DSC 6 Description

The DSC 6 is a heat-flux differential scanning calorimeter. Heat flow is determined by measuring a temperature difference over a very accurately known thermal resistance. The DSC 6 system permits the direct calorimetric measurement, characterization, and analysis of thermal properties of materials. Under the control of the Pyris Software for Windows on your PC, the DSC 6 is programmed from an initial to a final temperature through transitions in the sample material such as melting, glass transitions, solid-state transitions, or crystallization. Usually the DSC 6 is programmed to scan a temperature range at a linear rate for the study of these endothermic and exothermic reactions. Endotherms and exotherms can be shown as an upward or downward deviation from the baseline. The DSC 6 can also be used to perform isothermal experiments.

The DSC 6 has an integrated cooling system which accepts various types of cold liquids or gas. This allows operation of the DSC 6 from ambient to 450°C. It is recommended that normal tap water be used as a cooling agent. The tap water must be circulated through the heat exchanger at a low flow rate between 5.52 and 6.90 kPa (8 – 10 cc/min) during the operation of the analyzer.

For subambient operation to –120°C, cold gaseous nitrogen is required. A controlled-pressure liquid nitrogen dewar system is necessary. This maintains a constant pressure level of 6.90 – 20.7 kPa (1 – 3 psi).

For more discussion of the features of the DSC 6, click on one of the topics below:

- **Furnace and Sensor Design**
- **Display**
- **Cooling**
- **Modular Construction**

Furnace and Sensor Design

A low-mass furnace forms the heart of the DSC 6. The furnace is designed for ruggedness and resistance to chemical corrosion. Because of its low mass, the furnace can heat and cool rapidly.

A platinum resistance sensing device measures and controls the furnace temperature. This device, coupled with intrinsic "fuzzy logic" technology, provides very accurate furnace temperature control with minimal temperature overshoot. Fuzzy logic maintains tight temperature control because it incorporates intelligent decision-making capabilities.

The atmosphere surrounding the sample is controlled very efficiently since the gas enters above the sample crucible. This is extremely important when performing oxidative stability experiments. The purge gas is also preheated resulting in minimal disturbance of the signal.

The sample is placed on a precisely machined sensor disk constructed of 90% nickel/10% chromium. This rugged and durable disk provides high thermal conductivity and a very low time constant for superior instrument response. Highly sensitive thermocouples measure the temperature at both the sample and reference positions. The DSC 6 accommodates the complete range of Perkin–Elmer sample pans. Over two dozen different pan types are available.

DSC 6 Cooling

The DSC 6 incorporates a built-in cooling chamber that lies below and is attached to the furnace assembly. The chamber uses a simple in/out design which allows for the use of any noncorrosive circulating liquid or cooling gas. Cooling gases include liquid nitrogen and cooling liquids include water and ethylene glycol.

Cooling with liquid nitrogen gas is simple, fast, and inexpensive. It requires only a transfer line and a low-pressure LN_2 dewar — no extra accessories are needed. For cooling with liquids, many commercial circulating coolers are available that can be interfaced to the DSC 6.

Safety Precautions for the DSC 6

WARNING: Be sure that all instrument operators read and understand the following precautions. It is advisable to post a copy of these precautions on or near the instrument itself.

The following precautions must be observed before and when using the DSC 6 Differential Scanning Calorimeter:

- Before connecting the DSC 6 to the mains outlet, check the mains voltage setting and fuse.
- The DSC 6 requires a good earth ground that is common to the earth ground of the computer.
- Do not operate the instrument in a cold room. The ambient temperature and the temperature of the instrument should be between 10°C and 30°C.

WARNING: Never touch the outer and inner furnace lids. The temperature of the furnace can reach as high as 450°C. Use tweezers to remove the lids.

WARNING: Do not touch the inside of the furnace; it might be hot and internal pollution can cause loss of performance.

WARNING: Always ensure that there is adequate ventilation when operating the DSC 6. Operate the DSC 6 in a fume hood when running samples that give off toxic gases since the reaction gases escape through the furnace lids.

CAUTION: Do NOT expose the sensor surfaces to mechanical stress. If no external cooling is applied, do not operate the DSC 6 above 50°C.

CAUTION: Do not exert excessive forces on the sensor disks.

CAUTION: Care must be taken when handling the outer furnace lid so its ceramic lining is not damaged.

- Avoid contamination of the furnace, use high-purity purge gases to purge any reaction products out of the furnace, and heat the furnace to temperatures above 430°C ONLY when absolutely necessary.
- Check that the cooling liquid supply is circulating before starting a run.
- Ensure the purge gases are set to the recommended flow rates. A rate that is too high will disturb the inner furnace lid.
- Never open the furnace when it is at an elevated temperature.

Calibration of the DSC 6

The DSC 6 analyzer has been calibrated at the factory for both temperature and heat flow. Under normal conditions, the DSC 6 does not need temperature recalibration. As a result of the use of high-precision Pt100 sensors, the temperature accuracy does not vary over the temperature range and remains constant for long periods of time.

We recommend that the temperature calibration and heat flow calibration be checked using the precrimped samples of indium and zinc before obtaining data. We also recommend that you perform calibrations in the following order:

- **Baseline Correction**
- **Temperature Calibration**
- **Heat Flow Calibration**

Baseline Correction of the DSC 6

Generally, it is not necessary to perform a baseline correction on the analyzer. However, if you find that your baselines have too much slope (>3 mW at a scan of 20°C/min with no sample pans in the furnace), you can perform a baseline correction. The baseline correction eliminates a static offset and drift of the baseline during a temperature scan. The correction performed is a linear correction between two points.

To correct the baseline, perform the following steps:

1. Determine the temperature range over which the baseline is to be optimized. It is not necessary to optimize the baseline over the entire temperature range if most of your experiments will be performed over a smaller portion of the range.
2. Select the coolant or cooling accessory that will be used for your experiment.
3. Set the purge gas to a flow rate of approximately 20 – 30 cc/min.
4. Remove all sample and reference pans from the furnace. Make sure that the furnace lids are in place.
5. In the Pyris software, click on the DSC 6 button in the Pyris Manager to select the DSC 6.
6. From the Method Editor, select **File** followed by **Open Method**. A list of method file names is displayed.
7. Double click on **baseline.d6m**. This method is displayed on the screen. It can be used for obtaining routine baselines. If necessary, change any of the values, e.g., the temperature program.
8. Make sure the Sample Weight box contains 0.00.
9. Click on the **Start** button  in the Control Panel to start the run. The light on the DSC 6 will change from red to green indicating that the DSC 6 is in temperature control and data are being collected.
10. When the run is finished, save the data file by selecting **Save** in the File menu and entering a data file name in the dialog box.

The data file from this run is used to correct the baseline of subsequent runs. The two-point baseline correction is active over the whole temperature range used. To correct the baseline of a data file, you would select Use Baseline Subtraction in the Initial State page (click here to see page). This activates the other fields in the Baseline File box. From the Browse dialog box, select the file collected here.

Temperature Calibration

The DSC 6 has been calibrated at the factory for temperature. Under normal conditions the DSC 6 does not need temperature calibration. As a result of the use of high-precision Pt100 sensors, the temperature accuracy does not vary over the temperature range and remains constant for long periods of time.

