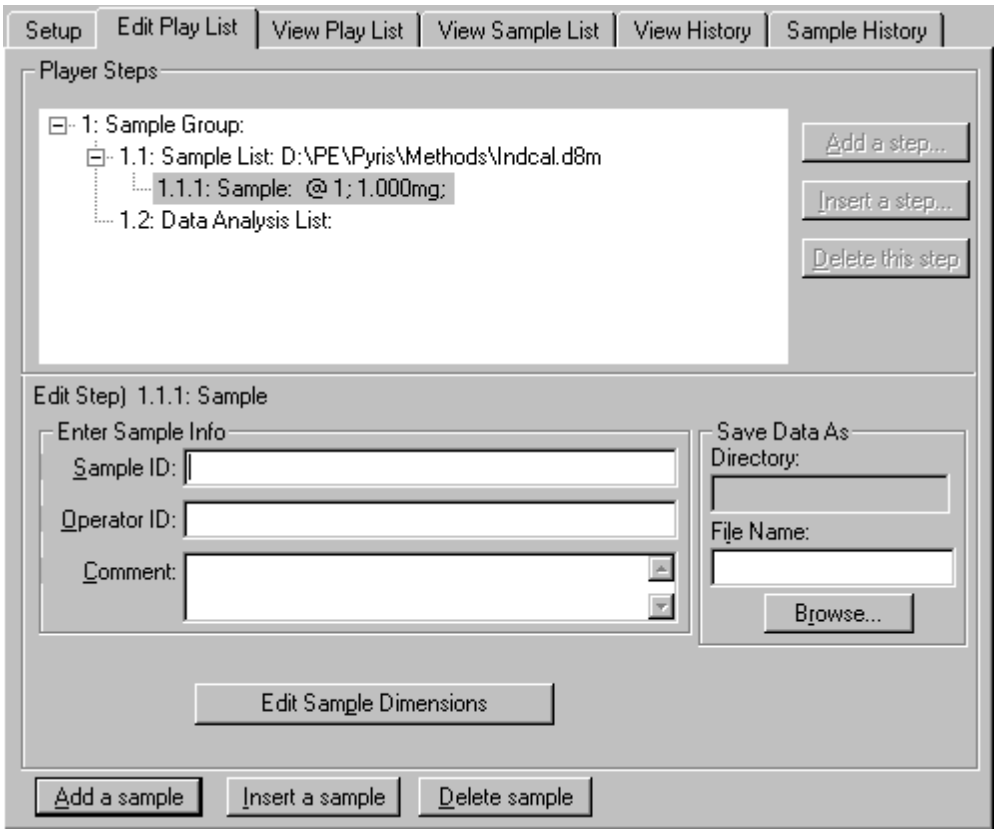
The Edit Step: Sample area is displayed when you click on **Add a sample** at the Sample Group window or the Sample List window. It is also displayed when the focused line is a Sample line in a Sample List. The items in this area vary with the analyzer you are using. Displayed below are the Edit Step: Sample areas for the Pyris 1 TGA with autosampler, a Pyris 1 DSC, and a DMA 7e, respectively:

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Setup
Edit Play List
View Play List
View Sample List
View History
Sample History
Player Steps
1: Sample Group:
1.1: Sample List: D:\PE\Pyris\Methods\NickCal.tg1m
1.1.1: Sample: @ 1, 1.000 mg, 0.000 mg: D:\PE\Pyris\Data\nic
1.1.2: Sample: @ 2, 1.200 mg, 0.000 mg:
1.1.3: Sample: @ 3, 1.213 mg, 0.000 mg:
1.1.4: Sample: @ 4, 1.034 mg, 0.000 mg:
1.1.5: Sample: @ 5, 1.056 mg, 0.000 mg:
Add a step...
Insert a step...
Delete this step
Edit Step) 1.1.1: Sample
Enter Sample Info
Sample ID: nickel sample at 1
Operator ID: abc
Comment:
Save Data As
Directory:
D:\PE\Pyris\Data
File Name:
nickel#.tg1d
Browse...
Sample Details
Weight: 1.000 mg
Zero: 0.000 mg
Weigh at Start of Run
Location: 1
Add a sample
Insert a sample
Delete sample
Tare This...
Weigh This...



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Enter Sample Info

Sample ID

Enter a sample name of up to 40 characters. The entry field scrolls horizontally as you type.

Operator ID

Enter the name of the person who will run the experiment; the name can be up to 40 characters. The entry field scrolls horizontally as you type.

Comment

Enter any additional information about the sample or run; the entry can be a maximum of 160 characters. The entry field automatically wraps horizontally and scrolls vertically. Start typing on a new line by pressing **Enter**.

Save Data As

File Name

This field is for the name of the file in which the data collected from the run will be saved. You can enter the file name directly in this field or in the Browse dialog box. A default file name is used if you do not specify a file name. The default name is taken from the entry made in the Save Preferences page. You can also use incremental file names, e.g., if you enter File# in the File Name field, the data file saved is FileXXX, where XXX starts at 001 and is incremented each time the method is used. This feature is useful when creating a Sample List.

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If when running the play list it is found that a file name already exists, the system will add a date/time stamp at the end of the new file name to distinguish it from the existing file.

Browse

If you want to select a directory other than the default (selected in the Save Preferences page) for storing the data file, click on the **Browse** button to display the Browse dialog box.

Directory

This field displays the drive and directory in which the data are saved after the run. This value reflects the disk\directory chosen in the Browse dialog box. If you do not select a specific directory, the default will be displayed.

Sample Details

Weight

For a DSC or TGA analyzer, you can enter the weight of the sample in milligrams. The default is 1.000 mg. If you have a Pyris 1 TGA or a Pyris 6 TGA with an autosampler, you can use the **Weigh This** button to have the instrument weigh the sample in the specified location of the autosampler. When you click on this button, the TGA Tare/Weigh System dialog box (see below) or the Pyris 6 AS Tare/Weigh System dialog box (see below) is displayed and the steps necessary to weigh the sample are performed automatically. The Weight value is entered into the Sample line of the Sample List automatically

Zero (TGA only)

The zero weight value is read directly from the analyzer by selecting the **Zero Weight** button in the control panel; the value appears in the Zero field. The default value is 0.000 mg. This value is the weight of the empty sample pan either on the hangdown wire for the TGA 7 or Pyris 1 TGA or on the sample holder for the Pyris 6 TGA. The value is used to tare the weight of the sample pan before loading the sample. If you have a Pyris 1 TGA or a Pyris 6 TGA with an autosampler, you can use the **Tare This** button to have the instrument weigh the empty crucible or sample pan in the specified location of the autosampler. When you click on the button, the TGA Tare/Weigh System dialog box or the Pyris 6 AS Tare/Weigh System dialog box is displayed and the steps necessary to tare the crucible or pan are performed automatically. The Zero value is entered into the Sample line of the Sample List automatically.

Weigh at Start of Run (TGA only)

Click on this box to have the weight of the sample determined immediately before the run begins. This is useful if the sample contains volatiles. There may be some evaporation between weighing the sample and the start of the run if the weight is taken at the creation of the play list.

Location

Indicates the position in the autosampler from which to take the sample for the run. If your analyzer does not have an autosampler, this field is irrelevant.

Edit Sample Dimensions (DMA/TMA only)

This button displays the Player Sample Dimensions dialog box.

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Add a sample button

Use this button to add a sample to the end of an existing sample list. A line is added to the Sample List, with the line number incremented. The location in the Edit Step area is incremented also. This can be edited if you want to use a sample from another position in the autosampler. All other items in the Edit Step: Sample area can be edited. The empty crucible in the new location can be tared and the sample weight can be determined using the **Tare This** and **Weigh This** buttons.

Insert a sample button

As soon as one sample is in the Sample List, the **Insert a sample** button appears. The inserted sample appears above the sample that was highlighted when you selected this button. The line numbers are adjusted accordingly and the location of the sample (indicated by the @X in the Sample line) takes on the same value as that of the sample above the inserted line. If your analyzer has an autosampler, you may need to edit the location number if it is different than where the sample is located in the tray.

Delete sample button

Highlight the sample you want to remove from the Sample List and click on **Delete sample**. If a following Data Analysis step (but not Data Analysis List step) depends on this sample, a Dependencies List dialog box is displayed. For example, if you try to delete a Display Curve step, you may be reminded that an Add Curve step below it depends on that step. The Data Analysis List items are not sample-dependent, i.e., you cannot add two curves from the sample list together. You have to create a Data Analysis section following the Sample Group and include and calculations that use two or more of the sample list items.

Tare This button

This button is relevant to Pyris 1 TGA and Pyris 6 TGA autosampler systems. When you click on this button, the TGA Tare/Weigh System dialog box or the Pyris 6 AS Tare/Weigh System dialog box is displayed. The system automatically performs the steps necessary to tare the empty crucible or sample pan in the specified autosampler location.

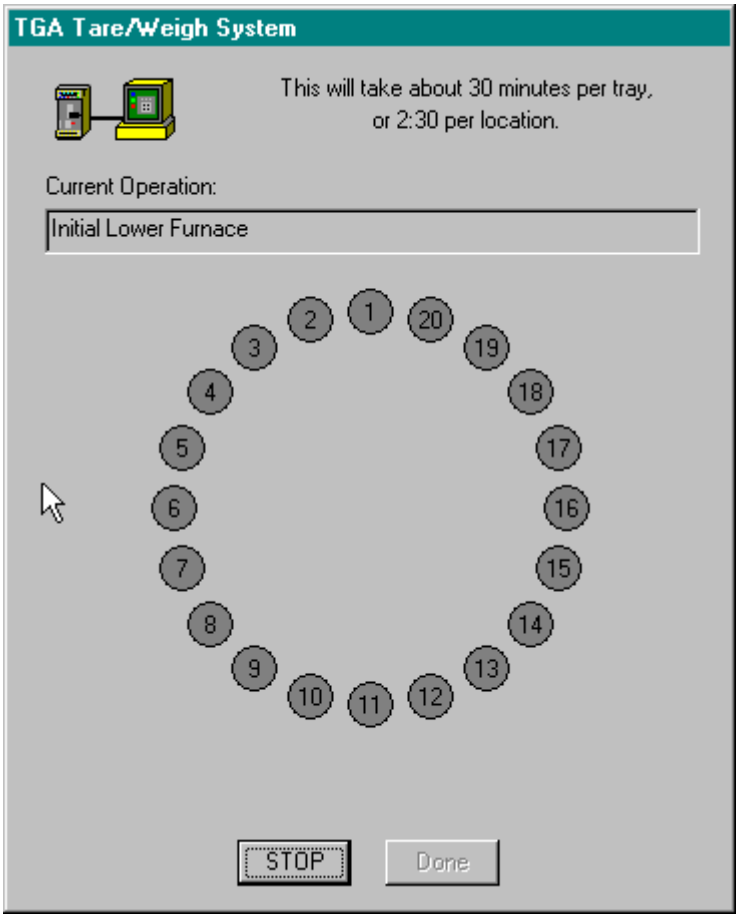
Weigh This button

This button is relevant to Pyris 1 TGA and Pyris 6 TGA autosampler systems. When you click on this button, the TGA Tare/Weigh System dialog box or the Pyris 6 AS Tare/Weigh System dialog box is displayed. The system automatically performs the steps necessary to obtain the weight of the sample in the crucible or sample pan at the specified location.

TGA Tare/Weigh System

When you click on the **Tare List** or **Weigh List** button when the Sample List line in a play list is highlighted or when you click on the **Tare This** or **Weigh This** button when a Sample line in a Sample List is highlighted, the TGA Tare/Weigh System dialog box is displayed. It also is displayed after clicking on **OK** in the Advanced Tare Options dialog box.

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The Current Operation field displays the step currently being executed.

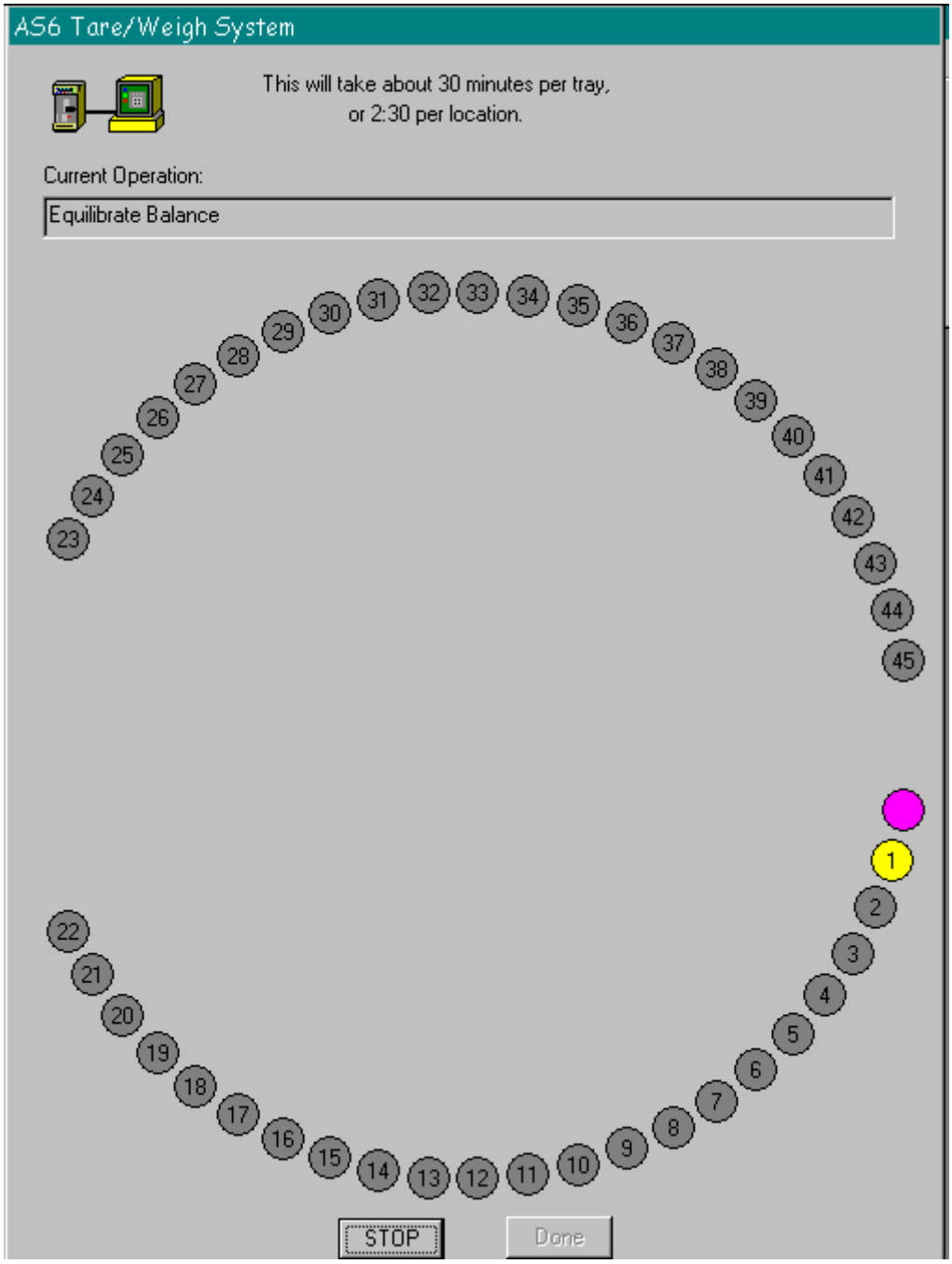
The autosampler position whose crucible or sample pan is being tared or whose sample is being weighed is indicated by a color: **yellow** means that the tare or weigh operation is in progress, **green** means that the tare or weigh operation completed successfully, **red** means that the operation failed. If there is a failure at a location, the procedure will continue.

You can stop the tare or weigh operation by clicking on the **STOP** button. The operation on the current location will go to completion. Click on **Done** when the operation is complete to close the dialog box.

Pyris 6 AS Tare/Weigh System

When you click on the **Tare List** or **Weigh List** button when the Sample List line in a play list is highlighted or when you click on the **Tare This** or **Weigh This** button when a Sample line in a Sample List is highlighted, the Pyris 6 AS Tare/Weigh System dialog box is displayed. It also is displayed after clicking on **OK** in the Advanced Tare Options dialog box.

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The Current Operation field displays the step currently being performed.

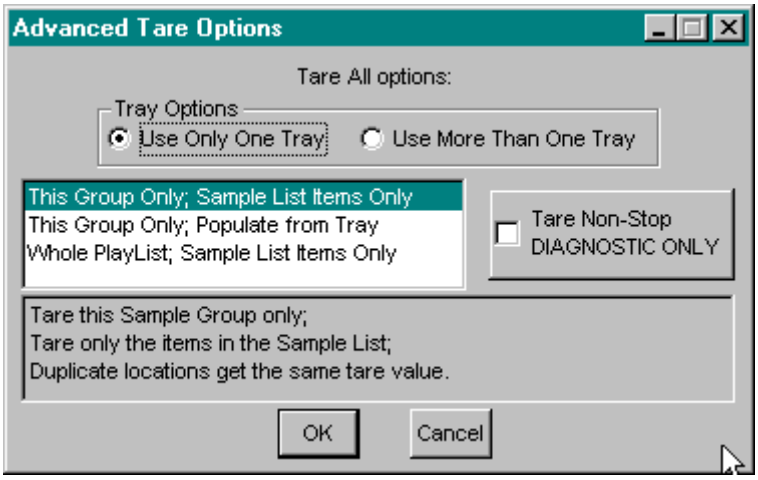
The autosampler position whose crucible or sample pan is being tared or whose sample is being weighed is indicated by a color: **yellow** means that the tare or weigh operation is in progress, **green** means that the tare or weigh operation completed successfully, **red** means that the operation failed. If there is a failure at a location, the procedure will continue. The zero position is indicated by **magenta**. You can select the **STOP** button to halt the tare or weigh procedure. The operation on the current location will go to completion. Click on **Done** when the operation is complete and the button becomes selectable.

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Advanced Tare Options

The Tare All/Weigh All system of the Pyris software is designed to handle processing of Pyris 1 TGA autosampler and Pyris 6 TGA with autosampler sample information in a play list. It is necessary to tare all sample pans that will be used in an autosampler run before the run begins. With the Pyris play list's Sample Group feature, there is an easy way to accomplish this. If your play list contains a Sample Group, you can have the system tare the sample pans and enter the tare weight automatically into the Zero field in the Sample Details field of the screen and in each Sample line of the Sample List.

With **Sample Group** the focused line in the play list — and you do not have to have any sample items entered into the Sample List yet — click on the **Tare All** button to display the Advanced Tare Options dialog box:



There are many ways in which to use the Tare All feature of a Sample Group.

First select the Tray option: You can use one tray for one or more Sample Groups or you can use a different tray for each Sample Group in the play list. You cannot change trays in the middle of a Sample Group. If you wish to use more than one tray, click on **Use More Than One Tray**. After the first tray's pans have been tared, a message telling you to change the tray is displayed.

Use Only One Tray

This Group Only; Sample List Items Only: The system will tare only the items listed in the Sample List of the current Sample Group and will fill in the same tare weight for a sample item everywhere it appears in the Sample List, i.e., if position 1 in the tray is listed three times, the same tare weight is entered at those three places. If there are no samples in the Sample List, then an error message is displayed in the TGA Tare/Weigh System dialog box.

This Group Only; Populate from Tray: The system will tare all the sample pans it finds in the sample tray. You do not have to have any Sample lines in the Sample List. This is a convenient way to create a Sample List without adding each line manually using the **Add a sample** button. As a sample pan is found and tared, that location is added to the Sample List. The Sample List created is for the current Sample Group only.

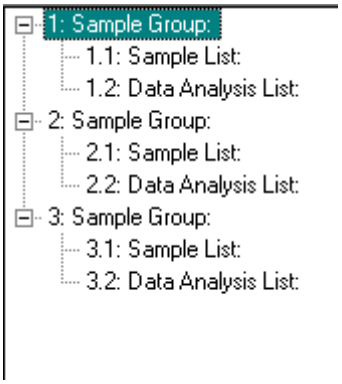
Whole Playlist; Sample List Items Only: The system will tare the sample pans at the locations specified in the Sample List for the current Sample Group and all subsequent Sample Groups in the play list. Duplicate sample locations will have the same tare value entered, i.e., if location 1 in the tray is listed in three different Sample Groups, the same tare weight is entered for each entry. If

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there are no samples in the Sample List, then an error message is displayed in the Tare/Weigh System dialog box.

Use More Than One Tray

Whole Playlist; Populate from Tray: You can create a skeleton play list similar to the following:



The system will tare the sample pans it finds in the first sample tray. The Sample lines are entered into the Sample List of the current Sample Group. Once all of the positions in the tray have been reached in the Tare/Weigh System dialog box, a pause message appears prompting you to change the tray. Click on **OK** in the message box to continue once the new tray is in place. The next Sample Group's Sample List is then populated with those locations in the new tray that have a pan. As a sample pan is found and tared, that location is added to the Sample List.

Whole Playlist; Sample List Items Only: This the same as that above except that you have to enter a Sample List for each Sample Group. The locations indicated in the Sample List should have sample pans. The tare weights of those pans are entered in the Zero field on the Edit Step: Sample area. At the end of one Sample List, you are prompted to change the sample tray. How the tray is populated should correspond to the Sample List of the next Sample Group.

Tare Non-stop: Diagnostic Only

This should be used by service engineers only. It is a convenient way to test the autosampler over an extended period of time. You can setup a Sample Group, fill the sample tray with pans or crucibles, select Tare Non-Stop and the analyzer will keep taring the pans over and over until you select **STOP** on the Tare/Weigh System dialog box.

Once you make your selections in the Advanced Tare Options box and click on **OK**, the TGA Tare/Weigh System dialog box for the Pyris 1 TGA or the Pyris 6 AS Tare/Weigh System for the Pyris 6 TGA, which shows the autosampler tray's locations and the current operation as each sample pan is tared, is displayed. After the operation, you are returned to the Edit Play List page and the tare weights will appear in the Zero field for each Sample line in the Sample List. If you chose to have the Sample List populated from the tray, then the Sample List will contain Sample lines for those positions in the tray that contain a sample pan. You can then select a Sample line and fill in pertinent information.

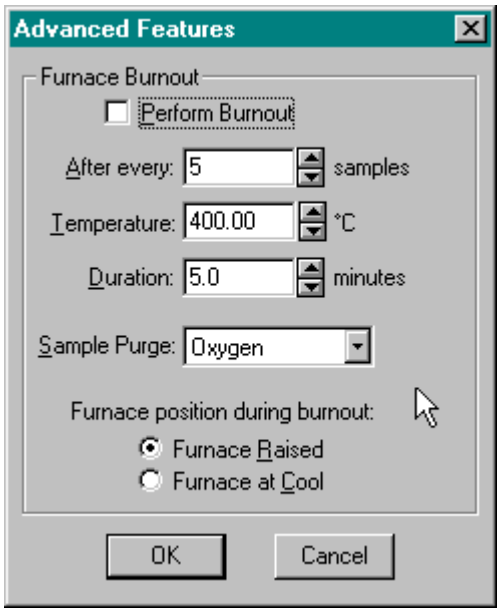
Furnace Burnout

For a Pyris 1 TGA autosampler and a Pyris 6 TGA autosampler system, there is feature associated with the Sample Group in a play list called Furnace Burnout. Click on the **Advanced** button while a Sample Group line is focused to display the Advanced Features dialog box. This feature tells the

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system to burn off at a specified temperature for the indicated time any volatile residue in the furnace after the specified number of samples in the Sample List have run. After you have set up this feature, furnace burnout will be performed after the allotted number of samples in the play list have run. After the burnout procedure is finished, the run will continue with the next sample in the Sample List.

NOTE: For DSC analyzers, the **Advanced** button appears on the Edit Play List page when Sample Group is highlighted. However, the Burnout Features dialog box is disabled when displayed. You cannot perform burnout for DSCs because of the possibility of the presence of a meltable item in the reference cell such as an aluminum sample pan.



To have the system perform the burnout procedure, click in the box next to **Perform Burnout**. Enter the frequency at which you want the burnout performed, the temperature at which to set the furnace, and how long you want the burnout to last. You can also specify a sample purge gas from the drop-down menu, or select No Gas. You do not have to use a sample purge gas.
If your analyzer is a Pyris 1 TGA with autosampler, you can specify whether to have the furnace in the Raised or Cool position during furnace burnout. It must be in the Raised position if you are running NOACK tests. These selections are grayed out for the Pyris 6 TGA with autosampler.

Pause

Pause is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button. Pause is handy in a play list for an instrument that does not have an autosampler. When the Pause line is reached and played, the message entered in the Comment field is displayed in a dialog box. For example, if you create a play list for the Pyris 6 TGA to be run by another operator, you can insert a Pause line with a Comment entry of “Fill Sample Pan 1/2 Full with AL2O3.” The operator reads the message and performs the operation. Clicking on **OK** clears the dialog box; the playback of the play list resumes.

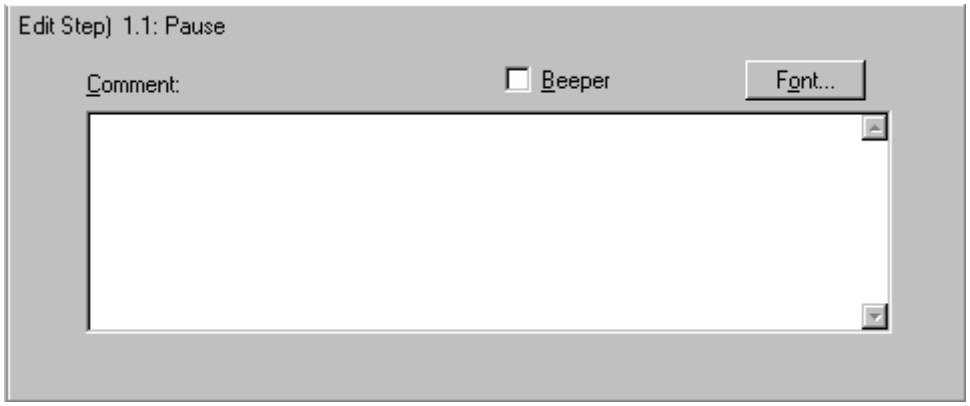
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Another use of Pause is after a Display Curve command. Display Curve will display the Data Analysis window with the selected curve or curves displayed but it does not remain on the screen. A Pause command is needed in order to keep the curve displayed until you click on the **OK** button in the Pause dialog box.

When Pause is added to the play list, the Edit Step area displays a Comment field in which you enter the comment to be displayed in the dialog box that appears when the Pause step is reached during playback.

Edit Step: Pause

The free-form text you enter in the Comment field appears in the Pause dialog box that is displayed when the Pause step is reached in playback. The message can describe what the next group of steps will do or give instructions to the operator, such as to remove or load the sample pan, lower or raise the probe, and so on. You can indicate the font to use for the display of the Pause dialog box by clicking on the Font button. The standard Font dialog box appears from which to select the font. Pyris can send a beep sound when this Pause line is reached in the playback if click on the Beeper box.



Start Method

Start Method is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for any analyzer. When Start Method is selected, the Method File Name dialog box appears from which you select the method that you want to run. When the method is selected, the Edit Step area below the Player Steps box displays fields that appear in the Sample Info page of the method. Access to the Initial State and Program pages of the method is through the **Edit Method** button. You can edit this method the same way as you would in Method Editor.

A method can be edited while the play list is running if the Start Method step has not been reached it yet:

1. Click on the **Pause** button on the Player toolbar.
2. When the play list playback has paused, click on the Start Method line.
3. Edit the fields you want to change; use the **Edit Method** button to access the Player Method Editor window.

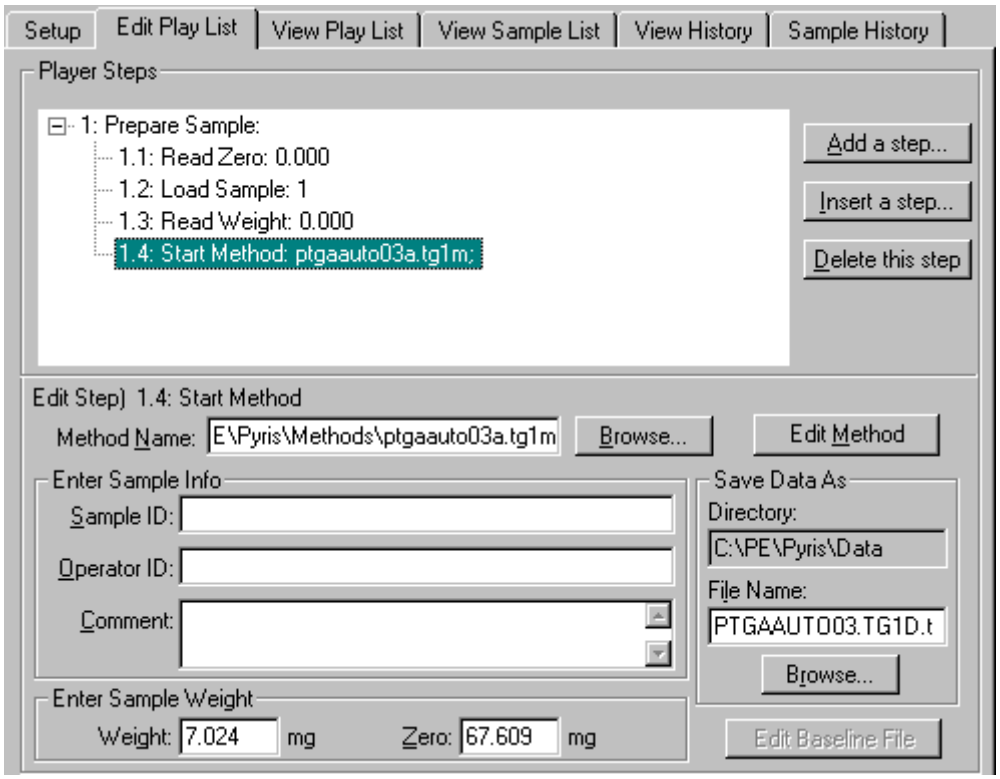
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4. Edit the Initial State and Program information.
5. Click on the **X** in the upper-right-hand corner of the window to save the changes and close the window. You return to the Edit Step area.
6. Select Save from the File menu to save the changes to the method.
7. Click on the **Resume** button on the Player toolbar to resume playback.

Edit Step: Start Method

When you select Start Method from the Select Player Options dialog box, the program adds the Start Method to the Player Steps and the Edit Step area displays the parameters of the method for editing. The Start Method Edit Step area is similar to the Sample Info page of the Method Editor. Click on the **Edit Method** button to access the other two pages of a method, Initial State and Program. When you are finished editing these two pages, click on the **Close** button (☒) in the upper-right-hand corner of the window to close the window and return to the Edit Play List page.

Here's an example of a Start Method Edit Step for a Pyris 1 TGA:



Method Name

Displays the full path, i.e., drive and directory, of the method selected when the Start Method item is added to the play list. You can change the method by typing in another method file name in the Method Name field or use the **Browse** button to find the file on your system.

Enter Sample Info

Sample ID

Enter a sample name for the sample to be run by this method. It can be up to 40 characters. The entry field scrolls horizontally as you type in the name.

Operator ID

Enter the name of the person who will run the experiment (or who starts playback of the play list). The name can be up to 40 characters. The entry field scrolls horizontally as you type.

Comment

Enter any additional information about the sample or the method. The entry can be a maximum of 160 characters.

Enter Sample Weight

Weight

Enter the weight of the sample in milligrams. The default value is 1.000 mg. For TGA methods, you can use the **Read Weight** command in a play list prior to the Start Method entry to have the system read the weight of the sample. This is equivalent to using the **Read Weight** button on the control panel to have the analyzer determine the weight.

Zero

For TGA analyzers, enter the weight of the empty sample pan in milligrams. The default value is 0.00 mg. You can use the Read Zero command in a play list prior to the Start Method entry to have the system read the weight of the pan. This is equivalent to using the Zero Weight button on the control panel. For DTA 7, this value is the difference in temperature between the sample and the reference thermocouples. You can use the Read Zero command in a play list prior to the Start Method entry to have the system read the temperature difference between thermocouples. This is equivalent to using the Read Zero button on the control panel.

Edit Sample Dimensions Button (DMA/TMA)

This button appears for DMA and TMA analyzers only and displays the Player Sample Dimensions dialog box.

Edit Method Button

Click on this button to access the Initial State and the Program pages. The fields in these pages are exactly the same as for a method in Method Editor.

Directory

Displays the drive and directory in which the data from the run is saved. This entry reflects the disk/directory chosen in the Browse dialog box. If you do not select a specific directory, the default will be displayed.

File Name

Enter the name of the file in which the data collected from the run will be saved. You can enter the file name directly into this field; the default Directory entry (C:\Program Files\Pyris\Data) will be displayed. If this method is run more than once in the play list, e.g., sample pans 1 – 10 in the sample tray are analyzed in succession with this method, the Pyris software appends a date and time stamp to the file name for each run in order to make each

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file name different. It is in the format: <*file name*>@120997221810.dcd. This Pyris 1 DSC data file was collected on December 9, 1997 at 10:18:10 p.m. You can also use incremental file names, e.g., if you enter <*filename*>### in the File Name field in each Sample List entry, the program appends 001, 002, etc., each time the method is used, i.e., for each sample in the sample list. If when running the play list it is found that a file name already exists, the system will add a date/time stamp at the end of the new file name to distinguish it from the existing file.

Browse

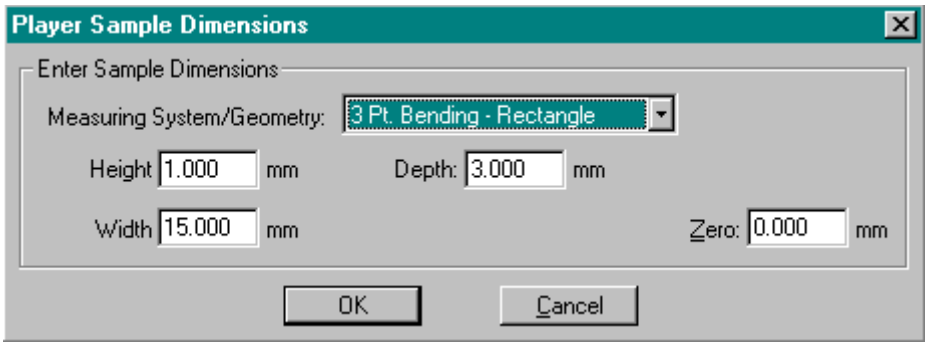
Change the drive and directory in which to save the data file by selecting the desired path in the Browse dialog box, displayed by clicking on the **Browse** button.

Edit Baseline File

Displays the Player Baseline File dialog box.

Player Sample Dimensions Dialog Box

For DMA and TMA analyzers, the Edit Step area for Start Method contains the **Edit Sample Dimensions** button which displays the Player Sample Dimensions dialog box:



Measuring System/Geometry

From the drop-down list displayed when you click on the down arrow, select the measuring system that you are using and the associated geometry of the sample. The other items in this dialog box depend on this selection.

Height

Enter the sample height. You can also enter a **Read Height** command in the play list above the Start Method entry to have the sample's height read from the instrument by the software. This value does not change when another method is used in the play list after this method. You must change the Height value.

Width

Enter the width of the sample. This value does not change with change of method unless done by the operator.

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Depth

Enter the depth of the sample. This value does not change with change of method unless done by the operator.

Length

Enter the length of the sample (for an extension measuring system). This value does not change with change of method unless done by the operator.

Diameter

Enter the diameter of the sample (for cylindrical or disk-shaped samples). This value does not change with change of method unless done by the operator.

Thickness

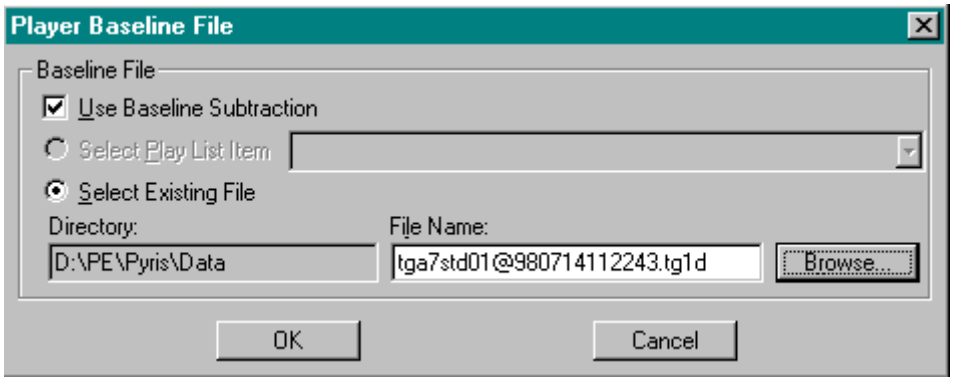
This dimension is the thickness of one of the two samples used with a shear measuring system. It is entered by the user. The maximum thickness is 3.000 mm. This value does not change with change of method unless done by the operator.

Zero

Enter the zero height value, i.e., the height of the probe without a sample in place. You can also enter a **Read Zero** command in the play list above the Start Method entry to have the zero height read by the software from the instrument. The instrument has to be prepared for the read, i.e., the probe must be down and there must be no sample on the platform. This value does not change with change of method unless done by the operator.

Player Baseline File Dialog Box

You can optimize the data as it is collected by subtracting a baseline file from the data. When you select the **Edit Baseline File** button in the Start Method Edit Step area, the Player Baseline File dialog box appears.



The fields in this box are similar those in the Initial State page of the Method Editor. The items is this dialog box are as follows:

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Use Baseline Subtraction check box

Click on the check box to activate Baseline Subtraction for the data collected by this method.

Select Play List Item

To use for baseline subtraction a data file created while running the current play list, click in this radio button. Select the play list item that creates the baseline subtraction file from the drop-down list. The available selections are the data files created before the play list reaches the step in which it used for baseline subtraction. The play list entry is displayed in this field.

Select Existing File

To select an existing file for baseline subtraction, click in this radio button and enter the file name in the File Name field or use the **Browse** button to find and select the file. The Directory field will display the drive and directory where the file resides.

Load Sample

Load Sample is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for any analyzer. When Load Sample is selected, the Edit Step area displays the Carousel Location field in which you enter the position in the sample tray of the autosampler from which to take the sample pan and load it into the sample holder or onto the hangdown wire. If your instrument does not have an autosampler, you can still select Load Sample for the play list. When playback reaches this step, the message "Automatic Pause" is displayed. The operator can then load the prepared sample into the instrument. When the sample is in place, click on **OK** to clear the message and continue playback of the play list.

NOTE: When the Load Sample command is encountered in a play list for a Pyris 1 DSC autosampler, the system does **NOT** check for the presence of a sample pan in the sample holder. Select **Use Initial Check** in the Autosampler page of Preferences to tell the system to check the sample holder before loading the first sample in a play list.

It is recommended that if your instrument does not have an autosampler, use the Pause entry instead of Load Sample in the play list. Enter an informative message in the Comment field for Pause telling the user to load the sample.

Return Sample

Return Sample is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for any analyzer. When Return Sample is selected, the Edit Step area displays the Carousel Location field in which you enter the position in the sample tray to which to return the sample pan from the sample holder or hangdown wire. If your instrument does not have an autosampler, you can still select Return Sample for the play list. When playback reaches this step, the message "Automatic Pause" is displayed. The

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operator can then remove the sample from the instrument. When the sample has been removed, click on **OK** to clear the message and continue the play list.

It is recommended that if your instrument does not have an autosampler, use the Pause entry instead of Return Sample in the play list. Enter an informative message in the Comment field for Pause telling the user to remove the sample.

Load Reference

Load Reference is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for DSC analyzers. When Load Reference is selected, the Edit Step area displays the Carousel Location field in which you enter the position in the autosampler's tray from which to take the sample pan and load it into the sample holder. This is relevant for the DSC 7 Robotic System and the Pyris 1 DSC Autosampler. If your analyzer does not have an autosampler attached, or you are using the Pyris 6 DSC, this entry is irrelevant. When playback of the play list reaches this step, the message "Automatic Pause" is displayed. The operator can then load the prepared reference sample into the instrument. When the reference is in place, click on **OK** to clear the message and continue playback.

NOTE: When the Load Reference command is encountered in a play list for a Pyris 1 DSC autosampler, the system does **NOT** check for the presence of a pan in the reference cell of the sample holder. Select the Use Initial Check feature in the Autosampler page of Preferences to tell the system to check the sample holder before loading the first reference sample in a play list.

It is recommended that if your instrument does not have an autosampler, use the Pause entry instead of Load Reference in the play list. Enter an informative message in the Comment field for Pause telling the user to load the reference.

Return Reference

Return Reference is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for a DSC analyzer. When Return Reference is selected, the Edit Step area displays the Carousel Location field in which you enter the position in the sample tray of the autosampler to which to return the reference pan from the sample holder. If your instrument does not have an autosampler, you can still select Return Reference for the play list. When playback reaches this step, the message "Automatic Pause" is displayed. The operator can then remove the reference from the instrument. When the reference pan has been removed, click on **OK** to clear the message and continue the playback of the play list.

It is recommended that if your instrument does not have an autosampler, use the Pause entry instead of Return Reference in the play list. Enter an informative message in the Comment field for Pause telling the user to remove the reference.

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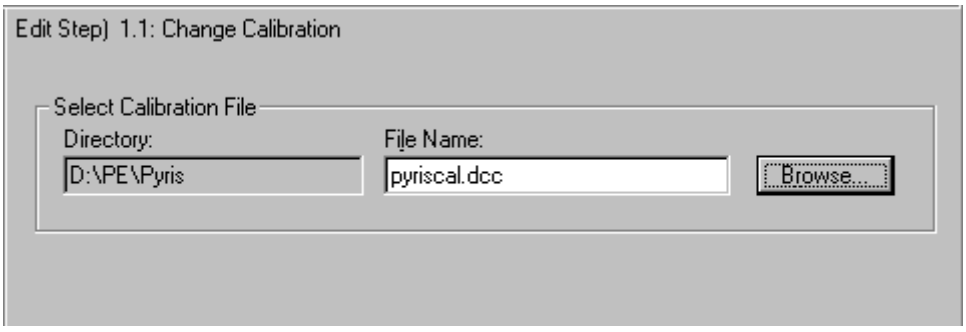
Carousel Location

When you select Return Sample or Load Sample from the Player Step Options dialog box to add or insert a step into the play list, the Edit Step area displays the Carousel Location field. If you have a DSC 7 Robotics System or a Pyris 1 DSC Autosampler, the loading and unloading of samples to and from the specified tray location is performed by the instrument. The default sample tray location is 1. Type in the position (from 1 to 48) from which you want to load a sample into the sample holder or to which to return a sample from the sample holder. You can also use the spin buttons to increase or decrease the location number. If you have a Pyris 1 DSC Autosampler, this field also applies to Load Reference and Return Reference.

If your instrument does not have an autosampler, Carousel Location is not relevant. When the Load Sample or Return Sample entry is reached in the play list, the message "Automatic Pause" is displayed. This gives the operator the opportunity to load or unload the sample as would be done when performing a run without the play list.

Change Calibration

Change Calibration is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for any analyzer. You may have different calibration files for different sample pans. If you want to change the calibration file before running a particular sample, just put this command in the play list and select the calibration file in the Edit Step section. When you select Change Calibration, the Edit Step area appears:



Enter the calibration file name in the File Name field. Use the **Browse** button to find the file on your computer or network. Once you select the calibration file, its name is displayed in the File Name field and the path is displayed in the Directory field.

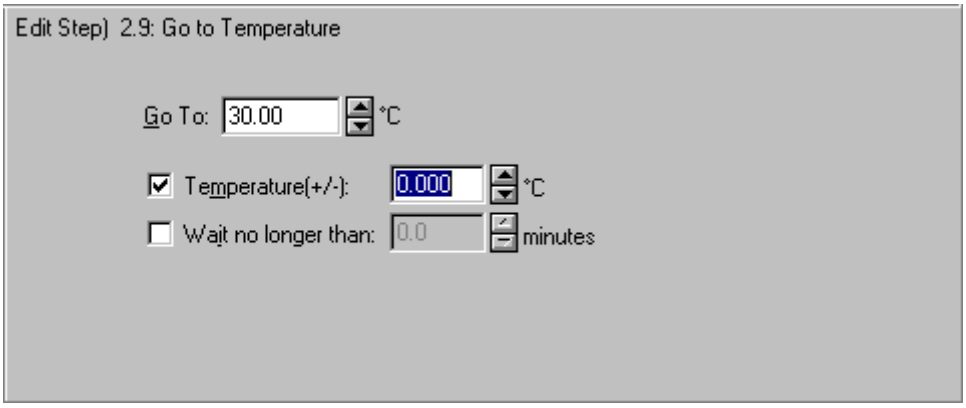
Go to Temperature

You can select Go To Temperature as an item in the play list for all analyzers. When this step is reached in the play list, it is equivalent to clicking on the **Go To Temperature** button on the control panel. Go To Temperature programs the analyzer to the temperature entered in Edit Step Go To field. For a DMA or a TMA, the stresses (or forces) and frequency will remain the same. If using an DSC autosampler, this item should be used in the play list to return the sample temperature to a value at which the suction tip of the robot arm can safely pick up the sample pan and return it to the sample tray. The temperature should be between 0 °C and 70 °C but it can safely pick up the sample pan when the temperature is 50 °C–55 °C. You would also need to heat up or

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cool down the sample temperature in order to open the protective cover of the autosampler or Robotics System.