It is recommended that a baseline correction be performed before temperature calibration.

At least two measured onset temperatures are necessary to calibrate the DSC 6. The calibration materials selected must have at least a 50°C temperature difference in their theoretical Onset temperatures. Indium and zinc are the recommended calibration materials. However, other reference materials can be used to calibrate the analyzer.

To calibrate temperature of the DSC 6:

1. While using the Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu. The Calibration window is displayed.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are going to perform a heat flow calibration, restore all default calibration values by selecting the **All** command.
3. Select the **Save and Apply** button in the Calibration window to send the default values to the analyzer and save the current calibration file. The Save As dialog box appears. Select **OK** to save the default file.
4. Select **Close** to close the Calibration window and begin using the new calibration values.

After you have done these steps, go on to the following topics to complete the temperature calibration of the DSC 6:

- **Performing Indium and Zinc Runs**
- **Performing a Peak Area with Onset Calculation**
- **Performing a Temperature Calibration**

Performing a Peak Area with Onset Calculation

For the indium and zinc runs that you performed, a Peak Area with Onset calculation is performed. The data file you just collected should be displayed in the Data Analysis window. Perform an onset calculation on the data as follows:

1. Select **Onset** from the Calc menu. The Onset Calculation dialog box appears.
2. Enter the Left Limit and the Right Limit. You can also click on the two red **X**'s that appear on the curve and drag them to the desired left and right limits. The Left Limit and the Right Limit fields will be filled in on the dialog box.
3. Click on the **Calculate** button.
4. Record the Onset value for temperature calibration and ΔH for heat flow calibration. If you performing a heat flow calibration, you will need the ΔH values.
5. Repeat steps 1 – 4 for the zinc calibration run.

Once you obtain the Onset values, you can perform a temperature calibration.

Performing Indium and Zinc Runs

In performing an indium and zinc run, you can use the precrimped, preweighed samples of indium and zinc or you can prepare your own samples for calibration. To perform the run, follow the steps below:

1. Place the sample pan containing indium or zinc in the sample area of the sample holder and place the reference sample pan in the reference area of the sample holder.
2. Using tweezers, cover the furnace with the inner and the outer furnace covers.
3. If not already displayed, select the Method Editor icon  from the toolbar.
4. From the File menu, select **Open Method**. The Open Method dialog box displays the methods available in the default methods directory.
5. Select the **IndCal.d6m** method if using indium or **ZincCal.d6m** if using zinc.
6. In the Sample Info page, fill in the sample information. Enter the weight of the indium or zinc sample and the file name under which the data file is to be saved.
7. Click on the **Start** button  in the DSC 6 Control Panel to start the run.

   The run starts. When finished, the data file will be saved with the file name that you entered in the method.

After performing the indium and zinc runs, you must perform a peak area with onset calculation.

Performing a Temperature Calibration

The measured Onset values for indium and zinc are used to calibrate the temperature of the DSC 6. To perform the temperature calibration, perform the following steps:

1. With either the Instrument Viewer or the Method Editor as the active window, select **Calibrate** from the View menu. The Calibrate dialog box is displayed.
2. Select the **Temperature** tab.
3. Enter the name of the reference material that you used.
4. Enter the expected onset temperature for the reference material in the Exp. Onset field (e.g., 156.60 for indium).
5. In the Meas. Onset field enter the onset temperature that you obtained when you performed the peak area with onset calculation.
6. Enter the method used for the calibration.
7. Select the check box in the Use column for each reference material used in the calibration.
8. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the current calibration file.
9. Go to the Heat Flow calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

Heat Flow Calibration

It is recommended that indium be used when performing a heat flow calibration. However, the heat flow calibration can be performed using any one of the following materials:

Calibration Substance	Heat of Fusion (J/g)
Cyclopentane	8.60
Gallium	79.87
Indium	28.45
Lead	23.01
Tin	60.46
Zinc	108.37

1. While using the Instrument Viewer or the Method Editor, select **Calibrate** from the View menu.
2. Restore the default Heat Flow calibration by selecting **Heat Flow** from the Restore menu. If you performed a Temperature calibration just prior to starting a Heat Flow calibration and selected **All** from the Restore menu, then you do not need to restore the default Heat Flow calibration here.
3. Select **Save and Apply**.
4. Select **Close** in the Calibration window.
5. In Data Analysis, use the same data file obtained from the run in Obtaining an Indium and Zinc Run, or perform a run using another reference material.
6. Perform a Peak Area with Onset calculation and note the ΔH result, or use the ΔH result recorded for the reference material when you performed this step for temperature calibration.
7. You are now ready to perform a heat flow calibration.

DSC 6 Help

Sample Handling for the DSC 6

The DSC 6 measures differential temperature and reports the heat flow to the sample as it is heated, cooled, or held isothermally. The following topics discuss the materials and techniques used to obtain data with a DSC 6. Some topics contain videos illustrating the subject.

- **Sample Preparation**
- **Sample Pans**
- **Sample Encapsulation**
- **Sample Atmosphere**
- **Loading the Sample into the DSC 6**

Sample Preparation for the DSC 6

For optimum results, observe the guidelines below when preparing your samples for analysis.

Sample Size

The size of samples that can be analyzed with the DSC 6 is limited only by the volume of the sample pan you are using. However, the sample size in conjunction with the scanning rate will affect the quality of your results.

With the DSC 6, running a large sample at a faster scanning rate will improve the apparent sensitivity. Running a small sample at a slower scanning rate will improve the resulting peak resolution.

Most samples run on the DSC 6 should be in the 5 – 30-mg range. Use small samples of 1 mg or less for decomposition studies and other analyses in which the sample may tend to contaminate the sensor disks. Small sample sizes are also recommended if the sample has not yet been investigated.

Temperature Range

The temperature range for your analysis depends on the sample and the type of experiment you are performing. The DSC 6 can be used to analyze samples from a temperature of –120°C to 200°C when used with cold nitrogen gas or from ambient to 450°C.

Heating Rate

The DSC 6 allows heating rates of 0.1 to 50°C/min in steps of 0.1°C/min. Proper selection of heating rate will increase efficiency of your analysis at the desired sensitivity.

Generally, slower heating rates improve the peak resolution while faster heating rates improve the apparent sensitivity. Heating rates greater than 40°C/min are generally used for rapidly heating or cooling the DSC 6 to the beginning temperature of the next segment or to selected isothermal temperatures in the temperature program. Typical experimental heating rates range from 5.0 to 20°C/min. Slower rates can be used for liquid crystals and purity analyses.

Samples

The DSC 6 analyzes both solid and liquid samples. Solid samples can be in film, powder, crystal, or granular form.

To learn how to prepare a solid polymer sample for encapsulation in a sample pan, see **Preparing a Solid Polymer Sample**

To learn how to prepare a liquid sample for encapsulation, see **Preparing a Liquid Sample.**

To learn how to prepare a powdered sample for encapsulation, see **Preparing a Powdered Sample.**

The prepared samples are encapsulated in sample pans. Your choice of sample pan depends on the nature of the sample and the temperature range of interest. A summary of sample pans and sealers (with their Perkin–Elmer part numbers) available from Perkin–Elmer is given in DSC 6 Sample Pans and Sealers.

NOTE: The standard aluminum sample pans and covers provided with your DSC 6 are suitable for most thermal analysis applications. Metal standards, such as indium, tin, lead, and zinc, must be encapsulated in aluminum pans because they will alloy with copper, gold, or platinum pans.

After the sample has been prepared and placed in a sample pan, it must be encapsulated. The method of

sample encapsulation most widely used is crimping the sample pan cover in place. The normal crimping procedure yields a tightly but not hermetically sealed sample. For a detailed discussion of how to encapsulate samples properly, see Sample Encapsulation.

Sample Atmosphere

The DSC 6 has a Dry Gas Inlet and a Purge Gas Inlet at the rear of the analyzer. The Dry Gas Inlet (external purge) purges the outer portion of the sample holder/heat exchanger assembly with dry gas. This must be used whenever working below ambient temperatures. The dry gas, which is preferably dry nitrogen, prevents condensation of water vapor on the measuring cell. A flow rate of 20 – 40 cc/min is recommended for the external purge.

NOTE: There is no flow restrictor built into the analyzer. Therefore, it is necessary to use either an H restrictor (P/N 0154-1498) or a Float Displacement Flow Meter (P/N 0290-1624) to obtain the proper flow rates. $70.00 each

NOTE: It is not necessary to connect a dry gas provided you do not operate the DSC 6 below ambient temperatures.

The Purge Gas Inlet (sample area) is used to purge the furnace. Argon or 99.9% minimum purity nitrogen is recommended for purging the sample holders. Other gases such as air or oxygen can also be used.

The gas must be dry. The required flow rate should be between 20 and 40 cc/min. Use a size 1A cylinder equipped with a pressure regulator that has a shutoff valve at the outlet.

When using both dry gas and purge gas, set the flow rates equal to each other (20 – 40 cc/min).

The TAGS (Thermal Analysis Gas Station) may also be used for automatic switching of four sample purge gases using a predefined operation program that you set up in the method. You are allowed up to ten gas changes per temperature program step with the TAGS.

1 cc/min ≡ 1 psi

∴ set @ 20-40 psi

H-restricter in the line 1/8 inch I.D.

Loading the Sample into the DSC 6

Prior to loading the sample into the DSC 6 sample holder, the sample must undergo sample preparation and encapsulation.

In addition to encapsulating the sample, it is recommended that a reference capsule be used in the reference furnace during the DSC 6 experiment. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated.

You load the sample into the DSC 6 as follows:

1. Open the sample holder by lifting off the two furnace covers with tweezers and placing them onto the stainless-steel ring that surrounds the outer furnace cover.

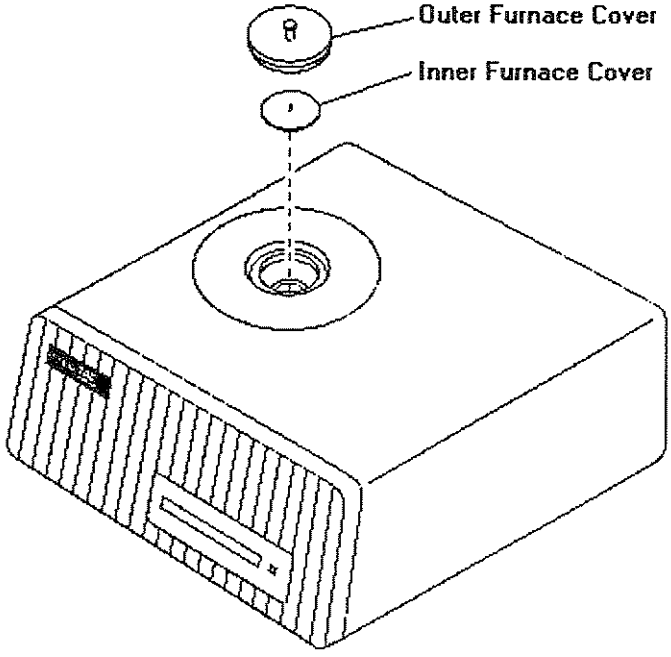
   Click here to view figure.

   CAUTION: Do NOT place the inner furnace cover onto the painted DSC 6 cover when it is hot. It might damage the paint on the analyzer.

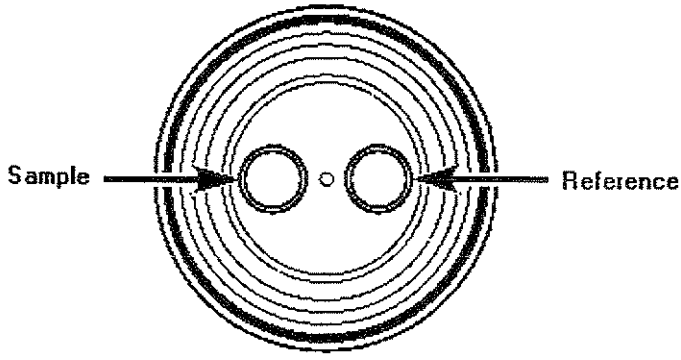
2. Using tweezers, place an encapsulated sample into the furnace on the sample side (left) and an empty pan and lid of the same type used to encapsulate the sample on the reference side (right). Make sure that the pans are centered on the sensors.

   Click here to see figure.

Removing Outer and Inner Furnace Covers



Placing Sample Pans into Furnace



DSC 6 Accessories, Replacement Parts, and Parts Provided

Supplies, accessories, and replacement parts for the DSC 6 analyzer can be ordered directly from Perkin-Elmer. **PE XPRESS**, Perkin-Elmer's catalog service, offers a full selection of high-quality thermal analysis supplies through the Thermal Analysis Supplies Catalog. To place an order, request a free catalog, or ask for information:

- If you are located within the U.S., call toll free **1-800-762-4002**, 8 a.m. to 8 p.m. EST.
- If you are located outside of the U.S., call your local Perkin-Elmer sales office.

For a list of the parts provided with your DSC 6, see the DSC 6 Spares Kit.

Technical Service 1-800-762-4002 8:30-5 M-F EST
Have serial # of product available
9032206 & 9032207

Rescaling

You can optimize the display of the data file by rescaling the X and Y axes of the Instrument Viewer or the Data Analysis window, thereby displaying only a portion of the curve(s) in the window. There are three ways to rescale:

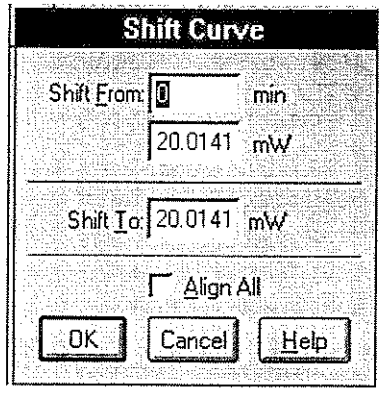
1. Create a box around the portion of the curve you want to display by clicking on the mouse and dragging it. While dragging, a box is created. Once the box is around the desired area, double click inside the box. Both X and Y axes of the active curve are automatically rescaled. If the Radar window also is displayed while rescaling, its box is redrawn to reflect the new scaling.