Edit Step: Go to Temperature



Go To

Enter the temperature to which the analyzer is to be set. The temperature for a Pyris 1 DSC must be in the range of –180°C to 730°C; for the Pyris 6 DSC, –120°C and 450°C; and for the DSC 7, –180°C and 730°C. For the TGA 7, the range is –20°C to 1030°C; for the Pyris 6 TGA, the range is –20°C to 1000°C; and for the Pyris 1 TGA, the range is 0°C to 1030°C. For the DMA 7e and the TMA 7, the temperature must be between –180°C and 500°C.

Temperature (+/–)

Set a range in which the temperature can fall by clicking on the Temperature (+/–) check box and entering the value in the field.

Wait no longer than

The value entered in this field is the time allotted for equilibration of the temperature.

Display Curve

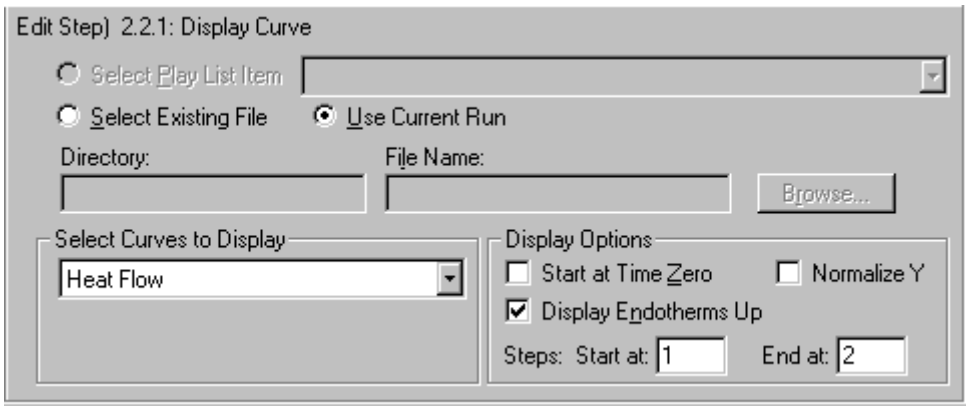
Display Curve is a selection for the play list when the most recent main-level item in the list is Data Analysis or Data Analysis List in Sample Group. After selecting a curve or curves to display, you can select functions from Math Options, Calculation Options, and Rescale Options from the Player Step Options dialog box to perform on the displayed curves.

NOTE: A Pause line should follow a Display Curve command in a play list in order to view the display of the curve. The Data Analysis window with the curve or curves is displayed quickly. The Pause command will keep the Data Analysis window displayed until you click on the **OK** button in the Pause dialog box.

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Edit Step: Display Curve

When you select Display Curve from the Player Step Options dialog box, the Edit Step area displays the following fields:



Select Play List Item

If you want to display a data curve from a preceding run in the play list, click in this radio button. All data files created by running the play list up to this point are available for display. Click on the down arrow to display the drop-down list of available curves; the line number and description in the play list are displayed.

Select Existing File

If you want to display a curve from an existing data file, perhaps for comparative purposes, click in this radio button. Enter the file name directly into the File Name field or use the **Browse** button to search for the file.

Use Current Run

When the Display Curve command is part of a Data Analysis List of a Sample Group, you can also select the curve from the current run for display. This button is not displayed for a Display Curve command in Data Analysis.

Directory

If you select an existing file, the full path, i.e., drive and directories, of that file is displayed in this field for information only.

File Name

If you select an existing file to display, enter the name of the file or select it from the Browse dialog box displayed by clicking on the **Browse** button. Once you select the file, the drive and directory of that file is displayed in the Directory field.

Select Curves to Display

Select the type of curve to display. The selection is analyzer dependent.

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Display Options

Click in the check box of each item you want to activate.

- If **Start at Time Zero** is selected, the data from each method step selected for display will be displayed as starting at zero minutes so that the data from different steps can be compared. The curves already on the screen are not affected by this selection.
- If **Display Endotherms Up** is selected (for DSC analyzers only), the Y axis increases from bottom to top. Curves already on the screen are not affected by a change in endotherm setting.
- For selecting the method **Steps** to include in the curve displayed, enter the start and end steps.
- **Normalize Y** is an additional display option for Heat Flow curves of DSC and DTA data and Probe Position for DMA data. Normalize Y changes the Y axis of the active curve to a normalized state. A curve is normalized by dividing the Y value by the sample weight. All curves associated with the active Y axis are also normalized.

Open Cover

This is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for a DSC analyzer with an autosampler. The cover of the autosampler's furnace area will open when this command is executed. There are no fields in the Edit Step area to fill.

Close Cover

This is a second-level entry for the Pyris Player play list. It is a choice in the Player Edit Options dialog box which is displayed when you click on the **Add a step** or the **Insert a step** button in the Edit Play List page. It is available for selection for a DSC analyzer with an autosampler. The cover of the autosampler's furnace area will close when this command is executed. There are no fields in the Edit Step area to fill.

Read Height

If you are creating a play list for the DMA 7e or the TMA 7, the Read Height command is a play list option. When the playback of the play list reaches this command, the analyzer must be ready to read the height of the sample on the sample platform. Therefore, you must have a Pause command before the Read Height line in order to pause playback and place the sample on the platform and lower the probe by pressing the **Probe Down** button on the analyzer's front panel. As soon as the analyzer is ready, click on the **OK** button in the Pause dialog box and the playback continues. The command goes out for the analyzer to read the height of the sample. The value appears in the Probe Position in the Status Panel (if displayed), the Height field in the Sample Info page of the Method Editor window, and in the Player Sample Dimensions dialog box when displayed.

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Read Zero

This option appears in the Player Step Options dialog box for a number of analyzers.

If you are creating a play list for the DMA 7e or the TMA 7, the Read Zero command applies to the sample height. When the playback of the play list reaches this command, the analyzer must be ready to read the probe position without any sample in place on the sample platform. This is done so that the height of the platform is not included in future sample height readings. You must have a Pause command before the Read Zero command to pause the playback of the play list. When the Pause dialog box is displayed, make sure that the analyzer is ready for a zero reading, i.e., no sample is on the sample platform and the probe is down. Click on **OK** in the dialog box and the Read Zero command is sent to the analyzer. The result is displayed in the Player Sample Dimensions dialog box and in the Sample Info page of the Method Editor window.

If you are creating a play list for the DTA 7, Read Zero refers to the difference in temperature between the sample and the reference thermocouples with no sample in the sample cup. The analyzer must be ready to take this reading when this step is reached. Place a Pause command before the Read Zero command so you can prepare the analyzer for the reading. As soon as you click on the **OK** button in the Pause dialog box, the reading is made. The result of the reading is displayed in the Zero field in the Method Editor and in the Enter Sample Weight box of the Edit Step area.

If you are creating a play list for the Pyris 6 TGA, TGA 7, or Pyris 1 TGA, Read Zero is used to read the weight of the empty sample pan. The weight of the sample pan is the zero weight; the pan's weight will not be included in sample weight readings. Place a Pause command before the Read Zero command in order to pause the playback and place the sample pan in the sample holder or load it on the hangdown wire. When it is place, click on **OK** in the Pause dialog box and the sample pan's weight is read. The result is displayed in the Zero field in the Method Editor and in the Enter Sample Weight box of the Edit Step area. If you have a Pyris 1 TGA with autosampler, you can use the Tare This or Tare List command from the Sample Group page to have the instrument tare all empty sample pans automatically.

Read Weight

This option appears in the Player Step Options dialog box for the Pyris 6 TGA, TGA 7, and Pyris 1 TGA. Read Weight is used to read the weight of the sample. This command should follow the Read Zero command. Place a Pause command after the Read Zero command and before the Read Weight command in order to pause the playback and place the sample in the sample pan and then place the pan in the sample holder. When it is place, click on **OK** in the Pause dialog box and the sample's weight is read. The result is displayed in the Weight field in the Method Editor and in the Enter Sample Weight box of the Edit Step area. If you have a Pyris 1 TGA with autosampler, you can use the Weigh This or Weigh List command on the Sample Group page to have the sample weights measured automatically.

Raise Furnace

The Raise Furnace option for the play list is available for the TGA 7 and the Pyris 1 TGA. This command will move the furnace to the Run position and lock it in place. (This is equivalent to using the **Raise Furnace** button on the control panel.) In order to perform a run, the furnace must be in this position. A Pause command should be placed before the Raise Furnace command in a play list in order to prepare the analyzer for the start of the run.

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If your analyzer does not have an autosampler, the typical play list for running a sample would be similar to the following:

1. Prepare Sample
2. Raise Furnace
3. Pause
4. Read Zero
5. Lower Furnace
6. Pause
7. Load Sample
8. Raise Furnace
9. Pause
10. Read Weight
11. Start Method
12. Lower Furnace
13. Return Sample

When the play list reaches the Load Sample command, an Automatic Pause dialog box is displayed since Pyris does not detect an autosampler. While the dialog box remains displayed, move the sample loading platform directly under the sample pan. Raise the platform up until the pan rests on the platform and the stirrup raises off the bend in the hangdown wire. Using tweezers, remove the sample pan and stirrup assembly. Place the sample in the sample pan. Place the pan and stirrup on the sample loading platform so that the stirrup will hook on the hangdown wire when the sample loading platform is lowered. Lower the sample loading platform. The sample pan and stirrup should be hanging on the hangdown wire. Swing the sample loading platform back into its safe position. Wait several seconds for the sample pan to stop swinging. Click on **OK** to clear the dialog box. The furnace will automatically rise to the Run position and lock into place.

Lower Furnace

The Lower Furnace option for the play list is available for the TGA 7 and the Pyris 1 TGA. This command will lower the furnace in order for you to gain access to the sample pan for loading or removing samples. (This is equivalent to the Lower Furnace button on the control panel.)

If your analyzer does not have an autosampler, the typical play list for running a sample would be similar to the following:

1. Prepare Sample
2. Raise Furnace
3. Pause
4. Read Zero
5. Lower Furnace
6. Pause
7. Load Sample
8. Raise Furnace
9. Pause
10. Read Weight
11. Start Method

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12. Lower Furnace
13. Return Sample

Since there is no autosampler attached to the TGA 7, the Return Sample command after the Lower Furnace command displays the Automatic Pause dialog box. While the dialog box is displayed, move the sample loading platform directly under the sample pan. Raise the sample platform up until the pan rests on the platform and the stirrup raises off the bend in the hangdown wire. Using tweezers, remove the sample pan and stirrup assembly. Click on **OK** to clear the dialog box.

Cool Furnace

The Cool Furnace option for the play list is available for the TGA 7 and Pyris 1 TGA. This command will automatically move the furnace inside the analyzer over a large cooling fan (TGA 7) or under a cooling fan (Pyris 1 TGA) in order to cool it quickly. This is equivalent to the **Cool Furnace** button on the control panel.

Math Options Drop-Down List

When Math Options is listed in the Player Step Options dialog box, the following items are available for selection for Display Curve in the play list:

Derivative

When Derivative is selected from the Math Options list, the Edit Step area displays the following field:

Select Active Curve

Select a curve from the drop-down list of curves currently available based on the play list up to this point. The curves may be previously existing data files or from the current run of the play list. Select the curve (data file) on which you want the derivative to be calculated.

Subtract

The **Subtract** command is used to subtract the Y values of a selected curve from the active curve. The Edit Step: Subtract area contains the following fields:

Select Active Curve

Select a focused curve from the drop-down list of active curves available (collected by the play list above this point).

Select the curve to be subtracted from the focused curve

This list contains all curves available (collected by the play list above this point) whose ordinate values can be subtracted from the ordinate values of the focused curve. Select one curve from the list to highlight it for subtraction.

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Add and Average

The **Add** command is used to add one or more curves to the active curve. The Average command takes one or more curves and calculates the average Y value with that of the active curve. The Edit Step area for either command contains the following fields:

Select Active Curve

Select a focused curve from the drop-down list of active curves available (collected by the play list above this point). The focused curve is the curve to which another curve is added or averaged.

Select the curves to be added to the focused curve

This list contains all curves available (collected by the play list above this point) whose ordinate values can be added to or averaged with the ordinate values of the focused curve. Select one or more curves.

Smooth

Smoothing removes noise from the focused curve. When you select **Smooth**, the Edit Step area containing the following fields appears:

Select Active Curve

Select a curve from the drop-down list of active curves available (collected by the play list above this point).

Left Limit

Enter the left X-axis limit for the smooth calculation.

Right Limit

Enter the right X-axis limit for the smooth calculation.

Algorithm

Select an smoothing algorithm:

- **Standard**: A simple sliding average algorithm. Each point is smoothed based on its current value and a group of points that make up the window centered on it. This type of smoothing results in moderate smoothing of noise.
- **Median**: A sliding average, like Standard smooth, but each point in the window is weighted differently, with the point in the center of the smooth window weighted heaviest and points on the ends weighted least. This results in moderate smoothing with better peak height preservation.
- **Average**: Another sliding average algorithm that results in better baseline smoothing than Standard or Median, but there may be some degradation of peak height. Use this algorithm for smoothing DDSC curves, using the size of the repeat step as the window size.

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- **Savitzky-Golay**: A weighted smoothing algorithm that is very good for smoothing baselines and maintaining peaks. However, there may be slight shifting in the peak value.

Window Size

Enter a window size (in points) to be used in the smooth calculation. The default window size is 5 points in all cases except DDSC Heat Flow curves. In this case, the default window size is the number of points in one repeat unit.

Calculation Options Drop-Down List

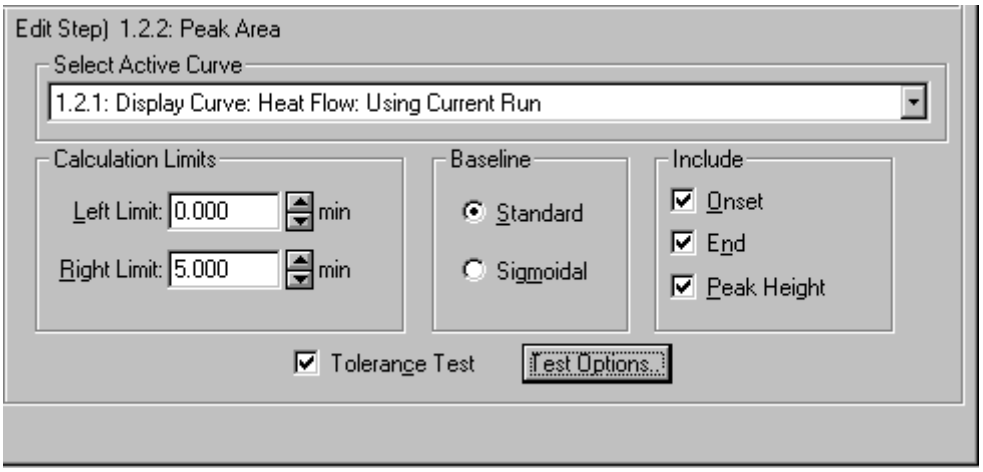
When Calculation Options is listed in the Player Step Options dialog box, the following items are available for selection for the curve displayed by the Display Curve entry. This option has its own drop-down menu which contains the following items:

- Peak Area
- Peak Search
- Onset
- Trigger
- OIT
- Delta Y
- Delta X
- Event
- Slope
- Step
- Tg
- Purity
- Specific Heat - Single Curve
- Specific Heat - Multi Curve
- Expansion Coefficient
- Enthalpy
- Create Table
- Noack Test

Edit Step: Peak Area

When you select **Peak Area** from the Calculation Options drop-down list for a Display Curve play list item, the Edit Step area contains the following fields:

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Select Active Curve

Select a focused curve from the drop-down list of active curves available.

Left Limit

Enter the left X-axis limit for the calculation.

Right Limit

Enter the right X-axis limit for the calculation.

Baseline

Select either a Standard or a Sigmoidal baseline to be used in the calculation.

Include

Select Onset to have the onset temperature calculated and included in the results. Select End to have the peak end value calculated and included in the results. Select Peak Height to have the peak height value calculated and included in the results. You can select one, two, or all three items. The value(s) will be displayed in the Data Analysis window and on the printout.

Tolerance Test

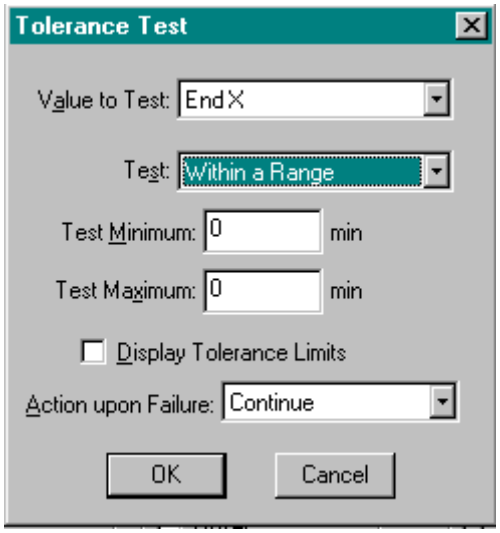
Click in the check box if you want a tolerance test performed on the results of the peak area calculation. A tolerance test checks whether the results are within a specific range. Unlike using tolerance test in a Peak Area calculation while in Data Analysis, you cannot specify the limits within which to test on the curve itself. You enter the values in the Tolerance Test dialog box.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

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Tolerance Test Dialog Box



Value to Test

The items displayed in this drop-down list are calculation-specific, e.g., for Peak Area your choices are Peak X, Onset X, End X, Peak Height, Peak Area, and Delta H. the fields that follow depend on the selection made here.

Test

The type of tolerance test that the data must pass is selected from this list. The data must be: Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Peak Area tolerance test, the units displayed for minimum and maximum are minutes.

Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

Action Upon Failure (for play list calculations only)

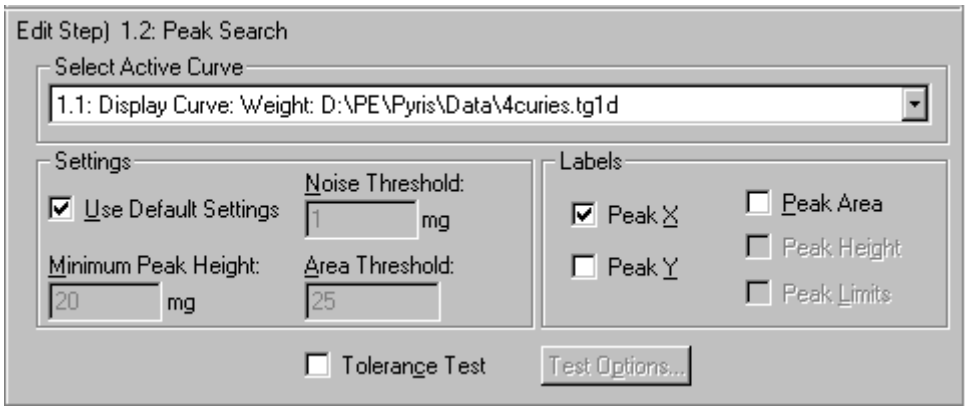
If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

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Edit Step: Peak Search

When you select **Peak Search** from the Calculation Options drop-down list for a Display Curve play list item, the Edit Step area contains the following fields:



Select Active Curve

Select a focused curve from the drop-down list of active curves available.

Use Default Settings

The default settings are 1 mg for Noise Threshold, 20 mg for Minimum Peak Height, and 25 for Area Threshold.

Noise Threshold

Enter a noise threshold in Y-axis units for which the Pyris software will search. This parameter enables Pyris to discriminate between baseline noise and peaks. If the vertical difference between the inflection point on the leading and trailing edges of the peak and the top of the peak exceeds the Noise Threshold, then Pyris recognizes the potential start of a peak. The Noise Threshold must be met on only one side of the peak. All peaks in the active curve whose heights are greater than this value will be identified. The lower the Noise Threshold value, the more sensitive peak detection is.

Area Threshold

Area Threshold is used to discriminate between noise spikes and peaks. This parameter is used after the Noise Threshold to confirm the potential start of peaks that pass the Noise Threshold test. After passing that test, the data points must continue to pass the test and the cumulative sum of the data points on the leading edge must eventually exceed the area threshold for the peak to be confirmed. The higher the area threshold value, the more difficult it is to confirm a peak.

Minimum Peak Height

Minimum Peak Height is used to avoid finding a false peak top because of noise. To find the top of a peak, Pyris tries to identify a local maximum bunched point value. When a bunched point is lower than the previous one, the previous point is considered to be the potential peak top. To avoid finding a false peak top because of noise, Pyris performs a confirmation test by summing the differences between the potential top and subsequent bunched points. If the sum exceeds two-

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thirds of the Area Threshold value, the potential peak top is confirmed. If a higher bunched point is found before the area test is passed, a new potential top is identified and the area test is restarted. Because of this top-of-the-peak test, the choice of an area threshold value affects both peak confirmation and how shoulders are detected on the leading edge of a larger peak.

Labels

Select either Peak X, Peak Y, or both to have the label displayed on each peak found. If you select Peak Area, then Peak Height and Peak Limits become selectable labels for each peak found.

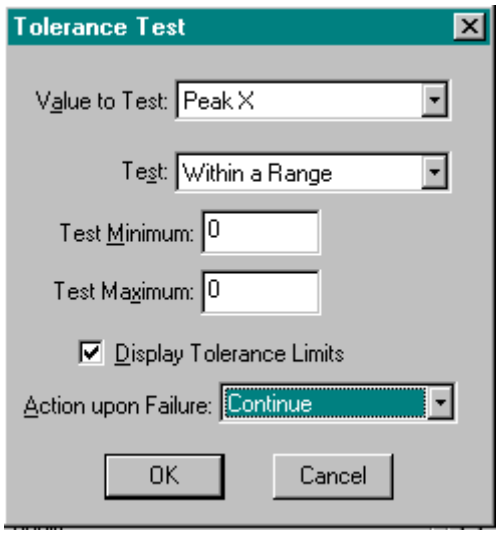
Tolerance Test

Click in the check box if you want a tolerance test performed on the results of the peak search calculation. A tolerance test checks whether the data are within a specific range. Unlike using tolerance test in a Peak Search calculation while in Data Analysis, you cannot specify the limits within which to test on the curve itself.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

Tolerance Test Dialog Box



Value to Test

Peak X is the only selection.

Test

Types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X values.

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Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

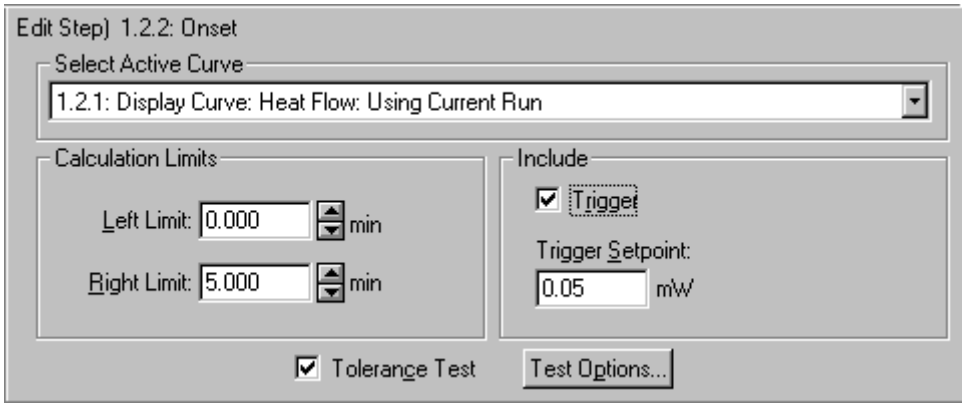
Action Upon Failure

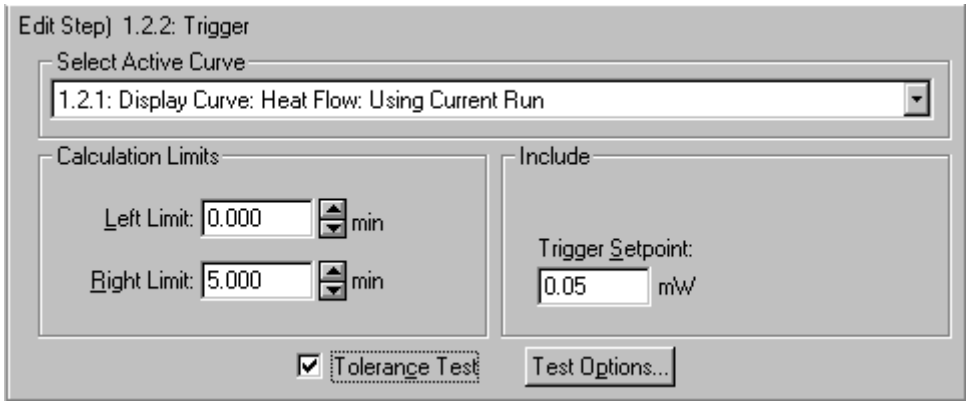
If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Edit Step: Onset, Trigger, and Oxidative Induction

An Onset calculation determines the beginning of any transition that is distinguished by a significant change from the baseline. A Trigger calculation determines the beginning of a transition without a clearly defined onset in the curve. This calculation is available for DSC analyzers only. The Oxidative Induction calculation is a special case of the Onset calculation. It must be used on a normalized heat flow curve displayed on a time scale. Oxidative induction time is an accelerated test used as a qualitative evaluation of the stability of a material. It is typically used to assess antioxidant formulations in plastics but is also applicable to many other materials.





Select Active Curve

Select a curve from the drop-down list of curves available.

Left Limit

Enter the left X-axis limit for the onset temperature calculation, the trigger calculation, or the oxidative induction calculation.

Right Limit

Enter the right X-axis limit for the onset temperature calculation, the trigger calculation, or the oxidative induction calculation.

Include Trigger check box

In the Onset and Oxidative Induction Edit Step areas, to include the trigger onset in the calculation, click in the Trigger box. The Trigger Setpoint entry field is then activated in which you enter the trigger value. The trigger value is calculated by locating the first time or temperature for which the vertical distance between the curve and the initial tangent line is greater than the value of the trigger. The initial tangent line is defined as the left-hand limit tangent line for a heating curve on temperature X axis or the right-hand limit tangent line for a cooling curve on a temperature X axis.

Trigger Setpoint

The Trigger setpoint default value is 0.05°C. This is displayed for information purposes only.

Onset

In the Oxidative Induction Edit Step area, to include the onset temperature in the OIT calculation, click in the Onset box.

Time Zero

For an Oxidative Induction calculation, you can indicate where in the data file to begin using data, e.g., start at 3 minutes and ignore the data collected from 0 to 3 minutes.

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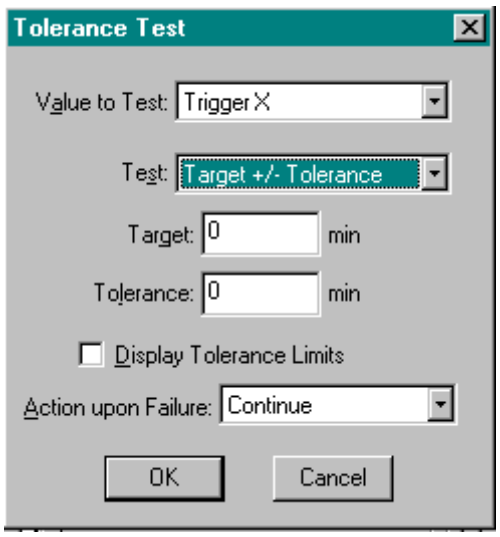
Tolerance Test

Click in the check box if you want a tolerance test performed on the results of the peak search calculation. A tolerance test checks whether the data are within a specific range. Unlike using tolerance test in an Onset, Trigger, or Oxidative Induction calculation while in Data Analysis, you cannot specify the limits within which to test on the curve itself.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

Tolerance Test Dialog Box



Value to Test

Onset X and Trigger X.

Test

Types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X values.

Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

Action Upon Failure

If the data fail the tolerance test, you can have the play list do one of the following:

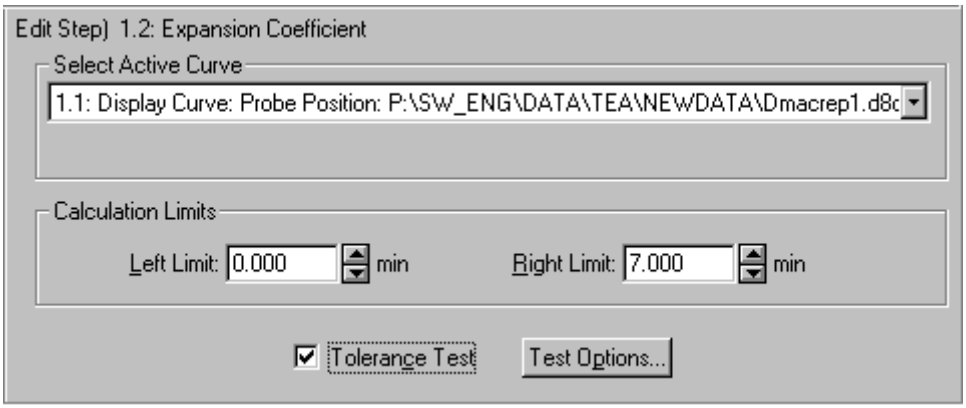
- Continue

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- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Edit Step: Expansion Coefficient, Slope, and Delta Y

The Expansion Coefficient calculation applies to DMA 7e data only. The Delta Y and Slope calculations are available for all analyzers. The coefficient of expansion is the change in volume of the sample per degree temperature increase from the initial temperature. It is derived using the volumetric expansion values that are calculated from the probe position. The displayed curve must be a Probe Position curve in order for Expansion Coefficient to be included in the Calculation Options drop-down menu. The Delta Y calculation determines the change in the ordinate axis after you select two points on the X axis for the active curve. The Slope command calculates the slope of a user-defined section of the focused data curve. The Edit Step area for all three selections has the same fields:



Select Active Curve

Select a curve from the drop-down list of active curves available.

Calculation Limits

Left Limit

Enter the left X-axis limit for the expansion coefficient, the slope, or the Delta Y calculation.

Right Limit

Enter the right X-axis limit for the expansion coefficient, the slope, or the Delta Y calculation.

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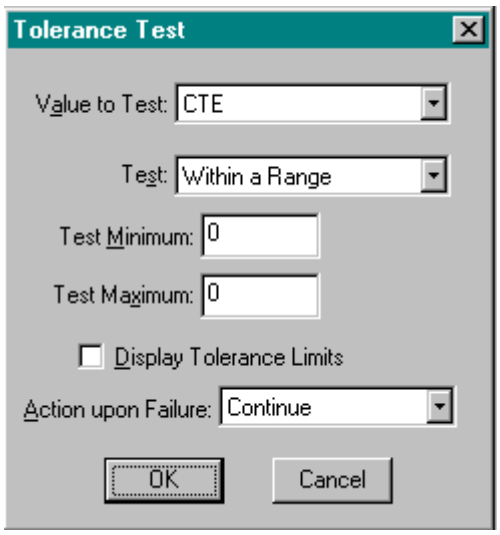
Tolerance Test

Click in the check box if you want a tolerance test performed on the results of the expansion coefficient, delta Y, or slope calculation. A tolerance test checks whether the results are within a specific range. Unlike using tolerance test in a while in Data Analysis, you cannot specify the limits within which to test on the curve itself.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

Tolerance Test Dialog Box



Value to Test

For expansion coefficient calculations is CTE, the available value for selection is the coefficient of expansion. For Delta Y it is Delta Y, and for Slope the values are Slope and Inverse Slope.

Test

Types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X value. For example, for a expansion coefficient tolerance test, there are no units displayed for Minimum and Maximum.

Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

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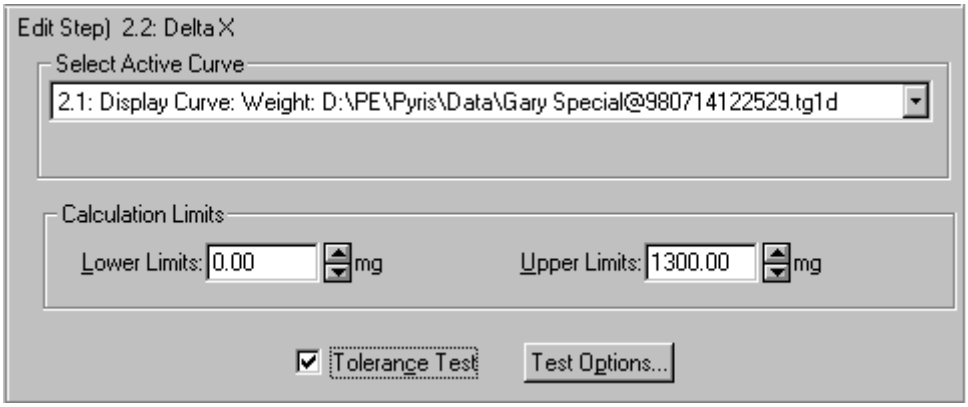
Action Upon Failure

If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Edit Step: Delta X

Delta X is an option in the Calculation Options drop-down menu for TGA Weight and Weight % curves and for all DMA 7e and TMA 7 curves. The Delta X calculation determines the change in the abscissa axis after you select two points on the Y axis for active curve.



Select Active Curve

Select a curve from the drop-down list of active curves available.

Upper Limits

Enter the Y value of the upper limit for the Delta X calculation. The default upper limit depends on the selection made for Y Data in the instrument's Preferences page.

Lower Limits

Enter the Y value of the lower limit for the Delta X calculation.

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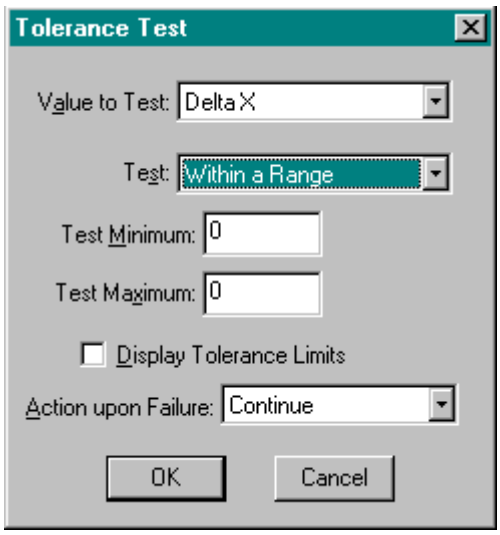
Tolerance Test

Click in the check box if you want a tolerance test performed on the results of the peak search calculation. A tolerance test checks whether the data are within a specific range. Unlike using tolerance test in a Peak Search calculation while in Data Analysis, you cannot specify the limits within which to test on the curve itself.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

Tolerance Test Dialog Box



Value to Test

Delta X only.

Test

Types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X values.

Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

Action Upon Failure

If the data fail the tolerance test, you can have the play list do one of the following:

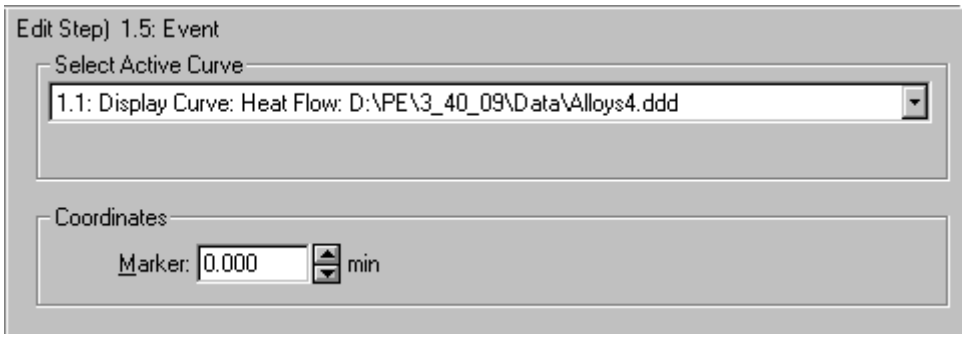
- Continue

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- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Edit Step: Event

Event lets you label the X and Y coordinates of an event on a curve. A tic mark appears at the user-selected position and the mark is annotated with the X value and unit and the Y value and unit. When you select Event from the Calculation Options drop-down list for a Display Curve play list item, the Edit Step area displayed contains the following fields:



Select Active Curve

Select a curve from the drop-down list of active curves available.

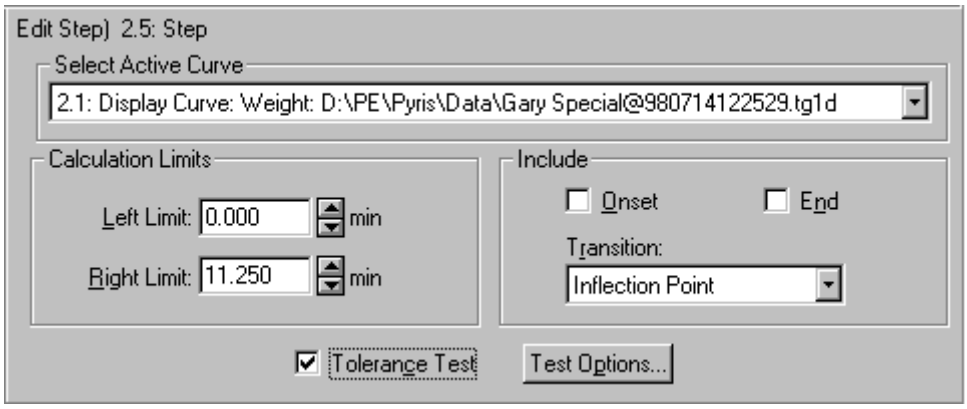
Marker

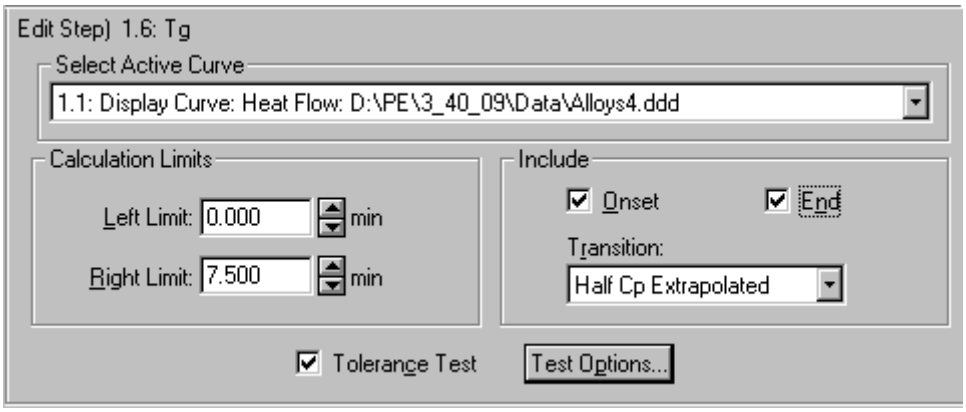
Enter the X value of the event you wish to mark.

Edit Step: Step and Tg

A Step calculation can be applied to TGA and DMA/TMA data. It finds the starting point, midpoint, and end point of a step transition. A Tg calculation is used for heat flow curves. It determines the starting point, midpoint, and end point of a glass transition.

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Select Active Curve

Select a curve from the drop-down list of active curves available.

Left Limit

Enter the left limit of the X axis for the step transition or the glass transition calculation.

Right Limit

Enter the right limit of the X axis for the step transition or glass transition calculation.

Include

Select Onset and End to have the onset and end temperatures calculated and included in the results. The onset value is calculated by finding the intersection of the extrapolated tangent at the first limit and the extrapolated tangent at the inflection point. The end value is calculated by finding the intersection of the extrapolated tangent at the second limit and the extrapolated tangent at the inflection point.

Transition

Select the transition type to use in the step transition or the glass transition calculation:

Inflection Point reports the point between the limits at which the slope of the curve changes from increasing to decreasing or vice versa.

Half Height (step transition only) reports the point on the curve that is halfway between the extrapolated tangent lines.

Half Width reports the point on the curve that is halfway between the onset and end points.

Half Cp Extrapolated (Tg only) reports the point on the curve where the specific heat change is half of the change in the completed transition.

Fictive Temperature (Tg only) reports the point on the enthalpy curve where the change of slope occurs.

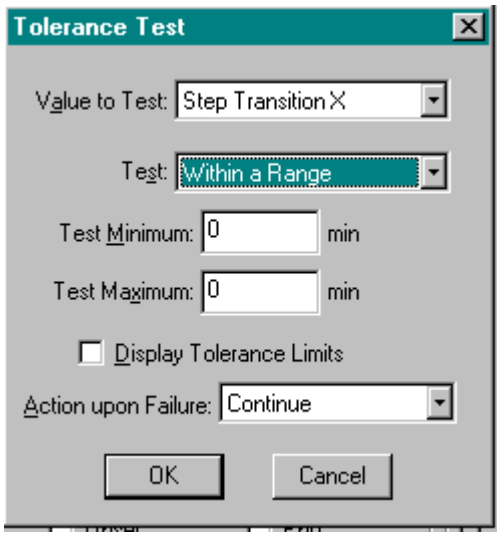
Tolerance Test

Click in the check box if you want a tolerance test performed on the results of the step transition or the glass transition calculation. A tolerance test checks whether the results are within a specific range. Unlike using tolerance test in a while in Data Analysis, you cannot specify the limits within which to test on the curve itself.

Test Options

In calculation dialog boxes as well as in Edit Step areas for calculations in a play list, you can elect to have the Pyris Software for Windows perform a tolerance test (pass/fail) on the results by clicking in the Tolerance Test check box. This activates the **Test Options** button. Click on the button to display the Tolerance Test dialog box where you specify the criteria and limits to be used for the test.

Tolerance Test Dialog Box



Value to Test

For Step Transition calculations, values available for selection are Step Transition X, Delta X, Onset X, and End X. For Tg they are Glass Transition X, Delta Cp, Onset X, and End X.

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Test

Types are Within a Range, Target +/– Tolerance, Greater Than, Greater Than or Equal To, Less Than, and Less Than or Equal To. The selection here affects the items displayed below it. For Within a Range, you enter the minimum and maximum X or Y values, depending on the Value to Test selection. For example, for a Step Transition X tolerance test, the units displayed for minimum and maximum are minutes.

Display Tolerance Limits

You can choose not to display the tolerance limits that you define on the curve. Sometimes the display may have so many annotations that it becomes difficult to read, so not displaying these limits may be helpful.

Action Upon Failure

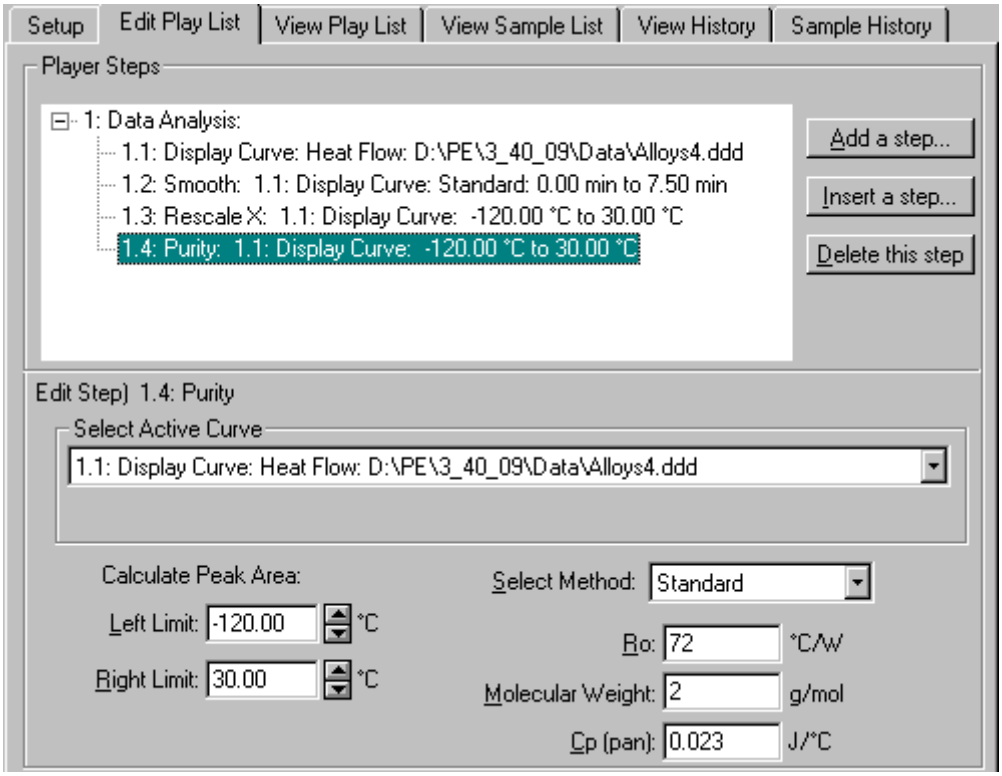
If the data fail the tolerance test, you can have the play list do one of the following:

- Continue
- Stop Playback
- Skip to Next Block: If the play list is currently in a Sample List of a Sample Group, this command tells the play list to jump out of the Sample Group and to the next main-level item in the play list.
- Pause
- Print and Continue
- Skip to Next Sample: This applies to Sample Lists in Sample Groups. Instead of skipping all the remaining samples in a sample list if a run fails the tolerance test, this command tells the play list to go to the next sample in the list.