   This action rescales the X axis for all curves and the Y axis of the active curve. All curves using the same Y axis as the active curve are also displayed on the new Y scale. All other curves are rescaled in the X direction only.
2. You can resize the existing box in the Radar window. The curve in the Data Analysis window will be rescaled accordingly.
3. Use the commands on the Display menu or the buttons in the Rescale Tools toolbar to rescale: Rescale X Axis, Rescale Y Axis, Full X Scale, Full Y Scale, Full Scale, and Previous Scale.

Shift Curves

You can shift any heat flow, baseline heat flow, or unsubtracted heat flow curve vertically along the Y axis using the **Shift Curve** button  on the Rescale Tools toolbar. This button is gray until a heat flow curve is displayed. Shifting a curve vertically makes it easier to see differences between curves that are overlapping. When you shift a curve, only the curve moves; the axes and scales do not change.

Select the curve you want to shift by making it the active curve, then select the **Shift Curve** button in the Rescale Tools toolbar. The Shift Curve dialog box appears and an **X** appears on the active curve.



The shift along the Y axis is defined by two points: the Shift From point and the Shift To point. The point at which the **X** intersects the active curve is the Shift From point. The Shift To point is the Y-axis value to which you want to move the Shift From point. Use the mouse to drag the **X** to the desired Shift From and Shift To points or type the values directly in the dialog box.

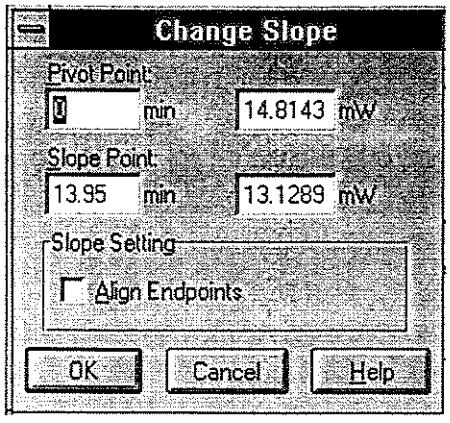
If you select the Align All check box, all curves in the active Data Analysis window that share the same Y axis will shift to the point on the Y axis defined by the **X**. This is a quick way to shift more than one curve.

Change the Curve's Slope

You can change the slope of any heat flow, baseline heat flow, or unsubtracted heat flow curve using the **Change Slope** button  on the Rescale Tools toolbar. When you change the slope of a curve, only the active curve is affected; the axes and scales do not change.

Select the curve whose slope you want to change, then select the **Change Slope** button  on the Rescale

Tools toolbar. The Change Slope dialog box appears:



A line representing the slope is placed 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**. Use the mouse to drag the pivot point and the slope point to the desired coordinates on the curve or type the X coordinates for each point directly in the Change Slope dialog box. The values in the dialog box update automatically when you move the pivot and slope point markers with the mouse. If you type the coordinates in the dialog box, the slope line on the curve does not update until you click on **OK**. The pivot point must always be to the left of the slope point.

Align Endpoints automatically assigns the slope point and the pivot point to the same Y value, forcing the slope line to be horizontal and the slope to be 0.

Annotate Curves

Another way to optimize the data curve display is to add informative and descriptive text labels. Either click on the **Annotate** button on the Rescale Tools toolbar or select **Annotate** from the Display menu to display the Annotations dialog box. The labels you add can be saved with the data or used for display only. You can create multiple labels for each curve. You can reposition, edit (e.g., change its font, color, and orientation), and delete an annotation.

Top

Math, Calc, and Curves Menus

You can display additional curves based on the data represented by the active curve in a Data Analysis window by using the items in the Curves, Math, and Calc menus. The new curve is added to the active Data Analysis window. You can also display other curves in the Instrument Viewer.

The data saved from a run includes analyzer signals pertaining to certain parameters. For example, the sample and program temperatures are saved for all analyzers. The heat flow is saved for DSC analyzers. Weight and weight % are saved for the TGA analyzers. Probe position, static and dynamic forces, frequency, amplitude, and phase angle are saved by the DMA 7e. In addition, for the DMA /TMA analyzers, other parameters such as expansion coefficient, volumetric expansion, and modulus are also saved and can be displayed as curves in the Data Analysis window. Delta T is saved for the DTA 7. These analyzer signal curves are on the Curves menu.

In the Data Analysis application for all analyzers, you can display curves that are calculated using the data in the active curve by selecting a command from the Math or Calc menu. The calculated curve is added to the window and becomes the active curve. You can perform such calculations as obtaining the derivative, adding curves together, subtracting, averaging, smoothing, peak area, glass transition etc. You can also smooth and normalize the focused curve.

You can display data from one or more method steps rather than all of the data in the active curve by selecting the **Step Select** command in the Curves menu. When you select this command, a checkmark appears next to the command in the menu. As long the checkmark is displayed, Step Select is "on." Each time you select a command in the Curves menu to display a different curve, the Step Select dialog box appears. Click on the **Select All** button to highlight and select all of the steps.

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 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.

In some context-sensitive help topics there may be a button at the bottom of the topic. Click on this to display a "how to" topic related to the current topic. For example, if the current topic is Temperature Calibration for the TGA 6, this button will connect you to the topic in Hardware Help that discusses exactly how to perform a temperature calibration for a TGA 6.

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.

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.

> 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.

Standard Window Components

The standard window components are as follows:

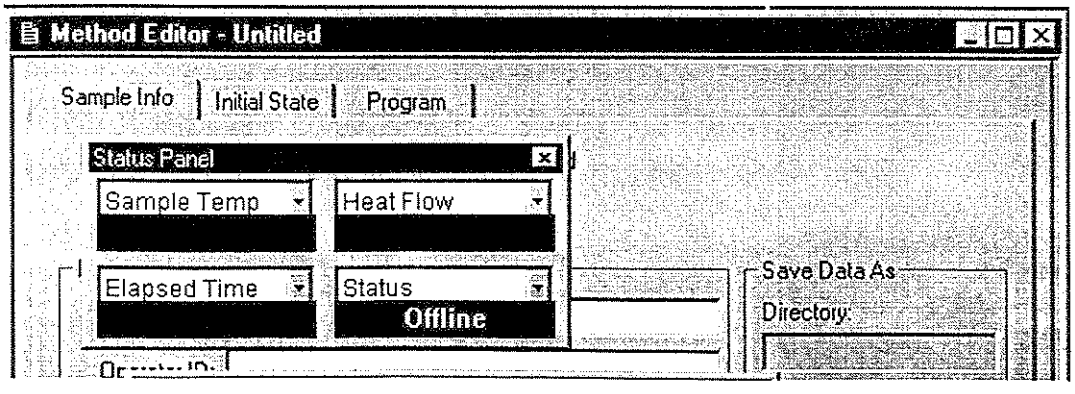
- Title Bar
- Status Bar
- Maximize Button
- Minimize Button
- Vertical Scroll Bar
- Horizontal Scroll Bar
- Left Window Border
- Right Window Border
- Top Window Border
- Top Left Window Border
- Top Right Window Border
- Bottom Window Border
- Bottom Left Window Border
- Bottom Right Window Border
- Print Preview Toolbar
- Context-Sensitive Help Button
- Control Menu