Edit Step: Purity

When you select Purity from the Calculation Options drop-down list for a Display Curve play list item, the Edit Step area displays parameters for calculating the purity of sample by fitting a portion of the data from a DSC analyzer to the Van't Hoff relationship. The data must be a heat flow curve and the X axis must be Temperature. The fit determines the reliability of the purity measurement for the sample. To perform a purity analysis on DSC heat flow data, you must use data that is subtracted, smoothed, or optimized. The screen below shows a play list that first smoothes the data file and then rescales the X axis to temperature in order to perform a Purity analysis. Purity will not appear in the Player Step Options Calculations drop-down list unless the data file meets these criteria.

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Select Active Curve

Select a curve from the drop-down list of active curves available.

Calculate Peak Area

Left Limit

Enter the left limit for the peak area calculation to be used in the purity calculation.

Right Limit

Enter the right limit for the peak area calculation to be used in the purity calculation.

Select Method

There are two methods for calculating the purity parameters: the standard method and the multiple linear regression method. The one you select depends on the baseline of the data curve that you are analyzing for purity.

If your DSC data is characterized by a conventional melting curve in which the melting peak is complete, the total area under the peak is calculated. Therefore, it is not necessary to create a new baseline. In this case, you can use either the standard or the MLR method. The standard method involves an iterative calculation of the x-correction, followed by a multiple linear regression on the two remaining purity parameters.

In some DSC analyses, the sample may decompose during the melting phase. This decomposition may produce a melting curve in which the melting peak is incomplete and the total area under the peak cannot be obtained. For these analyses, you must use the multiple linear regression method of calculating purity of the sample. This requires creating a new baseline for the data curve. The

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MLR method uses a 3 x 3 multiple linear solution which determines the three purity parameters simultaneously. The method does not require obtaining the total area under the peak.

R0 (Thermal Resistance Constant)

Enter the thermal resistance constant for your sample pan. The value depends on the type of pan you are using, the way the pan has been crimped, and the temperature of the reaction. For most experiments, use the default value of 72.00 °C/W. However, under certain conditions it may be necessary to calculate R0 for the sample pan:

- using a nonstandard sample pan such as graphite, alumina, or high-pressure capsule
- the reaction takes place over a high temperature range, e.g., 400°C

Molecular Weight

Enter the molecular weight (in g/mol) of your sample material. Purity calculations cannot be performed until the molecular weight has been entered.

Cp (pan)

Enter the heat capacity for the sample pan. This value depends on the type of pan used. The value is used to perform a minor correction in the purity calculation. Use the default 0.023 J/°C for most applications. However, if you are using high-pressure capsules, you may want to calculate the heat capacity of your sample pan material.

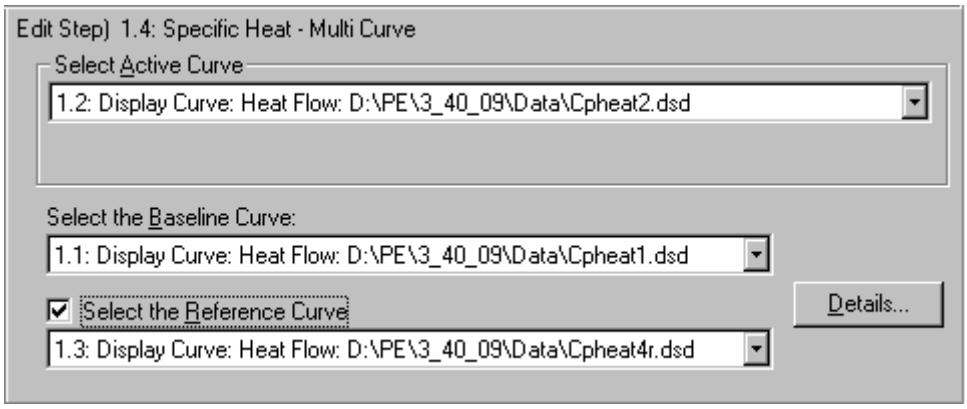
Edit Step: Specific Heat

The option of specific heat calculated using multiple curves is available on the Calculations drop-down menu if there are two or more curves displayed. The curves must be Heat Flow curves generated by a DSC 7, Pyris 6 DSC, or Pyris 1 DSC. When you select Specific Heat – Multi-Curve from the Calculation Options drop-down menu in the Player Step Options dialog box for Display Curve, if there is no curve available to select for the Baseline curve, you will see the message:

This item will be deleted. A baseline curve with zero sample weight is required for multiple curve Cp.

The Specific Heat entry will not be added to or inserted into the play list in this case. If a Baseline curve with zero sample weight is displayed through a Display Curve entry, then the Edit Step area displays the following fields:

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Select Active Curve

Select a curve from the drop-down list of active curves available. The curve must be a result of an Iso-Scan-Iso temperature program, where the scanning step could be either heating or cooling.

Select the Baseline Curve

Each curve above the Specific Heat entry that meets the following criteria is displayed in the drop-down list for Baseline Curve: The curve must (1) be a heat flow curve, (2) have the form of Iso–Scan–Iso, (3) have the leading and trailing isothermal steps and the scanning step be at least 1 minute long, and (4) have the starting and ending temperatures in the scan be the same in all curves. The baseline curve results from performing a run using an empty sample pan (baseline pan) of the same type used for the sample run and using the same method. Select the curve you want for the baseline curve by clicking on it. If you click on the Details button after selecting a Baseline curve but not a Reference curve, a compressed Details dialog box is displayed.

Reference Curve check box

If you want to use a reference curve in the calculation of specific heat, click in the box. (This box does not appear if there are only two curves available for the specific heat calculation.) When you select Reference Curve and click on the **Details** button, an expanded Details dialog box is displayed.

Select the Reference Curve

Each displayed curve that meets the following criteria is displayed in the drop-down list for Reference Curve: The curve must (1) be a heat flow curve, (2) have the form of Iso–Scan–Iso, (3) have the leading and trailing isothermal steps and the scanning step be at least 1 minute long, and (4) have the starting and ending temperatures in the scan be the same for all curves. The Reference curve results from performing a run with a reference material contained in a reference pan of the same type used for the sample run and using the same method. Select the curve you want for the reference curve.

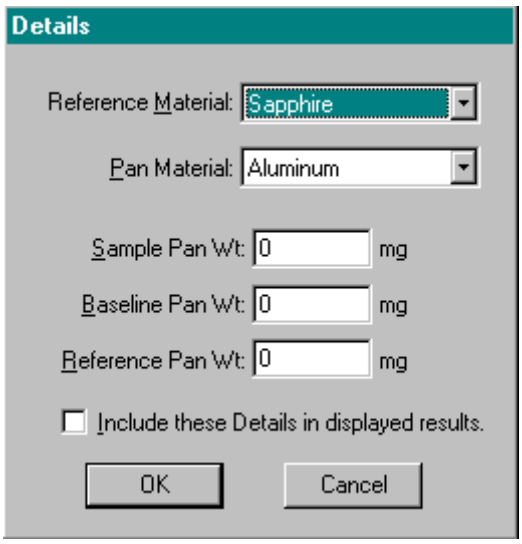
Details

To add details to the specific heat calculation and have them displayed, click on this button to display the Specific Heat Details dialog box.

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Specific Heat Details Dialog Box

This dialog box appears if you click on the **Details** button in the Pyris Player's Edit Step area for Multi-Curve Specific Heat calculation and a reference curve has been selected. Here you enter details on the sample pans and the reference material that were used in collecting the data curves to be used in the Specific Heat calculation.



R0

Enter the thermal resistance constant of the sample pan. This parameter appears in the Details dialog box for a two-curve specific heat calculation only.

Reference Material

Enter the name of the reference material used for the Reference curve, if applicable. You can also select the name from the drop-down menu.

Pan Material

Enter the name of the pan material used in the sample run, the baseline run, and the reference run. You can also select the name from the drop-down menu.

Sample Pan Weight

Enter the weight (mg) of the sample pan used for the sample run. This is used if the heat flow curve is not normalized.

Baseline Pan Weight

Enter the weight (mg) of the sample pan used for the baseline run.

Reference Pan Weight

Enter the weight (mg) of the sample pan used for the reference run, if applicable.

Include these Details in Displayed Results check box

Click in the check box to have these details annotated to the display in the Data Analysis window.

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Details Dialog Box

This dialog box appears if you click on the **Details** button in the Edit Step area for two-curve specific heat calculation for which there would be no reference curve or for a multicurve specific heat calculation with no Reference curve. It contains the following field:

R0 (Thermal Resistance Constant)

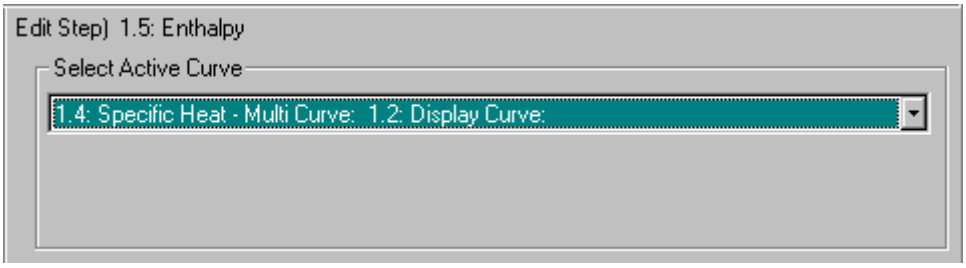
Enter the thermal resistance constant for your sample pan. The value depends on the type of pan you are using, the way the pan has been crimped, and the temperature of the reaction. For most experiments, use the default value of 25.00°C/W. However, under certain conditions it may be necessary to calculate R0 for the sample pan. These conditions are

Include these Details in Displayed Results check box

Click in the check box to have these details annotated to the display in the Data Analysis window.

Edit Step: Enthalpy

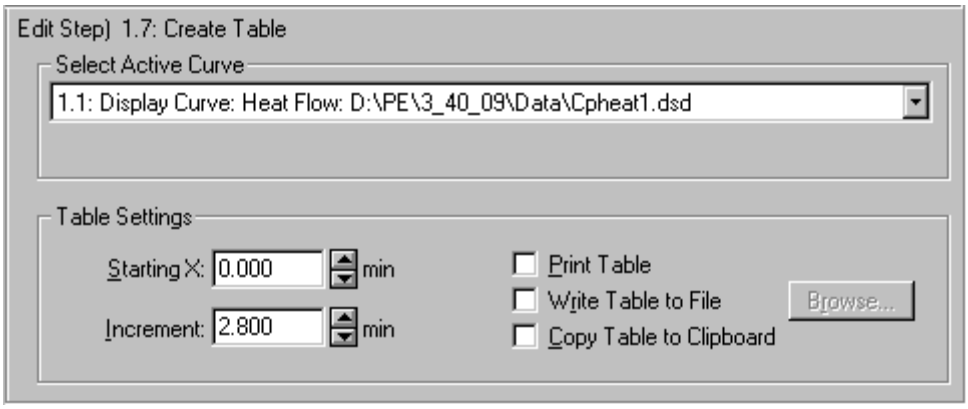
The Enthalpy calculation applies to specific heat curves only. It appears on the Calculation Options list in the Player Step Options dialog box when a specific heat curve is available at that position. When selected, the Edit Step area will display the Select Active Curve drop-down list which contains all the Specific Heat curves currently displayed. This option calculates the enthalpy change of the material which is the integral of the specific heat over the specified temperature range. Enthalpy curves are generally calculated from the total specific heat curve but can be generated from any other specific heat curve generated by Pyris.



Edit Step: Create Table

The Create Table command in the Calculation Options list is available for all analyzers. This command takes the data points of the selected curve and displays them in tabular form. When you select Create Table, the Edit Step area displayed contains the following fields:

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Select Active Curve

Select a curve from the drop-down list of active curves available.

Table Settings

Starting X

The X value of the first data point at which you want to start the table. The default value is the X value of the first data point of the file.

Increment

You can modify the number of data points in the table by choosing to use X values at a specified increment.

Print Table

Click on the box to have the table printed out.

Write Table to File

Click on the box to have the table saved to a file. The **Browse** button becomes active when this option is selected. Click on the **Browse** button to display the Write Table to File dialog box. In the File name field enter the file name for the table. The default file name is OUTPUT.TXT and the default directory is the same directory as the data file. Click on **Save**. The file is saved in ASCII format.

Copy Table to Clipboard

Click in this box to save the table to the clipboard. You can then paste it into another application.

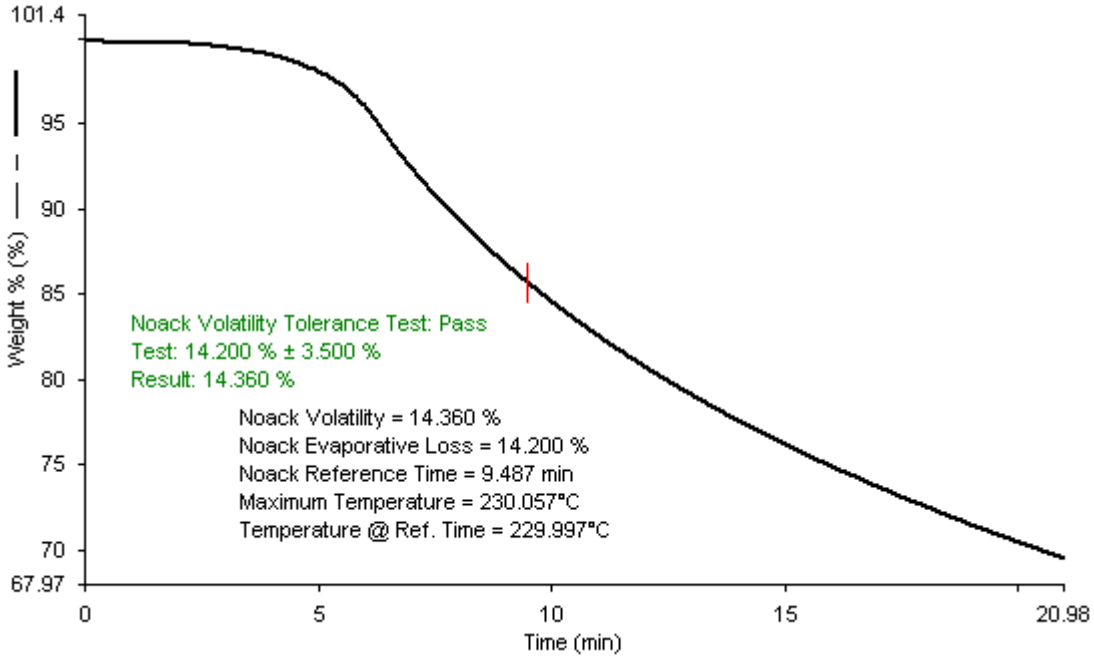
Edit Step: Noack Test

The Noack test is used for testing for results of oil sample runs. It is a Calculations option for play lists for TGA analyzers. It is available only in the Pyris Player environment. It will appear in the Calculations options drop-down list in the Player Step Options dialog box when your play list has at least two Display Curves items above the Noack test and each curve is a Weight or a Weight % versus Time. If any of the curves selected for use in the test are not Weight % or the X scale is not Time, the appropriate changes are made.

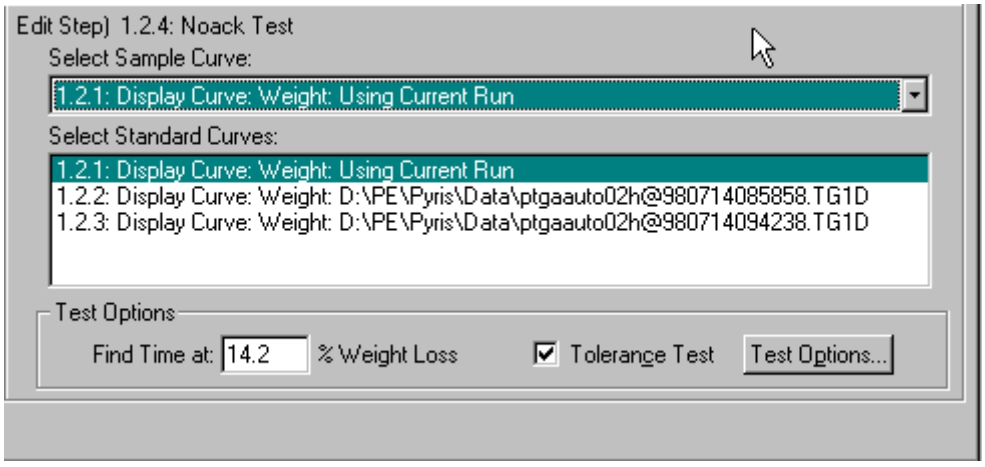
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To perform the test, one sample curve is selected along with one or more standard curves. The time for the given weight loss (Noack Evaporative Loss) is found. This weight loss is actually a change in weight % from the initial weight % in the curve. If the first point is at 99% and the Noack Evaporative Loss is 14%, then the time corresponding to 85% is found. The time is found for each standard curve selected and then averaged. That time (Noack Reference Time) is then used to find the weight loss % in the sample curve. This is called the Noack Volatility.

The results of the test display Noack Volatility, Noack Evaporative Loss, Noack Reference Time, Maximum Temperature, and Temperature at Reference Time on the curve display.



When Noack Test is selected from the Calculation Options, the Edit Step: Noack Test area appears:



Select Sample Curve

Select the curve from the play list on which to perform the Noack test. All Display Curve items above this point in the play list are available for selection as the sample curve. The curve selected in Display Curve can be from the current run if you are using the Noack Test in a Data Analysis List in a Sample Group. This way the results can be tested immediately. The sample curve can be one collected from the play back of a standard play list (no Sample Group) or it can be an existing file. Since the Noack Test is available in Pyris Player only, you can easily create a play list containing just Data Analysis as the main item with Display Curves calling in existing data files for use in the Noack Test.

The result of the Noack test calculation is saved in the sample curve data file. Therefore, if you use Copy to Clipboard after Noack Test and select Graph Image, the curve copied to the clipboard Noack test result but has the sample curve's file name.

Select Standard Curves

Select one or more standard curves against which the sample curve is tested. If there is more than one standard curve, then the Noack Reference Time for each curve is found and then they are averaged.

Find Time at

The default value of 14.2% is the Noack Evaporative Loss value. You can change this value, perhaps to see at what % the test fails.

Tolerance Test

Click in the Tolerance Test check box to activate the **Test Options** button. Click on the **Test Options** button to display the standard Tolerance Test dialog box. However, the only items that you can change are the Tolerance value and the Action Upon Failure. The default Tolerance is 3.5%.

Edit Step: Select Active Curve

In the Player Step Options dialog box, when you select Delete Curve, Derivative from the Math Options list, Single Curve Specific Heat or Enthalpy from the Calculation Options list, or Log Y from the Rescale Options list, the Edit Step area displays the following field:

Select Active Curve

This field's drop-down list contains all of the curves currently available based on the play list up to this point. The curves may be previously existing data files or from the current run of the play list. Select the curve (data file) on which you want an action (delete, calculate derivative, calculate specific heat, enthalpy, log Y) to be performed.

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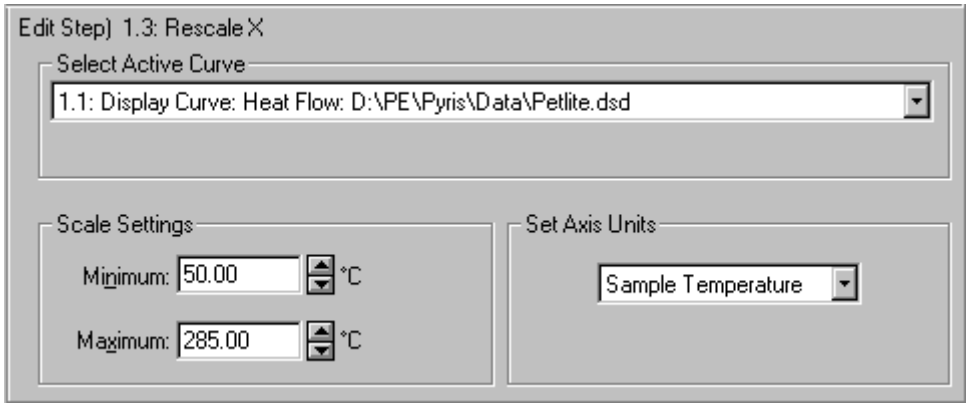
Rescale Options Drop-Down List

When Display Curve is the focused item in the play list, Rescale Options is listed as an option in the Player Step Options dialog box. This option has its own drop-down menu which contains the following items:

- Rescale X
- Rescale Y
- Full Scale
- Log X
- Log Y
- Shift Curve
- Slope
- Annotate

Edit Step: Rescale X

Rescale X is available from the Rescale Options drop-down list for all analyzers' play lists. There must be a Display Curve entry above the point at which you want to add/insert a Rescale X command. Rescaling the X axis involves changing the minimum and maximum values on the X axis. You can also change the axis units.



Select Active Curve

Select the curve to rescale from the drop-down list of available curves.

Scale Settings

Minimum

Enter a minimum X value or use the default current minimum value from the data file.

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Maximum

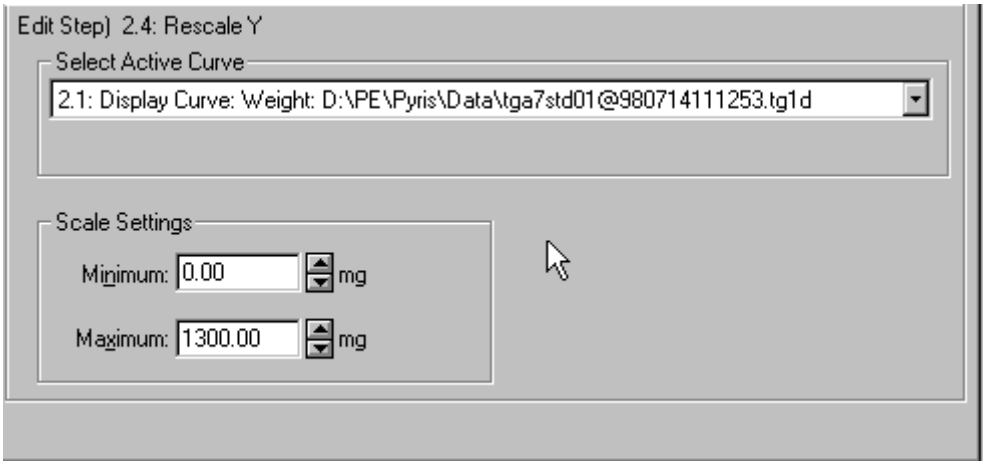
Enter a maximum X value or use the default current maximum value from the data file.

Set Axis Units

You can choose Time or Sample Temperature for the X axis unit. The default is the current unit.

Edit Step: Rescale Y

Rescale Y is available from the Rescale Options drop-down list for all analyzers' play lists. There must be a Display Curve entry above the point at which you want to add/insert a Rescale Y command. Rescaling the Y axis involves changing the minimum and maximum values on the Y axis.



Select Active Curve

Select the curve to rescale from the drop-down list of available curves.

Scale Settings

Minimum

Enter a minimum Y value or use the default minimum value from the data file.

Maximum

Enter a maximum Y value or use the default maximum value from the data file.

Full Scale

Full Scale in the play list tells the program to display the full contents of the focused curve's data file. If preceding play list commands have rescaled the file, Full Scale will return the full display of the curve.

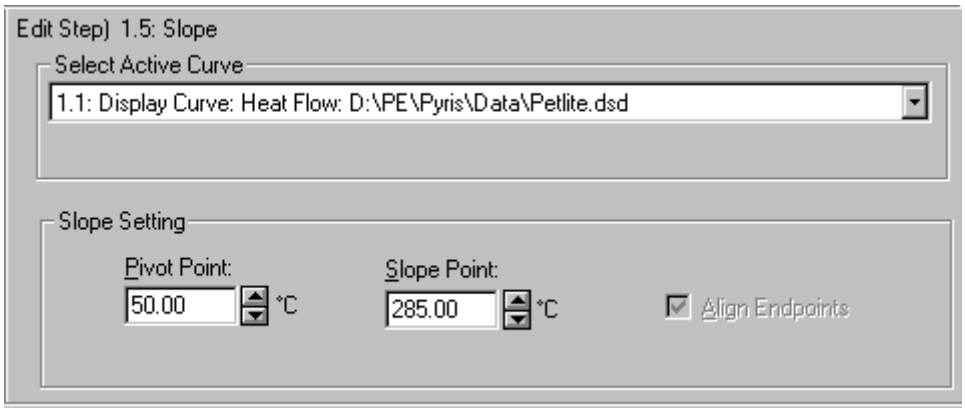
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Log X and Log Y

Log X and Log Y commands in the play list tell the program to display the selected curve on a logarithmic scale. They are available from Rescale Options in the Player Step Options dialog box for all analyzers' play lists. The Log X plot requires that the curve and the scale X axis minimum values be greater than 0. The Log Y plot requires that the curve and the scale Y axis minimum values be greater than 0. The Edit Step area of each contains no fields.

Edit Step: Slope

The Slope command is available from the Rescale Options drop-down list for heat flow curves (DSC and DTA). The Slope command changes the slope of the entire selected curve, either collected during playback of the play list or a previously existing file that is displayed. You can adjust the slope to compare a curve with a similar curve or curves.



Select Active Curve

Select the curve whose slope you want to change from the drop-down list of available curves.

Pivot Point

The left end of the slope line is the pivot point of the slope line. Enter the X value of the point you want to use as the pivot for the slope calculations.

Slope Point

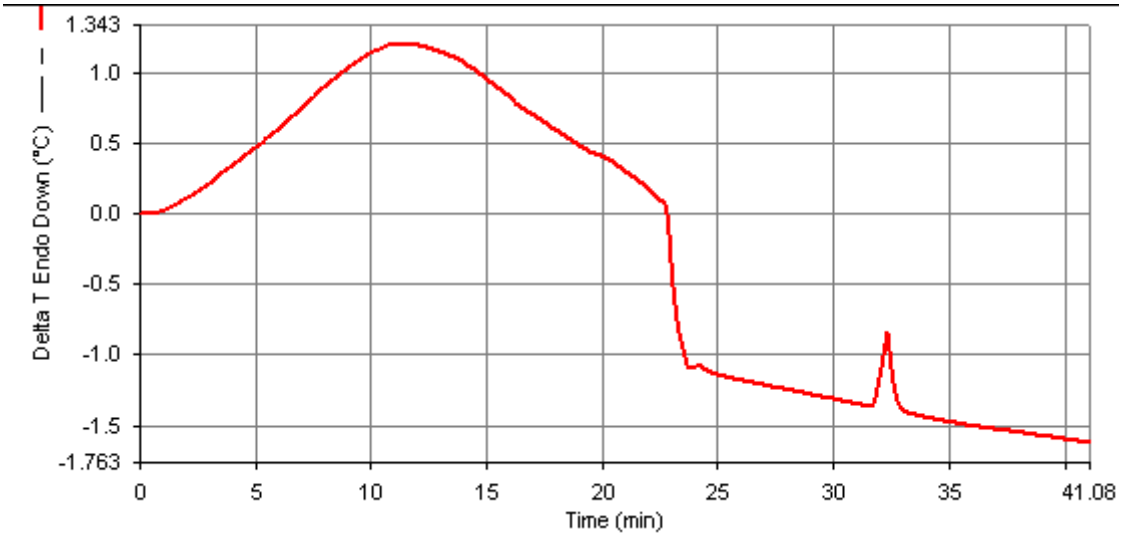
Enter the Y value of the point that you want to use to use for the slope line. The entire curve will be sloped accordingly.

Align Endpoints

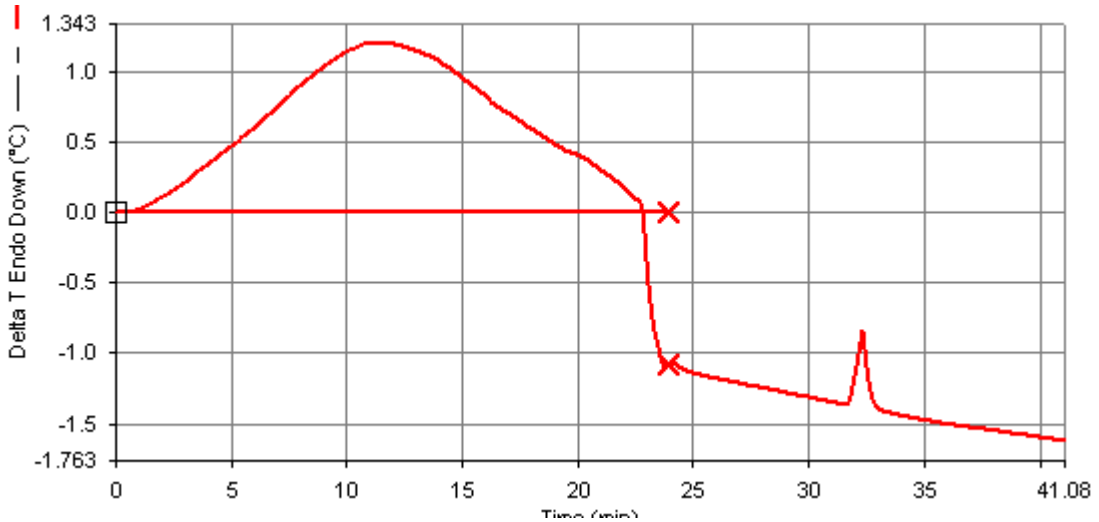
This is automatically "on" in Player since you cannot see the curve as you would in Data Analysis. This point in space gets its x value from the Slope Point and its y value from the Pivot Point.

The screens below show what happens with the Slope calculation. The first screen shows a Delta T curve for a DTA 7.

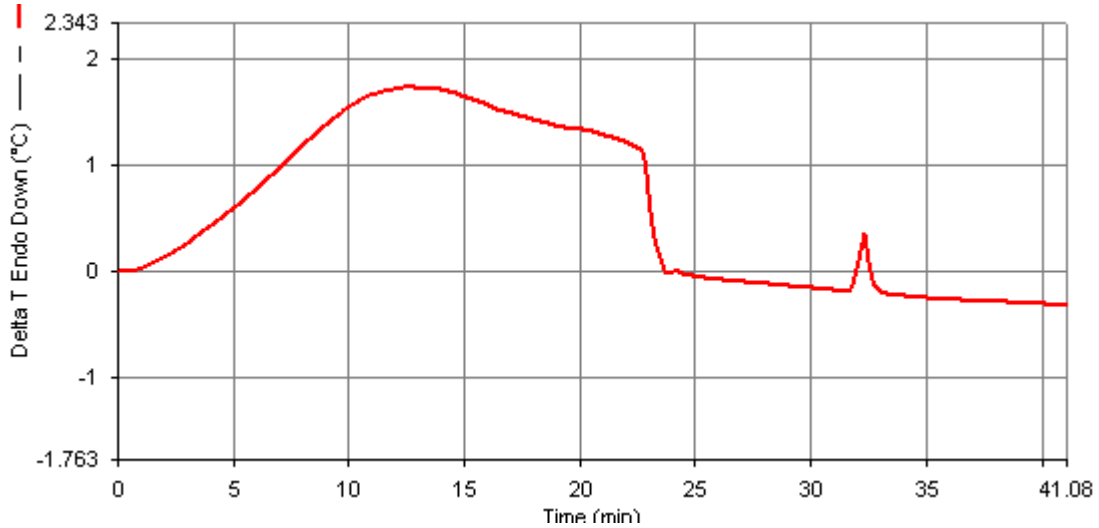
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The next screen shows the Align Endpoints:

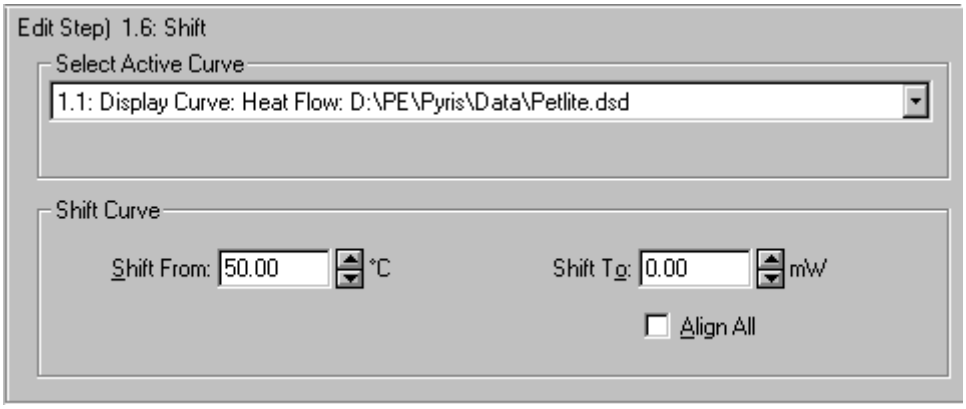


The last screen shows the results of the changed slope:



Edit Step: Shift Curve

Shift Curve is available for a play list from the Rescale Options drop-down list in Player Step Options for heat flow curves (DSC and DTA). Use Shift Curve to move the selected curve up or down along the Y axis, i.e., offset the curve. This is handy when you have two or more similar curves displayed and want to compare them.



Select Active Curve

Select the curve you want to shift from the drop-down list of available curves.

Shift Curve

Shift From

Enter the X coordinate of the curve that you want to use as the shifting point.

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Shift To

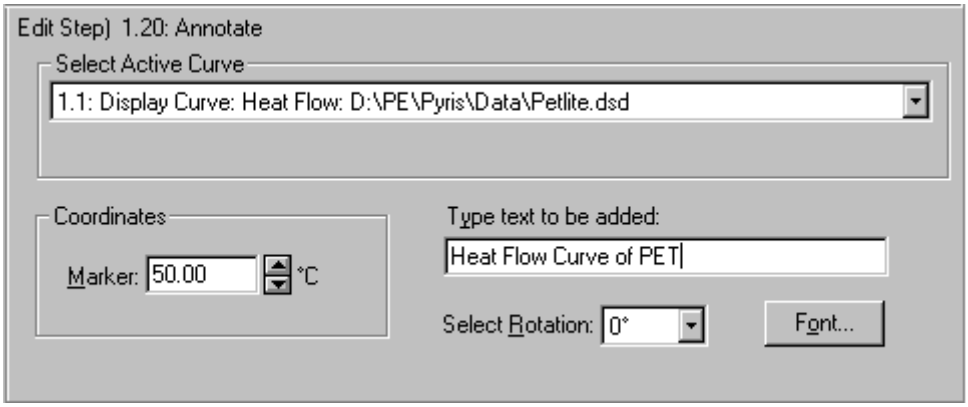
Enter the Y coordinate to whit to shift the active curve. The command takes the Y coordinate value of the Shift From X coordinate and shifts the curve from that Y value to the Shift To Y value.

Align All

Selecting this box shifts all curves that use the same Y axis as the active curve to the Shift To coordinate.

Edit Step: Annotate

Annotate in the play list adds a label to the selected curve. It is available from Rescale Options in the Player Step Options dialog box for all analyzers' play lists.



Select Active Curve

Select the curve to which you want to add a label from the drop-down list of available curves.

Marker

Enter the X value at which the label will appear near the curve.

Type text to be added

Enter the text (up to 40 characters) of the label.

Select Rotation

From the drop-down list, select the angle at which to display the annotated text label: 0°, 90°, 180°, or 270°.

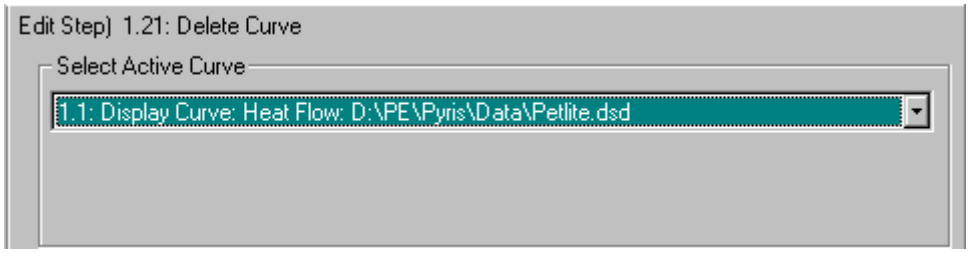
Font

This button displays the standard Windows Font dialog box in which you can select a typeface, type style, and type size for your text label.

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Delete Curve

Delete Curve is an option in the Play List Options dialog box when Data Analysis is the preceding main-level item. This option would be selected somewhere after a Display Curve entry in the play list. You may have your play list set up to display curves that are used in some mathematical or calculation option. Once the calculation is performed and the resulting curve is displayed, you may want to remove the curves used in the calculation from the Data Analysis window. Delete Curve is used for this purpose. When you select this option, the Edit Step area displays the Select Active Curve field. Delete Curve removes one curve at a time.



Copy to Clipboard

A feature of Pyris Player Data Analysis is Copy to Clipboard. If there is a Display Curve entry in the Data Analysis or Data Analysis List section of your play list, then Copy to Clipboard becomes an option in the Player List Options dialog box. Copy to Clipboard will copy either the method used to collect the selected data file, the X - Y data of the curve, or the graphic image of the curve to the clipboard. You can then paste the data into a third-party software such as PowerPoint or Word. The graphic image is the same as you get with Copy Image, i.e., no peripheral items from the screen, just the axes, labels, and the curves.

Select Active Curve

Select the curve you wish to copy to the clipboard from the list of curves available. All curves that are displayed, calculated, or collected by the play list above the current point in the play list are available.

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Method Used

The information on the method used to collect the selected data file is sent to the clipboard. An example of a method used pasted into a Word document is seen below:

Filename:	C:\Program Files\Pyris\tga7std01.tg1d
Operator ID:	
Sample ID:	
Comment:	
Serial Number:	
Data Collected:	7/14/98 2:36:14 PM
Validation	
Validated:	No
By:	
Date:	
Calibration Information	
Filename:	C:\Program Files\Pyris\kcgood.tg1c
Date/Time:	7/9/98 9:39:22 PM
Initial Conditions	
Temperature:	50.00°C
Sample Rate:	Standard
Purge Gas:	Nitrogen
Purge Gas Rate:	20.0 ml/min
Equilibrate Within	
Temperature:	1.00°C
Maximum Time:	10.00 min
Weight:	1.000 mg
Baseline Filename:	C:\Program Files\Pyris\Data\ptgabaseline.tg1d
End Condition:	Go To Temp
Method Steps:	
1)	Hold for 1.0 min at 50.00°C
2)	Heat from 50.00°C to 300.00°C at 40.00°C/min
3)	Cool from 300.00°C to 50.00°C at 40.00°C/min

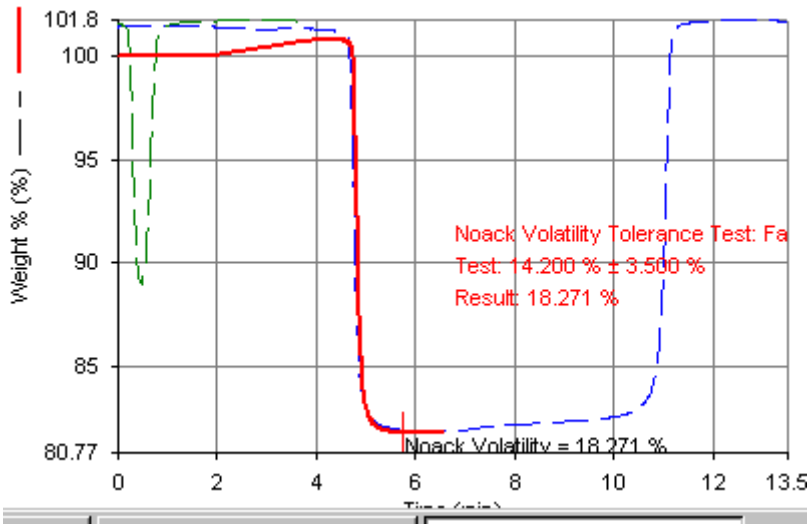
X - Y Curve Data

The data points that make up the data curve can be copied to the clipboard in ASCII format and pasted into another software package.

Graph Image

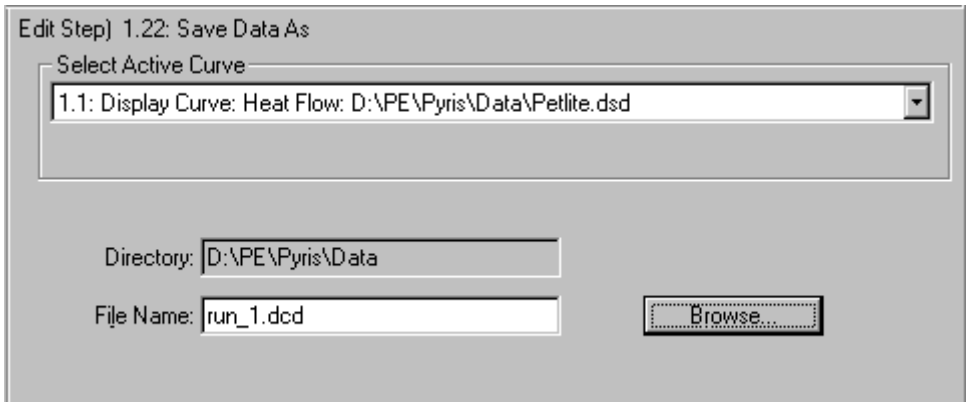
The curve, labels, and axes labels are copied to the clipboard. The peripheral items such as scroll bars and title bar are omitted. An example is shown by below:

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Save Data As

Save Data As is an option in the Play List Options dialog box when Data Analysis is the preceding main-level item. This option would be selected somewhere after a Display Curve entry in the play list. Your play list may perform some type of calculation whose resulting data curve you want to save in a new data file. For example, if your play list performs an Add Curves using two displayed data curves, the resulting curve replaces the selected active curve. If you select Save Data As, the resulting curve will be saved to a new data file, preserving the original data file.



Select Active Curve

This drop-down list displays all the curves displayed in the Data Analysis window that are available for selection, i.e., those created from running the play list and those existing files selected for display in Display Curve.

Directory

The drive and full directory path to which the selected curve will be saved is displayed for information only.

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File Name

Enter the file name to which to save the curve or use the Browse dialog box, displayed by clicking on the **Browse** button, to get to the desired directory and then enter the file name in this field. After entering the file name, the drive and directory are displayed in the Directory field.

Save All

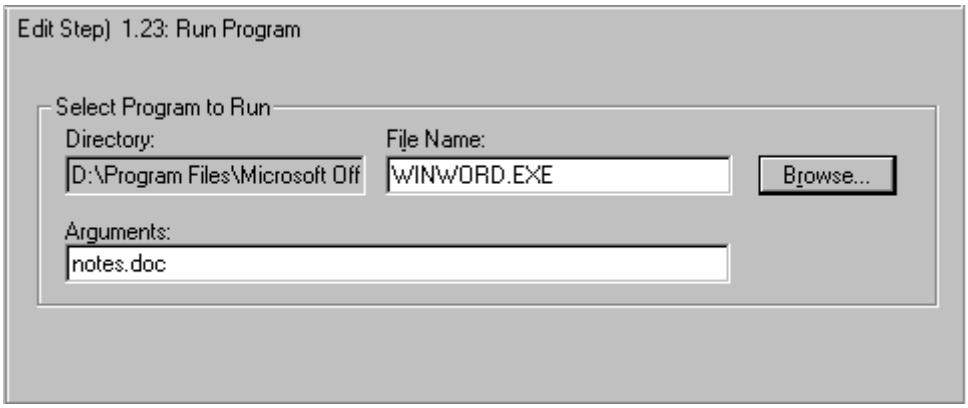
Save All is an option in the Play List Options dialog box when Data Analysis is the preceding main-level item. This option would be selected somewhere after a Display Curve entry in the play list. Your play list may perform some calculations whose resulting data curves are displayed in the Data Analysis along with the curves used for the calculations. Before deleting any curves from the Data Analysis window, use Save All to save all curves. There are no fields displayed in the Edit Step area. After Save All in the play list, you can use Delete Curve for each curve you want to remove from the Data Analysis window.

Print

The Print option is available in the Player List Options dialog box when the most recent main-level item is Data Analysis. The Print command tells the system to print the curves in the Data Analysis window at that point. There are no fields in the Edit Step area for Print.

Run Program

Run Program is available in the Player List Options dialog box when the most recent main-level item is Data Analysis. This option gives you access to third-party programs. Enter the file name of the executable file in the File Name field. Use the **Browse** button to display the Select Program to Run dialog box in which you can search for and select the executable file. The drive and directory path are displayed in the Directory field automatically. Enter the arguments needed for the program in the Arguments field.



Creating and Editing a Play List

When creating a play list for any analyzer, the items in the Player Step Options dialog box consists of Prepare Sample, Data Analysis, and Sample Group. The list of items in the Player Step Options dialog box when adding a line to or inserting a line into the play list depends on analyzer and the focused step at the time.

When you click on **Add a step**, the new step is always appended to the end of the play list, no matter which play list item is highlighted. When you click on **Insert a step**, the new step is inserted above the focused step. The choices displayed in the Player Step Options dialog box depend on what main-level item the added or inserted item will appear beneath: Prepare Sample, Data Analysis, or Sample Group. If you are adding or inserting a line beneath a Display Curve entry, which would appear under Data Analysis, the Player Step Options dialog box contains the same selections that appear in the Math, Calc, and Display menus.

The main-level items in a regular play list are numbered 1, 2, 3, etc. The second-level items are numbered x.1, x.2, x.3, etc., where x is the number of the main level item directly above. These items are indented. Dotted lines connect the main-level items to their second-level items. Since the area in which a play list's steps is small in the Edit Play List page, you can see more of the list in the View Play List page. You can get a better picture of the structure of the play list. As you add or insert steps into the play list, the Edit Step area below the Player Steps box displays information and fields specific to that step.

Another main-level item for a play list is Sample Group. This is a special feature of a play list that is meant for analyzing samples using an autosampler. A Sample Group comprises a Sample List and a Data Analysis List. The steps that you would have to enter into a regular play list are added automatically when a Sample List is run. A Sample Group can be embedded within a regular play list or it can stand alone. A play list can contain many Sample Groups. A Sample Group can be used with an analyzer without an autosampler. Automatic Pause dialog boxes are displayed at the appropriate points by the program. You would then have to remove or load a sample.

See Chapter 11, *Quick Help*, for four lessons on how to create a play list – two for instruments with and two for instruments without autosamplers.

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Chapter 10 Applications

Sample applications are in the following sections. Each application gives sample preparation, the method parameters, and the results.

DSC Applications

The following list is a cross section of the types of applications for which you can use a DSC analyzer:

- Oxidative Induction Time
- Oxidative Induction Time (OIT) of Lubricating Materials by HPDSC
- Quantitative Analysis of Semi-Crystalline Polymer Blends or Mixed Recyclate
- DSC Isothermal Crystallization
- Effect of Sample Weight on a DSC Run
- Determining Vapor Pressure by Pressure DSC

Oxidative Induction Time

Introduction

Oxidative induction time is an accelerated test used as a qualitative evaluation of the stability of a material. It is typically used to assess antioxidant formulations in plastics but is also applicable to many other materials.

Theory

An antioxidant prevents the propagation of oxidation reactions but is consumed in the process and, when completely consumed, the oxidation reactions proceed rapidly. Since these reactions are highly exothermic, the onset and the trigger point are easily determined by DSC.

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Instrument

Pyris 1 DSC or DSC 7 with GSA 7 or TAGS gas selector accessory

Procedure

The sample used to illustrate this application is polyethylene. The method involves heating to a preset temperature in a nitrogen atmosphere and equilibrating there. The atmosphere is then switched to oxygen and the time to the onset of degradation is measured. The isothermal temperature is typically between 180°C and 200°C for polyethylene.

Sample Preparation

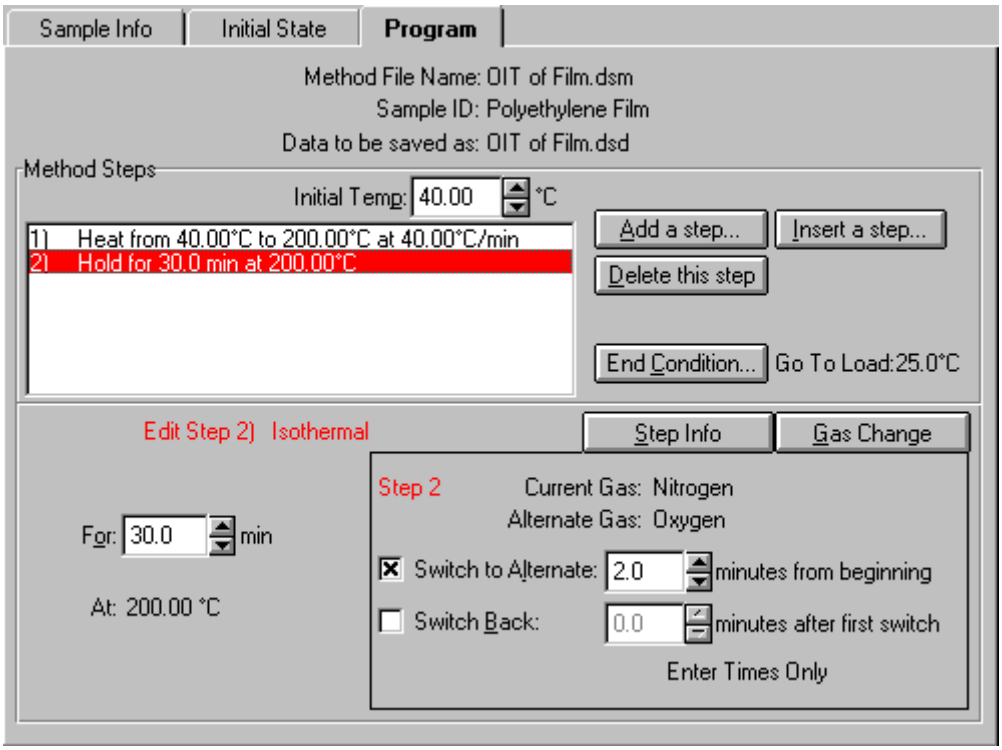
Consistent surface area and sample weight are very important for adequate reproducibility. Punch or razor cut samples and accurately weigh the sample. (For a discussion on sample preparation, including videos, click here.)

Place the sample in an open standard aluminum sample pan.

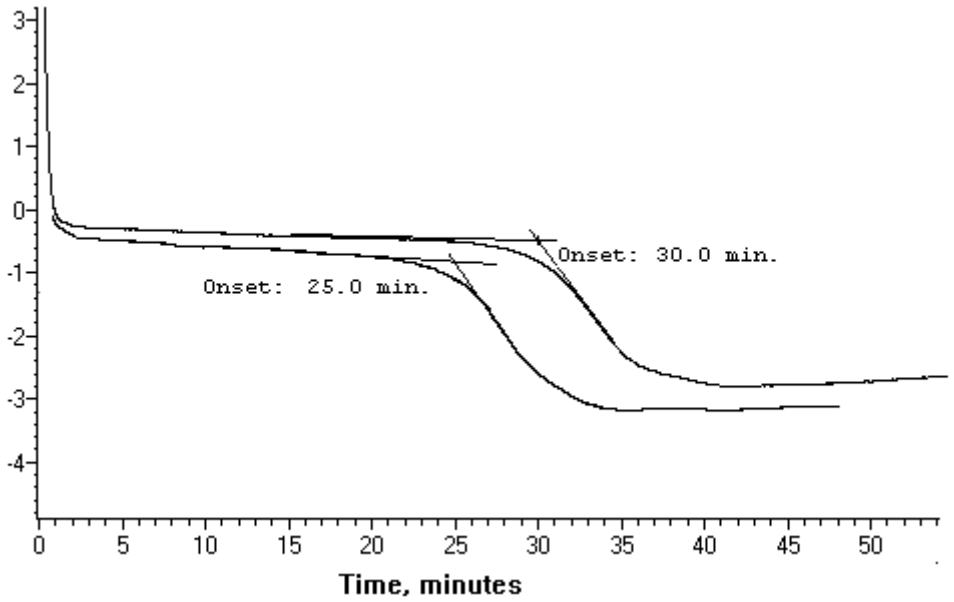
Method

The temperature range of the experiment is from 40°C to selected temperature (180°C – 200°C).

Program up to isothermal temperature at 40°C/min in nitrogen. Wait for sample equilibration at the selected isothermal for 2 minutes before changing to oxygen.



Results



Oxidative Induction Time of Lubricating Materials by High Pressure Differential Scanning Calorimetry

Introduction

Oxidative induction time by high-pressure differential scanning calorimetry (HPDSC) is an accelerated test to qualitatively assess the service life and stability of a lubricant. It is frequently used in evaluating antioxidants and characterizing these complex formulas.

Theory

An antioxidant prevents the propagation of oxidation reactions but is consumed in the process. When the antioxidant is completely consumed, the oxidation reactions proceed rapidly. Since these reactions are highly exothermic, the onset and the trigger point are easily determined by DSC.

Instrument

Pyris 1 DSC or DSC 7 with HPDSC accessory

Procedure

A small quantity of lubricant is placed in the center of a volatile sample pan cover and inserted into the calorimeter. The instrument is programmed to a predetermined temperature (between 130°C and 210°C) and held isothermally for the duration of the experiment. After the sample has equilibrated for 2 minutes at the selected temperature, the system is pressurized with oxygen at 3.5 MPa (500 psig). The OIT is measured from the time the system is pressurized to the onset time and/or trigger time of the exothermic reaction.

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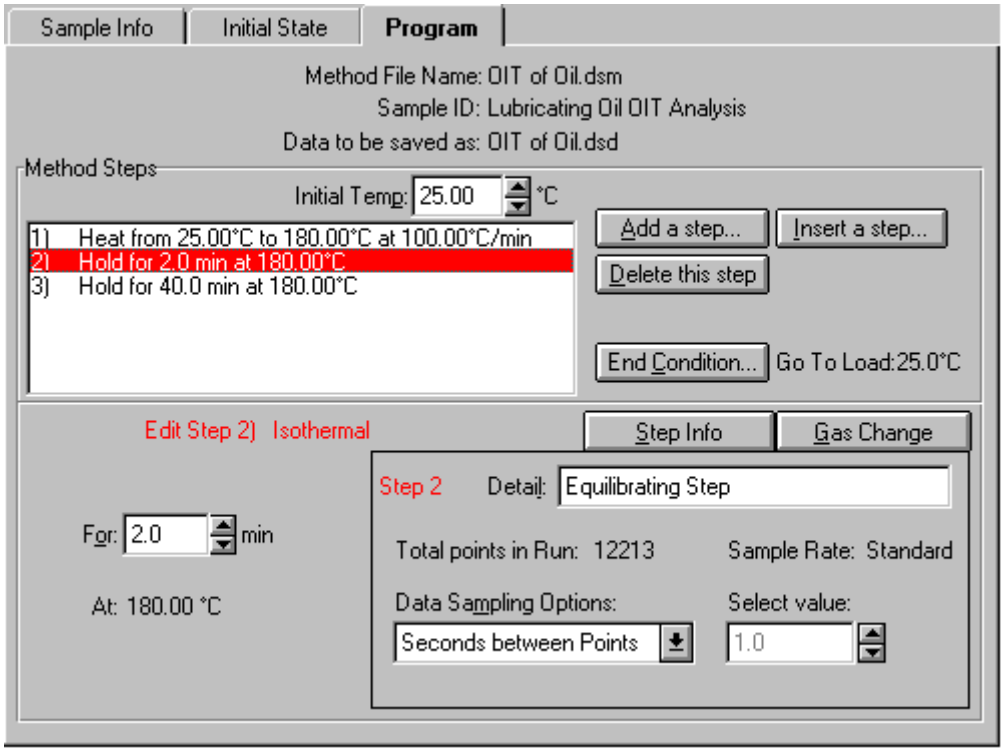
Sample Preparation

Distribute a thin layer (1 – 5 mg) of sample evenly in a volatile aluminum sample pan (0219-0062) cover and place in the HPDSC cell.

Method

The temperature range for this experiment is from ambient to selected temperature (210°C, 180°C, 155°C, or 130°C). Program up to isothermal temperature at 100°C/min. Wait for sample equilibration at selected isothermal for 2 minutes before pressurizing the system. (**NOTE:** If induction time occurs in less than 10 minutes, the next lowest isothermal temperature should be used.)

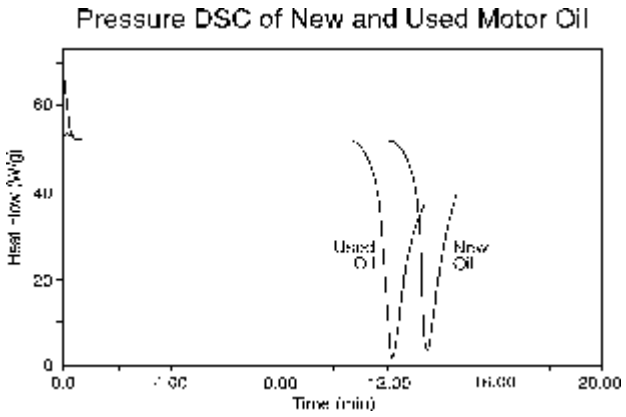
Atmospheric conditions are as follows: oxygen, minimum purity 99.5%, extra dry; pressurized to 500 psig (3.5 MPa), with a dynamic purge of 100 mL/min.



Results/Discussion

Oxidative induction time of oils and lubricants can be determined at the onset or at a trigger point. The more stable the material, the longer it will take to succumb to the oxidation process. The data in the figure below is that of a virgin oil and used auto oil (2500 miles) showing that the used oil takes less time to reach the exothermic reaction thereby indicating lower resistance to oxidation. This method is useful for predicting life expectancy and for characterizing and formulating lubricating products.

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References

1. “Analysis of Motor Oils by High Pressure DSC,” Perkin-Elmer Thermal Analysis Newsletter (PETAN) #46. The Perkin-Elmer Corporation, Norwalk, CT, 1994.
2. In-Sik Rhee, “Development of a New Oxidative Stability Test Method for Greases Using a Pressure Differential Scanning Calorimeter,” US Army Belvoir Research and Development Center, July 1991.
3. Noel, "Thermal Analysis of Lubricating Oils," Imperial Oil Enterprises Ltd., Thermochem. Acta (4), 1972.

Quantitative Analysis of Semicrystalline Polymer Blends or Mixed Recyclate

Introduction

The amount of a particular semicrystalline component in a mixture or blend can affect the processing conditions and the physical properties of the end product (1). For that reason suppliers and users of mixed recyclate and other blends often use DSC to quantify the amounts of the crystalline or semicrystalline components (2). Examples of materials that can be assayed in this manner include polyethylene, polypropylene, polyethylene terephthalate, and nylon.

Theory

The determination is based on measuring the apparent heat of fusion of the component in question. The resultant per gram energy is compared with the heat of fusion of the pure individual component under similar conditions, or to the apparent heat of fusion of this component in a known mixture. From this the amount of the component is calculated by ratios. The peak energy calculation method is consistent with the standard ASTM methods E793 and D3417 (3).

Instrument

DSC 7, Pyris 1 DSC, or Pyris 6 DSC

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Procedure

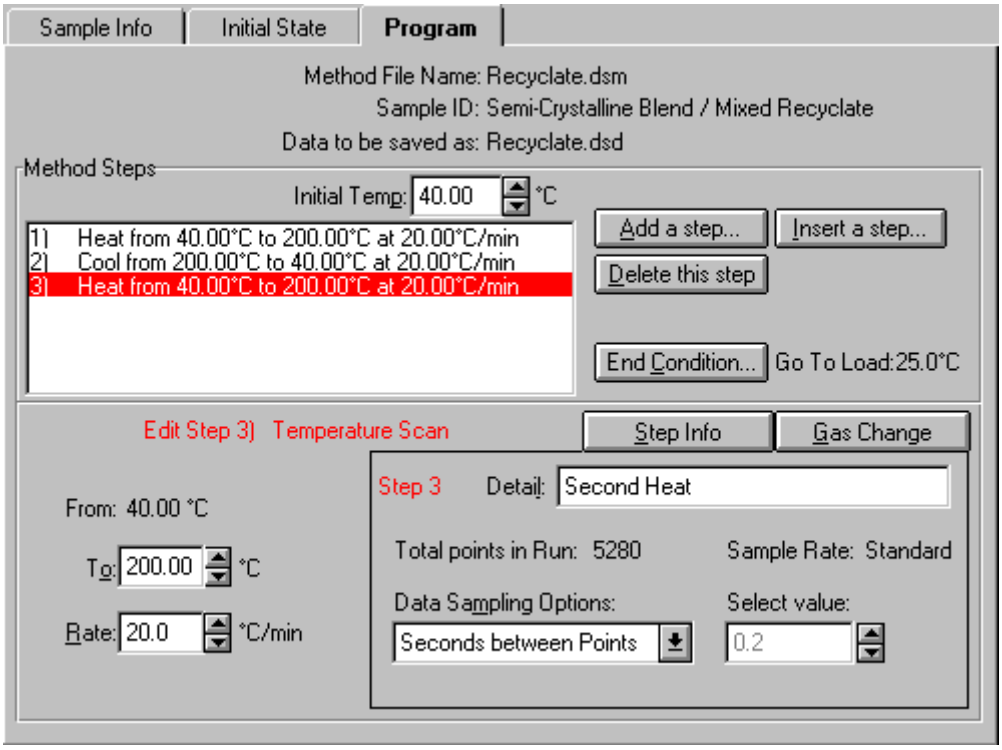
The sample used to illustrate this application is the determination of polypropylene in polyethylene curbside recyclate. The DSC method involves heating through the melting range of polypropylene in a nitrogen atmosphere.

Sample Preparation

Prepare a representative sample. Grind or mix in the melted state and pelletize. Other geometries can also be used. Cut a 5 – 10-mg sample and place it flat side down in a standard aluminum DSC pan. Crimp the sample pan with an aluminum lid to hold it securely.

Method

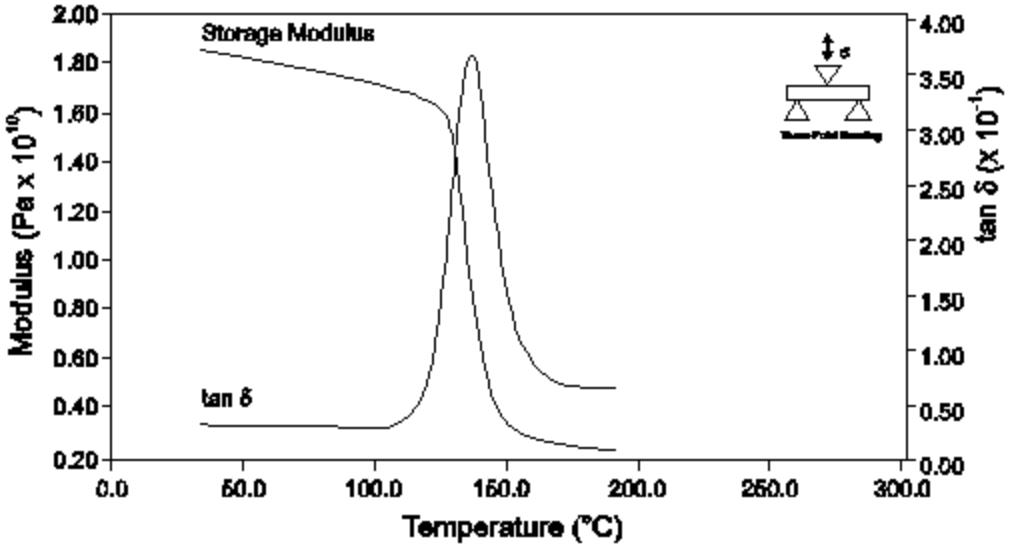
Heat from 40°C to 200°C at 20°C/min in nitrogen, cool at 20°C/min to 40°C, then reheat to 200°C. If the component peak is not well resolved from an adjacent peak, repeat at a slower scan rate. Select the curve that best isolates the melting or fusion of the component of interest.



Discussion

Duplicate the analysis above using a comparative standard consisting of a weighed specimen of the unblended component to be analyzed. In this case the sample could be polypropylene from bottle enclosures which are commonly mixed with polyethylene in mixed recyclate.

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Using the Peak calculation, determine the per gram peak area of the component in both the blended and unblended samples. The fraction of the component in the blend is the per gram peak area of the component in the mixture divided by the per gram peak area of the component in the unblended state. In the case of comparing with a blended reference, this result is multiplied by the weight fraction of the component in the blended reference.

References

1. "Applications of Thermal Analysis in the Automotive Industries," Perkin-Elmer Thermal Analysis Application Study (TAAS) #26. The Perkin-Elmer Corporation, Norwalk, CT, 1978.
2. B. Cassel, M.S. Feder, et al., "Quantifying the Quality of Recycled Plastics," Pi Quality, 2, 1992.
3. ASTM Annual Book of Standards, Philadelphia, PA, 1994, Vol. 14.02 and 08.01, respectively.

DSC Isothermal Crystallization

Introduction

Isothermal crystallization can be one of the most sensitive methods to determine small differences among semicrystalline polymers. Recognize, however, that this is not always an easy experiment to run. There is an element of trial and error involved. It is hoped that the following will reduce your experimental times. Isothermal crystallization is very sensitive to temperature (which must be chosen with care) and small differences in the polymer itself such as crosslinking, molecular weight, degree of branching, and the presence of additives among others.

Instrument

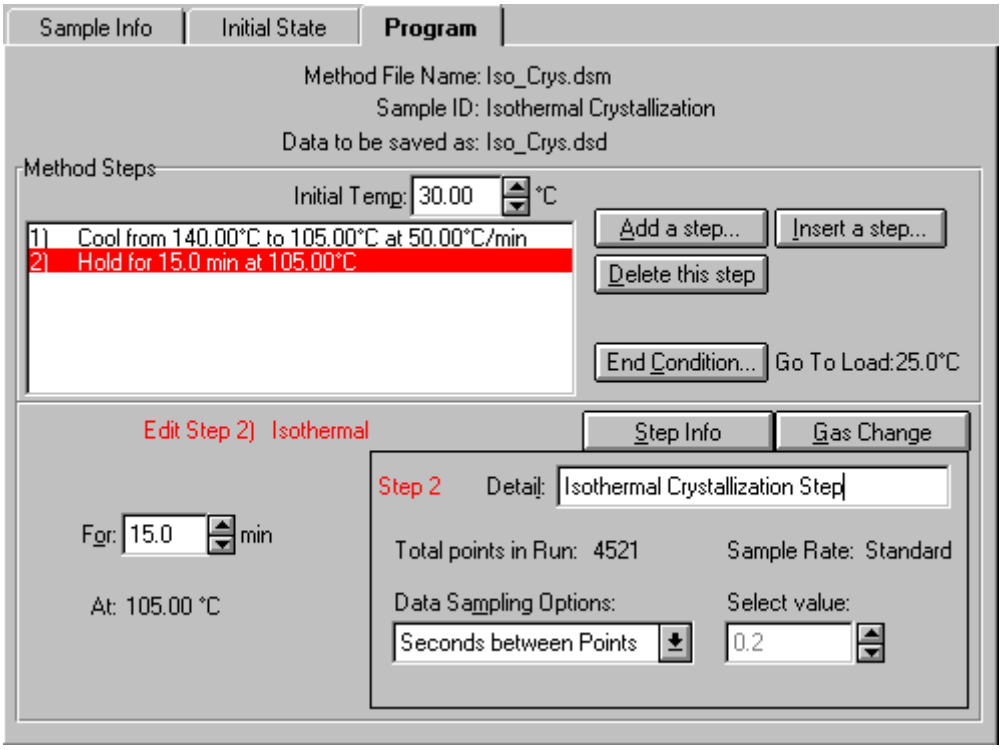
Pyris 1 DSC or DSC 7

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Procedure

Use between 5 and 15 mg of sample. Next determine a target temperature. If the temperature is too high, the sample may never crystallize; if the temperature is too low, crystallization may occur too quickly to observe. To determine a temperature to use:

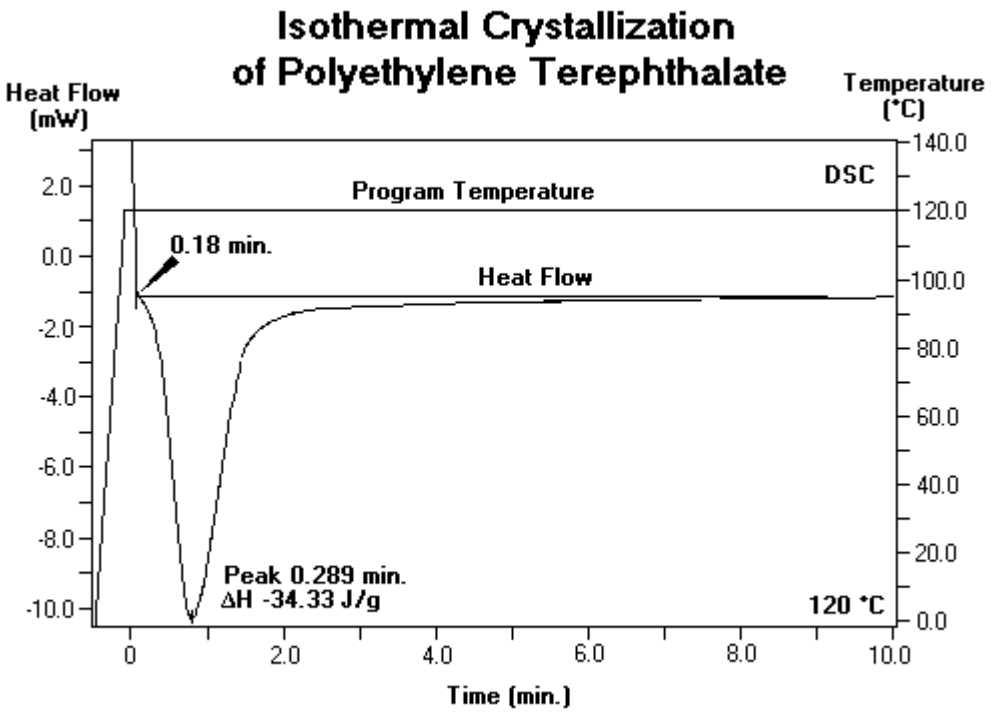
1. Melt the sample then perform a cooling experiment at 10°C/min to determine the onset of crystallization. To this temperature add 3°C – 5°C to determine your first target temperature.
2. The next step involves "balancing the heat capacity." Add one extra aluminum lid to the reference pan for each 5 mg of sample. (Do not forget a lid to match the one you already have on your sample pan). After melting the sample, use the Go To Temp entry field and button on the control panel to reduce the temperature to 20°C above your target temperature. Set up a run to cool the sample to the target temperature at 40°C – 50°C/min. (The most common mistake made here is to cool from a high temperature at the maximum cooling rate. That will virtually guarantee failure.)

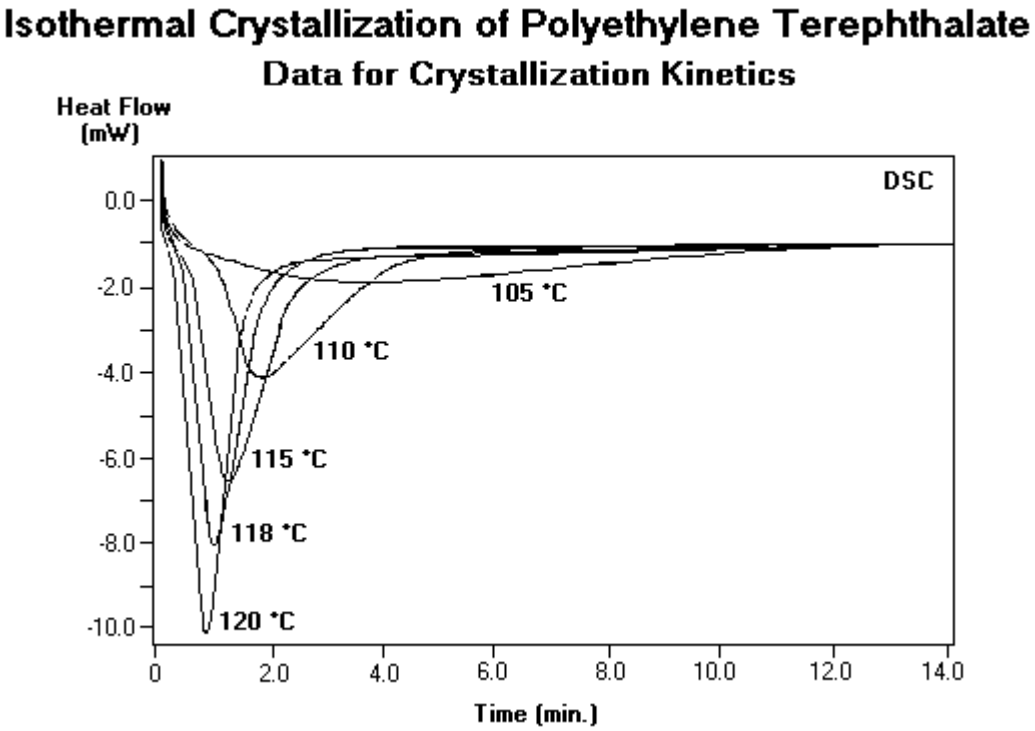


Results

Recall multiple curves and calculate peak maxima (times) for comparisons. Usually differences will be readily evident.

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Isothermal Crystallization
of Polyethylene Terephthalate
Heat Flow
(mW)
Temperature
(°C)
DSC
Program Temperature
0.18 min.
Heat Flow
Peak 0.289 min.
ΔH -34.33 J/g
120 °C
2.0
0.0
-2.0
-4.0
-6.0
-8.0
-10.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
0
2.0
4.0
6.0
8.0
10.0
Time (min.)


Isothermal Crystallization of Polyethylene Terephthalate
Data for Crystallization Kinetics
Heat Flow
(mW)
DSC
105 °C
110 °C
115 °C
118 °C
120 °C
0.0
-2.0
-4.0
-6.0
-8.0
-10.0
0
2.0
4.0
6.0
8.0
10.0
12.0
14.0
Time (min.)

References

1. "Determination of Kinetic Parameters for the Crystallization of PET," Perkin-Elmer Thermal Analysis Newsletter (PETAN) #50. The Perkin-Elmer Corporation, Norwalk, CT, 1994.
2. "Measurement of Kinetic Parameters for the Crystallization of PEEK," Perkin-Elmer Thermal Analysis Newsletter (PETAN) #66. The Perkin-Elmer Corporation, Norwalk, CT, 1995.

Effect of Sample Weight on a DSC Run

Introduction

The DSC can accommodate samples in a wide variety of shapes and configurations, including films, powders, liquids, and chunks. Sample pans are available to handle any type of sample that can be run: aluminum (standard, robotic, and volatile), copper, gold (standard, robotic, volatile, and high-pressure), platinum, alumina, graphite, stainless steel (standard and high-pressure), and titanium. Highly aerated samples should be compressed and covered with a platinum mesh in the sample pan. It is common practice to use one extra aluminum lid in the reference side per 5 mg of sample. This will balance the heat capacity between the sample and reference cups and provide for a flatter baseline.

Theory

Larger samples will produce larger transitions, so small changes in a sample are easier to see using large samples. However, issues such as thermal gradients in the sample must be taken into account when using larger samples.

Instrument

Pyris 1 DSC or DSC 7, AD-6 AutoBalance. Nitrogen purge at 30 cc/min. Commonly used purge gases are argon, nitrogen, air, and oxygen. Helium should be used when cooling the DSC with liquid nitrogen. Flow rates of 20 – 40 cc/min are generally used.

Sample Preparation

Generally, a sample weight of 5 – 10 mg is adequate. Large samples produce higher melting peaks but also reduced resolution.

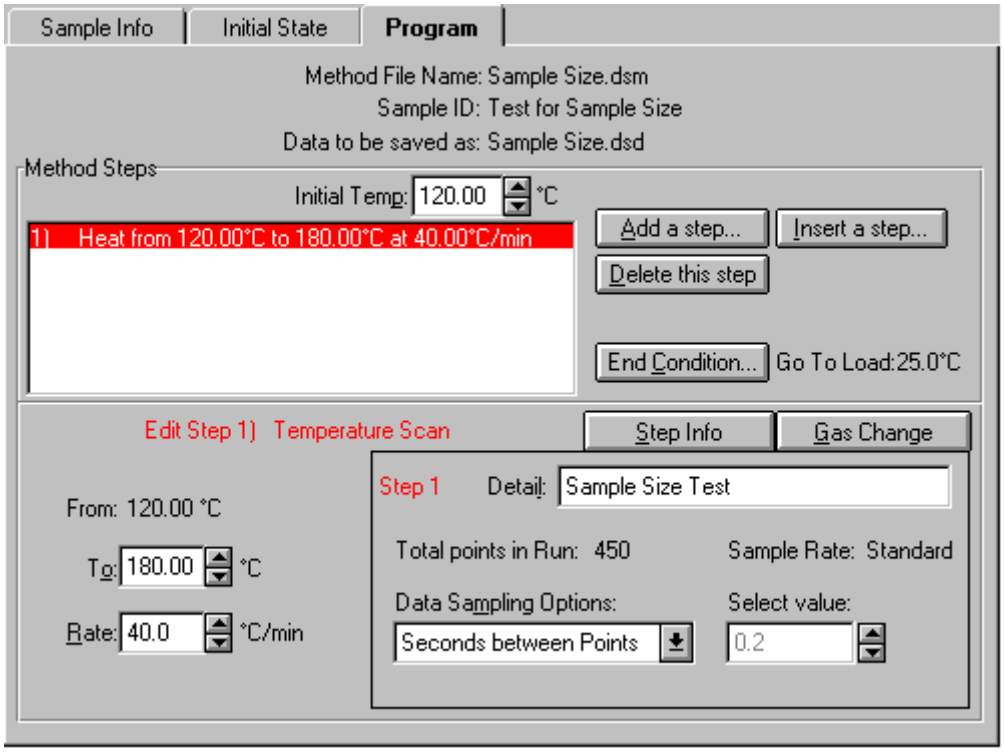
Method

For all samples, it is essential that good contact is made with the bottom of the pan. The pan bottom must be flat to ensure good contact with the sensor. This is especially critical when using large samples, since thermal gradient effects are potentially increased. This test was performed using several different weights of indium (1, 5, 10, and 20 mg).

Generally, the DSC should be allowed to equilibrate to within ±0.01°C of the starting temperature and within ±0.1 mW (to ensure a stable ordinate signal) before starting the run.

The instrument was purged with nitrogen at 20 cc/min and the sample scanned from 120°C to 10°C at 10°C/min for each run.

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Results/Discussion

Data should be viewed on both a temperature scale and a time scale to see the full effect of sample weight on a DSC run.

Onset and peak area calculations are done to verify independence with regard to sample weight.

References

1. "Characterization of (Epoxy) Adhesives Using Multiple Thermal Analysis Techniques," Perkin-Elmer Thermal Analysis Newsletter (PETAN) #47. The Perkin-Elmer Corporation, Norwalk, CT, 1993.
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3. "Some Applications of DSC for Polymer Films," Perkin-Elmer Thermal Analysis Application Study (TAAS) #15. The Perkin-Elmer Corporation, Norwalk, CT, 1974.
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Determining Vapor Pressure by Pressure DSC

Introduction

With greater awareness for reducing hazards in the workplace has come a need to quantify the volatility of laboratory substances in order to develop procedures for handling these materials safely. DTA, DSC, and TGA have been used in the past to determine boiling points and to estimate vapor pressure.

Theory

When a volatile sample, sealed in a capsule having a small pinhole, is heated at a slow rate, an equilibrium is established between material in the gas phase and in the condensed phase. If the hole is small enough, then very little material evaporates until the boiling point is reached. At that point the remaining material in the capsule goes into the vapor phase at a rate that is limited by the flow of heat from the DSC. The boiling point can be obtained as the onset of the isothermal boiling endotherm. The purpose of the DSC measurement is to obtain accurate boiling point temperatures at several imposed pressures (above and/or below ambient) and fit the data to an appropriate equation. This P–T curve can than be interpolated or extrapolated to obtain the equilibrium vapor pressure at some other temperature.

Instrument

Pyris 1 DSC or DSC 7, with a pressure DSC cell

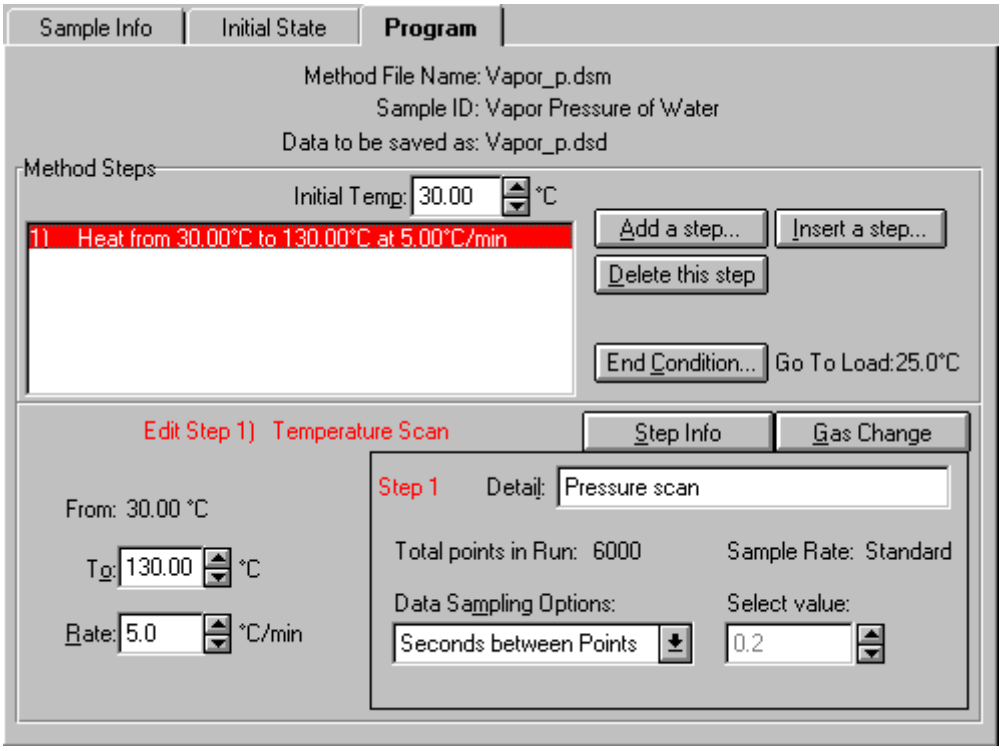
Sample Preparation

Approximately 3 – 5 mg of material is weighed into the specialized vapor pressure pan (N519-0788). The capsule is sealed using the volatile sealer assembly (Part No. 0219-0061) or the Sealing Insert Accessory (Part No. B014-4637) with the Universal Crimper Press (Part No. B013-9005).

Method

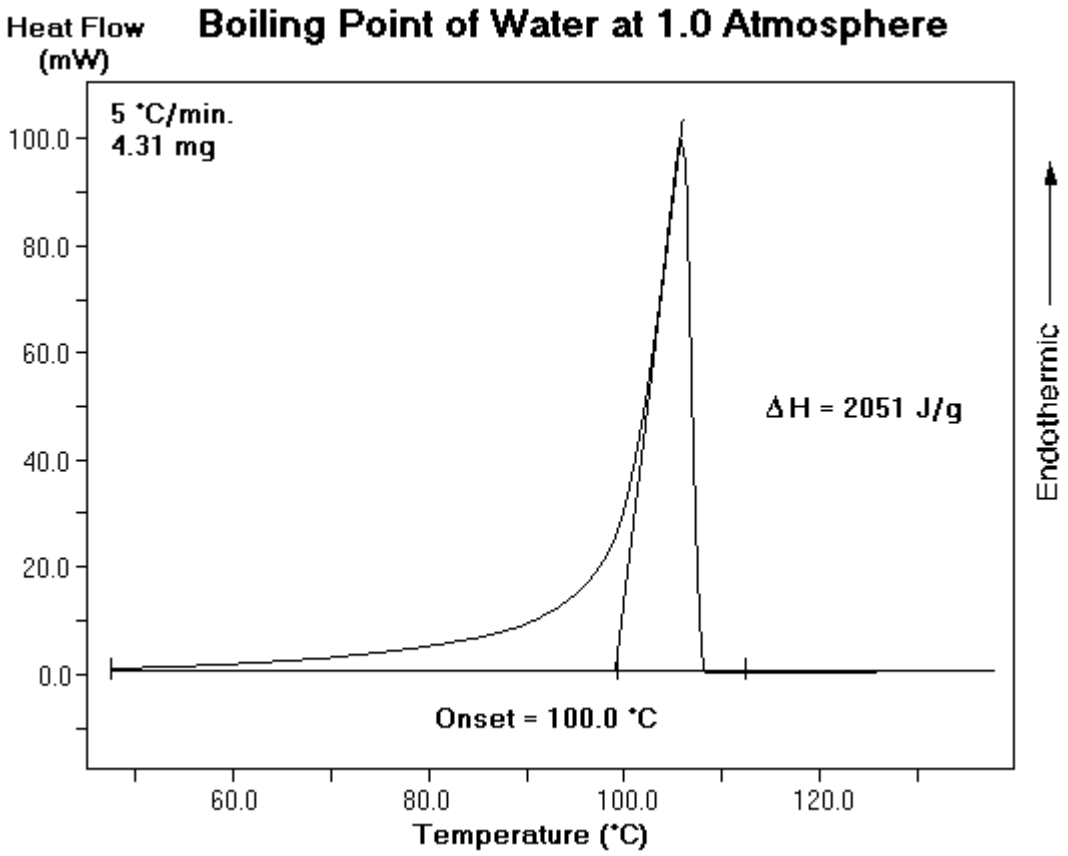
The DSC pressure accessory is set up at the desired external pressure by using the nitrogen purge gas cylinder to pressurize the cell or by using a vacuum pump to draw the pressure to the desired level. When the pressure is fairly stable, start the 5°C/min scan from an appropriate low temperature through the boiling point. Make note of the pressure on the gauge as the material starts to boil.

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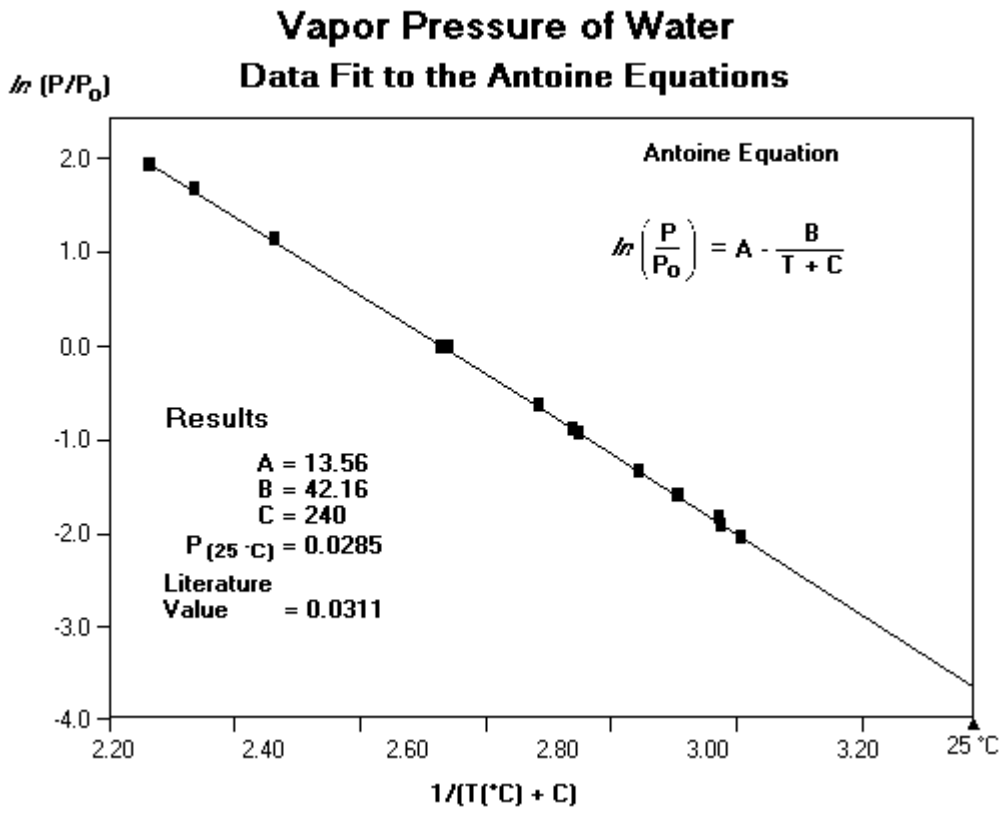
Results/Discussion

Determine the extrapolated onset, the boiling point, by using the Onset Calculation and selecting a point before the boiling starts and the steepest slope as the calculation inputs. If there has not been much evaporation prior to boiling, then the peak energy should be approximately the heat of vaporization.



Once the boiling temperatures have been determined at several pressures, fit the data to a smooth curve using the Antoine equation to determine the plot axes. If desired, solve for the parameters that fit the data. This can be accomplished using a spreadsheet program. To determine the vapor pressure at room temperature, extrapolate or interpolate the data to T = 25°C.

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Reference

"Determining Vapor Pressure by Pressure DSC," Perkin-Elmer Thermal Analysis Newsletter (PETAN) #49. The Perkin-Elmer Corporation, Norwalk, CT, 1994.

DMA Applications

The following list is a cross section of the types of applications for which you can use your DMA 7e:

- Glass Transition Analysis of Epoxy–Glass Composite Using DMA
- Fast Mechanical Characterization of an Epoxy Composite
- Isothermal Cure of an Epoxy by DMA
- Softening Temperature Determination Using the DMA 7e
- DMA 7e Modulus Reported by Each Measuring System
- DMA 7e Flexural Modulus Determination
- DMA 7e Compressive Modulus Determination
- DMA 7e Tensile Modulus Determination
- PID Factors for Position Control
- Isothermal Modulus Determination Using Position Control
- Thermal Characterization of a Thin Film Using Position Control

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Glass Transition Analysis of Epoxy–Glass Composite Using DMA

Introduction

At temperatures below the glass transition (Tg), a composite behaves like a glassy material. At higher temperatures, the composite behaves like a viscoelastic material.