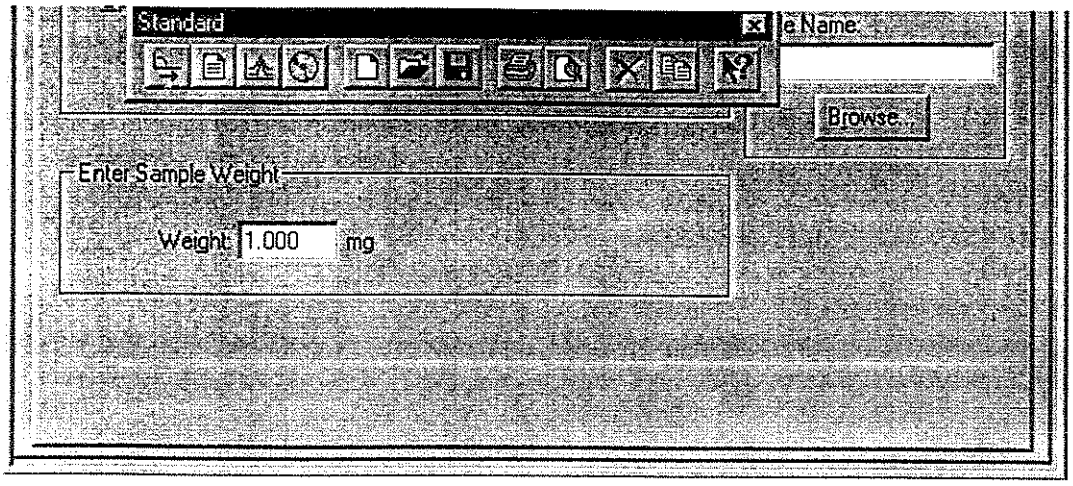
Customizing Pyris

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.

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. The control panel can be attached to the left or right side of the Pyris window. When a toolbar, the status panel, or the control panel floats, it 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.

Customizing the Curves Display

Pyris Software for Windows supports right-click menus in the graphics displays, i.e., Instrument Viewer and Data Analysis. You can customize the display using the items available on these menus.

Using the Pyris Manager

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.

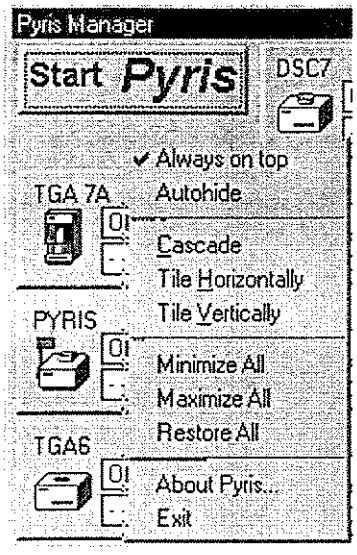
To open the Pyris Manager

Double click on Pyris Manager in the Pyris Software for Windows folder in the Programs menu which is accessed via the Windows **Start** button. A shortcut to the Pyris Manager may be on the desktop and/or in the Start menu if either or both of these options were selected during installation.

Displaying the Pyris Manager

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.

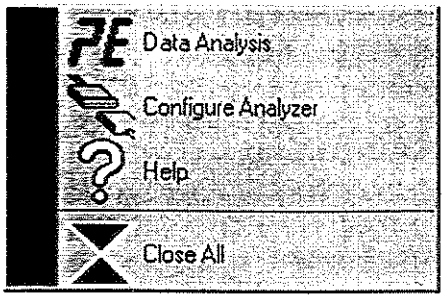
Place the cursor within the Pyris Manager and click on the right mouse button to display the Pyris Manager popup menu:



- Select **Always on top** to keep the Pyris Manager available for display. How it behaves depends on whether Autohide is activated. When Always on top is activated, a checkmark appears next to item.
- Select **Autohide** to have the Pyris Manager "hide" when in Pyris or another application such a word processor. Always on top must be selected. When both are activated, the Pyris Manager will roll off of the screen so that it does not take up room on your display but it is always available. For example, if it is displayed horizontally and moved to the bottom of the screen and you move the cursor up into the screen, the Pyris Manager will roll off of the screen. Bring the cursor back to the bottom of the screen, by the Windows taskbar, and Pyris Manager will reappear. If you select Autohide when Always on top is **not** selected, the Pyris Manager will close automatically when you select an Instrument Application. When activated, a checkmark appears next to the item in the popup menu.
- You can change the display of all your open Pyris windows by selecting Cascade, Tile Horizontally, or Tile Vertically. These are the standard Windows features. Selecting any of these options does affect non-Pyris windows.
- You can minimize all open Pyris windows to the taskbar at the bottom of the screen by clicking on **Minimize All**. Redisplay in maximum size all Pyris windows that are indicated on the taskbar by clicking on **Maximize All** in the popup menu. **Restore All** restores all Pyris windows to their original position and size before they were minimized or maximized.

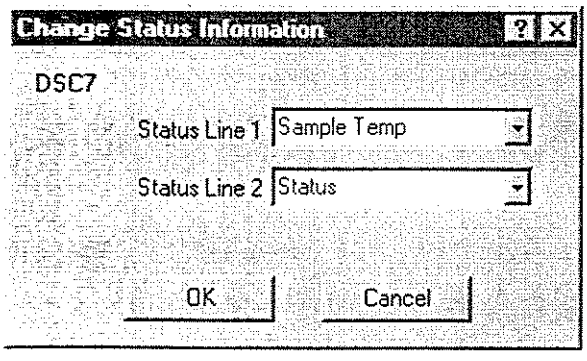
Features of the Pyris Manager

The Pyris Manager contains a **Start Pyris** button and a button for each configured instrument on your system. 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 Task menu:



- Access Data Analysis Applications from here rather than from the Programs menu.
- Open and run Pyris Configuration to dynamically add an instrument to or remove an instrument from the system. Each configured thermal analyzer is represented by a button on the Pyris Manager.
- Open online Pyris Help by clicking on Help.
- Close all Pyris windows via Close All.

The instrument buttons on the Pyris Manager are used to enter the Instrument Application. They are also used to monitor the status of the instrument. If you place the cursor within the button but not in a display field and click on the right mouse button, the Change Status Information dialog box appears:



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.

Quick Help

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.

Pyris 1 DSC Configuration

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.

Hardware Help

DSC 6 Configuration

The DSC 6 Configuration dialog box appears when you select the **Edit** button in the Pyris Configuration dialog box when the DSC 6 analyzer is highlighted in the Analyzers list. It is also displayed when there is a DSC 6 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 6, 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 DSC 6, the only accessory is for purge gas. Click on the drop-down arrow to display the available choices:

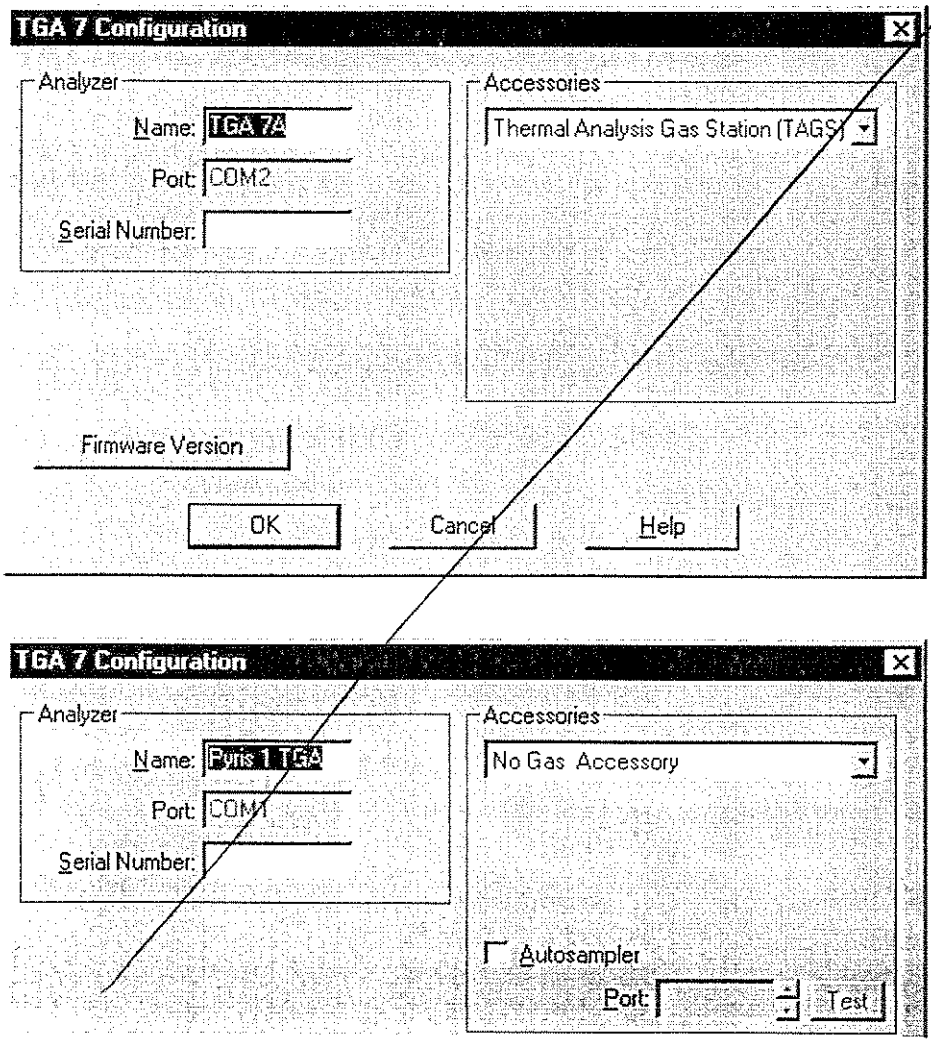
- **Thermal Analysis Gas Station (TAGS):** Select this accessory if a TAGS is attached to your analyzer. The TAGS can control up to 4 purge gases. Selection of this accessory is reflected in the Gas Change box on the Program page of the Method Editor. The gas program can have the TAGS switch from one 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:



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

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DSC 6 Instrument Application

The main parts of the DSC 6 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 DSC 6 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.

Method Editor

The DSC 6 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. The Method Editor comprises the following pages, each of which contains its own set of parameters:

- Sample Info Page
- Initial State Page
- Program Page

Another feature of the Method Editor is the Thermal Program Window.

Data Analysis

The Data Analysis window is opened one of two ways: First, you can select Data Analysis from the **Start Pyris** 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 DSC 6 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 sselect 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 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, **Minimize** and **Maximize** buttons, and a border that can be used for resizing.

Pyris Player

Pyris Player is used to create play lists or sequences of commands that automate data collection and postrun data analysis functions. You can create a play list that prompts you through the steps necessary ro 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. Pyris Player's Sample Group feature, which includes a Sample List and a Data Analysis List, simplifies creation of a play list. For each Sample line in the Sample List, the program will pause in order for you to load or remove the sample since it does not detect an autosampler. The Sample List is a good way to group similar samples together to be analyzed by the same method. 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 Analysi in a play list.

While a play list is running, you can perform other functions on your computer or pause the play list to edit entries below the current line. A complete history file is generated that contains the record of success or failure of 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 Lists in the current play list.

See the following topics for more details on the parts of Pyris Player:

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

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TGA 7 Instrument Application

To learn more about the main parts of the TGA 7 Instrument Application, see each of the topics below:

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 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**.

Quick Help | DSC 7 Hardware Help

DSC 6 Calibration

There are two calibration routines for a DSC 6:

- **Temperature**
- **Heat Flow**

It is recommended that a baseline correction be performed before temperature calibration.

The DSC 6 analyzer has been calibrated at the factory for both temperature and heat flow. Under normal conditions, the DSC 6 does not need temperature recalibration. Temperature calibration and heat flow calibration should be checked using the precrimped samples of indium and zinc before obtaining data. Some conditions that could affect the current calibration of the DSC 6:

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 DSC 6 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 DSC 6, 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 6. 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 6 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.

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 6 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.

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.

Running Samples

In Pyris Software for Windows, each analyzer's Instrument Application contains a Method Editor, Control Panel, Instrument Viewer, and Status Panel. To run a sample using any analyzer, each of these parts of the software is used.

Using the Pyris Player feature of Pyris Software for Windows, you can create play lists that automate data collection and analysis for an analyzer with an autosampler. Play lists can also be created for analyzers without autosamplers. These lists would automate postrun data analysis on existing data files.

In general, the procedure for a typical experiment includes the following steps:

1. Start the Pyris system.
2. From the Pyris Manager, start the specific analyzer's Instrument Application.
3. In the Method Editor, select an existing method or create a new method.
4. Load the sample in the analyzer.
5. Start the run from the Control Panel.
6. Monitor the run's data collection in the Instrument Viewer and using the Status Panel.
7. Save the data.
8. Perform postrun analysis.

A play list for an analyzer with an autosampler can automate steps 3 through 8. All you need to do is prepare the sampler, place samples in the sample tray, set up the play list, and start the run.

See how parts of the Pyris software are involved in running samples:

- **Methods**
- **Pyris Player**
- **Control Panel**

Quick Help

Methods

A method is a set of parameters that determine how the system will collect data on a given sample. Any number of methods can be generated and stored so that the parameters can be optimized to handle different analyses. A default method is included with Pyris Software for Windows. You can edit this method and then save it under a new method name.

Methods are created and managed in the Method Editor. The Method Editor can be opened in both an Instrument Application and the Data Analysis Application by selecting the **Method Editor** button in the toolbar (). In an Instrument Application, you can edit only those methods that control that instrument. In the Data Analysis Application, you can edit a method for any instrument attached to your system. You can load a method for another instrument type while in Data Analysis by using the **Open Method** command from the File menu. In the Open Method dialog box, select the instrument type from the List Files Type box and then select a file from the list of available methods.

Methods can also be part of a play list created with Pyris Player. Just as with individual runs, the methods in the list contain the information necessary to run the sample that has been loaded into the sample holder via a Load Sample entry in the play list.

The Method Editor contains three tabbed pages in which you enter information needed for the sample run. The parameters displayed on each page depend on the analyzer. The three pages of the Method Editor are

Sample Info Page

This page contains information about the sample to be run such as operator ID, sample name, weight (for TGA analyzers), dimensions (for DMA/TMA analyzers), measuring system and geometry of the sample (DMA/TMA analyzers), and location and name of the file in which to save the run's data. If you do not fill a location and file name, Pyris will use the defaults entered in the Save page of Preferences.