Theory

The glass transition is often used to identify the temperature range of the glassy to viscoelastic transition.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel 3-point bending measuring system with a 20-mm bending platform and a 10-mm knife edge.

Sample Preparation

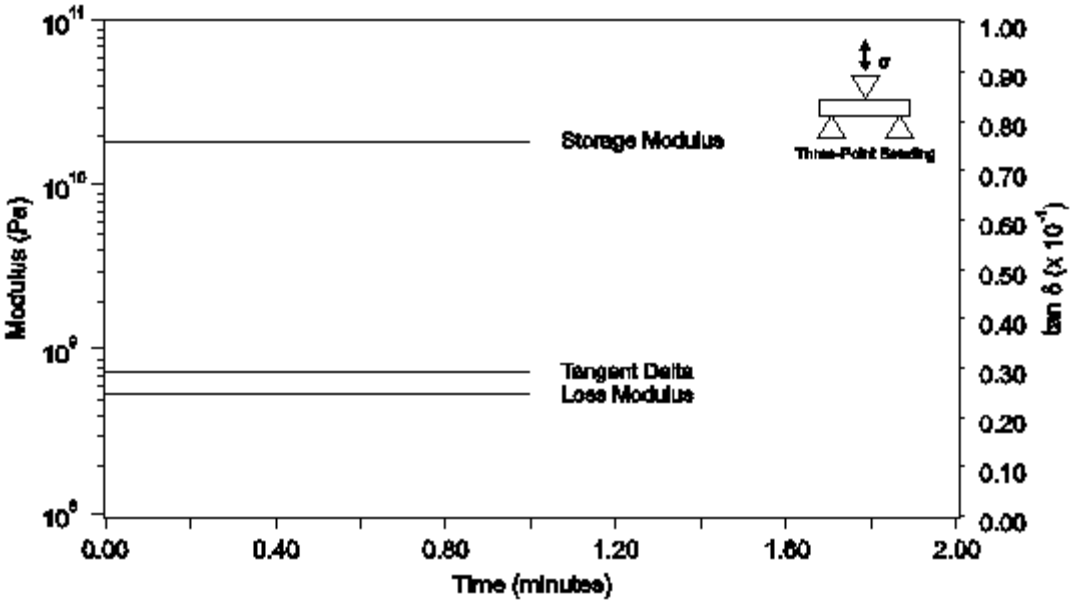
Cut a sample that measures 23 mm long by 3 mm deep. Using tweezers, place the sample directly on the 3-point bending platform. No further sample preparation or clamping is necessary.

Method

Program the temperature scan from 50°C to 225°C at a heating rate of 2°C/min. A typical method is as follows:

Sample:	Epoxy-Glass Composite
Instrumental:	Analyzer: DMA 7e Measuring System: 3-Point Bending Geometry: Rectangle
Environmental:	Purge: Nitrogen Coolant: Ice water
Parameters:	Method: Temperature Scan Static Force: 550 mN Dynamic Force: 500 mN Frequency: 1 Hz Heat from 50°C to 225°C at 2°C/min
Controls:	Static Force Dynamic Force

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Results/Discussion

The figure above shows a plot of storage modulus and tangent delta versus temperature. The change in the curves indicates the glass transition region in the 120°C – 175°C range. The onset of softening of the storage modulus (i.e., indicative of the material storing energy) occurs at approximately 125°C; the peak of tan delta occurs at approximately 136°C.

A decrease in the storage modulus at approximately 120°C indicates a decrease in the stiffness of the composite. The decrease occurs because the composite softens from its glassy state, a state dominated by the resin and fiber composite structure. At higher temperatures, the storage modulus finally reaches a minimum value as the contribution from the resin decreases and the contribution from the fiber increases.

The methodology outlined in this application example can be applied to a wide variety of thermosets, thermoplastics, and composites. No special sample preparation is necessary. Further analysis includes parallel plate analysis for the neat resin to determine the gel point or characterize cure. Additional testing may include examination of the beta and gamma transitions as well as frequency dependence. Also, TGA can be used to evaluate the moisture content or the effect of the outgassing that occurs at these temperatures while DSC can be used to confirm the thermal transitions.

References

1. Brennan, W.P., Cassel, R.B., “Applications of Thermal Analysis in the Electrical and Electronics Industries,” Thermal Analysis Applications Study No. 25. The Perkin-Elmer Corporation, Norwalk, CT, 1978.
2. Cassel, R.B., “Characterization of Thermosets,” Thermal Analysis Application Study No. 19. The Perkin-Elmer Corporation, Norwalk, CT, 1977.
3. Gray, A.P., “Establishing a Correlation Between the Degree of Cure and the Glass Transition Temperature of Epoxy Resins,” Thermal Analysis Applications Study No. 2. The Perkin-Elmer Corporation, Norwalk, CT, 1972.

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Fast Mechanical Characterization of an Epoxy Composite

Introduction

Storage modulus has been widely accepted as a tool to characterize performance and processability of composites. The storage modulus has also been used to characterize impact properties, dimensional stability (warpage), the degree of cure, effects of modifiers, toughenners, extenders, fire retardants, and other additives.

Theory

Examination of a material's modulus at room temperature is a deceptively simple and effective test that can yield useful information about a composite.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel 3-point bending measuring system with a 15-mm bending platform and a 10-mm knife edge.

Sample Preparation

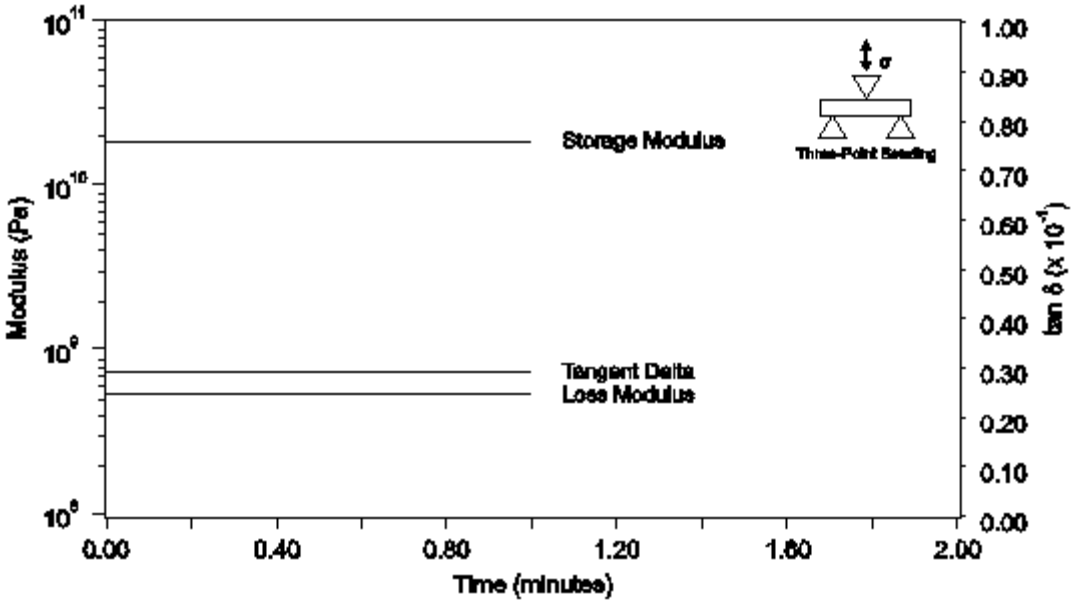
Cut a sample measuring approximately 23 mm long by 3 mm deep. Using tweezers, place it directly on the 3-point bending platform. No further sample preparation or clamping is necessary.

Method

Program an isothermal scan at 25°C for 3 min. A typical method is as follows:

Sample:	Epoxy-Glass Composite
Instrumental:	Analyzer: DMA 7e Measuring System: 3-Point Bending Geometry: Rectangle Sample height (y): 0.865 mm Sample width (x): 15.000 mm Sample depth (z): 3.016 mm
Environmental:	Purge: Nitrogen (30 cc/min) Coolant: Ice water
Parameters:	Method: 1-min Isothermal Scan Static Force: 550 mN Dynamic Force: 500 mN Frequency: 1 Hz
Controls:	Static: Force Dynamic: Force Temperature: 25°C

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Results/Discussion

The figure above shows the plot of storage modulus for the composite sample (log Pascal scale) versus time. The storage modulus of 1.92 x 10^{10} Pa agrees with the manufacturer's specification and is indicative of the material's ability to store energy. It can be increased by the effects of crystallization, degradation, annealing, internal stresses, further curing, advancement, or postcure.

The loss modulus (log Pascal scale) versus time plot is also seen in the figure above. The loss modulus of 5.31 x 10^{8} Pa agrees with the manufacturer's specification and is indicative of the material's ability to dissipate energy, often in the form of heat or molecular rearrangements. It can be increased by the effects of increased damping in the sample. Damping in a composite sample can be affected by free mobility, uncrosslinked epoxy, additives, plasticizers, and toughenners.

The figure also shows the plot of tan delta versus temperature. Tangent delta for this material is 0.0290 + 0.005 at 25°C, is independent of sample dimensions, and is indicative of the properties of a composite. It is not necessary to measure the sample and enter sample dimensions if this is the only curve to be analyzed.

The methodology outlined in this application example can be applied to a wide variety of thermosets, thermoplastics, and composites. No special sample preparation is necessary. Parallel plate analysis of the neat resin for determining the gel point or for characterizing cure can also be performed. Additional testing includes examination of the beta and gamma transitions as well as frequency dependence. TGA can be used to evaluate the moisture content or effect of outgassing that occurs at these temperatures while DSC can be used to confirm the thermal transitions.

References

1. Brennan, W.P., Cassel, R.B., "Applications of Thermal Analysis in the Electrical and Electronics Industries," Thermal Analysis Applications Study No. 25. The Perkin-Elmer Corporation, Norwalk, CT, 1978.
2. Cassel, R.B., "Characterization of Thermosets," Thermal Analysis Application Study No. 19. The Perkin-Elmer Corporation, Norwalk, CT, 1977.

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3. Gray, A.P., “Establishing a Correlation Between the Degree of Cure and the Glass Transition Temperature of Epoxy Resins,” Thermal Analysis Applications Study No. 2. The Perkin-Elmer Corporation, Norwalk, CT, 1972.

Isothermal Cure of an Epoxy by DMA

Introduction

As applications broaden or service requirements become more demanding, epoxy materials are reformulated to meet these demands. For example, the epoxy gel used in this experiment is a thick material often used in vertical or overhead applications because it has limited sag or drip. The DMA 7e provides a fast and accurate way to characterize the curing process to obtain faster cure cycles, lower internal stresses, and higher degrees of cure. The extended force range of the DMA 7e enables characterization of epoxy and similar materials from the uncured low-modulus (liquid) state through to the fully cured high-modulus state without changing the sample geometry.

Theory

Epoxy adhesives cure and adhere via a chemical reaction between the resin and hardener. The reaction results in high-strength bonds that are resistant to most chemicals. Epoxy adhesives are specifically formulated for a number of applications.

Instrument

Use the DMA 7e equipped with the cup-and-plate measuring system. It has a 13-mm-diameter cup (other sizes available include 8, 18, and 21.5 mm) mounted at the base and a 10-mm-diameter top parallel plate (other sizes available include 1, 3, 5, 13, 15, and 18 mm).

Sample Preparation

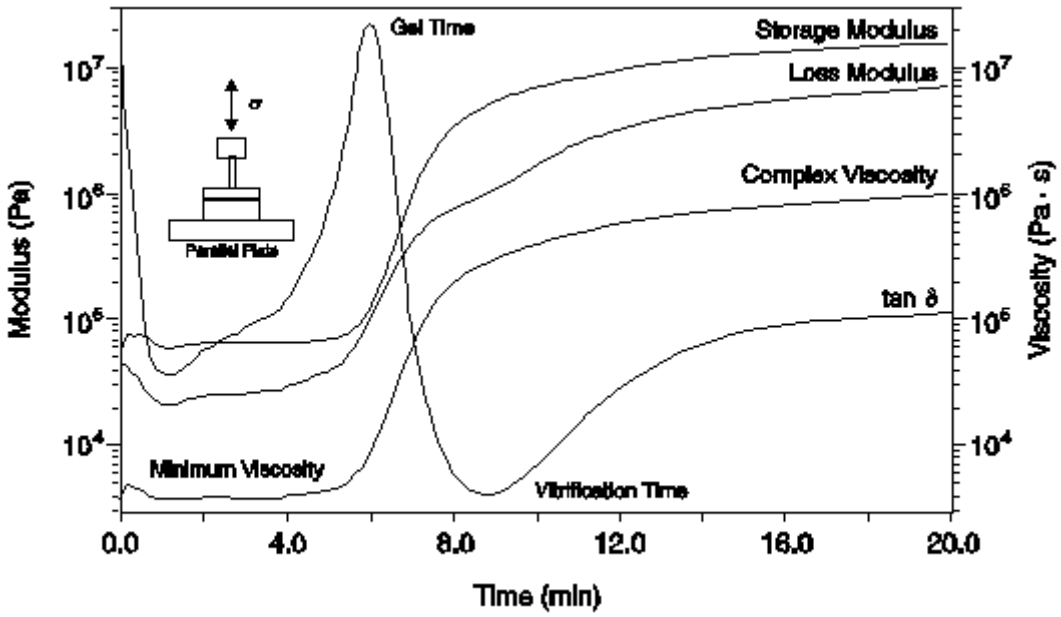
The specimen is a “5-minute epoxy gel” with a mixing indicator that cures to an opaque product. Mix the specimen in a crucible according to the manufacturer’s specifications. Lower the furnace and, using a spatula, transfer the specimen from the crucible to the center of the cup. Fill the cup about half-way. Lower the top plate so that it touches the specimen but does not penetrate it.

Method

Program an isothermal run at 25°C for 120 min with both static and dynamic stresses of 0 Pa at a frequency of 1 Hz. Set the “static position control” to “current” and the “dynamic amplitude control” to 10 µm. Quickly raise the furnace around the specimen; this activates the static position control and dynamic amplitude control. Allow the system to equilibrate to the new stress values and then start the run. A typical method is as follows:

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Sample:	5-min Epoxy: 2-part gel
Instrumental:	Analyzer: DMA 7e Measuring System: Cup and Plate Geometry: Parallel Plate Sample Height (y): 4.579 mm Sample Diameter (d): 10.000 mm
Environmental:	Purge Gas: Helium (30 cc/min) Coolant: Tap Water
Parameters:	Method: Isothermal Scan Temp 1: 25°C for 120.0 min Static Force: 0 Pa Dynamic Force: 0 Pa Frequency: 1.00 Hz
Controls:	Static Position Control: Current Dynamic Amplitude Control: 10 µm



Results/Discussion

The figure above shows plots of storage modulus and loss modulus (log Pascal scale), complex viscosity (log Pascal second scale), and tangent delta (linear scale) versus time.

Observe the high tangent delta value at the beginning of the run (0.5 min). The specimen was equilibrating and flowing into the cup. Applied forces must be kept at a minimum (<10 mN) to ensure that the crosslinking process is not disturbed by the stresses of the experiment. The DMA 7e dynamic amplitude control automatically maintains a 10- µm amplitude and easily provides low enough forces to analyze low-modulus semisolid-to-flowing materials, such as an uncured epoxy, without changing the sample geometry.

Observe the minimum in the tangent delta at approximately 1.5 min. This resulted from stress relief and cessation of flow. There is very little change in the storage modulus and complex

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viscosity. The complex viscosity is used to identify the minimum viscosity. At approximately 1.75 min the tangent delta begins to increase from the minimum value, and viscous properties and chemical equilibrium dominate the physical behavior of the polymer. During this period the reaction is diffusion controlled and some volatilization may occur.

At about 4 min the slope of the tangent delta curve changes. The reaction rate increases and is controlled by localized physical and chemical equilibria. At approximately 5.5 min, there is an onset increase in the storage modulus, loss modulus, and complex viscosity as well as a peak in the tangent delta. The complex viscosity onset or the peak of the tangent delta can be used to identify the gel time. The slope of the storage modulus increase is proportional to the reaction rate, which is controlled by the onset of the storage modulus and complex viscosity. The slope indicates a dominance of the physical equilibrium over the chemical equilibrium.

At approximately 8 min there is a second change in the slope of the storage modulus and complex viscosity, indicating that the specimen is beginning to set. Setting is caused by primary microgels interconnecting with each other to form a secondary microgel with increased mechanical integrity. The reaction rate slows as the concentration of the reactant species diminishes. A minimum tangent delta of 0.01 is observed at about 9 min. Vitrification occurs when the polymer's glass transition temperature (Tg) exceeds the cure temperature. The tangent delta minimum is used to identify the vitrification time.

At approximately 14 min there is a change in the slope of the tangent delta and a much slower rate of increase in the storage modulus, loss modulus, and complex viscosity. These changes result from a slowdown in the curing reaction caused by reduced mobility of the reactant species. Localized and side chain reactions cause the storage modulus to slowly increase to ultimately reach the fully cured state. The DMA 7e has a maximum force of approximately 8 N. This provides forces high enough for the analysis of many cured epoxies without changing sample geometry.

References

1. Brennan, W.P., Cassel, R.B., "Applications of Thermal Analysis in the Electrical and Electronics Industries," The Perkin-Elmer Thermal Analysis Applications Study No. 25. The Perkin-Elmer Corporation, Norwalk, CT, 1978.
2. Gray, A.P., "Establishing a Correlation Between the Degree of Cure and the Glass Transition Temperature of Epoxy Resins," Perkin-Elmer Thermal Analysis Applications Study No. 2. The Perkin-Elmer Corporation, Norwalk, CT, 1972.
3. Neilsen, L.E., *Mechanical Properties of Polymers and Composites.* Marcel Dekker, New York, 1974.
4. Ferry, J.D., *Viscoelastic Properties of Polymers*; 2nd ed. Wiley & Sons, New York, 1970.

Softening Temperature Determination Using the DMA 7e

Introduction

The DMA 7e method described here is optimized for performing vicat softening temperature determinations for a broad range of thermoplastics, elastomers, and some thermosets in the rigid and semirigid states. Samples in the form of disks or rectangles are mounted using the 1-mm parallel plate (compression) measuring system. The softening temperature is the temperature at which a 1-mm probe penetrates 1 mm into the sample. This measurement is made while the sample is heated and a 5000-mN force is applied.

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NOTE: The instrument can be modified to accommodate an oil bath, but good temperature precision is obtained using the standard furnace system. Temperatures can be verified using primary melting standards. A force of 500 gF (gram force) was used in this test. A force of 1000 gF can be achieved using weights in the weight tray.

Theory

Softening temperature determinations provide a qualitative measure of the temperature resistance of a material. Standard test methods have been created by ASTM and ISO to define the vicat softening temperature determination (1–3).

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel parallel plate measuring system with a 1-mm plate probe tip.

Sample Preparation

Cut the sample, an injection-molded 2-mm-thick sheet of styrene-butadiene, into disks using a heavy-duty 6-mm hole punch. Tare the weight and zero the height. Mount the sample by placing two disks on the bottom plate, lowering the probe, and visually aligning it with the top plate.

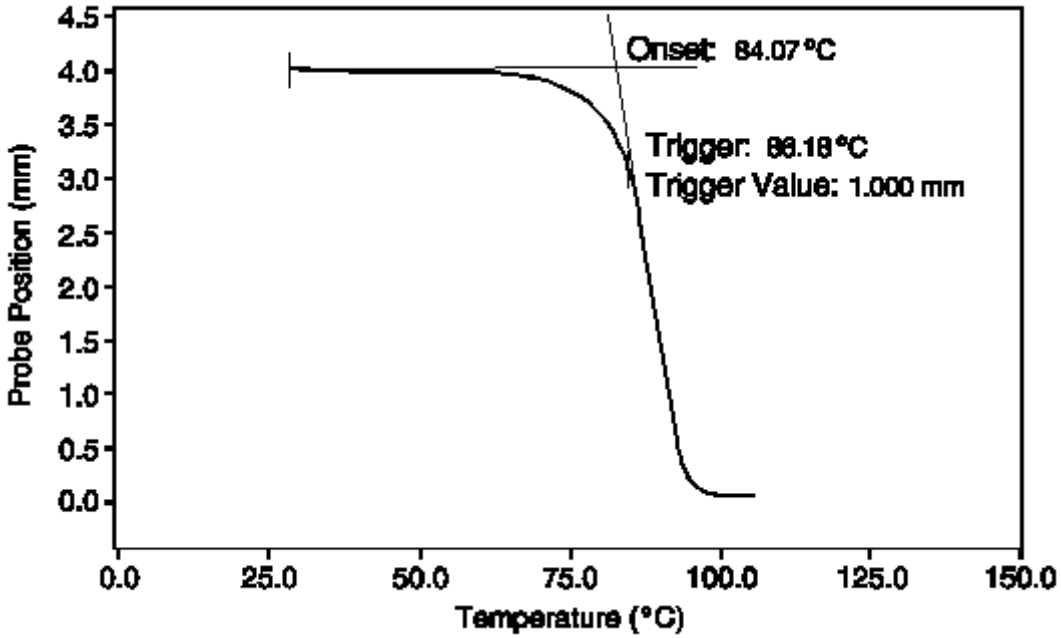
Method

Program the temperature scan from 30°C to 250°C at 2°C/min (120°C/h). Use a faster heating rate if all the samples are exposed to the same temperatures and the temperature is controlled to within + 1°C. Enter a static force of 5000 mN (6.37×10^6 Pa), a dynamic force of 10 mN (1.27×10^4 Pa), and a frequency of 10 Hz in the Initial State page. The dynamic force and frequency are included to enhance probe position sensitivity. Raise the furnace and allow the sample, analyzer, and furnace to equilibrate for about 2 min or until the probe position stabilizes. Read the sample height and start the run. When the probe has penetrated more than 1 mm, stop the run by pressing the **Probe Up** button on the analyzer.

A typical method for this application is as follows:

Sample:	Styrene Butadiene
Instrumental:	Analyzer: DMA 7e Measuring System: 1-mm Parallel Plate Geometry: Disk Sample Height (y): 4.054 mm Sample Diameter (d): 1 mm
Environmental:	Purge Gas: Nitrogen (20 cc/min) Coolant: Tap Water
Parameters:	Method: Temperature Scan Static Force: 5000 mN Dynamic Force: 10 mN Frequency: 10 Hz Heat from 30°C to 250°C at 2°C/min

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Results/Discussion

The figure above is a plot of probe position (linear millimeter scale) versus temperature for the SBR sample. There is a decrease in the probe position resulting from the slow penetration of the probe as the sample is heated and the stress placed upon it. Above 50°C, the probe position begins to decrease rapidly as the sample approaches its melting temperature. A few degrees above 80°C the sample melts and the probe position indicates 0 mm.

Perform an onset calculation on the probe position curve. Place the left tangent on the original sample height at 30°C and the right tangent on the midpoint of melting, and include a trigger value in the calculation. The onset of the melt and the softening temperature of this sample occur at 84.07°C. The 1-mm trigger is reported as 86.18°C.

References

1. “Standard Test Methods for Vicat Softening Temperature of Plastics” ASTM Standard D1525-91, Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.01.
2. “Vicat Softening Temperature of Thermoplastics,” ISO 306; ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 2.
3. “Vicat Softening Temperature of Unplasticized PVC Pipes and Fittings,” ISO 2507; ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 3.

DMA 7e Modulus Reported by Each Measuring System

Introduction

The DMA 7e measuring systems, combined with the many sample geometries available, provide an unequaled versatility in modulus determination. The measuring systems allow you to determine the modulus of samples in various physical states from viscous liquids to melts, semisolids, solid

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bars, films, and fibers. The modulus obtained by each measuring system is identified by its own symbol, as shown in the table below. Standard terminology has been established in the literature and by ASTM and ISO that defines the moduli reported by DMA measuring systems (1–9).

Each measuring system applies stresses differently resulting in a different displacement of the sample and, thus, the calculation of a different type of modulus. The modulus obtained from one geometry may not relate to that obtained by another geometry.

Theory

A modulus is defined as the stress divided by the sample strain. Modulus is typically determined within the linear region (or elastic limit) of the test material. This region can be defined by finding the first region where the modulus curve approaches linearity, making certain that there are no anomalies in the stress vs. strain curve.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer to determine modulus.

Discussion

The DMA 7e consists of measuring systems used to determine different moduli. The table below lists the types of moduli and the measuring systems used to determine them. Flexural modulus (E) is determined by using a flexural deformation that is composed of compression on the top of the sample, tension on the bottom side of the sample, and gradient shear through the center of the sample. Tensile modulus (M) is determined by using uniaxial, unidirectional tensile deformation. Shear modulus (G*)* is ideally determined by using a cubical element in simple shear. Here, shear modulus is approximated by using flexural, tensile, or compressive deformations and applying Poisson's ratio. For example, a flexural modulus of 9×10^9 Pa gives a shear approximation of 3×10^9 Pa. Bulk (compressive) modulus (K) is determined by using bulk volumetric (dilatometer) deformations. Compressive modulus (L) is determined by using a uniaxial, (longitudinal) unidirectional compressive deformation.

Type of Modulus	Symbol	Sample Geometry	Measuring System	Part Number
Flexural	E	Bar or rod	3-Point Bending	Standard
Flexural	E	Bar or rod	4-Point Bending	Standard
Flexural	E	Bar or rod	Dual Cantilever	N539-0131
Flexural	E	Bar or rod	Single Cantilever	N539-0131
Flexural	E	Bar or rod	3-Point Bending	N539-0136
Tensile	M	Film	Extension	N539-0132
Tensile	M	Fiber	Extension	N539-0132
Tensile	M	Film	Quartz Extension	N539-0134
Tensile	M	Fiber	Quartz Extension	N539-0134
Bulk	K	Volumetric	Dilatometer	N539-0763
Shear	G	Flowing	Shear	Standard
Compressive	L	Disk or rectangle	Parallel Plates	N539-0133
Compressive	L	Disk or rectangle	Cup and Plates	N539-0464
Compressive	L	Disk or rectangle	Plate and Tray	N539-0469
Compressive	L	Disk or rectangle	Sintered Plates	N539-0460
Compressive	L	Disk or rectangle	Quartz plates	N539-0135

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References

1. “Standard Terminology Relating to Dynamic Mechanical Measurements on Plastics,” ASTM Standard D4092-90, Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.02.
2. “Terminology and Symbols,” ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 2.
3. “Standard Terminology Relating to Plastics,” ASTM Standard D883-93, Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.01.
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9. Ferry, J.D., *Viscoelastic Properties of Polymers.* Wiley & Sons, New York, 1970.

DMA 7e Flexural Modulus Determination

Introduction

The DMA 7e method described here is optimized to perform flexural modulus determinations for a broad range of materials including thermosets, thermoplastics, elastomers, and samples that are reinforced, nonreinforced, impact-modified, or filled and in the solid and the semisolid states. In flexural modulus determinations, samples in the form of rectangular bars or cylindrical rods are mounted using the three-point bending (flexural) measuring system. (Four-point bending, single- and dual-cantilever systems also may be used.)

Theory

Standard test methods have been created by ASTM and ISO to define the flexural modulus determination (1,2). Flexural modulus (E) is a measure of the resistance of the sample to flexural deformation and can be used as an indicator of stiffness or rigidity. Flexural deformation that is composed of compression on the top of the sample, tension on the bottom of the sample, and gradient shear through the center of the sample. Flexural modulus is similar to but does not necessarily equate to Young's modulus of elasticity.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel 3-point bending measuring system with a 20-mm bending platform and a cylindrical knife edge.

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Sample Preparation

With the micrometer measure and cut the sample, in this case a commercial grade bisphenol-A-type epoxy resin composite with four layers of E-type woven glass reinforcement, to the following dimensions: approximately 1 + 0.5 mm high by 23 + 3 mm wide by 3 + 1 mm deep. Cut the sample uniformly to prevent glass fiber splinters, shattered resin, or burrs.

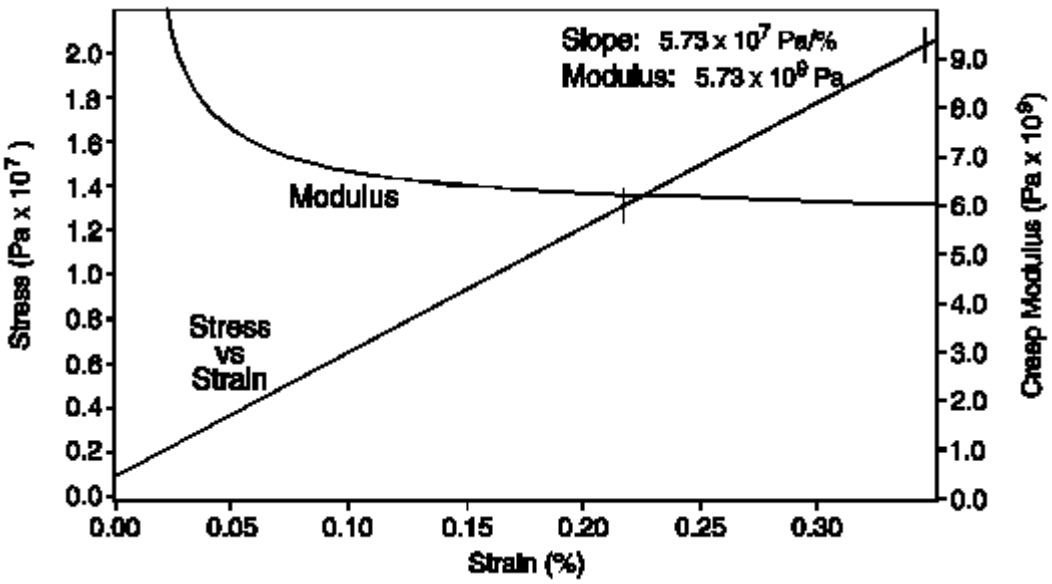
Method

Program a static stress scan at 25°C for 3 min (see the rest of the parameters of the method below). Mount the sample on the bending platform and visually align it. Before starting the run, allow the furnace to equilibrate at 23°C for about 2 min or until the probe position stabilizes. Hold the sample in place initially with a minimal static stress, then increase the static stress. Use the response of the sample (static strain) to calculate the static flexural modulus.

NOTE: The flexural modulus is determined in the linear region of stress and strain. The proportional limit, yield, drawing, ultimate strength, and other results can be determined using other methods

Sample:	Epoxy-Glass Composite: Rectangular bar
Instrumental:	Analyzer: DMA 7e Measuring System: Three Point Bending Geometry: Rectangle Sample Height: 1.096 mm Sample Width: 20.000 mm Sample Depth: 2.991 mm
Environmental:	Purge Gas: Helium (20 cc/min) Coolant: Tap Water
Parameters:	Method: Static Stress Scan Temperature Program: 23°C for 4.8 min Recovery Force: 100 mN Creep Ramp Rate: 500 mN/min Creep Force: 2500 mN End Condition: Go To Load

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Results/Discussion

The figure above shows the plot of stress and flexural modulus versus strain. As stress increases, the sample strain increases. As the stress increases above 0.20 x 10^7 Pa, the strain increases. This is typically due to stress relief between the sample and measuring system. A second run would minimize this effect. As the stress increases above 1.40 x 10^7 Pa, there is a linear relationship between stress and strain. This is the first region of the modulus curve where there is a linear relationship and where the stress is well-behaved. (**Note:** This is the linear region for this sample *at this temperature*.)

Above a stress of 2.00 x 10^7 Pa, the system produces an increasing and nonproportional strain as the linear region is exceeded and the proportional limit is reached. This is a result of the sample sliding over the platform knives, alignment, end effects, and edge effects.

The flexural modulus (E) is defined as the slope of the flexural stress versus the flexural strain in the linear region. The slope of the stress/strain curve in this region is 5.73 x 10^7 Pa/%. This is multiplied by 100 to cancel the unit of % strain, yielding a flexural modulus of 5.73 x 10^9 Pa in the linear region for this sample at 23°C.

The Pyris software can also calculate the flexural modulus and present it as a curve as seen in the figure above. Above a stress of 1.40 x 10^7 Pa, the flexural modulus is approximately 5.73 x 10^9 Pa.

References

1. "Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials [Metric]," ASTM Standard D790M-92, Annual Book of ASTM Standards. ASTM, Philadelphia, Vol. 08.01.
2. "Determination of Flexural Properties of Rigid Plastics," ISO 178, ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 1.

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DMA 7e Compressive Modulus Determination

Introduction

This DMA 7e method is optimized to perform compressive modulus determinations for a broad range of materials including thermosets, thermoplastics, elastomers and samples that are vulcanized, unvulcanized, cured, modified, extended, or filled, in the solid and semi-solid state.

In compressive modulus, determination, samples can be mounted in the form of rectangles or cylindrical disks using the parallel plate (compressive) measuring system.

Theory

Standard test methods have been created by ASTM and ISO to define the compressive modulus determination (1,2). Compressive modulus (L) is a measure of the resistance of the sample to compression and is an indicator of stiffness or rigidity. Compressive modulus is determined by using a uniaxial (longitudinal), unidirectional compressive deformation. The compressive modulus is similar to but does not necessarily equate to Young's modulus of elasticity. It provides complementary information to hardness and durometer tests.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel parallel plate measuring system with 10-mm plates.

Sample Preparation

Run a crosslinked silicone elastomer disk as received. Tare the weight of the probe and zero the height.

Method

Program a static stress scan at 25°C for 3 min (see the rest of the parameters of the method below). Place the sample on the bottom plate of the parallel plate measuring system. Lower the probe while visually aligning the sample with the top plate. The top plate is 10 mm in diameter. (This is used as the sample diameter for parallel plate measurements.) Before starting the run, allow the system to equilibrate at 23°C for about 2 min or until the probe position stabilizes. Read the sample height again and start the run. The sample is held in place initially with a minimal static stress, then the static stress increases. The response of the sample (static strain) is used to calculate the static compressive modulus.

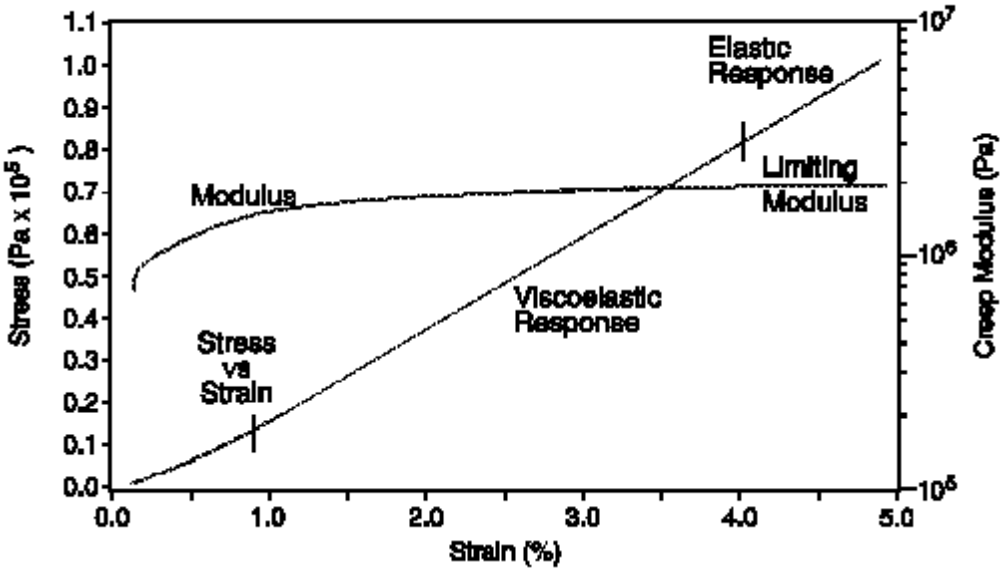
NOTE: The compressive modulus is determined in the linear region of stress and strain. The proportional limit, yield, ultimate strength, and other results can be determined using other methods.

Sample:	Silicone Elastomer Disk
Instrumental:	Analyzer: DMA 7e Measuring System: Parallel Plate Geometry: Disk Sample Height: 3.189 mm Sample Diameter: 10.00 mm
Environmental:	Purge Gas: Helium (20 cc/min) Coolant: Tap Water

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Parameters:

Method: Creep Ramp
Temp 1: 23°C
Time 1: 15.8 min
Recovery Force: 100 mN
Creep Ramp Rate: 500 mN/min
Creep Force: 8000 mN
End Condition: Go To Load



Results/Discussion

The figure above shows the plots of stress and compressive modulus versus percent strain. The sample exhibits a 0.175% strain as a result of a stress of 0.013 x 10^5 Pa (100 mN) applied before the run. As the stress begins to increase from 0.013 x 10^5 Pa to 0.138 x 10^5 Pa, the strain increases from 0.175% to 0.923%. This increase can be attributed to sample stress relief. As the stress increases from 0.138 x 10^5 Pa to 0.816 x 10^5 Pa, the strain increases to 4.000%. This is a result of the viscoelastic behavior of the sample.

As the stress increases from 0.816 x 10^5 to 1.018 x 10^5 Pa, the strain again increases to 4.897%. In this region, the sample shows elastic behavior only. This is known as Hookean behavior, similar to a perfect spring. As the stress increases to 1.018 x 10^5 Pa, the strain does not significantly increase, while the modulus becomes more linear. This is because of the limiting modulus as the sample becomes more compressed.

The compressive modulus (L) is given by the slope of the compressive stress versus the compressive strain in the linear region. For compressive tests, the modulus can be taken from either the linear region or the region of the limiting modulus. For this sample, the compressive modulus in the limiting modulus region at 23°C is 2.256 x 10^6 Pa.

The Pyris software can calculate the compressive modulus and present it as a curve. This modulus curve is shown in the figure versus percent strain. In the region from 4.0% to 5.0% strain, the limiting modulus is approximately 2.2 x 10^6 Pa.

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References

1. “Standard Test Methods for Compressive Properties of Rigid Plastics [Metric],” ASTM Standard D695M-91, Annual Book of ASTM Standards. ASTM, Philadelphia, Vol. 08.01.
2. “Determination of Compressive Properties,” ISO 604, ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 1.

DMA 7e Tensile Modulus Determination

Introduction

The DMA 7e method described here is optimized to perform tensile modulus determinations for a broad range of materials including PET, polyurethane, polypropylene, and samples in the solid and the semisolid states. In the determination of tensile modulus, samples in the form of rectangular films or cylindrical fibers are mounted using the extension (tensile) measuring system.

Theory

Standard test methods have been created by ASTM and ISO to define the tensile modulus determination (1–4). Tensile modulus (M) is a measure of the resistance of the sample to deformation and is an indicator of stiffness or rigidity. Tensile modulus is determined using uniaxial, unidirectional tensile deformation. The tensile modulus is similar to but does not necessarily equate with Young's modulus of elasticity

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel extension measuring system.

Sample Preparation

Cut a length of processed, consumer, photographic film as cleanly as possible from one edge up to the edge with the track holes. Measure the dimensions of the sample with a micrometer. In this example, the sample was approximately 2.380 mm wide, 0.145 mm deep, and 30 mm long. Tare the weight and zero the height.

Method

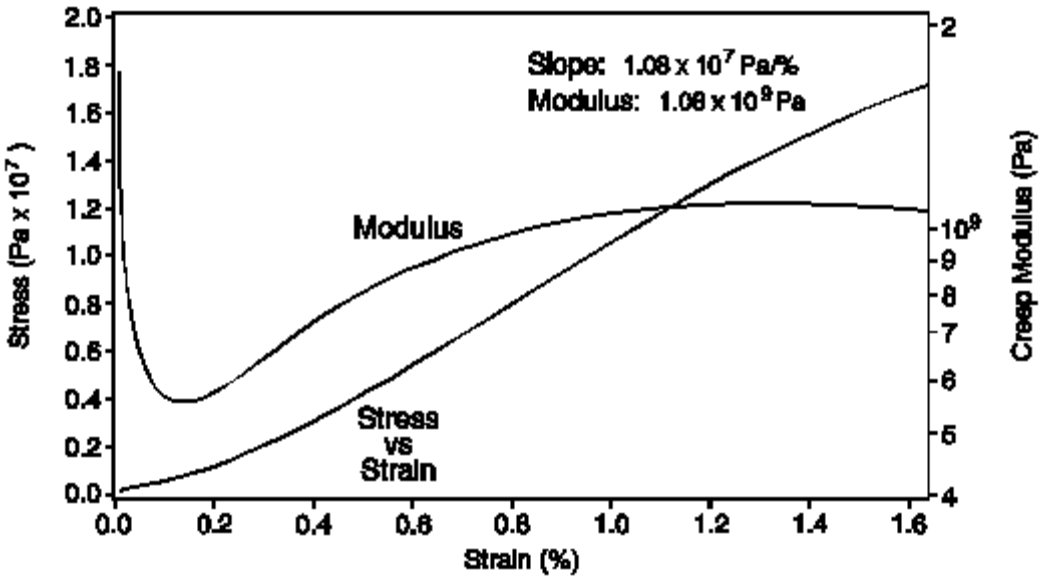
Program a static stress scan at 25°C for 3 min. Mount the sample in the top clamp first. Raise the top clamp to approximately 10 mm above the bottom clamp. Loosen the bolts of the top clamp and thread the sample through the clamp. Tighten the two bolts alternately to about 3600 g cm (50 oz in.) while keeping the sample in the center of the clamp. Thread the sample through the bottom clamp and tighten in the same manner, making certain that it is aligned.

Raise the furnace and allow the sample, analyzer, and furnace to equilibrate for about 2 min or until the probe position stabilizes. Read the sample height and start the run. During the run, the sample is held in place with a minimal static stress, then the static stress is linearly increased. The response of the sample, represented by static strain, is used to calculate the static tensile modulus.

NOTE: The tensile modulus is determined in the linear region of stress and strain. The proportional limit, yield, drawing, ultimate strength, and other results can be determined using other methods for high modulus materials.

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Sample:	Photographic film
Instrumental:	Analyzer: DMA 7e Measuring System: Extension Geometry: Film Sample Height: 5.946 mm Sample Width: 2.380 mm Sample Depth: 0.145 mm
Environmental:	Purge Gas: Helium (20 cc/min) Coolant: Tap Water
Parameters:	Method: Static force scan Temperature Program: 23°C for 12.8 min Recovery Force: 100 mN Creep Ramp Rate: 500 mN/min Creep Force: 6475 mN End Condition: Go To Load



Results/Discussion

The figure above shows the plots of stress and tensile modulus versus strain. As the stress increases above 0.01 x 10^7 Pa, the apparent modulus decreases due to sample mounting effects and stress relief between the sample and the measuring system. As the stress increases above 0.20 x 10^7 Pa, the strain increases as the sample becomes more elongated.

As the stress increases above 1.00 x 10^7 Pa, the sample exhibits Hookean behavior, i.e., it behaves like an ideal spring. In this region, increasing stress produces increasing and proportional strain. This is the linear region for this sample *at this temperature*.

The tensile modulus (M) is the slope of the tensile stress versus the tensile strain in the linear region. Like flexural modulus, the linear region for tensile modulus is that part of the modulus curve that approaches a linear relationship with strain, given a well-behaved stress–strain relationship. Tensile modulus also requires that the sample shape must not change (i.e., the sample cannot begin to draw or elongate). The slope of the stress–strain curve in this region is 1.08 x 10^7 Pa/%. This result is multiplied by 100 to cancel the % strain units. The tensile modulus in the linear region for this sample at 23°C is 1.08 x 10^9 Pa.

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The Pyris software can calculate the tensile modulus and present it as a curve. This modulus curve is shown in the figure above versus strain. Above a stress of 1.00 x 10^7 Pa, a tensile modulus of approximately 1.08 x 10^7 Pa is observed.

References

1. "Standard Test Methods for Tensile Properties of Plastics (Metric)," ASTM Standard D638M-93, Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.01.
2. "Standard Test Methods for Tensile Properties of Thin Plastic Sheeting," ASTM Standard D882-91, Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.01.
3. "Determination of Tensile Properties," ISO 527, ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 1.
4. "Determination of Tensile Properties by Use of Small Specimens," ISO 6239, ISO Standards Handbook 21-Plastics. ISO, Geneva, 1990, Vol. 1.

PID Factors for Position Control

Introduction

When using Position Control with the DMA 7e, the probe is maintained at a constant position using a "Proportional + Integral + Derivative" motor control system. The motor control system compares the measured sample position to the program position (setpoint) and generates a motor command that is related to the difference between these positions (1). The motor command is then multiplied by the user-selected values. These values are selected in the PID Controls page in Preferences. The motor command is then used to program the amount of force applied by the motor to the sample.

Theory

Constant length or fixed length mechanical analysis is often used to evaluate shrinkage force of films and fiber samples.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel extension measuring system.

Sample Preparation

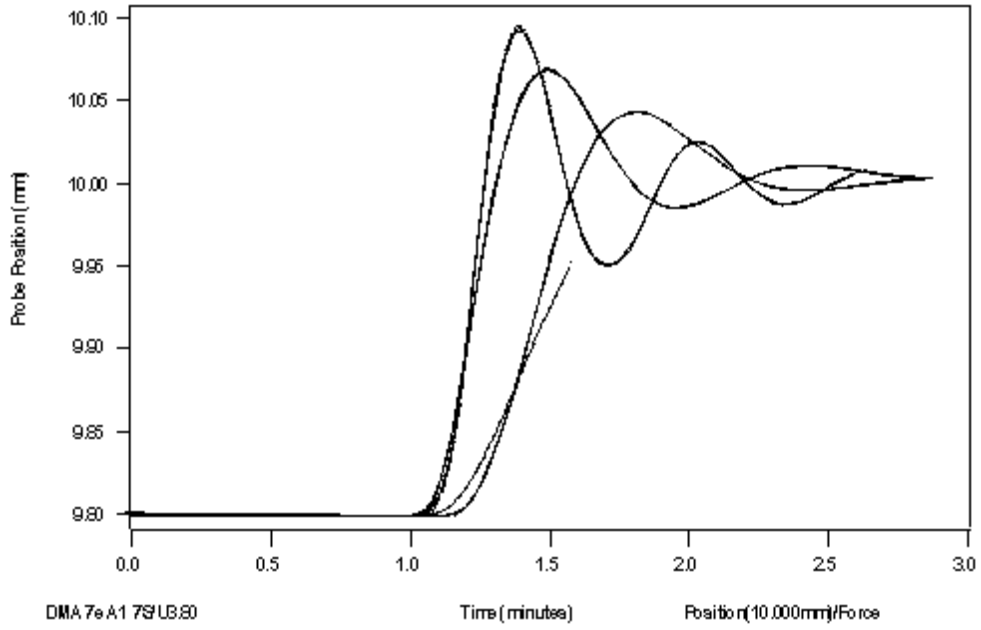
Cut the sample to size using a razor blade.

Method

Program an isothermal scan at 25°C for 10 min. A typical method for this application is as follows:

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Sample:	Thin Film
Instrumental:	Analyzer: DMA 7e Measuring System: Extension Geometry: Film
Environmental:	Purge Gas: Nitrogen (30 cc/min) Coolant: Ice water
Parameters:	Method: Isothermal Scan Static Force: 550 mN Dynamic Force: 500 mN Frequency: 1 Hz
Controls:	Position Control = ON: Current Dynamic: Force Temperature: 25°C

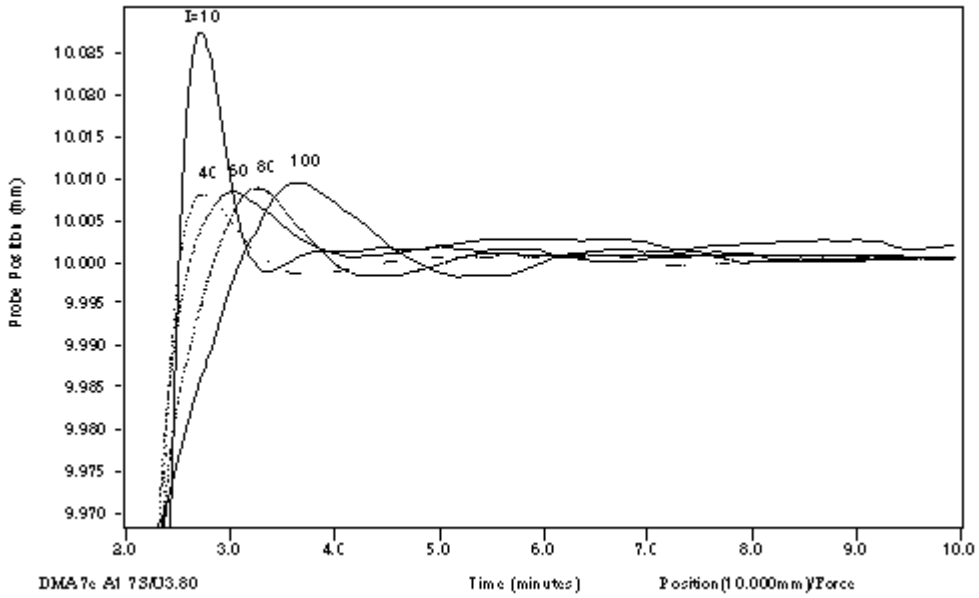


Results/Discussion

The figure above is a plot of the probe position for four runs of the same sample using different P values selected for PID gains versus time. (P values are entered in the PID Controls page in Preferences.) Initially, the sample is 9.8 mm long; after 1 minute Position Control is turned on and extends the sample to the setpoint of 10.00 mm. The first term of the PID control equation produces a correction that is proportional ("P") to the difference between the user-selected setpoint and the current probe position. A control equation that uses only proportional control will typically maintain a probe position within a close range of the setpoint; however, it typically does not converge to the setpoint and there is often some constant offset. The higher the proportional gain, the greater the response of the control system and it is likely to overshoot the setpoint. The effect of selecting P gains of 60, 30, 20, and 10 is shown in the figure above. A P gain of 60 caused the control system to overshoot the 10.000-mm setpoint. P gains of 20 and 10 caused the control system to react more slowly. P gain of 30 caused the control system to react quickly with less than 0.5% overshoot. The correction of this overshoot using the “D” gain is described below.

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The third term produces a correction based on the time derivative ("D") of the difference between the setpoint and the probe position. This control minimizes overshoot and undershoot of the setpoint by controlling the slope of the position versus time curve. This component of the control typically responds more slowly and can eliminate any constant offset between the probe position and the probe setpoint. The higher the D gain the more accurately it will converge on the setpoint with time. The effect of selecting D gains of 10, 40, 60, 80, and 100 is shown in the figure below. A D gain of 10 caused the control system to overshoot the 10.000-mm setpoint. A D gain of 40 greatly reduced the overshoot but did not converge to the setpoint as quickly. D gains of 80 and 100 caused the control system to react even more slowly. A D gain of 60 caused the control system to react quickly with less than 0.1% overshoot and converge very quickly on the desired 10.000 mm sample height.



If less than 0.1% overshoot is acceptable, then the PID gains of 30, 60, 60 are optimum and will stabilize within 3 minutes. If no overshoot is required, then motor PID gains of 20, 60, 60 may be selected, but these gains may require more time to stabilize for this sample, measuring system, and temperature.

Hints for Optimum Performance

If performing heating or cooling experiments, heating and cooling rates should not exceed the time required for the Position Control to react. When Position Control adjustments are made, there may be modulus and tangent delta transitions as a results of the new forces that are applied. The transition will be proportional to the adjustment and not indicative of sample properties.

How Motor Control Is Used

Motor controls may be turned on or off at any time before or during the analysis. When the motor control is off, the force control is used to control the motor.

Motor Control Gains (PID)

Motor control motor gains determine the way the motor control operates. The values used for these motor gains depend on the users' requirements. The motor gains are selected in the PID

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Controls page in Preferences. The software reads these values whenever the motor control is turned on.

Reference

Cassel, R.B., Twombly, B., Goodkowsky, S.F., Proceedings of the 21st NATAS Conference, Atlanta, GA; North America Thermal Analysis Society, 1992, Paper 106.

Isothermal Modulus Determination Using Position Control

Introduction

Position Control holds the sample in a constant position selected by the user by increasing or decreasing the static force applied to the sample. Position Control is also called PID control (Proportional + Integral + Derivative). The sensitivity of this control must be adjusted for the specific sample or analysis to be run. This application presents a procedure for determining the storage modulus and tangent delta of a thin film at a fixed sample length of 10 mm. It also provides instructions on selecting the optimum settings (gains) for the Position Control function.

Theory

The isothermal modulus of a sample may be determined with a fixed sample length using Position Control.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel extension measuring system.

Sample Preparation

Cut a photographic film sample and mount it in the extension probe. Click here to see how to mount your sample.

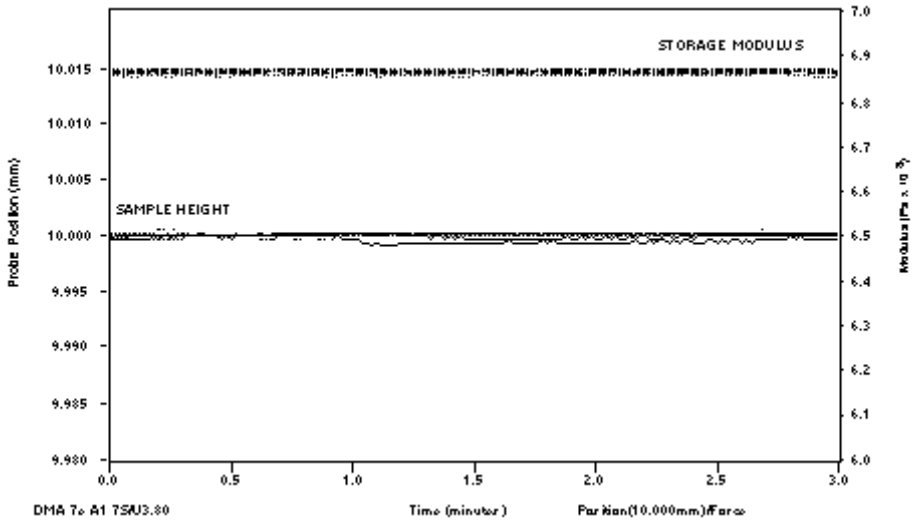
Method

Install the dewar and fill with liquid nitrogen. Supply a helium purge gas at 30 cc/min. Set the P and D gains to 30 and 60, respectively, in the PID Controls page of Preferences and program an isothermal run +25°C for 3 min. Program the furnace to heat to 25°C and raise it around the measuring system. Turn on Position Control in the Initial State page, allow to equilibrate for about 2 min, and then start the analysis. The total equilibrate and run time is 5 min. A typical method for this application is as follows:

Sample:	Photographic film
Instrumental:	Analyzer: DMA 7e Measuring System: Extension Geometry: Film
Environmental:	Purge Gas: Helium (30 cc/min) Coolant: Liquid Helium
Parameters:	Method: Isothermal Temperature Program: 25°C for 3 min Static Force: 550 mN

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	Dynamic Force: 500 mN Frequency: 1 Hz
Control:	Position Control = ON: 10 mm



Results/Discussion

The figure shows results for six runs. The sample was maintained at the 10.000 mm height within 0.1% for all six runs. The resulting modulus of 6.87 x 10^8 was reproduced for all six runs.

Hints for Optimum Performance

If performing heating or cooling experiments, heating and cooling rates should not exceed the time required for the Position Control to react. When Position Control adjustments are made, there may be modulus and tangent delta transition resulting from the new forces that are applied. The transition will be proportional to the adjustment and not indicative of sample properties.

How Motor Control Is Used

Motor controls may be turned on or off at any time before or during the analysis. When the motor control is off, the force control is used to control the motor.

Motor Control Factors

Motor control gains determine the way motor control operates. The values that you assign to these motor gains depend on your requirements. The motor gains are selected in the PID Controls page in Preferences in the Tools menu. The software reads these values whenever the motor control is turned on.

Reference

Cassel, R.B., Twombly, B., Goodkowsky, S.F., Proceedings of the 21st NATAS Conference, Atlanta, GA; North America Thermal Analysis Society, 1992, Paper 106.

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Thermal Characterization of a Thin Film Using Position Control

Introduction

When using Position Control with the DMA 7e, the probe is maintained at a constant position using a “Proportional + Integral + Derivative” motor control system. The motor control system compares the measured sample position to the program position (setpoint) and generates a motor command related to the difference between these positions (1). The motor command is then multiplied by the user-selected values entered in the PID Controls page of Preferences. The motor command then is used to program the amount of force to be applied by the motor to the sample.

Theory

Modulus is often used to describe physical properties of samples. Modulus is typically determined within the linear region (or elastic limit) of the test material. If a material has a limited linear region, the sample length should not exceed this region. Under these conditions it may be necessary to hold the sample at a fixed length to obtain an accurate determination of its modulus. The DMA 7e measuring systems, combined with the numerous geometries and instrument controls, extend the analytical range of the instrument and give the operator additional versatility.

Instrument

Use the DMA 7e Dynamic Mechanical Analyzer equipped with the stainless-steel extension measuring system.

Sample Preparation

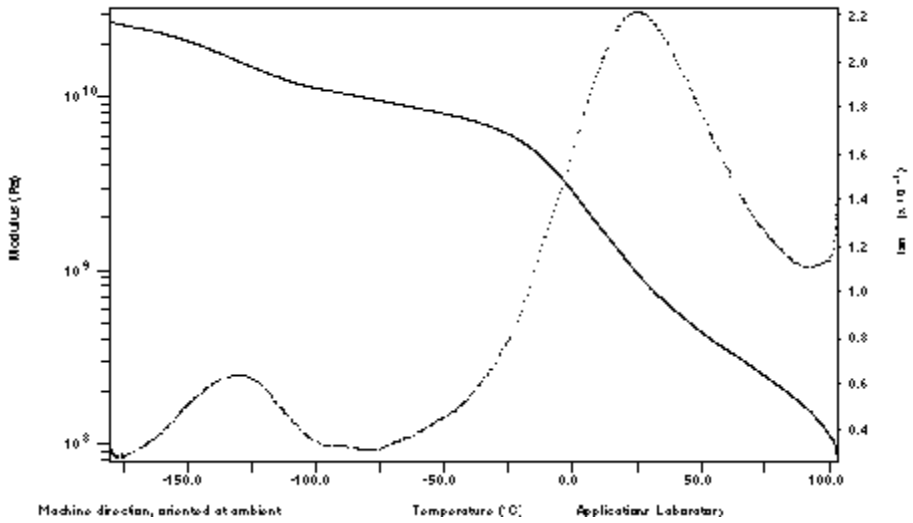
Cut the sample to size using a razor blade. Mount the sample in the extension measuring system. Click here to see how to mount the sample.

Method

Program a heating experiment from –170°C to +100°C at a heating rate of 10°C/ min. Program the furnace to heat to –170°C and raise it around the measuring system. Turn on Position Control in the Initial State page, allow to equilibrate for about 2 min, and then start the analysis.

Sample:	Epoxy-Glass Composite
Instrumental:	Analyzer: DMA 7e Measuring System: Extension Geometry: Film
Environmental:	Purge Gas: Nitrogen Coolant: Ice water
Parameters:	Method: Temperature Scan Static Force: 550 mN Dynamic Force: 500 mN Frequency: 1 Hz Heat from –170°C to 100°C at 10°C/min
Controls:	Position Control = ON: current Dynamic Force

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Results/Discussion

The figure above shows storage modulus and tangent delta versus temperature. As the sample is heated from –170°C to –75°C, the storage modulus exhibits a mild decrease and tangent delta shows a peak. These characteristics result from segmental molecular motion while the sample is in the glassy state (referred to as the beta transition region).

As the sample is heated from –75°C to +75°C, the storage modulus exhibits a very large decrease and the tangent delta shows a large peak. These characteristics result from the sample passing through its glass transition region (Tg). The glass transition is the temperature range where the sample changes from a “glassy” state to a “rubbery” state.

As the sample is heated from +75°C to 100°C, there is little change in mechanical properties in this “rubbery” region.

As the sample is heated above 100°C, it begins to soften again as the sample passes out of the rubbery region into the melt region. When the sample is no longer in the solid state the run is concluded.

How Motor Control Is Used

Motor controls may be turned on or off at any time before or during the analysis. When the motor control is off, the force control is used to control the motor. The optimum setup of the motor control system will control the sample position at the program position (set point) without excessive delay or correction. Optimum settings for any experiment depend on the individual experimental requirements.

Motor Gain Files

Motor control gains determine the way motor control operates. The values that you assign to these motor gains depend on your requirements. The motor gains are selected in the PID Controls page in Preferences in the Tools menu. The software reads these values whenever the motor control is turned on.

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Reference

Cassel, R.B., Twombly, B., Goodkowsky, S.F., *Proceedings of the 21st NATAS Conference*, Atlanta, GA; North America Thermal Analysis Society, 1992; Paper 106.

TGA Application

TGA for the Determination of Percent Carbon Black

Introduction

Carbon black is often found in plastics as an additive for the purpose of improving its properties: physical, chemical, or mechanical. The amount of carbon black can often be assessed using the Thermogravimetric Analyzer.

Theory

Carbon black content can be determined by TGA by evaluating the weight loss data. A plastic is heated to 600°C in an inert atmosphere (nitrogen or argon). While at 600°C, the atmosphere is switched from inert to an oxidizing atmosphere (air or oxygen). At this elevated temperature with an oxidizing environment, the carbon will burn yielding a weight loss equal to the percent carbon.

Instrument

TGA 7 with GSA 7 or TAGS gas selector accessory

Sample Preparation

Samples can be sliced, diced, or pulverized in order to increase the surface area. Once the sample has been prepared, it should be placed into the TGA sample pan and distributed evenly across the pan bottom. The standard platinum sample pan (0319-0264) as supplied with the analyzer (standard furnace) or platinum sample pan (N519-0280) with the high temperature furnace is used for this application. After the furnace has been raised, it is important to allow the system to purge with the inert gas for about 10 minutes to ensure an air-free environment.

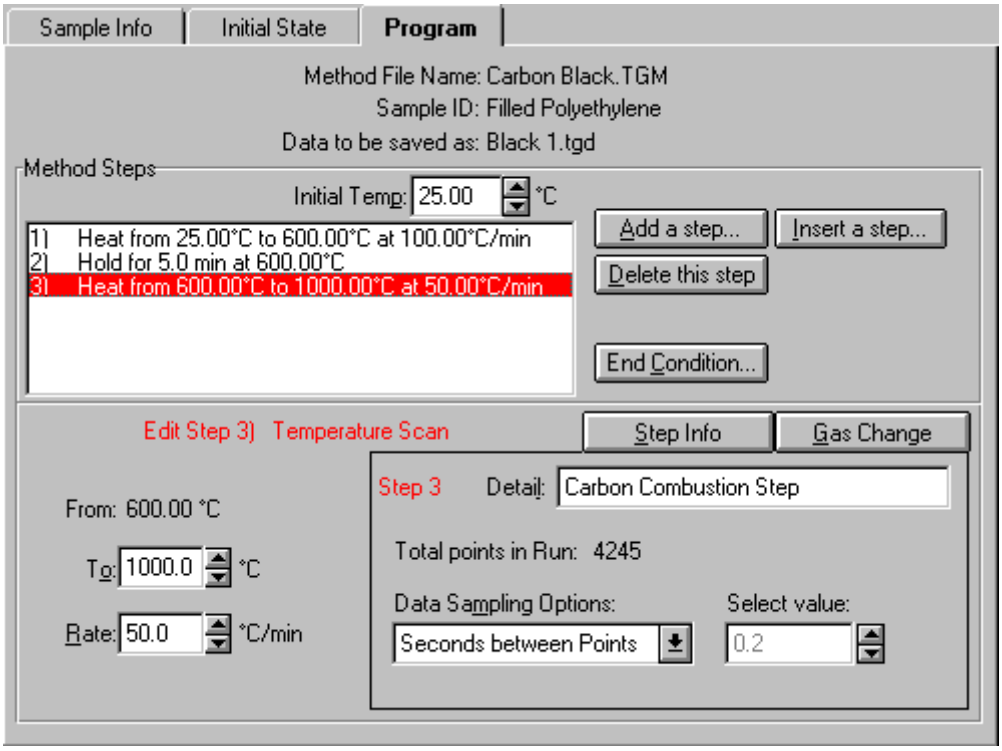
Method

The temperature range is from room temperature to 600°C. After switching the purge gas, increasing the temperature to 1000°C will help the carbon combustion.

A relatively quick temperature scan of 100°C/min from room temperature to 600°C is used. At 600°C the analyzer can be held isothermally for 5 minutes. This should give a plateau where no weight is being lost. After 5 minutes at 600°C, the purge gas is switched from inert to active and the analyzer can be heated to 1000°C at 50°C/min. This facilitates the combustion and will shorten the analysis time.

Nitrogen or argon is required for the pyrolysis part of the experiment. Oxygen or air is necessary for the carbon combustion. Pure oxygen will facilitate a faster, cleaner burn at lower temperatures.

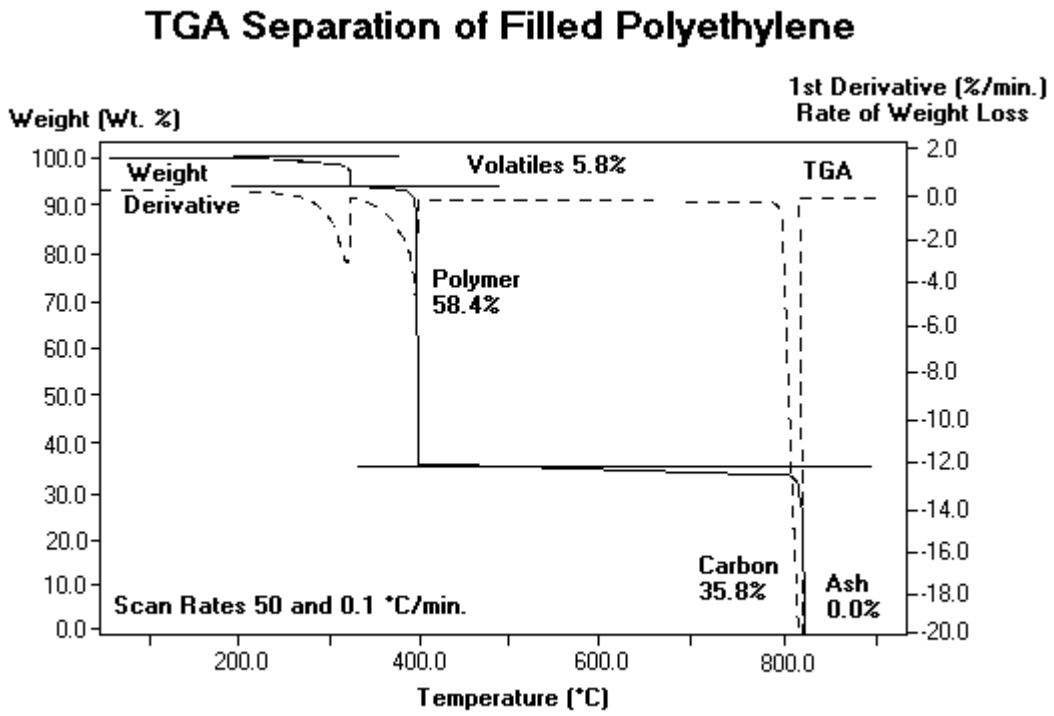
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Results/Discussion

Percent carbon is calculated directly from the weight loss data. This is taken from where the data plateaus at 600°C while still under an inert atmosphere and the final weight value after the carbon has been burned.

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References

1. "Characterization and Quality Control of Engineering Thermoplastics by Thermal Analysis," Perkin-Elmer Thermal Analysis Application Study (TAAS) #22. The Perkin-Elmer Corporation, Norwalk, CT, 1977.
2. "Applications of Thermal Analysis in the Automotive Industries," Perkin-Elmer Thermal Analysis Application Study (TAAS) #26. The Perkin-Elmer Corporation, Norwalk, CT, 1978.
3. "Separation of Filled Polyethylene Using Auto-Stepwise Mode," Perkin-Elmer Thermal Analysis Newsletter (PETAN) #57, The Perkin-Elmer Corporation, Norwalk, CT, 1994.

TMA Application

TMA 7 Vicat Softening Temperature Determination

Introduction

The TMA 7 method described here is optimized for performing vicat softening temperature determinations for a broad range of thermoplastics, elastomers, and some thermosets in the rigid and semirigid states. Samples in the form of disks or rectangles are mounted using the 1-mm parallel plate quartz measuring system (penetration probe). The softening temperature is the temperature at which a 1-mm probe penetrates 1 mm into the sample. This measurement is made while the sample is heated and 5000-mN force is applied.

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NOTE: The instrument can be modified to accommodate an oil bath, but good temperature precision is obtained using the standard furnace system. Temperatures can be verified using primary melting standards. A force of 500 gF (gram force) was used in this test. A force of 1000 gF can be achieved using weights in the weight tray.

Theory

Softening temperature determinations provide a qualitative measure of the temperature resistance of a material. Standard test methods have been created by ASTM and ISO to define the vicat softening temperature determination (1–3).

Instrument

Use the TMA 7 Thermomechanical Analyzer equipped with the quartz (1 mm) penetration probe measuring system.

Sample Preparation

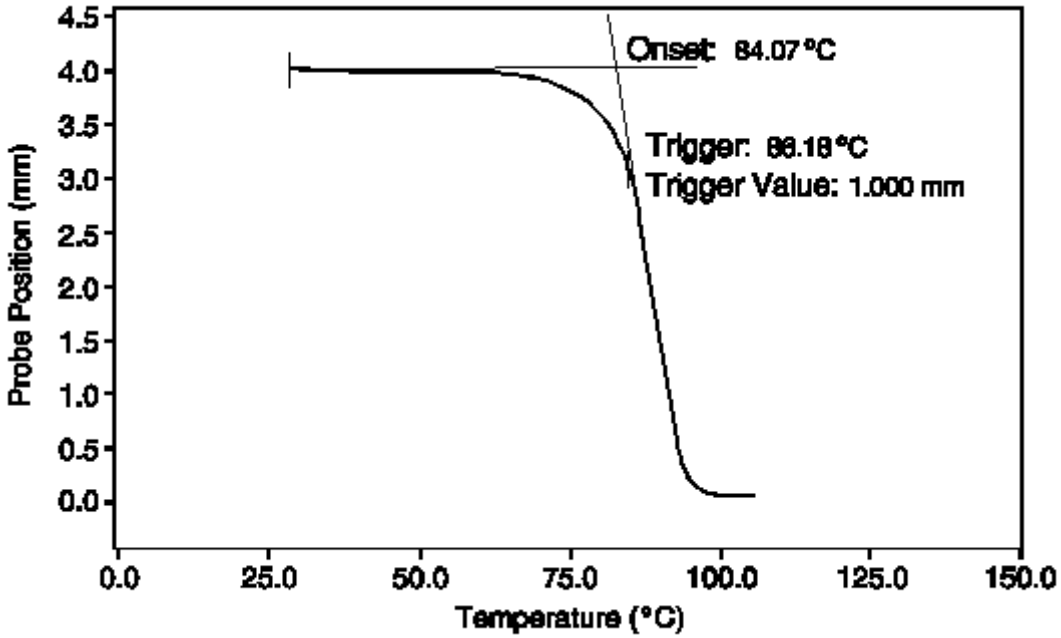
Cut the sample, an injection-molded 2-mm-thick sheet of styrene-butadiene, into disks using a heavy-duty 6-mm hole punch. Tare the weight and zero the height. Mount the sample by placing two disks on the bottom plate, lowering the probe, and visually aligning it with the top plate.

Method

Program the temperature scan from 30°C to 250°C at 2°C/min (120°C/h). Use a faster heating rate if all the samples are exposed to the same temperatures and the temperature is controlled to within + 1°C. Enter a static force of 5000 mN (6.37×10^6 Pa), a dynamic force of 10 mN (1.27×10^4 Pa), and a frequency of 10 Hz in the Initial State page. The dynamic force and frequency are included to enhance probe position sensitivity. Raise the furnace and allow the sample, analyzer, and furnace to equilibrate for about 2 min or until the probe position stabilizes. Read the sample height and start the run. When the probe has penetrated more than 1 mm, stop the run by clicking on the **Stop** button on the control panel.

Sample:	Styrene Butadiene Disk
Instrumental:	TMA 7 Analyzer Probe: Penetration Sample Height (y): 4.054 mm
Environmental:	Purge Gas: Helium, (20 cc/min) Coolant: Tap Water
Parameters:	Method: TMA Temperature Scan Temperature Program: 30°C to 250°C at 2°C/min Static Force: 5000 mN

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Results/Discussion

The figure above shows a plot of probe position (linear millimeter scale) versus temperature for the SBR sample. There is a decrease in the probe position resulting from the slow penetration of the probe as the sample is heated and the stress placed upon it. Above 50°C, the probe position begins to decrease rapidly as the sample approaches its melting temperature. A few degrees above 80°C the sample melts and the probe position indicates 0 mm.

Perform an onset calculation on the probe position curve. Place the left tangent on the original sample height at 30°C and the right tangent on the midpoint of melting, and include a trigger value in the calculation. The onset of the melt occurs at 84.07°C. The 1-mm trigger and the softening temperature of this sample occur at 86.18°C.

References

1. "Standard Test Methods for Vicat Softening Temperature of Plastics," ASTM Standard D1525-91; Annual Book of ASTM Standards. ASTM, Philadelphia, 1994, Vol. 08.01.
2. "Vicat Softening Temperature of Thermoplastics," ISO 306; ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 2.
3. "Vicat Softening Temperature of Unplasticized PVC Pipes and Fittings," ISO 2507; ISO Standards Handbook 21 - Plastics. ISO, Geneva, 1990, Vol. 3.

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4.

Chapter 11
Quick Help

NOTE: Throughout Quick Help there is reference to videos, which would be run if you were in online help. In order to see the videos that are associated with the text while reading this manual, you will have to insert the Pyris CD in the CD drive of your computer. Display the Multimd directory's contents in Windows Explorer. Double click on the .avi file indicated in the text below; the file should play on the computer monitor.

Explore the Software

Using the Pyris Manager

Opening the Pyris Manager  (START.AVI)

1. Click on the Windows **Start** button in the lower-left-hand side of the screen. This displays the **Start** button menu.
2. Bring the cursor up to Programs and click on it. This displays the Programs menu.
3. Bring the cursor down to Pyris Software for Windows and click on it. This displays the Pyris menu.
4. Click on Pyris Manager to open the Pyris Manager. The Pyris Manager bar will be displayed across the top of the screen or wherever it was last displayed.

Hiding the Pyris Manager  (MANAGER.AVI and MGRSIDE.AVI)

To have the Pyris Manager "hide" off the screen while not needed, you have to activate the Autohide feature.

1. Position the cursor on the Start Pyris button on the Pyris Manager or in the space beneath it.
2. Click on the right mouse button to display the Pyris Manager popup menu.

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3. Click on Autohide. The menu automatically closes.
4. Bring the cursor off of the Pyris Manager and it will automatically roll off of the top of the side of the screen.
5. Bring the cursor close to the edge of the screen and the Pyris Manager will automatically reappear.

Repositioning the Pyris Manager

The Pyris Manager can be displayed across the top of the screen (the default position) or on the side. When in either position, the full Pyris Manager bar is displayed. It can also be positioned anywhere else on the screen as just the **Start Pyris** button.

To reposition the full Pyris Manager  **(REPOSIT.AVI)**

1. Make sure that Autohide is off.
2. Position the cursor in the blank space beneath the **Start Pyris** button.
3. Click on the left mouse button and keep your finger on the button.
4. Slightly move the cursor down and to the left. The Pyris Manager will be redisplayed vertically on the left-hand side of the screen.
5. To bring the bar back to the top of the screen, place the cursor in the blank space, depress the left mouse button and move the cursor slightly up and to the right.

To reposition the Start Pyris button  **(REPOBTN.AVI)**

1. Make sure that the Autohide feature is on.
2. Position the cursor in the space beneath the **Start Pyris** button.
3. Click on the left mouse button and keep your finger on the button.
4. Drag the Pyris Manager down. The bar instantly shrinks. You can take your finger off of the mouse button when the **Start Pyris** button is positioned where you want it.
5. Position the cursor in the space beneath the **Start Pyris** button, depress the left mouse button, and drag the box up to the top of the screen. The full Pyris Manager will automatically redisplay.

Start the Software

To start Pyris Software for Windows, you first must open either the Data Analysis application or the Pyris Manager. From the Pyris Manager you can open an Instrument Application.

To start, do one of the following:

1. Select Pyris Manager from the Pyris Software for Windows menu which is displayed by clicking on the Windows **Start** button, selecting Programs, and then selecting Pyris Software for Windows. This will display the Pyris Manager bar.

OR

2. Click on the Pyris Manager icon on the Windows desktop if that option was selected during installation. This will display the Pyris Manager bar.

OR

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3. Select Data Analysis from the Pyris Software for Windows menu which is displayed by clicking on the Windows **Start** button, selecting Programs, and then selecting Pyris Software for Windows. This will display the Data Analysis window with the last data file that was displayed.

To open an Instrument Application:

The Pyris Manager bar contains a button for each instrument that is configured into the system. Click on the button of the analyzer you wish to use. The software is loaded and the Method Editor window is displayed.

To open a Data Analysis Application:

Besides opening the general Data Analysis, in which you can open data files from any analyzer, from the Pyris Software for Windows menu, you can also access Data Analysis from the Pyris Manager.

1. Open the Pyris Manager.
2. Click on the **Start Pyris** button to display the Task menu.
3. Select Data Analysis from the menu. The Data Analysis window appears with the last data file that was displayed.

Configure Your System

Your analyzer(s) should be configured when the Perkin Elmer service engineer installs them. However, you may add or remove an analyzer later, or you may add or remove an accessory for an existing analyzer. You can do this with the Pyris Configuration software.

To add a new instrument:

1. Make sure the analyzers to be configured are installed, connected to the PC, and powered on.
2. Select Pyris Configuration from the Pyris Software for Windows menu which is displayed by clicking on the Start button, selecting Programs, and then selecting Pyris Software for Windows. Or open the Pyris Manager and select Configure Analyzers from the **Start Pyris** button's task menu.
3. Select the **Add Analyzer** button to add a new analyzer to the system.
4. Select the communications port where the new analyzer is connected, then select the **Add** button.
5. Edit the analyzer name, if desired. Click on the check boxes for any accessories that are to be used, then select the **OK** button.
6. Repeat steps 3, 4, and 5 for each analyzer.
7. Select **Close** when all analyzers are configured.

You do not have to reboot the system; the configuration is dynamic and takes effect automatically.

To remove an instrument:

1. In the Pyris Configuration window, highlight the analyzer you wish to remove.
2. Select the **Remove** button.

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3. You are asked if you are sure you want to remove this analyzer. By clicking on **Yes**, you remove the analyzer from the configuration files and the instrument's button is removed from the Pyris Manager. You do not have to physically detach the analyzer from the computer if you might need to reconfigure the analyzer back onto the system later.

Monitor the System Status

To monitor the status of your Pyris analyzers:

1. Look at the Pyris Manager.
2. Each analyzer button on the Pyris Manager displays the analyzer's current temperature, status, and method information, if the analyzer is performing a run. You can change the information displayed in the button by right-clicking on the button to display the Change Status Information dialog box.
3. In an Instrument Application, look at the Status Panel. (If the Status Panel is not displayed, select the **Status Panel** command in the View menu.)
4. The Status Panel displays the current value of analyzer parameters that you choose to display. You can adjust the Status Panel to display more or less parameters.
5. While in an Instrument Application, look at the analyzer's real-time signal in the Instrument Viewer by selecting the **Monitor** command in the View menu.

Perform a Run

The steps involved in performing a typical run vary from instrument to instrument. There is a general outline, however, that applies to a typical run on any analyzer.

1. Start the system by powering on
   - computer
   - autosampler (if present)
   - any accessory such as GSA 7 or TAGS
   - analyzer
   - TAC (if present)
   - printer
2. Start the Pyris software.
3. Open the Method Editor. The Method Editor is opened automatically when you open an analyzer's Instrument Application.
4. Prepare and load your sample and reference material.
5. Create a new method or load and edit an existing method. Enter information about the sample, initial values, and a temperature – time program.

   You could also create a play list using the Pyris Player Editor. The play list should contain instructions to load samples (either manually or using an autosampler), run methods, and display and analyze results.
6. Start the run by clicking on the **Start** button on the control panel.

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7. Monitor the run in Instrument Viewer. At the end of the run the data is saved in the file specified in the Method Editor.
8. Perform any data analysis in the Data Analysis application or perform another run.

Look at the Data

1. To look at data **during** a run, look at the Instrument Viewer in the analyzer's Instrument Application. The Instrument Viewer displays the real-time curve.
2. To look at data already collected and saved:
   - If the Instrument Viewer or the Method Editor is displayed, select the **Data Analysis** button from the toolbar and select the desired file from the dialog box. This displays the data file in the instrument-specific Data Analysis window.
   - Select Data Analysis from **Start Pyris** button's task menu and then select the desired file from the dialog box. This displays the data file in the general Data Analysis Application.

Print a Curve, Method, or Calibration File

To print the displayed curve(s):

1. Use the Print Preview button  on the toolbar or the Print Preview command in the File menu to see what the printout will look like before you print.
2. You can select the Print button on the Print Preview window, the Print button  on the Pyris toolbar, or the Print command in the File menu. The Print dialog box appears.
3. Select the printer, any properties (e.g., landscape or portrait orientation), and number of copies you want. Click on OK.

To print a method from the Method Editor:

1. Use the Print Preview button  on the toolbar or the Print Preview command in the File menu to see what the printout will look like before you print.
2. You can select the Print button on the Print Preview window, the Print button  on the Pyris toolbar, or the Print command in the File menu. The Print dialog box appears.
3. Select the printer, any properties (e.g., landscape or portrait orientation), and number of copies you want. Click on OK.

To print a method from Data Analysis:

1. With the desired data file displayed, select the **Method Used** button  on the toolbar or the **Method Used** command in the View menu. The View Method Properties window appears.
2. Select the **Print** button in any of the pages. Select **OK** from the Print Data File dialog box.

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To print a calibration file:

1. Display the Calibration window in an Instrument Application by selecting Calibrate from the View menu.
2. Use the **Print Preview** button  on the toolbar or the **Print Preview** command in the File menu to see what the printout will look like before you print.
3. You can select the **Print** button on the Print Preview window, the **Print** button  on the Pyris toolbar, or the **Print** command in the File menu. The Print dialog box appears.
4. Select the printer, any properties (e.g., landscape or portrait orientation), and number of copies you want. Click on **OK**.

Shut Down the System

To shut down the system:

1. Select the Exit command in the File menu of an open application. Select Close All from the **Start Pyris** button's task menu to exit Pyris Software for Windows.

OR

2. Select Close All from the Start Pyris button task menu. This will close all Pyris-related windows. If any files were changed and not saved, you will be asked if you want to save them.
3. Turn off the printer.
4. Turn off all TAC controllers in your system.
5. Turn off all Pyris analyzers in your system.
6. Turn off all accessories in your system (GSA 7, printers, etc.).
7. Turn off the autosampler if present.
8. Shut down the computer.

Calibrate an Analyzer

The typical calibration routines for each analyzer are presented below.

Apply existing calibration file values:

When you open the Calibration window, the current calibration file's values are displayed. Suppose you are going to run some samples in a different temperature range than the one you have been using and that you calibrated the instrument for that range previously and saved the file. Select Open from the File menu, select the desired calibration file, click on **Save and Apply** to apply the calibration file's values to the analyzer, and click on **Close**.

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Calibrate a Pyris 1 DSC

To perform a Pyris 1 DSC Temperature Calibration:

1. In Instrument Viewer or Method Editor, select **Calibrate** from the View menu. The Calibration window appears.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are going to perform all of the calibration procedures, restore all the default calibration values by selecting the **All** command.
3. Select **Save and Apply** to send the default calibration values to the analyzer and save the calibration file.
4. Click on **Close** in the Calibration window.
5. Perform the sample run for each reference material to be used, under the same conditions that you run your samples.
6. For each run, perform a peak area calculation and include the onset temperature. Record the ΔH and onset results.
7. Select **Calibrate** from the View menu.
8. Select the Temperature tab in the Calibration window.
9. Enter the name of the reference material used, expected onset temperature, the measured onset temperature, and the method used for each run.
10. Click in the check box in the Use column for each reference that you want to use for the calibration.
11. Select the **Save and Apply** button to save the values and send them to the analyzer.
12. Select the **Close** button or select the Heat Flow tab to perform a Heat Flow calibration.

To perform a Pyris 1 DSC Heat Flow Calibration:

1. Perform a sample run for the reference material to be used or use the results from one of the sample runs done for the Temperature calibration.
2. If you performed a new run, calculate the peak area; otherwise, you can use the ΔH recorded during the Temperature calibration.
3. In the Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
4. Select the Heat Flow tab in the Calibration window.
5. Enter the sample name and weight, expected heat flow, and method for the reference material to be used.
6. Enter the calculated ΔH into the Calibration table.
7. Select the **Save and Apply** button to save the values and send them to the analyzer.
8. Select the **Close** button or select the Furnace tab to perform the Furnace calibration.

To perform a Pyris 1 DSC Furnace Calibration:

NOTE: A Furnace calibration should be done after a Temperature calibration.

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. If applicable, perform the Temperature calibration.

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3. Remove any sample pans from the sample and reference holders.
4. Select the Furnace tab in the Calibration window.
5. Enter the minimum and maximum temperature limits. These should be below and above your normal operating region.
6. Select the **Begin Calibration** button.
7. Wait the designated time for completion of the Furnace calibration.
8. Select **Save and Apply**.
9. Select **Close**.

Calibrate a DSC 7

To perform a DSC 7 Temperature Calibration:

1. In Instrument Viewer or Method Editor, select **Calibrate** from the View menu. The Calibration window appears.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are performing all of the calibration procedures, restore all the default calibration values by selecting the **All** command.
3. Select **Save and Apply**.
4. Click on **Close**.
5. Perform a run for each reference material to be used under the same conditions that you run your samples.
6. After each run, perform a peak area calculation and include the onset temperature. Record the ΔH and the onset temperature results.
7. Select **Calibrate** from the View menu.
8. Select the Temperature tab in the Calibration window.
9. Enter the name of the reference material used, the expected onset temperature, the measured onset temperature, and the method used for the run.
10. Click on the check box in the Use column for each reference that you want to use for the calibration.
11. Select the **Save and Apply** button to save the values and send them to the analyzer.
12. Select the **Close** button or select the Heat Flow tab to perform the Heat Flow calibration.

To perform a DSC 7 Heat Flow Calibration:

1. Perform the run for the reference material to be used or use the results from one of the sample runs done for the Temperature calibration.
2. If you performed a new run, calculate the peak area; otherwise, you can use the ΔH recorded during the Temperature calibration.
3. In Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
4. Select the Heat Flow tab in the Calibration window.
5. Enter the name of the reference material and the weight, expected heat flow, the measured heat flow, and the method used for the run.

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6. Select the **Save and Apply** button to save the values and send them to the analyzer.
7. Select the **Close** button or select the Furnace tab to perform the Furnace calibration.

To perform a DSC 7 Furnace Calibration:

NOTE: This calibration is available only if the DSC 7 has the DDSC accessory installed. This calibration should be done after a Temperature calibration.

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. If applicable, perform the Temperature calibration.
3. Remove any sample pans from the sample and reference holders.
4. Select the Furnace tab in the Calibration window.
5. Enter the minimum and maximum temperature limits. These should be below and above the normal operating temperature range.
6. Select the **Begin Calibration** button.
7. Wait the designated time for completion of the Furnace calibration.
8. Select **Save and Apply**.
9. Select **Close**.

Calibrate a Pyris 6 DSC

To perform a Pyris 6 DSC Temperature Calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu. The Calibration window appears.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are performing all of the calibration procedures, restore all the default calibration values by selecting the **All** command.
3. Select **Save and Apply**.
4. Select **Close**.
5. Perform a run for each reference material to be used under the same conditions that you run your samples. (See the Hardware manual for the Pyris 6 DSC for information on running reference materials.)
6. After each run perform a peak area calculation and include the onset temperature. Record the ΔH and the onset temperature.
7. Select **Calibrate** from the View menu.
8. Select the Temperature tab in the Calibration window.
9. Enter the name of the reference material, the expected onset temperature, the measured onset temperature, and the method used for the run.
10. Click on the check box in the Use column for each reference that you want to use for the calibration.
11. Select the **Save and Apply** button to save the values and send them to the analyzer.
12. Select the **Close** button.

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To perform a Pyris 6 DSC Heat Flow Calibration:

1. Perform the run for the reference material indium or use the results from one of the sample runs done for the Temperature calibration.
2. Perform a peak area calculation and include the onset temperature or use the result from the temperature calibration.
3. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
4. Select the Heat Flow tab in the Calibration window.
5. Enter the name and weight of the reference material, the expected heat flow, the measured ΔH, and the calibration method used.
6. Select **Save and Apply** to save the values and send them to the analyzer.
7. Select the **Close** button.

Calibrate a TGA 7 or a Pyris 1 TGA

To perform a TGA 7 or a Pyris 1 TGA Temperature Calibration:

1. In Instrument Viewer or Method Editor, select **Calibrate** from the View menu. The Calibration window appears.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are performing all of the calibration procedures, restore all the default calibration values by selecting the **All** command.
3. Select **Save and Apply** to send the default calibration values to the analyzer and save the calibration file.
4. Click on **Close.**
5. Perform a run for each reference material to be used under the same conditions that you run your samples.
6. For each run, perform an onset calculation at the end of the Curie point transition and note the Onset result.
7. Select **Calibrate** from the View menu.
8. Select the Temperature tab in the Calibration window.
9. Enter the name of the reference material, expected onset temperature, the measured onset temperature, and the method used.
10. Click on the check box in the Use column for each reference that you want to use for the calibration.
11. Select the **Save and Apply** button to save the values and send them to the analyzer.
12. Click on the **Close** button.

To perform a TGA 7 or a Pyris 1 TGA Weight Calibration:

NOTE: The following instructions apply to a Pyris 1 TGA without an autosampler attached. Instructions for analyzer with an autosampler follow these instructions.

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.