Initial State Page

This page contains the settings to be used at the start of the run such as temperature, forces, force controls, purge gas selection, equilibration, and baseline file.

Program Page

This page is where you set up the temperature and time program and the gas program for your sample run. Some information displayed on this page reflects entries made on the Initial State page. The main section of this page is Method Steps. You add, insert, or delete individual steps to build the temperature and time program. Steps are selected from a drop-down menu. The method steps are listed in order as you create the program. For a DDSC, when you select DDSC Repeated Scan step, the entire program created up to that point is replaced by the Repeated Scan step. For a DMA, if you select a Frequency, Dynamic Force, or Static Force Scan step, the entire program is replaced by that step. You can also create a program and then select Repeat Steps from the menu and have the program repeated. The page also contains scan rate, end conditions, and the purge gas program.

The method's file name (or "Untitled" if the method has not been saved), the sample ID, and the data's file name are displayed at the top of each page of the Method Editor.

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, and View History.

Setup Page

If you open Pyris Player while in Data Analysis, the Setup page allows you to select which instrument you would like to control with the player. If you open Pyris Player while in Instrument Application, the Player is used for the current instrument only.

Edit Play List Page

This page is where you create and edit play lists. The list is displayed in a format like that used in Windows Explorer. The folders are Prepare Sample, Sample Group, and Data Analysis. These folders contain items such as Load Sample, Start Method, Pause, or Display Curve. Sample Group contains two sections: sample list and data analysis list. If a folder item has a plus sign next to it, click on the plus sign to display the folder's contents; the plus sign changes to a minus sign. Click on the minus sign and the item folds up its list. An item's relation to a folder and the folder's relation to the entire list is shown by dotted lines. Use the **Add a step**, **Insert a step**, and **Delete this step** buttons to create and edit the play list.

View List Page

This page displays the play list in a manner similar to that shown in the Edit Play List page, i.e., use the "+" and "–" signs to open and close parts of the list. Since the page does not have the Edit Steps section, there is more room for displaying the play list. The View List Page is for viewing purposes only.

View Sample List

This page displays in spreadsheet format the list of samples in a sample group in the current play list. The columns in the spreadsheet are sample ID, file name, method used, carousel location of the sample, and the sample's weight.

View History Page

This page displays the history of the playback of the play list. Each time the play list is run, the system records the success or failure of each step. The play list is displayed in the same format as in the Edit Play List and View Play List pages except that the "+" and the "–" signs are replaced with other symbols that indicate if the item was completed successfully. A history file can be deleted from the list.

View Sample History Page

This page is the same spreadsheet as seen in View Sample List except for the additional column before Sample ID. This column contains a code that indicates the status of the sample's run: O = OK, S = Skip, and X = Error.

When you enter the Pyris Player, the standard toolbar is replaced by the Pyris Player standard toolbar and a

control bar.

Top

Quick Help

Control Panel

Each Instrument Application window contains a Control Panel on the right side. This panel is dockable and its display can be toggled on and off from the View menu. It contains buttons that directly control parameters specific to the analyzer. Some parameters can be set in both the Method Editor and the Control Panel. A change in one place is reflected in the other. Use the Control Panel to start and stop a run, change the furnace temperature, change the purge gas, zero the weight, zero the height, apply forces, hold the probe, or hold the temperature.

See the topics below to learn about the Control Panel for each analyzer:

- **Pyris 1 DSC Control Panel**
- **DSC 7 Control Panel**
- **DSC 6 Control Panel**
- **TGA 7 Control Panel**
- **TGA 6 Control Panel**
- **DMA 7e Control Panel**
- **TMA 7 Control Panel**
- **DTA 7 Control Panel**
- **Pyris 1 TGA Control Panel**

Monitoring Data Collection

While a method is running, the data collected is presented in graphical form in the Instrument Viewer. You can also see the status of selected instrument parameters or signals as curves if Monitor is activated. The status of the instrument's parameters is seen as well in the Status Panel. If you have multiple analyzers running, you can use the instrument buttons on the Pyris Manager to monitor the status of each analyzer while you do other work.

Instrument Viewer

The Instrument Viewer is a graphical window that displays the collected data in real-time during the run, as well as the status of selected analyzer signals in graphical form. For example, for a DMA 7e, you can display the Probe Position, Sample Temperature, Dynamic Force, and other parameters as a function of time. When a method is not running, the Instrument Viewer can display real-time baseline signals from the analyzer. You select what signals to display from the Curves menu. You can remove a curve from the display by selecting the curve and then pressing the **Shift + Delete** keys simultaneously. Stop the display of all selected curves in the Instrument Viewer by selecting **Monitor** in the View menu or the **Reset Monitor** button on the control panel. You can change the color, line style, and plot type of the curve by right-clicking on the curve and selecting the appropriate option from the popup menu displayed. You can change the axes labels and titles by right-clicking near the X or Y axis and selecting the option from the popup menu. You can toggle the grid lines on and off by right-clicking within the Instrument Viewer and selecting the option.

You can use the rescaling tools on the Rescale Tools toolbar or the rescaling options in the Display menu to redisplay the selected analyzer information and the real-time data.

Status Panel

The Status Panel is a dockable panel that appears in each Instrument Application window and Data Analysis

window. It is made up of boxes each of which displays a parameter and its current value, e.g., Probe Position for a DMA 7e. Select the parameter to be displayed in the box from a drop-down list of parameters. As soon as you select a parameter, its current value is displayed. You can also highlight the display field and type in the first letter of a parameter. For example, if you using a DTA 7 and type in "f" in a Status Panel box, one possible parameter that can be displayed is Furnace Lock. Type "f" again and Furnace Cover or Furnace Status will be displayed.

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.

You can hide the Status Panel to allow more room for other windows that are displayed beneath the Status Panel, e.g., the Initial State page in the Method Editor. Select Status Panel from the View menu to toggle the panel off.

Click here to see an example of the Status Panel while a run is in progress.

Using the Monitor

You can monitor the status of an analyzer in the Instrument Viewer before the start of a run by selecting the Monitor command in the View menu. The Monitor command toggles the monitoring feature on and off. A checkmark appears next to the command in the View menu when the Monitor is on.

You can select what to monitor in the Instrument Viewer by selecting an item from the Curves menu. For example, for the DMA 7e you can display curves for probe position, static and dynamic forces, frequency, sample and program temperatures, amplitude, and phase angle. You can remove a curve from the monitor by selecting it and pressing **Shift + Delete** simultaneously. The Y-axis label is also removed.

Select **Auto-Rescale** in the Display menu to have Pyris scale the curve automatically. If you want to rescale the curve, use the Rescale Tools toolbar or the items in the View menu.

To clear the data in the Instrument Viewer, select the **Reset Monitor** button  in the Control Panel. The Instrument Viewer will clear and the Monitor will restart.

Pyris Manager

You can use the Pyris Manager to monitor the status of multiple analyzers. Go to the Pyris Manager and display the Pyris Manager popup menu by placing the cursor in the background area of the bar and clicking the right mouse button. Select Always On Top to keep the Pyris Manager bar displayed on top of all open windows. Reposition the Pyris Manager to a more convenient place on the screen by clicking and dragging.