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2. Select the Weight tab in the Calibration window.
3. Enter the weight of the reference material in the Calibration table.
4. Select the **Begin Calibration** button.
5. Remove any sample from the sample pan, load the sample pan onto the hangdown wire, and select **OK** in the dialog box.
6. After the empty sample pan weight reading (tare weight) is stable, click **OK** in the Read Zero dialog box.
7. Bring the sample loading tray underneath the sample pan so that the pan rests on it, place the reference weight into the sample pan, and then move the sample loading tray away. Select **OK** in the dialog box.
8. After the reference weight reading is stable, click on **OK** in the dialog box. The calibration is complete.
9. The weight is entered in the Measured field in the Weight Calibration page.
10. Select **Save and Apply** to send the new calibration values to the analyzer and save the calibration file.
11. Click on **Close**.

To perform a Pyris 1 TGA with autosampler weight calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Enter the weight of the reference material in the Calibration table.
4. Select the **Begin Calibration** button.
5. The autosampler should be in the Safe position (to the right and away from the furnace). Place an empty crucible in position 1 of the autosampler tray.
6. Click on **OK** in the dialog box. The autosampler will swing to the Load position, the crucible will be loaded onto the hangdown wire, the autosampler will move to the Safe position, and the furnace will be raised.
7. Once the reading is stable, click on **OK** to accept the reading. The furnace will be lowered, the autosampler will swing to the Load position, and the crucible will be unloaded into position 1. The autosampler will move back to the Safe position.
8. The next dialog box instructs you to place the reference weight into crucible in position 1.
9. Click on **OK** in the dialog box. The autosampler will swing into the Load position, the crucible will be loaded onto the hangdown wire, the autosampler will move to the Safe position, and the furnace will be raised.
10. Once the reading is stable, click on **OK** to accept the reading. The furnace will be lowered, the autosampler will swing to the Load position, and the crucible will be unloaded into position 1. The autosampler will move back to the Safe position.
11. The weight of the reference material will be entered in the Measured field in the Weight Calibration page.
12. Select **Save and Apply** to send the new calibration value to the analyzer and save the calibration file.
13. Click on **Close**.

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To perform a TGA 7 or a Pyris 1 TGA Furnace Calibration:

NOTE: Furnace calibration must be performed AFTER a temperature calibration.

1. Make sure the thermocouple is functioning properly. Raise the furnace. Enter 100 in the Go To Temp entry field and click on the Go To Temp button on the control panel.
2. Check that sample temperature displayed in the status panel is at or below the intended minimum calibration temperature. Click on the **Go To Load** button on the control panel.
3. While in Instrument Viewer or Method Editor, select Calibrate from the View menu.
4. If applicable, perform the Temperature calibration.
5. If present, remove any sample from the sample pan.
6. Select the Furnace tab in the Calibration window.
7. Enter the minimum and maximum temperature limits. These should be below and above your normal operating conditions. Maximum minus minimum temperature must be greater than 100°C.
8. Select the **Begin Calibration** button.
9. Wait the designated time for completion of the Furnace calibration.
10. Select **Save and Apply**.
11. Select **Close**.

Calibrate a Pyris 6 TGA

To perform a Pyris 6 TGA Furnace Calibration:

NOTE: Furnace calibration must be performed BEFORE temperature calibration.

1. Check that the thermocouple is functioning properly. Enter 100 in the Go To Temp field and click on the **Go To Temp** button in the control panel.
2. Check that the current sample temperature displayed in the status panel is at or below the intended minimum calibration temperature. Click on the **Go To Load** button in the control panel.
3. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
4. Make sure there is no sample pan in the sample holder.
5. Select the Furnace tab in the Calibration window.
6. Enter the minimum and maximum temperature limits into the Calibration table. When selecting temperatures, make sure they encompass the temperature range in which you plan to operate.
7. Select the **Begin Calibration** button.

   A message stating that the system defaults will be used for all temperature calibrations if you perform a furnace calibration is displayed. Click on **OK** to continue.
8. Wait the designated time for completion of the Furnace calibration. It takes about one hour.

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9. Select **Save and Apply**.
10. Select **Close**.

To perform a Pyris 6 TGA Temperature calibration:

NOTE: Temperature calibration must be done AFTER furnace calibration.

1. In Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Restore the default temperature calibration by selecting **Temperature** in the Restore menu. If you are going to perform a Weight calibration, select the **All** command to restore temperature and weight default calibration values.
3. Select **Save and Apply**.
4. Click on **Close** in the Calibration window.
5. Complete a scan for two or three of the four reference materials supplied – perkalloy, alumel, nickel, and iron – under the same conditions that you run your samples.
    a. From the Method Editor select **Open Method** from the File menu. Double click on the method file for the reference material you are using. The method is displayed.
    b. Place an empty sample pan on the sample holder that is already in place in the furnace; make sure that it is centered.
    c. Place the lid on top of the analyzer.
    d. Click on the **Zero Weight** button on the control panel. The weight of the empty sample pan is displayed in the Zero field in the Sample Info page.
    e. Without removing the sample pan, place 1 – 2 mm of the reference material in the pan; place the lid on top of the analyzer. Position the magnet over the center of the lid.
    f. Click on the **Sample Weight** button on the control panel. The weight appears in the Weight field in the Sample Info page.
    g. Fill in the other information on the Sample Info page. Check that the scanning rate is 5°C/min for the first run of the reference material.
    h. Click on the **Start Method** button to start the run.
    i. After the run is complete, remove the sample pan and repeat steps a through g using the same reference material but a scanning rate of 50°C/min.
    j. After a second run of a reference material, repeat steps a through h for the other reference materials you choose to use.
6. For each data file collected, open the Data Analysis window and perform an onset calculation at the end of the Curie point transition and note the Onset result.
7. Select **Calibrate** from the View menu.
8. Select the Pyris 6 TGA Temperature Calibration tab.
9. Enter the reference material names and expected and calculated onset values into the Calibration table.
10. Enter the scanning rates used and click on the radio button next to the appropriate Use References.
11. Select the **Save and Apply** button to save the values and send them to the analyzer.

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12. Select the **Close** button.

To perform a Pyris 6 TGA Weight Calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Enter the weight of the reference material in the Reference Weight column of the Calibration table. (The reference weight is in the Spares kit.)
4. Select the **Begin Calibration** button.
5. A dialog box appears instructing you to prepare the analyzer for the zero reading. Carefully place an empty sample pan (crucible) onto the sample holder (which is already in the furnace), making sure that it is centered. Replace the furnace cover on the analyzer. Click on **OK** in the dialog box.
6. When the zero reading is stable, as displayed in the Read Zero dialog box, click on **OK**.
7. The Weight Calibration dialog box tells you to place the reference weight into the sample pan. Return the furnace cover to the analyzer. Click on **OK** in the dialog box.
8. When the weight reading is stable, as displayed in the Read Value dialog box, click on **OK**.
9. The weight is entered in the Measured column in the Weight Calibration page.
10. Select **Save and Apply** to send the new calibration values to the analyzer and save the calibration file.
11. Select **Close**.

Calibrate a DMA 7e

To perform a DMA Calibration:

1. Install a 3-point bending measuring system in the DMA 7e.
2. Make sure that the DMA 7e has been on for at least 40 minutes.
3. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
4. Select the Calibrate DMA tab.
5. Enter your name in the Operator field.
6. Click on **Begin Calibration** to initiate the DMA calibration.
7. Remove any samples from the analyzer.
8. Press the **Probe Down** button on the analyzer.
9. Press the furnace locking mechanism and raise the furnace assembly until it locks in place.
10. Click on **OK**. Calibration begins.
11. Click on **OK** when calibration is complete.
12. Select **Save and Apply**.
13. Click on **Close**.

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To perform a Height calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select **Height** from the Restore menu to restore the default calibration value.
3. Select **Save and Apply**.
4. Select the Height tab from the Calibration window.
5. Enter your name in the Operator field and the height of the displacement standard in the Ref. Height field.
6. Click on **Begin Calibration**.
7. Press the furnace locking mechanism and lower the furnace assembly to the base of the analyzer. Make sure that the furnace locks in place.
8. Press the **Probe Up** button on the analyzer.
9. Make sure that there are no samples on the sample platform.
10. Press the **Probe Down** button to lower the probe so that it rests on the empty sample platform. Click on **OK** to clear the dialog box. After about 30 seconds, the Y signal reading is displayed in the Read Zero dialog box.
11. When the Y signal stabilizes, select **OK** to accept the zero value.
12. Press the **Probe Up** button.
13. Place the sapphire height displacement standard on the sample platform.
14. Press the **Probe Down** button and lower the probe so that it rests on the displacement standard. Click on **OK** to clear the dialog box.
15. After about 30 seconds, the Y signal reading is displayed in the Read Height dialog box.
16. When the Y signal stabilizes, select **OK** to accept the height value and clear the dialog box.
17. Click on **Save and Apply**.
18. Click on **Close**.
19. Press the **Probe Up** button.
20. Remove the displacement standard.

To perform a Force calibration:

1. Locate the 50-g calibration reference material and weight platform.
2. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
3. Select Force from the Restore menu to restore the default force calibration value to the analyzer.
4. Click on **Save and Apply**.
5. Select the Force tab from the Calibration window.
6. Enter your name in the Operator field.
7. Click on **Begin Calibration**.
8. Remove the dust cover, remove any sample from the sample platform, install the weight tray, and lower the probe. Click on **OK** to clear the dialog box.
9. Place the 50-g weight on the tray. Calibration is over in a short time.

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10. Remove the weight and the tray.
11. Replace the dust cover.
12. Click on **Save and Apply**.
13. Click on **Close**.

To perform an Eigendeformation calibration:

NOTE: You must perform a height calibration and a force calibration BEFORE an eigendeformation calibration.

1. Locate the steel eigendeformation calibration cylinder.
2. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
3. Select **Eigendeformation** from the Restore menu.
4. Click on **Save and Apply**.
5. Select the Eigendeformation tab in the Calibration window.
6. Enter your name in the Operator field.
7. Click on **Begin Calibration**.
8. Remove any sample from the platform.
9. Press **Probe Down** on the analyzer and wait a minute.
10. Press the **Probe Up** button.
11. Insert the steel reference material.
12. Press **Probe Down**; the calibration is completed after a short time.
13. Press **Probe Up**.
14. Remove the steel reference material.
15. Click on **Save and Apply**.
16. Click on **Close**.

To perform a Temperature Calibration:

1. Install the large bending platform and 3-mm sphere probe tip onto the analyzer. (Click here for instructions.)
2. While in Instrument Viewer or Method Editor, select **Calibrate** in the View menu.
3. Select **Temperature** in the Restore menu.
4. Click on **Save and Apply**.
5. Click on the **Close** button.
6. Perform a run using the indium reference material. (See the Hardware manual for the DMA 7e for instructions.)
7. Select **Onset** from the Calc menu. Enter the Left and Right Limits in the dialog box.
8. Click on the **Calculate** button. Record the Onset temperature.
9. Select **Calibrate** from the View menu.
10. Select the Temperature tab in the Calibration window.

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11. Enter your name in the Operator field, the name of the reference material used, the expected onset temperature, and calculated onset temperature.
12. Click on the check box in the Use column.
13. Select **Save and Apply**.
14. Select **Close**.

To perform a DMA 7e Furnace Calibration:

NOTE: You must do a temperature calibration BEFORE a furnace calibration. Prior to performing the Furnace calibration, verify that the sample temperature is at or near the program temperature and that it is at or below the intended minimum calibration temperature. The sample platform must be empty.

You must use a coolant that will maintain a constant block temperature below 45°C throughout the calibration.

1. While in the Instrument Viewer or Method Editor, select **Calibrate** in the View menu.
2. Select the Furnace tab in the Calibration window.
3. Press the furnace locking mechanism and raise the furnace until it locks in place.
4. Enter your name in the Operator field. Enter the minimum and maximum temperatures.
5. Click on **Begin Calibration**. Calibration begins automatically.
6. Click on **Save and Apply**.
7. Select **Close**.

Calibrate a TMA 7

To perform a Height Calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select **Height** from the Restore menu.
3. Click on **Save and Apply**.
4. Select the Height tab from the Calibration window.
5. Enter your name in the Operator field and the height of the displacement standard in the Ref. Height field.
6. Click on **Begin Calibration**.
7. Press the furnace locking mechanism and lower the furnace assembly to the base of the analyzer. Make sure that the furnace locks in place.
8. Press the **Probe Up** button on the analyzer.
9. Make sure that there are no samples on the sample platform.
10. Press the **Probe Down** button to lower the probe so that it rests on the empty sample platform. Click on **OK** to clear the dialog box. After about 30 seconds, the Y signal reading is displayed in the Read Zero dialog box.

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11. Once the Y signal stabilizes, select **OK** if you want the current value entered as the new zero value.
12. Press the **Probe Up** button.
13. Place the sapphire height displacement standard on the sample platform.
14. Press the **Probe Down** button and lower the probe so that it rests on the displacement standard. Click on **OK** to clear the dialog box. It will take 20 – 30 seconds for the Y signal to be displayed in the Read Value field in the Read Height dialog box.
15. When the Y signal stabilizes, select **OK** to accept the height value and clear the dialog box.
16. Click on **Save and Apply**.
17. Click on **Close**.
18. Press the **Probe Up** button.
19. Remove the displacement standard.

To perform a Force Calibration:

1. Locate the 50-g calibration reference material and weight platform.
2. Select **Calibrate** from the View menu.
3. Select **Force** from the Restore menu.
4. Click on **Save and Apply**.
5. Select the Force tab from the Calibration window.
6. Enter your name in the Operator field.
7. Click on **Begin Calibration**.
8. Remove the dust cover, remove any sample from the sample platform, install the weight tray, and lower the probe. Click on **OK** to clear the dialog box.
9. Place the 50-g weight on the tray. Calibration is over in a short time.
10. Remove the weight and the tray.
11. Replace the dust cover.
12. Click on **Save and Apply**.
13. Click on **Close**.

To perform a Eigendeformation calibration:

NOTE: You must perform a Height calibration and a Force calibration BEFORE an eigendeformation calibration.

1. Locate the steel eigendeformation calibration cylinder.
2. Select **Calibrate** from the View menu.
3. Select **Eigendeformation** from the Restore menu.
4. Click on **Save and Apply**.
5. Select the Eigendeformation tab in the Calibration window.
6. Enter your name in the Operator field.

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7. Click on **Begin Calibration**.
8. Remove any sample from the platform.
9. Press **Probe Down** on the analyzer and wait a minute.
10. Press the **Probe Up** button.
11. Insert the steel reference material.
12. Press **Probe Down**; the calibration is complete after a short time.
13. Press **Probe Up**.
14. Remove the steel reference material.
15. Click on **Save and Apply**.
16. Click on **Close**.

To perform a Temperature calibration:

1. Install the TMA 7 penetration probe and, if necessary, the standard furnace tube.
2. While in the Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
3. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the **All** command.
4. Click on **Save and Apply**.
5. Click on **Close**.
6. Perform a sample run using the indium reference material. (See the Hardware manual for the TMA 7 for instructions on how to run the reference material.)
7. Select **Onset** from the Calc menu.
8. Enter the Left and Right Limits in the dialog box.
9. Click on the **Calculate** button. Record the onset temperature value.
10. Select **Calibrate** from the View menu.
11. Select the Temperature tab in the Calibration window.
12. Enter your name in the Operator field, the name of the reference material used, the expected onset temperature, and calculated onset temperature.
13. Click on the check box in the Use column.
14. Select **Save and Apply**.
15. Select **Close**.

To perform a TMA 7 Furnace calibration:

NOTE: You must do a temperature calibration BEFORE a furnace calibration. Before the furnace calibration, verify that the sample temperature is at or near the program temperature and that it is at or below the intended minimum calibration temperature. The sample platform must be empty.

You must use a coolant that will maintain a constant block temperature below 45°C throughout the calibration.

1. While in the Instrument Viewer or Method Editor, select **Calibrate** in the View menu.
2. Select the Furnace tab in the Calibration window.
3. Press the furnace locking mechanism and raise the furnace until it locks in place.
4. Enter your name in the Operator field. Enter the minimum and maximum temperatures. They must encompass the temperature range in which you will run your samples.
5. Click on **Begin Calibration**. Calibration begins automatically.
6. Click on **Save and Apply**.
7. Select **Close**.

Calibrate a DTA 7

To perform a Temperature calibration:

1. While in the Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select **Temperature** from the Restore menu.
3. Click on **Save and Apply**.
4. Click on **Close**.
5. Perform a sample run using the aluminum standard under the conditions that you run your samples. (See the DTA 7 Hardware manual for instructions on how to run a standard.)
6. Perform a peak area calculation and include the onset temperature. Record the ΔH and Onset results.
7. Perform a sample run using the gold standard. (See the DTA 7 Hardware manual for instructions on how to run a standard.)
8. Perform a peak area calculation and include the onset temperature. Record ΔH and Onset results.
9. Select **Calibrate** from the View menu.
10. Select the Temperature tab. Enter the reference material, expected Onset values, and measured Onset results.
11. Click in the Use check box for each material.
12. Select **Save and Apply**.
13. Click on **Close**.

To perform a Heat Flow calibration:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select **Heat Flow** from the Restore menu.
3. Select **Save and Apply**.
4. Select **Close**.
5. Complete a scan of each of the two reference materials provided (aluminum and gold) or use the data from the runs performed for the Temperature calibration.
6. Perform a peak area calculation (with the X axis as time) and note the peak area in °C s.

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7. Select **Calibrate** from the View menu.
8. Select the Heat Flow tab.
9. Enter the reference materials if not already displayed, the expected ΔH, the measured peak area in °C s, the expected melting point, and the weight of the reference material.
10. Select **Save and Apply**.
11. Select **Close**.

To perform a Furnace calibration:

NOTE: Furnace calibration should be performed AFTER temperature calibration.

1. While in Instrument Viewer or Method Editor, select Calibrate from the View menu.
2. If necessary, complete a Temperature calibration.
3. Remove the cups from the sample and reference cup holders.
4. Select the Furnace tab in the Calibration window.
5. Enter minimum and maximum temperatures. They should encompass the temperatures at which you will run your samples.
6. Select **Begin Calibration**. Furnace calibration takes about 2.5 hours.
7. Select **Save and Apply**.
8. Click on **Close**.

Restore Calibration

To restore calibration defaults:

When you use the Restore option on the Calibration menu bar, you are loading in the default calibration file in order to have its values applied to the analyzer. You may have calibrated the analyzer under different conditions than those currently in use and may wish to revert back to those original conditions. Rather than recalibrate, you can use the already saved calibration file.

1. Select **Calibrate** from the View menu.
2. Select **Restore** from the menu bar to display the Restore menu.
3. Select the calibration type to restore or restore all of the default calibration values.
4. Select **Save and Apply** to send the default calibration values to the analyzer and save the calibration file.

Prepare for Data Collection

Before performing a run on your samples, you need to set up a method which is a set of parameters that are used by the analyzer while collecting data. A method is set up by loading it into the Method Editor and editing the parameters, if necessary. The parameters in the method depend on the conditions used, the type of sample, the type of information you are looking for, and so on. Once the method is ready, you click on the Start Method button on the control panel and the run begins.

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Create a New Method

You can create your own methods to run samples using the Method Editor. Create a new method in one of two ways:

One way –

Make sure the Method Editor is the focused window.

1. Click on the **New Method** button  on the toolbar or select the **New Method** command in the File menu. The Method Editor displays a "blank" Sample Info page with a default data file name (QSAVE or whatever was entered in the Preferences page) and directory displayed.
2. Enter the desired values in the fields on the three tabbed pages.
3. Select **Save Method** from the File menu and enter a file name for the new method.

Another way –

1. Select **Open Method** from the File menu while in Method Editor.
2. In the Open Method dialog box choose either the default method or an existing method that you know is close to what you need to run your sample.
3. Edit the fields in the three pages of the method as needed.
4. Select **Save Method As** from the File menu to save the new method and retain the default method or the existing method for future use.

Edit the Method Pages

You can create a new method by editing the default method or another existing method that you created and saving it under a new file name by using **Save Method As** in the File menu. To edit an existing method, already loaded by using **Open Method** in the File menu, you can do any of the following steps:

1. Change the sample information on the Sample Info page of the Method Editor to uniquely identify your sample run. Move from field to field by pressing the **Tab** key or place the cursor in the desired field by using the mouse and clicking.
2. If you want to store the data file in a directory other than the default data directory, click on the **Browse** button to select an existing directory in which your data file will be stored.
3. After filling in the Sample Info page, click on the Initial State tab.
4. Move about this page using the **Tab** key or the mouse, filling in the necessary fields.
5. For analyzers other than DMA or TMA, if you want to use baseline subtraction with your data, click the box next to Use Baseline Subtraction. An "X" will appear in the box, indicating that baseline subtraction will be done during data collection.

   Click on the **Browse** button and select a baseline file from the list of available files. If you do not want to use baseline subtraction for your sample run, click on the check box to remove the "X."
6. If you have a DMA, the Initial State page contains a **Set Controls** button. Click on it to display more entry fields that define the initial state of the force controls for the analyzer.
7. Select the purge gas for your sample run. The gases displayed are those set up in Preferences.

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8. Enter equilibration threshold values in the Equilibrate Within section. The method will not begin until at least one of equilibration thresholds is reached.
9. After filling in the Initial State page, click on the Program tab.
10. Fill in the Program page.

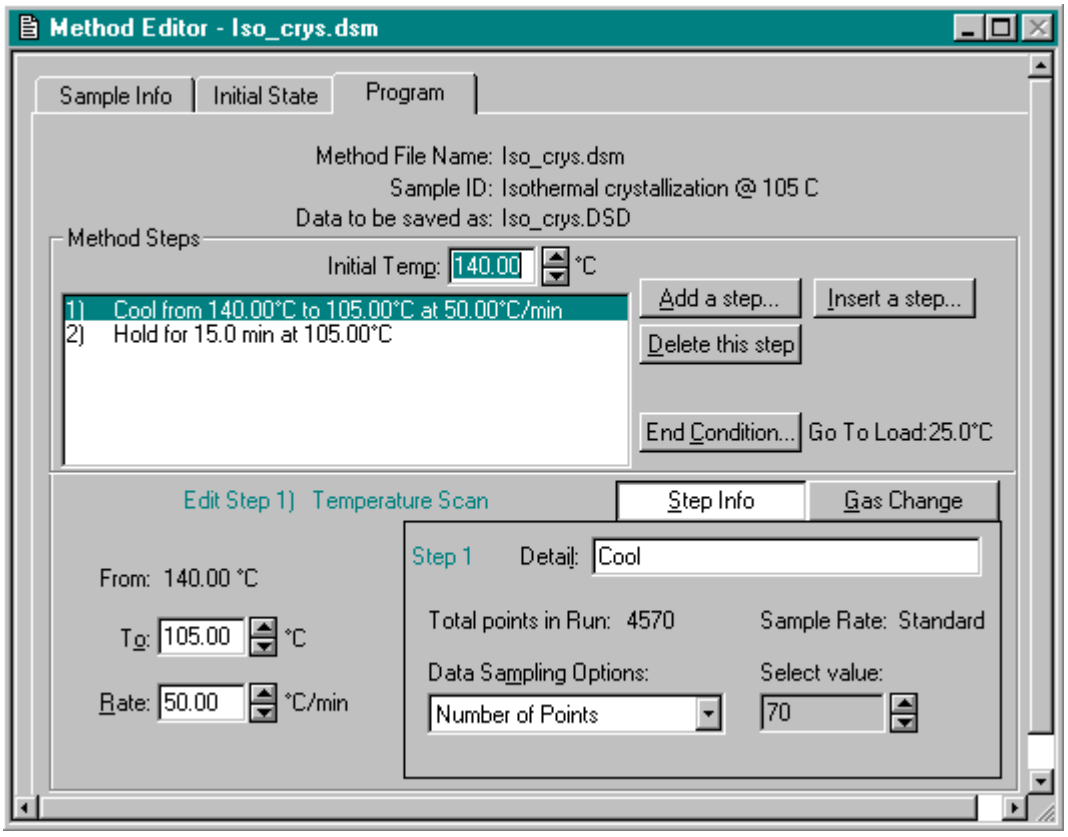
Edit the Program Page

To edit the Program page you can do any of the following:

Change the starting temperature:

The initial temperature displayed on the Program page is taken from the Initial State page. This can be changed by entering a new temperature in the Initial Temp field. If you go back to the Initial State page, you will see the new starting temperature displayed.

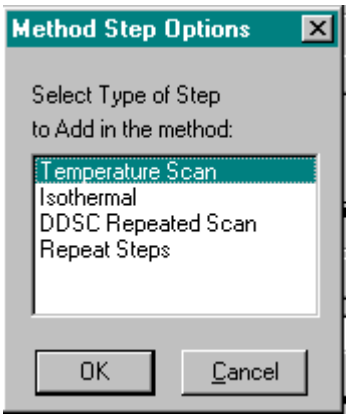
Method Program Page



Insert or add a new method step:

You can insert or add steps to your method program. Inserting a step places it immediately before the currently highlighted step in the Method Steps section. Adding a step places it at the end of the program. In some cases, the step you select will replace the entire program. When you select either **Add a step** or **Insert a step**, the Method Steps Options dialog box listing the types of steps available is displayed.

Method Step Option Dialog Box



Delete a method step:

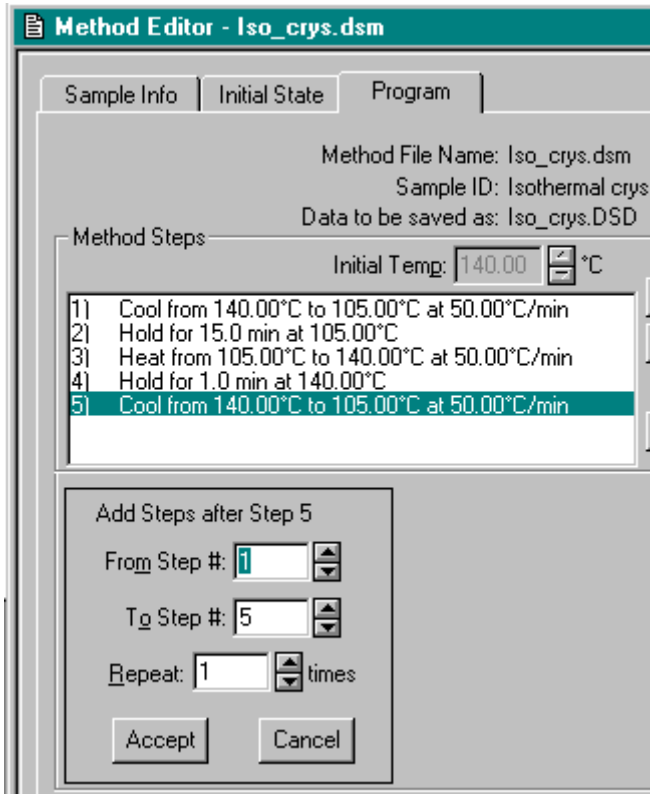
On the Program page, highlight the method step that you want to delete. Click on the **Delete this step** button. If the resulting temperature program is valid, the step is deleted and the parameters of the remaining steps are updated automatically.

Repeat steps:

If your method program contains Temperature Scan and Isothermal Scan steps, you can use the Repeat Steps feature. You must have at least two steps in the program already. Click on the **Add a step** or **Insert a step** button to display the Method Steps Options dialog box. Select Repeat Steps and click on **OK**. In the Repeat Steps area enter the numbers of the first and last steps you want to repeat and the number of times you want to repeat them. Click on **Accept**. If the resulting program is not valid, a message explaining why is displayed.

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Repeat Steps Area



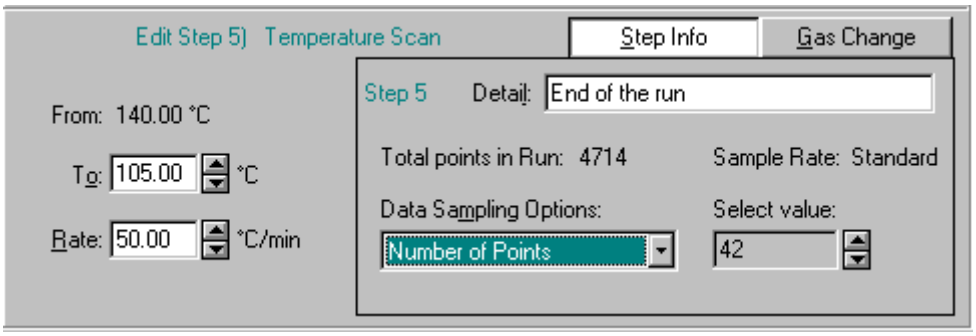
Edit a method step:

Edit the temperature and time parameters of a step by highlighting the step, then editing the values in the Edit Step section. To edit the temperature – time parameters of a method step, you must select the step in the Method Steps section of the Program page. You do this by clicking the mouse pointer on the step. The selected step will appear "highlighted" (in inverse video). To select a different method step, use the mouse or the up and down cursor keys. You can highlight only one method step at a time.

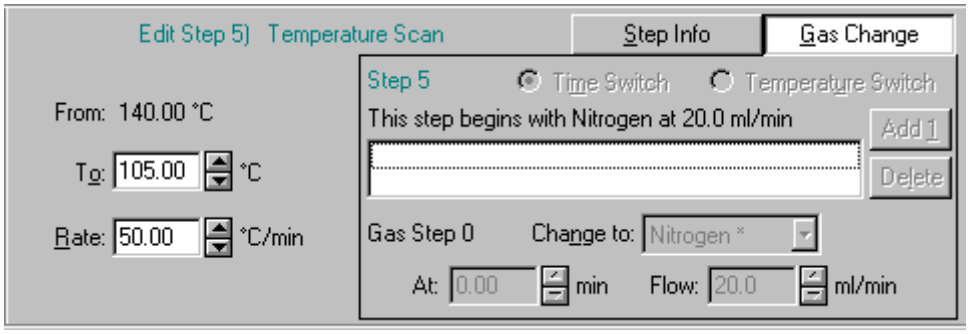
Change step details:

Highlight the step you want to change. The Step Info section should appear; if not, click on the Step Info button. Enter any text you want into the Detail field. Depending on the analyzer, you can select sampling options from the Data Sampling Options box. Select the number of points or seconds between points to be collected in the highlighted step. Click on the **Gas Change** button. You can change the gas program if you are using a TAGS or GSA 7.

Step Details Area



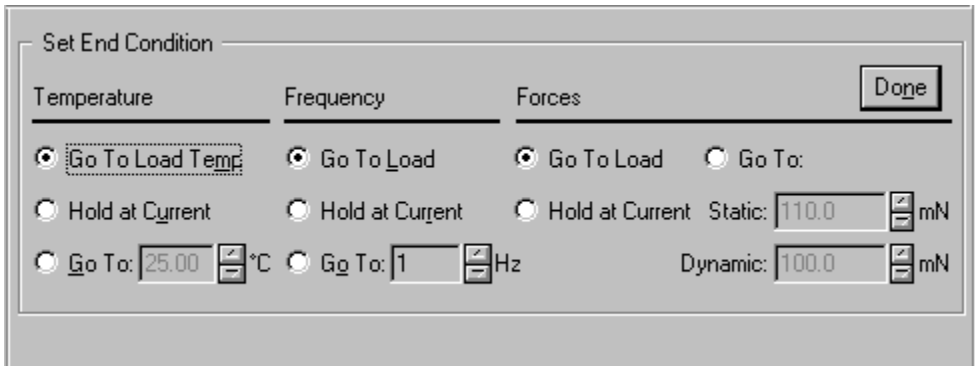
Gas Change Area



If the step is a Temperature Scan, click on the **Temperature Switch** radio button to have the purge gas switched at a specified temperature during the step, or click on the **Time Switch** button to have the purge gas switched at a specified time during the step. You can also enter a different flow rate.

Change the end condition:

Click on the **End Condition** button to display the Set End Condition section. You can tell the analyzer to go to the load temperature, hold at the current temperature, or go to a user-defined temperature at the end of the run. For a DMA or a TMA, you can also set end conditions for force and frequency.



Select an Existing Method

Instead of creating a new method, you select an existing method:

1. Select **Open Method** from the File menu or click on the **Open Method** button  on the toolbar.
2. Choose the desired method file from the dialog box.
3. Change the fields on the three pages as needed.
4. Click on the **Start Method** button in the control panel to start the run.

   You may want to save the method under the new file name by using Save Method As in the File menu.

Run a Sample Using Baseline Subtraction

1. Select the Initial State page of the Method Editor.
2. Select the Use Baseline Subtraction check box.
3. Enter a file name to be used as the baseline or select **Browse** to choose a file.

Performing Data Collection

Once you have set up the method to use for your run, you can begin data collection. Some hardware manuals contain detailed instructions on how to perform runs for calibration. Those instructions give you a good idea of what a routine run is like for that instrument. Other topics to see for information on data collection are the sample preparation and sample loading in the hardware manuals.

View Your Data

Look at Collected Data During a Run

While in the Instrument Application for the Pyris analyzer currently running, click on the **Instrument Viewer** button  on the toolbar to display the Instrument Viewer. The real-time data are displayed in the Instrument Viewer. This display can be changed by using the items in the Display menu.

Open a Data Analysis Window

Saved data files are displayed as curves in Data Analysis windows. Open a Data Analysis window in an Instrument Application or in a Data Analysis Application. You can have more than one Data Analysis window open at one time.

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Open a Data Analysis window one of three ways:

1. If you are in an Instrument Application, select the **Data Analysis** button on the toolbar. The Open Data File dialog box appears.
2. Select Data Analysis from the Pyris Software for Windows menu. This is displayed by selecting Programs on the Windows **Start** button menu and then selecting Pyris Software for Windows. The Data Analysis window is displayed automatically.
3. Select Data Analysis from the **Start Pyris** button on the Pyris Manager. The Data Analysis window is displayed automatically.

Display a Curve

To display a curve, you can do any of the following:

1. If you are in Data Analysis in an Instrument Application or in the Data Analysis Application, select the **Open Data** command in the File menu. The Open Data File dialog box appears. Select a data file. The selected curve is displayed in a new Data Analysis window. The other open Data Analysis window remains open.
2. If you are in Instrument Application and do not already have a Data Analysis window open, select the **Data Analysis** button on the toolbar. The Open Data File dialog box appears. Select a data file. The selected curve will be displayed in a new Data Analysis window.
3. If you are in Data Analysis in an Instrument Application or in the Data Analysis Application, select **New Data** from the File menu. Select a data file. The selected curve will replace the currently displayed curve.
4. If you are in Data Analysis in an Instrument Application or in the Data Analysis Application, select **Add Data** from the File menu. Select a data file. The selected curve will be added to the Data Analysis window and it becomes the active curve.
5. Select Data Analysis from the Pyris Software for Window menu or from the **Start Pyris** button's task menu. The Data Analysis Application opens automatically. Use **Open Data** or **New Data** from the File menu to display a curve.

Open a Data File

To open a data file in a Data Analysis Application:

1. Select the **Open Data** command in the File menu.
2. In the Open Data File dialog box, select the data file you want to open.

To open a data file in an Instrument Application:

1. Select the **Data Analysis** button in the toolbar.
2. In the Open Data File dialog box, select the data file you want to open.

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Add a Data File to the Window

To add a data file to a Data Analysis window:

1. Select the Data Analysis window to which you want to add a data file.
2. Select the **Add Data** command in the File menu.
3. In the Add Data File dialog box, select the data file you want to add. The curve is added to the Data Analysis window and becomes the focused curve.

Select Steps to Display

To select which method steps' curves to display in a Data Analysis window:

1. Select **Step Select** in the Curves menu. It is activated when a checkmark is displayed next to the item in the Curves menu.
2. Select a curve type from the Curves menu to be added to the display.
3. In the Step Select dialog box, highlight the steps that are to be displayed.
4. Select the **OK** button.

Change the Active Curve

To change the focused curve, do one of the following:

1. Click in the Data Analysis window on the curve you want to make the focused curve.
2. Click in the Legend window on the curve you want to make active.

Display Results

To display results from performing a calculation function on the Calc menu:

1. Display the desired data file in the Data Analysis window. Make sure it is the focused curve.
2. Select **Results** from the View menu. Any calculations that had been performed on the data are listed.
3. In the Results dialog box, highlight the calculation results to display.
4. Select the **View Results** button. The results are displayed in the Data Analysis window.

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Optimize Your Data

Rescale the X or Y Axis

1. Select the **Rescale X** or **Rescale Y** command in the Display menu or the **Rescale X** button  or the **Rescale Y** button  on the Rescale Tools toolbar. The Rescale X or Rescale Y dialog box appears.
2. Type the minimum value of the new X-axis scale or Y-axis scale in the Minimum entry field or use the spin buttons to increase or decrease the displayed value. The Increment field can be changed to adjust the increment used by the spin buttons.
3. Type the maximum value of the new X-axis scale or Y-axis scale or use the spin buttons to increase or decrease the displayed value. The Increment field can be changed to adjust the increment used by the spin buttons.
4. For the new X scale select the units you want in the Set Axis Units drop-down list box.
5. Select the type of scale you want (linear or logarithmic) by clicking on the appropriate radio button.
6. Select the **Rescale** button to close the dialog box and display the active curve using the new X-axis scale. Select the **Cancel** button to close the dialog box without changing the X-axis or Y-axis scale.

Display the X Axis, Y Axis, or Both Axes at Full Scale

1. Select the **Full X Scale** button  or the **Full Y Scale** button  on the Rescale Tools toolbar. The active Data Analysis window or the Instrument Viewer will be redrawn with the X axis or the Y axis displayed at full scale. In the Data Analysis window the full contents of the data file with respect to the X axis or the Y axis is displayed. In Instrument Viewer, the X axis or Y axis reverts to the default settings.
2. Select the **Full Scale** button  on the Rescale Tools toolbar. The active Data Analysis window or the Instrument Viewer will be redrawn with both axes displayed at full scale.

Use the Radar Window to Rescale a Curve

The Radar window is a movable, sizable window that shows all curves using the active Y axis at full scale. The current scale is shown as a boxed area in the Radar window.

To rescale the focused curve using the Radar window:

1. Select the **Radar** command in the View menu or the **Radar Window** button  on the Rescale Tools toolbar to display the Radar window.
2. Click and drag the mouse inside the Radar window to draw a box that defines the new scale.
3. Release the mouse button to redraw both axes using the new scale.

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Shift a Curve

Certain curves can be shifted or offset along the Y axis. If the **Shift Curve** button on the Rescale Tools toolbar is gray, the feature is not available for the active curve's type.

To shift a curve along the Y axis:

1. Select the **Shift Curve** button  on the Rescale Tools toolbar. The Shift Curve dialog box appears and a movable crosshair appears on the curve.
2. The Shift From entry fields display the X and Y coordinates of the starting point. The Shift To entry field displays the Y coordinate, which is the ending point for the shift.
3. Using the mouse, drag the crosshair to define the Shift From and Shift To coordinates. Drag it up or down to the Y value and then horizontally to the X axis position. There are two **X**'s: the one on the curve is the From point and that above or below the curve is the To point.
4. If you prefer, type the starting and ending points in the entry fields in the dialog box.
5. Select the Align All check box to shift all curves using the same Y axis as the active curve to the Shift To point.
6. Select the **OK** button to close the dialog box and shift the curve. Select the **Cancel** button to close the dialog box without shifting the curve.

Change the Slope of a Curve

Certain curves can have their slopes changed. If the **Change Slope** button on the Rescale Tools toolbar is gray, the feature is not available for the active curve's type.

To change the slope of the focused curve:

1. Select the **Slope** button  on the Rescale Tools toolbar. The Change Slope dialog box appears and a red line representing the current slope appears on the curve.

   The left end of the line is the pivot point and is identified by a small box. The right end of the line is the slope point and is identified by an **X**. The X and Y coordinates of the two points are displayed in the dialog box.
2. Type the desired X or Y coordinate for the pivot point, if you wish to change it. Use the **Tab** key to move the text cursor between entry fields.
3. Use the mouse to drag the slope point to the desired location. When you release the mouse, the values in the dialog box are updated automatically. Also, a second **X** appears at the point on the curve that corresponds to the new slope point.

   Alternatively, use the keyboard to type the X or Y coordinate in the Slope Point entry field.
4. Select the Align Endpoints check box to align the slope point and the pivot point on the same Y-axis point.
5. Select the **OK** button to close the dialog box and redraw the curve with the new slope. Select the **Cancel** button to close the dialog box without changing the slope of the curve.

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Perform a DMA Analysis

This is a step-by-step outline for performing a DMA analysis:

1. Check purge gas, cooling device, and coolant.
2. Select and install a measuring system.
3. Tare the probe.
4. Zero the probe.
5. Select the type of test method and enter parameters in Method Editor.
6. Measure sample dimensions and mount the sample.
7. Start the run and calculate and plot results.

Check Purge Gas, Cooling Device, and Coolant

1. Use helium for the purge gas. Helium has the best thermal conductivity characteristics but any inert gas can be used. Use the same purge gas for calibration and analysis.
2. Purge gas flow rate should be held constant at 30 mL/min from run to run and should be checked regularly.
3. Select the purge gas restrictor to use: An “A” restrictor (P/N 0154-1496) allows a flow rate of 10 mL/min and an “H” restrictor allows a rate of 1 mL/min.
4. Select a cooling device to use.
5. The temperature range you want to work in also affects the choice of furnace to use in the DMA 7e.

Cooling Choices

Cooling Device	Minimum Temperature (°C)	Maximum Temperature (°C)
Liquid nitrogen dewar	–170	200
Intracooler 2	–65	500 – 1000
Intracooler 1	–30	500 – 1000
Circulating ethylene glycol	–10	500 – 1000
Circulating water	40	500 – 1000
Ice water	25	200 – 1000

Furnaces

Two furnaces are available for the DMA 7e and the TMA 7. The large furnace is standard equipment for the DMA 7e and the small furnace is standard on the TMA 7. If high temperatures or high heating rates are required, the small furnace on the DMA 7e can be used. Measure the inside diameter to determine which furnace you are using.

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	Large Furnace (DMA)	Small Furnace (TMA)
Inside diameter (mm)	28	15
Maximum temperature (°C)	500	1000
Minimum temperature (°C)	–170	–170
Maximum heating rate (°C/min)	40	100
Minimum heating rate (°C/min)	0.1	0.1

Select and Install a Measuring System

Measuring systems hold the sample in place for analysis. There are many measuring systems available to accommodate different sample moduli, shapes, and sizes. Select the measuring system to use based on observed sample behavior.

Samples and Measuring Systems

Observed Physical Behavior	Modulus Range	Measuring System Description	Measuring System Kit Part No.
hard	10^5 – 10^{11} Pa	3-point bending 18 mm quartz	N539-0136
rigid	10^5 – 10^{11} Pa	3-point bending 15 mm	N539-0101
semirigid	10^5 – 10^{10} Pa	3 point bending 10 mm	N539-0137
film or fiber	10^3 – 10^{12} Pa	extension, stainless	N539-0132
fiber or film	10^3 – 10^{12} Pa	extension, quartz	N539-0134
semirigid	10^3 – 10^8 Pa	dual cantilever high modulus	N539-0131
pliable	10^3 – 10^7 Pa	dual cantilever low modulus	N539-0131
semirigid	10^3 – 10^6 Pa	parallel plate 10 mm	N539-0135
soft	10^3 – 10^4 Pa	parallel plate 15 mm	N539-0133
very soft	below 10^4 Pa	parallel plate 20 mm	N539-0133

Tare the Probe

To tare the probe means to find the weight (mass) of the probe when zero force is applied. The mass of the core rod is also known as the tare. The tare weight is measured when the probe makes contact with a firm surface after one second of free travel and when the measuring system you are using is selected in the software. The tare weight of the electromagnetically suspended core rod is used to calculate mass and initial effects necessary to apply forces to the sample accurately. You must tare the weight when you change the measuring system and before mounting a sample.

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To tare the probe:

1. Click on the **Tare Probe** button on the control panel. The Tare Probe dialog box is displayed.
2. If there is a sample on the sample platform, remove it.
3. Insert the zero block or some suitable object that will cause the probe to rest at the height of a mounted sample.
4. Raise the probe to the top by pressing the **Probe Up** button on the analyzer.
5. Press the **Probe Down** button to lower the probe until it touches the zero block. Observe the probe as it travels down. If it falls, hesitates, or vibrates, the tare is not valid.

   Observe the tare weight displayed in the dialog box as Current Reading.
6. Repeat this procedure until at least three tare values are within 0.1 g.

NOTE: The default tare for the DMA 7 is 60.00 g and for the DMA 7e 80.00 g. If the tare value is slightly above the default value, it means that the force has been calibrated. The typical tare value for the 10-mm knife, 15-mm 3-point bending is about 83 g for the DMA 7 and about 120 g for the DMA 7e.

Zero the Probe

To zero the probe means to set the LVDT to zero, i.e., read the height of the probe with no sample in place.

To zero the probe:

1. Set the current static force to 100 mN in the Initial State page.
2. Make sure there is no sample in the analyzer.
3. Press the **Probe Down** button on the analyzer.
4. Click on the **Read Zero** button on the Control Panel. The Zero Height value is displayed in the Zero field in the Sample Info page of the Method Editor.

The analyzer is now zeroed for height. The sample zero reflects the difference between calibration zero and the current probe position. When a sample is mounted in the analyzer, the reported height should be close to the sample height.

Select Test Method and Enter Parameters in Method Editor

1. Open an existing method or the default method and edit the parameters as necessary.
2. In the Method Steps section of the Program page, select the type of scan you wish to perform. See example methods for some of the test methods available for a DMA 7e:
   - **Temperature Scan**
   - **Frequency Scan**
   - **Isothermal Scan**
   - **Dynamic Stress**
   - **Static Stress**
   - **Creep-Recovery**

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DMA Temperature Scan Method

Use this method to identify the glass transition of an epoxy–glass sample. The sample temperature is varied with time. You can heat, cool, or hold the temperature. Static force, dynamic force, and frequency are held constant. The sample position, amplitude, and phase lag are measured versus temperature.

Measuring System/Geometry:	3-Point Bending/rectangle
Sample Height:	0.1 – 3 mm (up-down)
Sample Width:	15 mm (platform)
Sample Depth:	1 – 10 mm (front-back)
Temperature:	50°C
Static Force:	550 mN
Dynamic Force:	500 mN
Frequency:	1 Hz
Method Step:	Temperature Scan
Edit Step To:	150°C
Edit Step Rate:	5.00°C/min

In Data Analysis, calculate the onset of the storage modulus decrease. Report the log scale storage modulus and linear scale tangent delta versus linear scale (sensor) temperature. If the transition is not clear enough, the probe position may be substituted for storage modulus curve.

TMA Temperature Scan Method

Use this method to identify the glass transition of a laminated thermoset. The sample temperature is varied versus time. You can heat, cool, or hold the temperature. Static force is held constant. The change in sample position is measured.

Measuring System/Geometry:	Expansion/Disc
Height:	0.1 – 3 mm
Diameter:	3 mm
Temperature:	50°C
Static Force:	50 mN
Method Step:	Temperature Scan
Edit Steps To:	150°C
Edit Steps Rate:	10°C/min

In Data Analysis, calculate the onset increase of the probe position. This gives the glass transition.

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Frequency Scan Method

A frequency scan is for viscoelastic samples. The frequency of the dynamic force applied to the sample is varied automatically. The static force, dynamic force, and temperature are held constant. The sample position and displacement amplitude are measured.

To characterize a pelletized thermoplastic polymer sample, use the parameters below.

Measuring System/Geometry:	Parallel plate/disk
Sample Height:	0.5 – 3 mm (up-down)
Sample Diameter:	15 mm
Temperature:	290°C
Static Force:	0 mN
Dynamic Force:	50 mN
Frequency:	10 Hz
Method Step:	Frequency Scan
Edit Step To:	1 Hz

In Data Analysis, find the point where the storage modulus and complex viscosity cross each other. This point is very sensitive to changes in molecular weight and molecular weight distribution for thermoplastics at processing temperatures.

Isothermal Scan Method

Use this method to characterize the isothermal cure of a liquid epoxy. The sample temperature, static force, dynamic force, and frequency are held constant. The sample position and displacement amplitude are measured versus time.

Measuring System/Geometry:	Parallel Plate/Disk
Sample Height:	0.1 – 3 mm (up-down)
Sample Diameter:	10 mm
Temperature:	23°C
Static Force:	0 mN
Dynamic Force:	50 mN
Frequency:	1 Hz
Method Step:	Isothermal Scan
Edit Step For:	10 min

In Data Analysis, calculate the peak of the tangent delta curve to give the gel point. Using the Curves menu, display the tangent delta and storage modulus versus time.

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Dynamic Force/Stress Scan Method

Use this method to characterize a single filament (fiber. The amplitude of the dynamic force (sinusoidal oscillation) is varied using a dynamic stress scan. The static force can be programmed to change proportionally to the increasing dynamic force to assure that the sample remains in tension throughout analysis. The frequency and temperature are held constant. The sample position and displacement amplitude are measured.

Measuring System/Geometry:	Extension/Fiber
Sample Length:	5 – 10 mm (up-down)
Sample Diameter:	0.01 – 2 mm (plates)
Temperature:	23°C
Static Force:	11 mN
Dynamic Force:	10 mN (initial)
Static Force Control:	Tension, 110%
Method Step:	Dynamic Stress Scan
Edit Step To:	1100 mN
Edit Step Rate:	250 mN/min

In Data Analysis, calculate the slope of the stress vs. strain curve (in the viscoelastic region); this gives the modulus.

Static Force/Stress Scan Method

Use this method to characterize a thin film. The program static force (stress) is varied using a static force scan. There is no dynamic force or frequency. The temperature is held constant. The sample position (strain) is measured.

Measuring System/Geometry:	Extension/Film
Sample Length:	5 – 10 mm (up-down)
Sample Width:	0.5 – 3 mm (left-right)
Sample Depth:	0.01 – 2 mm (front-back)
Temperature:	23°C
Static Force:	1100 mN
Dynamic Force:	Click on Set Controls and turn off Dynamic Force
Method Step:	Static Force (Stress) Scan
Edit Step To:	1100 mN (recovery force)
Edit Step Rate:	250 mN/min

Display the stress versus strain on linear scales. The strain is calculated from the change in sample length (height) compared with the original sample height, relative to sample zero.

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Creep/Recovery Scan Method

Use this method to characterize the flow rate of an elastic disk. The equilibrium mechanical behavior of thermoplastics, thermosets, and elastomers is characterized for a specific stress at a specific temperature. The sample is taken from an equilibrium state with respect to static stress and temperature to a new static stress. The time-dependent displacement response is measured. There is no dynamic force or frequency and the temperature is held constant. The sample position is measured.

Measuring System/Geometry:	Parallel Plate/Disk
Sample Height:	0.5 – 3 mm (up-down)
Sample Diameter:	5 mm (plates)
Temperature:	23°C
Static Force:	1100 mN
Dynamic Force:	Click on Set Controls and turn off Dynamic Force
Method Step:	Creep Recovery
Edit Step:	
Equilibrate Time:	0.0 min
Creep Force:	1100 mN
Creep Time:	5 min
Recovery Force:	100 mN
Recovery Time:	20 min

The sample strain is reported versus time. The strain is calculated from the change in sample length (height) compared with the original sample height, relative to sample zero.

Measure Sample Dimensions and Mount the Sample

1. It is important to measure the sample correctly to get accurate data from your DMA experiment. Depending on the measuring system and the sample geometry chosen, you will measure the height, width, depth, length, and/or thickness of the sample. You may also need to measure the diameter of the probe plate if using the parallel plate measuring system. In some cases, the sample height can be measured automatically by the analyzer. See the measuring instructions in the specific measuring system's Instruction Manual for which dimensions to measure.
2. Enter the dimensions into the Sample Info page of the Method Editor.
3. Mount the sample according to the measuring system's Instruction Manual.
4. If you are using a 3-point bending, parallel plate, or extension measuring system and you decided to let the analyzer measure the sample height or diameter, then perform the steps necessary to take that measurement after mounting the sample according to the measuring system's Instruction Manual.

Start the Run and Calculate and Plot Results

1. Carefully raise the furnace assembly all the way up, making sure that it locks in place.
2. Place the diffusion cap around the sample tube at the top of the furnace.

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3. Click on the **Start Method** button in the Control Panel.
4. Monitor the progress of the run and the status of various parameters using the Instrument Viewer and the status panel.
5. When the run is complete, use the Data Analysis window to display results and perform calculations using the options on the Calc menu. These calculations can be saved with the data file. Different curves can be displayed via the Curves menu. Use View Results to display saved calculations.

Perform a Purity Analysis

Purity analysis of DSC data comprises the following steps:

1. Prepare samples and data
2. Performing the Purity calculation
3. Reading the results

Prepare Samples and Data

The preferred method of encapsulating DSC samples involves hermetically sealing the sample between two volatile sample pans or placing a thin disk of aluminum on top of the sample before sealing the pan. Items used for encapsulating DSC samples for Purity analysis include a Volatile Sample Sealer (P/N 0219-0061) and Volatile Sample Pans and Covers (P/N 0219-0062). The data for a Purity analysis must be from the melting of a fairly pure organic or inorganic material but not a polymer.

1. To prepare your data for Purity analysis, perform a sample run with your DSC 7, Pyris 6 DSC, or Pyris 1 DSC.
2. For best results, use a slow scanning rate (e.g., 1 °C/min or less) and use an average sample size (e.g., 1 – 3 mg). This should produce a peak height of approximately 2 – 10 mW.
3. Make sure that the X axis is **Temperature** not Time. Use Rescale X to change if necessary.
4. Click on the **Data Analysis** button  on the toolbar to open the Data Analysis application for that analyzer on which the data were collected.
5. In the Open File dialog box select the data file from the run you just performed.

Performing the Purity Calculation

With the data file from your Purity analysis run displayed in the Data Analysis window:

1. Select Purity from the Calc menu. The Calculate Peak Area dialog box is displayed and two X's appear on the curve.
2. Select left and right peak limits by either entering the values directly into the fields in the dialog box or by clicking on and dragging the X to the desired position on the curve.
3. Click on Next. The Select Calculation Method dialog box is seen next.

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4. Select the Calculation Method: Standard or MLR.
5. Enter the thermal resistance constant R0 of the sample.
6. Enter the molecular weight of the sample.
7. Enter the heat capacity Cp of the sample pan. The heat capacity of the sample pan depends on the type of pan used. This value is used to perform a minor correction in the equation. Therefore, use the default value of 0.023 J/°C for most applications. However, if you are using high-pressure capsules, you may wish to calculate the heat capacity of your sample pan material.
8. Click on the Next button. If you selected the Standard method, the next screen is Adjust % Area Limits. If you selected the MLR method, you see Adjust Baseline.
9. If you must adjust the baseline for an MLR method, enter the limits into the fields or move the two X's on the curve. Click on the Next button.
10. To adjust the % Area Limits, enter the limits directly into the fields in the dialog box or move the vertical red bars on the curve.
11. Click on the Next button. The View Curves dialog box is next.
12. To show how the data fits the Van't Hoff relationship, select T vs 1/F by clicking in the check box.
13. Click on the Finish button.

The results of the calculation are displayed on the screen.

Reading the Results

The results of a Purity analysis are as follows:

Limit 1 – lowest temperature used for the purity calculation

Limit 2 – highest temperature used for the purity calculation

Delta Hf – heat of fusion (kJ/mole)

Tm – melting point of the displayed data

T0 – melting point of theoretically pure sample

X correction – correction for melting below Limit 1

Purity – percent of pure material in the displayed data

The inputs are also displayed with the results:

Method – standard or MLR

X1 – left baseline limit

X2 – right baseline limit

R0 – thermal resistance constant of the sample pan

Molecular Weight – molecular weight of the sample material

Cp (pan) – heat capacity of the sample pan

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In the T vs 1/F curve, the corrected data points are shown as red circles and the line drawn through them shows how the data fit the Van't Hoff relationship. The blue boxes represent the uncorrected values calculated for 1/F (fraction melted) at given temperatures.

Determine Lag or Rate Compensation

In thermal analysis, thermal lags affect the accuracy of the temperature of the scanning data. As a sample is heated or cooled, the heat flowing into it produces temperature gradients in the sample and the instrument. Therefore, it is recommended that for the highest temperature accuracy, the analyzer must be calibrated at the conditions to be used for the experiment, including the scan rate.

To determine the lag or scan rate compensation for a DSC 7 or a Pyris 1 DSC:

1. Restore all calibration values to their default values by selecting **Calibrate** from the View menu and then **Restore All** from the Restore menu.
2. Set Lag Compensation to its default value of 0 by selecting the DSC 7 or Pyris page in Preferences (on the Tools menu) and entering 0 in the Lag Compensation field. Record the current lag compensation value so you can return your system to its current state if necessary.
3. Click on **OK** to save your entry and exit Preferences.
4. Prepare an indium sample of weight 5.0 + 0.5 mg and place in a sample pan that you plan to use for routine operation of your analyzer.
5. Perform heating scans on the indium sample using the following parameters:

   - Purge gas = gas you use for routine operation
   - Start temperature = 120°C
   - End temperature = 170°C
   - Scan rates = 5°C/min and 20°C/min
   - Load temperature = 50°C

   Be sure to cool to the Load temperature after the first indium scan and hold the temperature there for at least 5 minutes prior to starting the second indium scan.
6. Perform a Peak Area calculation on each indium melting scan.
7. Perform the following calculation to determine the lag compensation value for your analyzer:

   $$\text{Lag Comp.} = (T1 - T2) / (R1 - R2)$$

   where T1 is peak onset temperature for the 20°C/min scan
   T2 is peak onset temperature for the 5°C/min scan
   R1 is 20°C/min
   R2 is 5°C/min
8. Multiply the lag compensation value just calculated by 1000 and enter that value in the DSC 7 or Pyris page in Preferences.
9. Click on **OK** to save the Lag Compensation value and exit Preferences.

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10. Recalibrate the analyzer using the calibration procedures described in Calibration of the DSC.

After determining the lag compensation, this value will be applied automatically to all future experiments until you change the value in Preferences.

Display Curve Data in Third-Party Software

To display the data of a specific curve in Microsoft Excel:

1. Display or focus (highlight by clicking on) the curve of interest in the Data Analysis application.
2. From the Edit menu, select **Copy**.
3. Switch to the Excel application.

Notice the heavy black outline around the first cell in the upper-left-hand corner of the spreadsheet. Leave the outline on the box.

4. Select **Paste** from the Edit menu.
5. From the Format menu, select **Column** and then **Autofit Selection.**
6. Unhighlight the data and scroll down the spreadsheet to review the data, deleting any unwanted rows.

To display a curve's data in Word for Windows:

1. Display or focus the curve of interest in the Data Analysis application.
2. From the Edit menu select **Copy**.
3. Switch to Word for Windows.
4. Select **Paste** from the Edit menu.
5. Highlight the two columns of numbers.
6. Select **Convert Text to Table** from the Table menu.
7. In the dialog box, choose **Tabs** for Separate Text At and **2** for the Number of Columns; click on **OK**.
8. With the columns still highlighted, go to Cell Height and Width in the Table menu and adjust the width of the columns as necessary.
9. Place the cursor outside of the table and click to unhighlight the data. Then scroll down the table to review, deleting any unwanted rows.

Display Entire Data File in Third Party Software

To display all data of a data file in Microsoft Excel:

1. Open the data file in the Data Analysis application.
2. Click on the **Method Used** button in the toolbar.
3. Click on the **Create** button. The Create Data File dialog box is displayed.

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4. Select Include Data Points.
5. Record the path to which the .TXT file is to be saved and click on **OK**.
6. Click on the **OK** button to return to the Data Analysis window.
7. Switch to the Excel application.

Notice the heavy black outline around the first cell in the upper-left-hand corner of the spreadsheet. Leave the outline on the box.

8. Select **Open** from the File menu.
9. In the Open dialog box, display the drop-down menu under List Files of Type and select Text File.
10. Follow the path recorded in step 5 and find the file that was created in Pyris; highlight it and click on **OK**.
11. The Text Import Wizard box is displayed next. The responses to be entered to this wizard and subsequent screens are as follows (these are the default entries):

Delimited | Next | Tab | Next | General | Finish

12. Click on the blank button that is next to the "A" column header and above the numbered row column. This will highlight the entire document.
13. From the Format menu, select Column and AutoFit Selection.
14. Unhighlight the table and scroll down to review the data, deleting any unwanted rows.

To display all of a data file's data in Word for Windows:

1. Open the data file in the Data Analysis application.
2. Click on the **Method Used** button in the toolbar.
3. Click on the **Create** button. A dialog box is displayed.
4. Select Include Data Points.
5. Record the path to which the .TXT file is to be saved and click on **OK**.
6. Click on the **OK** button in the Options page to return to the Data Analysis window.
7. Switch to Word for Windows.
8. Select **Open** from the File menu.
9. In the Open dialog box, display the drop-down menu under List Files of Type and select Text File.
10. Follow the path recorded in step 5 and find the file that was created in Pyris; highlight it and click on **OK**.
11. Highlight the two columns of numbers.
12. In the Table menu select Convert Text to Table.
13. In the dialog box, choose **Tabs** for Separate Text At and **2** for the Number of Columns; click on **OK**.
14. With the columns still highlighted, go to Cell Height and Width in the Table menu and adjust the width of the columns as necessary.
15. Place the cursor outside of the table and click to unhighlight the data. Then scroll down the table to review the data, deleting any unwanted rows.

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Create a Play List

We give four examples of creating play lists: two for nonautosampler analyzers which contain commands for analyzing a data file, and two for analyzers with an autosampler. These are fairly simple play lists but they will give you an idea of the power and flexibility of play lists.

Create a Pyris 6 TGA Play List

1. Open the Pyris 6 TGA Instrument Application by selecting the Pyris 6 TGA button the Pyris Manager.
2. While in Method Editor, Instrument Viewer, or Data Analysis, click on the Pyris Player button  on the toolbar. The Pyris Player Setup page is displayed. Pyris 6 TGA should be displayed in the Analyzer Type field.
3. Click on the Edit Play List tab to display the Edit Play List page. If there had been a play list displayed in the previous session in the Pyris Player, it will be displayed automatically. If the Edit page was left blank in the preceding session, it will be displayed empty.
4. If an existing play list is displayed, select New Player from the File menu to clear the Player Steps box.
5. Click on the **Add a step** button.
6. From the Player Step Options dialog box, select Data Analysis.
7. Click on **Add a step**. Select Pause.
8. In the Comment field, type:

The data file will now be recalled.

9. Click on **Add a step**. Select Display Curve.
10. In the Edit Step area, the Select Existing File radio button should be selected. Using the **Browse** button, find the desired data file. It will be displayed in the File Name field.
11. In the Select Curves to Display field, select the curve type you wish to use. In this example, we use Unsubtracted Weight. Keep the default values for Start at Step and End at Step (step 1 of the Method program), Start at Time Zero is off.
12. Click on **Add a step**. Select Pause.
13. In the Commend field, type:

Now we will convert the X scale from time to temperature.

14. Click on **Add a step** and select Rescale Options. From the drop-down menu, select Rescale X and click on **OK**.
15. The curve selected in step 1.2 should be displayed in the Select Active Curve field. This is the curve for which you want to change the X-axis scale. In the Set Axis Units field, select Sample Temperature from the drop-down list. Keep the default temperatures in the Minimum and Maximum fields.
16. Click on **Add a step** and select Pause.
17. In the Comment field, enter

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Rescale the Y axis.

18. Click on **Add a step**, select Rescale Options, and select Rescale Y from the drop-down list. Click on **OK**.
19. The curve selected in step 1.2 should be displayed in the Select Active Curve field. In the Maximum field, change the value to 20 mg.
20. Select **Add a step** and select Pause.
21. In the Comment field, type

After rescaling, perform on Onset calculation.

22. Select **Add a step** and choose Calculation Options. Select Onset from the drop-down list and click **OK**.
23. The curve selected in step 1.2 should be displayed in the Select Active Curve field. Change the Left Limit to 100°C and the Right Limit to 340°C.
24. Select **Add a step** and choose Pause.
25. In the Comment field, enter

Another useful calculation is Delta Y.

26. Add a step and select Calculation Options, Delta Y, and click on **OK**.
27. The curve selected in step 1.2 should be displayed in the Select Active Curve field. Change the Left Limit to 50°C and the Right Limit to 900°C.
28. Add another Pause line and enter the following into the Comment field:

A first derivative curve will be calculated and displayed.

29. Add a step, select Math Options, and Derivative from the drop-down list. Click on **OK**. The curve selected in step 1.2 should be displayed in the Select Active Curve field. There are no fields to change in the Edit Step area.
30. Add a Pause step and enter into the Comment field:

Since the derivative is so sensitive, it has to be smoothed.

31. Add a step, select Math Options, and Smooth. Click on **OK**.
32. The only field to change in the Edit Steps area is the Window Size. Enter 50.
33. Finally, add a Pause step with the Comment:

A general analysis of the Pyris 6 TGA curve is now complete.

This comment is displayed after the curves are displayed in the Pyris Data Analysis window during playback of the play list.

34. Select **Save Player** from the File menu. Since this is a new play list, the Save Player As dialog box is displayed. Go to the directory to which you want to save the file (C:\Program Files\Pyris\Player Lists). Enter the file name; the .t6p extension is appended automatically.

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35. Click on the **Start at Top of Play List**  button on the Player toolbar. The commands in the play list will be executed. The results of each step are displayed on the Player Data Analysis window. After reading each message in the Pause dialog box, click on **Continue** to clear the dialog box and have the play list continue. If you want to edit the play list below its current position, click on **Pause to Edit** and return to the Edit Play List page. Make any corrections to the list and save. Click on the **Resume**  button on the Player toolbar to resume playback.

Create a Pyris 1 DSC Play List

1. Open the Pyris 1 DSC Instrument Application by selecting the Pyris 1 DSC button on the Pyris Manager.
2. While in Method Editor, Instrument Viewer, or Data Analysis, click on the **Pyris Player** button  on the toolbar. The Pyris Player Setup page is displayed. Pyris 1 DSC should be displayed in the Analyzer Type field.
3. Click on the Edit Play List tab to display the Edit Play List page. If there had been a play list displayed in the preceding session in Player, it will be displayed automatically. If the Edit page was left blank in the preceding session, it will be displayed empty.
4. If an existing play list is displayed, select New Player from the File menu to clear the Player Steps box.
5. Click on the **Add a step** button.
6. From the Player Step Options dialog box, select Prepare Sample.
7. Click on **Add a step** and select Load Sample.
8. In the Carousel Location field, if you have an autosampler, enter the number of the sample tray location in which the sample pan that you want analyzed is located. If there is no autosampler, the default is 1.
9. Click on **Add a step** and choose Start Method.
10. In the Edit Step area, use the **Browse** button to find the method you want to use. In this case, select polye.dsm which is in C:\Program Files\Pyris\Methods.
11. Enter the sample weight in the Sample Weight field.
12. Enter a file name for the data (POLYE.DSD).
13. Click on **Edit Method** to access the other pages of the method. Edit them as needed. Return to the Edit Step area by clicking on the Close Window button (in the upper-right-hand corner) to close the Program or Initial State page.
14. Select **Add a step** and choose Return Sample. The number in the Carousel Location should be the same number as selected for Load Sample.
15. Click on **Add a step** and select Data Analysis.
16. Add a Pause line, In the Comment field, type the following:

    Polymers are almost always heated (to destroy thermal history), cooled (to give a common or known thermal history), then reheated. This technique allows you to compare materials.

17. Add a Display Curve.

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18. In the Edit Step area, the Select Play List Item radio button should be selected. The curve collected in step 1.2 Start Method should be displayed.
19. In the Select Curves to Display field, select Heat Flow. Turn on Display Endotherms Up, start at step 1 and end at step 6, and Start at Time Zero should be off.
20. Add a Delete Curve step to delete the curve and all unsaved results.
21. Add Pause and enter in the Comment field:

    We will now look at the second heat of the experiment.

22. Add a Display Curve.
23. In the Edit Step area, the Select Play List Item radio button should be selected. The curve collected in step 1.2 Start Method should be displayed.
24. In the Select Curves to Display field, select Heat Flow. Turn on Display Endotherms Up, start at step 6 and end at step 6, and Start at Time Zero should be off.
25. Add a Pause line to read:

    The data will be redisplayed in temperature.

26. Add a step and select Rescale Options. From the drop-down menu, select Rescale X and click on **OK**.
27. The curve collected in step 1.2 is displayed in the Select Active Curve field. In the Set Axis Units field, select Sample Temperature from the drop-down list. The Minimum field should read 50°C and the Maximum 150°C.
28. Add a step and select Shift from the Rescale Options drop-down list. Click on **OK**.
29. In the Shift From field change 25 to 50°C.
30. Add a Pause line and enter the following:

    It is often useful to slope the curve and align the beginning and endpoints. The curve will be sloped when you press CONTINUE.

31. Add a step and select Calculation Options. From the drop-down list select Slope and click on **OK**.
32. The Heat Flow curve should be displayed in the Select Active Curve field. Enter 50 in the Pivot Point field and 150 in the Slope Point field. Align Endpoints should be selected.
33. Add a Pause line to read:

    Several more data sets will be recalled and optimized.

34. Add a step and select Display Curve.
35. In the Edit Step area, the Select Existing File radio button should be selected. Use the **Browse** button to select the data file poly1.dsd.
36. In the Select Curves to Display field, select Heat Flow. Turn on Display Endotherms Up, start at step 6 and end at step 6, and Start at Time Zero should be off.
37. Add a step and select Calculation Options. From the drop-down list select Slope and click on **OK**.

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38. The Heat Flow curve selected in step 35 should be displayed in the Select Active Curve field. Enter 50 in the Pivot Point field and 150 in the Slope Point field. Align Endpoints should be selected.
39. Add a step and select Display Curve.
40. In the Edit Step area, the Select Existing File radio button should be selected. Use the **Browse** button to select the data file poly2.dsd.
41. In the Select Curves to Display field, select Heat Flow. Turn on Display Endotherms Up, start at step 5 and end at step 5, and Start at Time Zero should be off.
42. Add a step and select Calculation Options. From the drop-down list select Slope and click on **OK**.
43. The Heat Flow curve selected in step 40 should be displayed in the Select Active Curve field. Enter 50 in the Pivot Point field and 150 in the Slope Point field. Align Endpoints should be selected.
44. Add a step and select Display Curve.
45. In the Edit Step area, the Select Existing File radio button should be selected. Use the **Browse** button to select the data file poly3.dsd.
46. In the Select Curves to Display field, select Heat Flow. Turn on Display Endotherms Up, start at step 5 and end at step 5, and Start at Time Zero should be off.
47. Add a step and select Calculation Options. From the drop-down list select Slope and click on **OK**.
48. The Heat Flow curve selected in step 45 should be displayed in the Select Active Curve field. Enter 50 in the Pivot Point field and 150 in the Slope Point field. Align Endpoints should be selected.
49. Add a Pause and comment:

    Aligning the data sets places the starting point for each at the same mW value.

50. Add a step and select Rescale Options. Select Shift from the drop-down list and click on **OK**.
51. Enter 50 in the Shift From field and click on Align All.
52. Add a Pause with the comment:

    Calculations are also possible. A peak calculation will now be performed.

53. Add a step and select Calculation Options. Select Peak Search from the drop-down list and click on **OK**.
54. The Minimum Peak Height should read 1 mW and the Peak X label box should be checked. Note that the search is over the original scale of the curve: 25°C to 150°C.
55. Add a final Pause comment:

    The analysis is complete.

    This line is displayed after the curves are displayed in the Player Data Analysis window during “play back” of the play list.

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56. Select Save Player from the File menu. Since this is a new play list, the Save Player As dialog box is displayed. Go to the directory to which you want to save the file (e.g., C:\Program Files\Pyris\Player Lists). Enter the file name; the .dsp extension is appended automatically.

57. Click on the **Start at Top of Play List**  button on the Player toolbar. First the autosampler will position the sample pan from position 1 in the sample tray in the sample holder. The cover closes and the run begins. After the data are collected, the sample pan is returned to position 1 in the tray. Data analysis starts. The results of each command are displayed in the Player Data Analysis window. After reading each Pause box, click on the **Continue** button to clear the dialog box and have the play list continue. If you want to edit the play list below its current position, you can click on **Pause to Edit** and return to the Edit Play List page. Make any changes to the list and save. Click on the **Resume**  button on the Player toolbar to have the playback continue.

Create a Pyris 1 DSC with Autosampler Play List

This lesson shows you how to create a typical play list to be used with a Pyris 1 DSC autosampler. It uses the time-saving feature of Sample Group which automates creation of the play list to run samples that are similar using the same method. The method must be created beforehand in order to select it for the sample group. It can be edited while in the Player, however. You must have prepared and weighed the samples before starting the playback of the play list.

NOTE: In order to see the video clips that are associated with this section, you will have to insert the Pyris CD in the CD drive of your computer. Display the Multimd directory's contents in Windows Explorer. Double click on the .avi file indicated in the text below; the file should play on the computer monitor.

1. Open the Pyris 1 DSC Instrument Application by selecting the Pyris 1 DSC button on the Pyris Manager bar.
2. While in Method Editor, Instrument Viewer, or Data Analysis, click on the Pyris Player button  on the toolbar. The Pyris Player Setup page is displayed. Pyris 1 DSC should be displayed in the Analyzer Type field.
3. Click on the Edit Play List tab to display the Edit Play List page. If there had been a play list displayed in the preceding session in Player, it will be displayed automatically. If the Edit page was left blank in the preceding session, it will be displayed empty.
4. If an existing play list is displayed, select **New Player** from the File menu to clear the Player Steps box.

    **(Play1.avi)**

5. Click on the **Add a step** button.
6. From the Player Step Options dialog box, select Sample Group. Three lines are added to the Player Steps area. These are the default lines of a Sample Group.
7. With Sample Group highlighted, enter a comment into the Comment field in the Edit Step: Sample Group area. For example, we are going to run samples of polyethylene that are in sample pans in carousel locations 1 through 5.
8. Click on Sample List in the Player Step box. The Edit Step: Sample List area is displayed.

9. Click on the **Browse** button to display the Open Methods display box to select your method. This method will be used to run each sample to follow. The method file name appears in the Method Name field.
10. Click on the **Edit Method** button. The Initial Values and Program pages of the method become available for editing.
11. The method program needs editing. In our video we added 5 additional lines that heat, cool, and reheat the sample.
12. Save the method by selecting **Save Method** from the File menu.
13. Close the Method Editor by clicking on the Close Window **X** in the upper-right-hand corner.
14. Click on **Add a sample** at the bottom of the window. A new line is added below Sample List. The indentation and dotted line indicate that it is a sublevel of Sample List. This sample belongs to the list and will be run using the method in Sample List.

 **(Play2.avi)**

15. In the Edit Step: Sample area, enter a Sample ID, Operator ID, and any comments you want to use to describe the sample.
16. Enter the carousel location at which the sample pan is located.
17. Enter the sample weight in the Weight field.
18. Enter a sample name in the File Name field. If your samples run in a sample list are similar, you may want to use the increment feature of Pyris. At the end of a file name type the pound sign #. This tells Pyris to append a number at the end of the file name and increment it for each sample entry. For example, for sample 1, the file name will be poly1, for the second sample poly2, and so on to poly5.
19. Click on the entry line in the Player Steps area and the line is redisplayed with additional information. Also, the default path for the data file is displayed in the Directory field.

 **(Play3.avi)**

20. Click on the **Add a sample** button. Another sample line is added to the Sample List. Fill in the Sample ID, Operator ID, and Comments fields as needed. If you used the # feature at the end of the previous sample's file name, you do not need to edit the File Name field. Edit the Weight field. The Location field is incremented automatically. Your samples must be in consecutive positions in the autosampler.

 **(Play3b.avi)**

21. The last three samples for the sample list are going to added using the Copy and Paste commands from the Player toolbar. Highlight line 1.1.2 and click on the **Copy** button  to copy the line to the clipboard.
22. Click on the **Paste** button on the toolbar to paste the copied line into the Sample List. Click on the button two more times to insert three sample lines. Note that the lines are exact copies of Sample 2; they need to be edited.
23. Click on line 1.1.3; edit the Weight field for the sample at location 3 of the autosampler. Edit the Sample ID and any other field as needed. The File Name will be poly#.dcd. Do the same for lines 1.1.4 and 1.1.5.

 **(Play4.avi)**

24. Click on Data Analysis List. The Edit Step: Data Analysis List area is displayed.
25. Enter any comment you wish about the data analysis about to take place.
26. Click on **Add a step**. From the Player Step Options dialog box, select **Display Curve**.
27. In the Edit Steps area, make sure that **Use Current Run** is selected. The rest of the area is grayed out except for Normalize Y.
28. Click on **Add a step** and select **Calculation Options**.
29. Select **Peak Area**.
30. Click on the Test Options button to display the Tolerance Test dialog box. We want to test the Peak Area within the range of 0 to 16 minutes. If the run fails the test, the playback of the play list will stop.
31. The play list is complete. Click on the View Play List tab to see the list without the Edit Step area or buttons around it.
32. Select Save Player from the File menu. In the Save Player As dialog box enter a file name for your new play list.

The play list can now be run or "played back" by clicking on the **Start at Top** button  on the Player toolbar.

Create a Pyris 1 TGA with Autosampler Play List

This lesson shows you how to create a typical play list to be used with a Pyris 1 TGA that has an autosampler. Here we use the Sample Group feature and the Tare This and Weigh This features. The method you want to use to analyze the samples in the autosampler must be created before you can select it in Pyris Player. However, you can edit the method once it is selected.

NOTE: In order to see the video clips that are associated with this section, you will have to insert the Pyris CD in the CD drive of your computer. Display the Multimd directory's contents in Windows Explorer. Double click on the .avi file indicated in the text below; the file should play on the computer monitor.

1. Open the Pyris 1 TGA Instrument Application by selecting the Pyris 1 TGA button on the Pyris Manager bar.
2. While in Method Editor, Instrument Viewer, or Data Analysis, click on the Pyris Player button  on the toolbar. The Pyris Player Setup page is displayed. Pyris 1 TGA should be displayed in the Analyzer Type field.
3. Click on the Edit Play List tab to display the Edit Play List page. If there had been a play list displayed in the preceding session in Player, it will be displayed automatically. If the Edit page was left blank in the preceding session, it will be displayed empty.
4. If an existing play list is displayed, select **New Player** from the File menu to clear the Player Steps box.
5. Click on the **Add a step** button.
6. From the Player Step Options dialog box, select Sample Group. Three lines are added to the Player Steps area. These are the default lines of a Sample Group.

 **(TGAPLAY1.AVI)**

7. With the Sample Group line highlighted, enter a comment into the Comment field in the Edit Step: Sample Group area.
8. Click on the Sample List line in the Player Step box. The Edit Step: Sample List area is displayed and a message stating that the method for running the sample list has not been specified. Click **OK** to clear the message box.
9. Click on the **Browse** button to display the Open Methods display box to select your method. This method will be used to run each sample to follow in the Sample List. The method file name selected appears in the Method Name field.
10. Click on the **Edit Method** button. The Initial Values and Program pages of the method become available for editing.
11. We edited the "heat to" temperature to 700°C in line 2. This automatically changes the "heat from" value in line 4.
12. Click on Save Method in the File menu.
13. Click on the Close Window **X** in the upper-right-hand corner of the Method Editor window to return to the Edit Play List page.
14. Click on the **Add a sample** button at the bottom of the screen.
15. Enter Sample ID, Operator ID, and Comments as needed. These are not required fields. Sample ID may be helpful in giving better identification to the sample beyond the file name.
16. If the crucible that you are going to use for this sample has not been tared yet, you can do so now. (In our video, we tared the crucibles before starting the build of our play list. We show how to weigh the sample.) The empty crucible must be in position (location 1). Click on **Tare This** and the Tare/Weigh System box appears. The system will tare the crucible and enter the weight in the Zero field.
17. With the autosampler in the Safe position (to the right), remove the autosampler ring containing the crucibles by placing the thumb and pointing finger of each hand on the ring's edge and carefully lifting it up and back a bit to clear the grippers. Once over the grippers, place the ring on the bench and place the sample in crucible #1. Return the ring to the autosampler, aligning the hole near location 8 with the standout on the autosampler.
18. With the sample now in place, click on **Weigh This** on the bottom of the screen. The Tare/Weigh System box appears and the program begins the steps to weigh the sample. The button representing location 1 turns and remains yellow during the procedure. When complete, the button should turn green if the weighing is successful. The weight is entered into the Weight field.

     **(TGAPLAY2.AVI)**
19. Click on **Add a sample** to add line 1.1.2 to the play list. The location defaults to position 2 in the carousel. This crucible has not been tared yet so we click on the Tare This button. The Tare/Weigh System box appears again and the taring procedure occurs. (Make sure that the empty crucible is in place first!)
20. Remove the autosampler ring and place the sample into crucible #2 and return the ring to the autosampler.
21. Click on **Weigh This** to obtain the weight of the sample.

     **(TGAPLAY3.AVI)**
22. Click on the sample line to display the weight in the line.

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23. Click on the Data Analysis List line. Enter a comment in the Comment field in Edit Step: Data Analysis area.
24. Click on the **Add a step** button to display the Player Step Options dialog box.
25. Select Display Curve and **OK**. The Display Curve line is added to the play list. The default curve is the heat flow curve of the current run.
26. Click on **Add a step** again and from the Player Step Options box, select Pause.
27. In the Comment line for Pause, enter your text. The Pause line is important here. It gives you the opportunity to view the curve of the current run before the playback of the list proceeds to run the next sample. If your Data Analysis List contains calculations that involve a Tolerance Test, you may want to view the results.
28. The Pause line is the last line of the play list. Select Save Player from the File menu.
29. Enter a file name in the Save Player As dialog box.

The play list can now be run or "played back" by clicking on the Start at Top button  on the Player toolbar.

Specific Heat Analysis

The determination of specific heat capacity by a power-compensated DSC analyzer is an important tool in thermal analysis. Heat capacity is an intrinsic property of a material. It is the amount of energy needed to increase a unit quantity (e.g., 1 g, 1 mole) of material 1°C. All materials have specific heat. If there is no phase transition or reaction, the specific heat is a positive number that gradually changes with temperature.

Specific heat is fully normalized output of a DSC, i.e., the displacement during a scan is equal to the product of the heat capacity times the sample weight times the scanning rate plus the no-sample baseline. DSC data is sometimes misinterpreted because just one heat flow run was performed. The data from that run may contain information about the instrument itself such as the specific heat of the reference side of the sample holder. This problem can be fixed by performing a baseline run with no sample using the same conditions to be used for the sample. This constitutes the classical two-curve Cp method. This is illustrated here by finding the specific heat of the reference standard sapphire using the Pyris 1 DSC.

The following steps were performed in order to obtain the specific heat of sapphire:

1. Prepare the Pyris 1 DSC according the suggestions in Suggestions for Accurate Cp.
2. Place an empty standard aluminum sample pan and lid in each sample holder in order to run a no-sample baseline.
3. Enter the parameters into the Preferences pages and the Method Editor. Use a Sample Weight of 0 for the baseline run.

Purge Gas	Nitrogen at 20 cc/min
Cooling Device	Ice bath
Sample Weight	28.12 mg
Initial Temperature	50°C
Y Initial	0 mW
Equilibrate Temp.	0.01°C
Heat Flow	0.01 mW

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Wait no longer than	15 min
Method Program	Isothermal – Scan – Isothermal
Heating/Cooling Rate	10°C/min
Temperature Range	50 – 100°C, 100 – 150°C, . . . , 250 – 300°C

4. Click on the **Start Method** button to start the baseline "no sample" run.
5. Find the sapphire standard in the Specific Heat kit (P/N 0219-0136) and encapsulate it in the standard aluminum sample pan used in the baseline run.
6. Enter the sample weight of 28.12 mg into the Method Editor.
7. Click on the **Start Method** button to start the sample run.
8. After data collection is complete, click on the **Data Analysis** button on the toolbar.
9. Using Add Data in the File menu, select the two data files you just collected. The baseline curve should be the lower curve and the sample curve should be the top curve.
10. The Step Select feature should be activated. Make sure that a checkmark appears next to Step Select in the Curves menu. If it does not, click on Step Select.

    If you have data from multiple scanning steps, the Start Time at Zero feature should not be checked in the Curves menu.
11. The data is now in the form such that you can calculate the specific heat for each iso–scan–iso step.
12. Click here to see an example of alignment of the baseline and sample curves.
13. Make sure that the sample curve is the active curve (heavy line display). Click on Heat Flow in the Curves menu. The Step Select dialog box is displayed.
14. From the list of steps, select adjacent iso–scan–iso steps to create a segment. Click on **OK**. If your method had 11 steps, you should be able to define 3 segments.
15. Repeat steps 11 and 12 for the baseline curve but use Baseline Heat Flow from the Curves menu.
16. Click on a segment of the sample curve and choose Multiple Curve from the Specific Heat submenu in the Calc menu.
17. The default selection for the baseline curve should be the segment of the curve that coincides with the segment of the sample curve selected. Click on **OK** or press **Enter** to accept the selection.

    The specific heat curve and its ordinate scale is added to the display.
18. In the case of multiple-step data, repeat steps 15 and 16 for each scanning step (iso–scan–iso segment).
19. Change the X scale to Temperature using Rescale X in the Display menu or in the toolbar.

You are finished finding the specific heat of sapphire.

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Chapter 12
Troubleshooting

If a problem should occur while using Pyris Software for Windows, first check the Release Notes that are installed on your computer when Pyris is installed. In the Pyris Software for Windows group in the Programs menu, select **Pyris Readme**. The text file will be displayed in Wordpad on your screen. At the end of the notes there is information on existing known problems and workarounds.

Emergency Repair Disk

The Repair Disk utility of Windows NT saves all the current system settings to an Emergency Repair Disk. You can then use the disk to restore your computer if files become damaged. The Repair Disk should be updated whenever there is a change in the configuration of the workstation, including adding or removing software, printer drivers, and hardware (e.g., tape drive, sound card, multiport card, etc.). The ERD is usually created when Windows NT is installed. You can update the repair disk by running RDISK.EXE, which is located in the SYSTEM32 subdirectory of your Windows directory (usually \WINDOWS\SYSTEM32 or \WINNT35\SYSTEM32). You can also access it by going to the Windows online Help from the **Start** button menu and then displaying the help topic for Emergency Repair Disk or Repair Disk Utility. Both topics have hotspots to the utility.

Security

If you have trouble installing security for Pyris Software for Windows:

- check that the security button holder is plugged into the parallel port
- if a printer is connected to the security holder, turn the printer on while installing security
- check the Pyris Readme Notes that accompany the software. You can read the notes by double clicking on Pyris Readme in the Pyris Software for Windows menu. The notes may contain new information about security holders that was not incorporated into the online help.

To install security after the software is already installed, begin the install procedure as you would to install the Pyris software. When the Welcome screen is displayed without any errors, security

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has been installed successfully. You may then exit from the Setup procedure and begin operating Pyris Software for Windows. (You must reboot the system to activate the security drivers.)

If you find that the security holder is not found, check that the printer connected to LPT1: is online. If it is powered on but offline, the software may not recognize the security holder. Put the printer online and try operating the software again.

Security Holder and Buttons

Pyris Software for Windows includes a CD, Configuration disk, 7 Series/UNIX Data Conversion disk, and a security holder with two buttons: the Main button (which has tape over it) and the Applications Add On button The buttons should be in the security holder already. The Main button contains the Main Applications Permissions Set. This set allows you to run Data Analysis. All other security permission bits are transferred to the Main button from the Applications Add On button.

NOTE: In order for the Pyris system to work properly, the Main button must NEVER be removed from the security holder.

Although the Applications Add On button has all the additional applications permissions, only those activated will be transferred to the Main button. On a new system, there are only two activated permissions: Instrument Control, which allows control of all analyzers, and Pyris Revision X.X, which allows the operation of the most recent version of Pyris software. During the software installation, the two activated permissions are transferred to the Main button and are removed from the Applications Add On button. The button can then be removed.

When additional software packages are purchased, the permissions for those packages are transferred to the Main button from the supplied Applications Add On button with those permissions activated. The button is inserted in the hole on the security holder. The permissions are transferred by using the floppy disk supplied with the application.

Multi-User Configuration

When dealing with the multiuser configuration of a Pyris system for Remote Monitor, all the computers involved must have Windows NT 4.0 installed as well as the same revision of Pyris Software for Windows. The computer that will be used to control the analyzers should be identified as the primary computer. The security holder that contains the two buttons must be plugged into LPT1 of the primary computer **before** software installation. The Main button contains Data Analysis permissions, the second button (Applications Add On) contains Instrument Control permissions. These permissions are transferred to the Main button during installation. The Instrument Control button can be removed after transfer.

A multiuser system includes one security holder with **two** buttons (Main and Applications Add On) and additional security holders, one for each additional computer. Each holder has only one button. The additional holders are installed on the remote computers.

Advanced packages contain a permissions button and a diskette. The Remote Monitor package must be installed on the remote computers.

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XFERPERM and DIAGPERM

To help determine if a security holder is good and what permissions are on the buttons in the holder, use the two diagnostic programs on the PE\PYRIS directory: XFERPERM and DIAGPERM. Access these programs using Windows Explorer. XFERPERM displays a series of dialog boxes that step through determination of button contents. DIAGPERM is a program that steps you through determining if the security holder is functioning properly.

Double-click on XFERPERM.EXE or DIAGPERM.EXE in Windows Explorer to start the program.

Instrument Communication

There may be an occasion when the computer loses communication with the analyzer. For example, the TAC 7/DX may be inadvertently turned off or unplugged. Should this occur, the **Start** button on the Control Panel will change to a **Reset** button:



and the Status will be listed as “Offline” or “Communications Error.”

If the status is “Offline,” check that the cables and power cords are in place and that there is power to the analyzer (and the TAC 7/DX). If the status is “Communications Error,” the problem may be caused by two applications trying to use the same communications port or by a hardware problem.

After fixing the problem, click on the **Reset** button in the control panel to reset communications with the analyzer.

Long File Names

Pyris Software for Windows supports long file names. The maximum length of the file name is 255 characters, including the drive and path name. However, some networks do not support long file names, even if you are connected to the network on a Windows NT or Windows 95 workstation. The network will not allow a long file name to be saved to it, and when you begin a run, a short file name will not be generated automatically by the operating system.

If a method is set up to save the data in a long file name on a network drive, Pyris Software for Windows will save the data to the root directory of the hard drive where the software resides. This is done to ensure that the data is saved properly. You can then move the data file anywhere after the run is over.

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