Each analyzer button on the Pyris Manager has two status lines. The default items for display are temperature and analyzer status. When there is no run in progress, the first status line displays "Offline." You can change the items displayed in these lines by using the Change Status Information dialog box.

The color of the LED in the upper-right-hand corner of each button indicates the status of the analyzer as follows:

Green: analyzer is on but no method is running

Red: analyzer is heating (method is running)

Yellow: analyzer is holding (method is running)

Blue: analyzer is cooling (method is running)

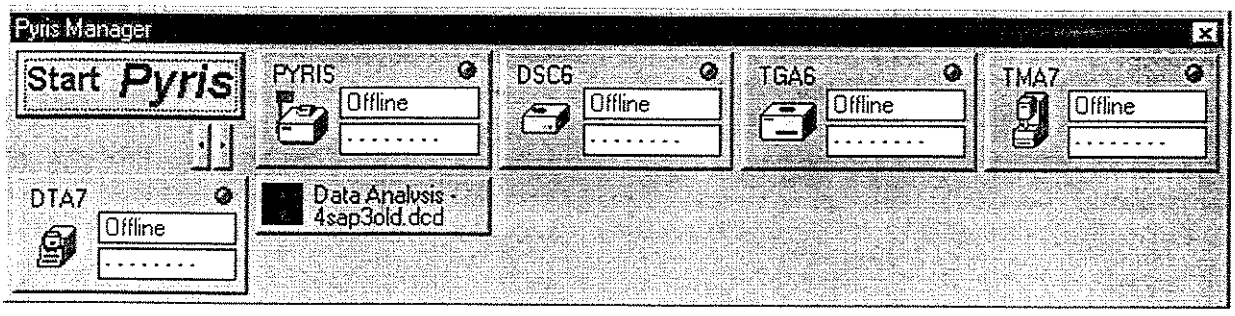
Dark Gray: analyzer is off

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.

Display Curves

In order to analyze your data that has been collected in a run and saved to a data file, you must first display it in a Data Analysis window. More than one curve can be displayed in a window and more than one Data Analysis window can be open at the same time.

Select the Data Analysis Application

After a run, click on the **Data Analysis** button on the toolbar  to open Data Analysis. This Data Analysis Application is associated with the particular Instrument Application you are using. You can also open a Data Analysis Application for another analyzer by selecting Data Analysis from the Pyris Manager Task menu.

Select the Data File

In the Open Data File dialog box, select a data file for display. The curve is displayed as it was last displayed before closing the file.

Use the Menus

Use the items in the View menu, Display menu, and Rescale Tools toolbar to change the display of the data. Use items in the Curves, Math, and Calc menus to display different curves and manipulate the data. Use the File menu items to add a curve to the display, open another window, or close the window.

Optimizing the Data Display

A curve displayed in the Instrument Viewer and the Data Analysis windows can be optimized a number of ways, mainly by using the rescale tools on the Rescale Tools toolbar. Some of those tools are also on menus.

Display Language

Select the language in which the LED 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 DSC 6 Data Sheet shipped with the instrument. Enter the value from the sheet here.

Heat Flow Conversion

The heat flow conversion (or calorimetric sensitivity) is calculated at the factory and is supplied on the DSC 6 Data Sheet shipped with the instrument. Enter the value from the sheet here.

Environment

Cooling Device

Select the cooling device attached to your DSC 6 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.

High Range

Sets the full-scale range of Y values (weight in milligrams) to 0 – 1300 mg.

Ordinate Filter Factor

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 PE 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.

Liquid Nitrogen in Use

When using the Liquid Nitrogen Subambient Accessory or the CryoFill LN2 Cooling System for cooling, select this box.

DSC 6 Instrument Page

The DSC 6 Instrument page appears when you select the DSC 6 tab in the Preferences window. The DSC 6 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 DSC 6 to the beginning temperature or the next program step or to selected isothermal temperatures in the temperature program.

Data

Viewing Methods and Results

After displaying, optimizing, and performing calculations on the data curves, you can view and printout those results in several ways.

View Results

When you perform calculations, the results are not saved unless you save the data file. When a data file with calculations is recalled, the results are not automatically displayed on the screen. Use the **Results** command in the View menu to display the calculation results. When you select this command, the View Results dialog box appears. All of the calculation results saved with the active curve or performed during the current session and which can be displayed are listed. Select one or more results, then select the **View Results** button. The calculations whose results cannot be displayed on the current axes are listed separately.

Use the **Delete Results** button in the View Results dialog box to delete calculation results from the active curve's data file or results obtained during the current session, e.g., the Van't Hoff curve for purity. The results deleted from the dialog box list will be deleted from the data file only when you save the file.

Printing Curves

Use the **Print** command or the **Print** button on the toolbar to print the active Data Analysis window. Before printing you can use **Print Setup** in the File menu to set up the page(s) on which the output will be printed. The Page Setup dialog box contains three tabbed pages in which you define a title, margins, and header and a footer for your printout. To see what your printout will look like before printing it, select the **Print Preview** command in the File menu or the **Print Preview** button on the toolbar.

When you select the **Print** command, the Print dialog box appears. Select the printer and the number of copies to print, or select the **Properties** button to display the Print Properties dialog box. In the Print Properties dialog box, you can access a number of different print options, including whether to print in Landscape or Portrait mode.

View Methods

You can view information about the method used to collect the data displayed in the Data Analysis window. Make the desired data curve the active curve, then select the **Method Used** command in the View menu. The View Method Properties window appears. This window comprises six pages which display the following information:

- **Sample information**
- **Initial conditions**
- **Equilibration parameters**
- **Method step information**
- **Validation Information**
- **Calibration information**

You can change such parameters as the Sample ID, Operator ID, and Comment for your data file. The other information is for display only; it cannot be changed.

Using the **Create** button on any of the pages, you can save the method as an ASCII file with the same file name as the method but with a .TXT extension.

Print Methods

There are two ways to print a method:

- With the Method Editor as the active window, use the **Print** command or the **Print** button on the toolbar to print the method currently displayed in the Method Editor.
- With a Data Analysis window as the active window, select the **Method Used** command in the View menu. Click on the Print button on any of the 6 pages of the View Method Properties window .

To see what your printout will look like before printing it, select the **Print Preview** command in the File menu or the **Print Preview** button on the toolbar.

Copy Methods

You can export your methods from Pyris Software for Windows by copying them to an ASCII file and then using the file in third-party software. Use the **Method Used** command in the Data Analysis window View menu to perform this function. When you select this command, the View Method Properties window appears. Select the **Create** button to create the ASCII text file. The file has the same file name as the method but has a .TXT extension. It is saved in the same directory as the method.

Include the data points collected in the run and/or the Calibration Information by selecting the appropriate check box before creating the ASCII file. The data points are saved as an ASCII table of values.

Saving Data

To save what is displayed in the Data Analysis window , select the **Save** button in the toolbar or **Save Data** from the File menu. The derived curves currently displayed in the Data Analysis window along with the calculation results stored with these curves and the original data and method are saved. When you open the data file again, the data will be displayed as you saved it. Use **Save Data As** from the File menu to save the data under a different file name. The original data file still exists.

If you delete a curve from the display and then save the data, the curve and all results calculated on that curve are not retrievable.

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. Also see the following topics:

- Emergency Repair Disk
- Security
- Instrument Communication
- Long File Names

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