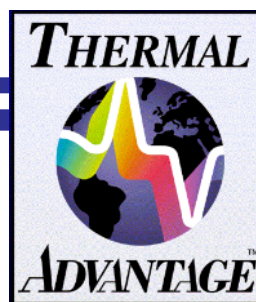


Thermal Advantage



User Reference Guide

© 1999, 2000 by TA Instruments
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New Castle, DE 19720

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Temperature Calibration Type	2-25
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Notes, Cautions, and Warnings

The following conventions are used throughout this guide to point out items of importance to you as you read through the instructions.

A NOTE highlights important information about equipment or procedures.



CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.



WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Chapter 1

Getting Started with Instrument Control

Overview

Before you begin this section, we recommend that you familiarize yourself with the basic operation of the Windows NT® system. Refer to the documentation for the operating system.

This chapter provides you with a brief explanation of Instrument Control and general guidelines for setting up an experiment. For further details refer to the online help that accompanies the *Thermal Advantage*™ software.

Definition of Thermal Analysis

Thermal Analysis is defined as the measurement of changes in chemical or physical properties of a sample as a function of temperature or time in a controlled atmosphere.

In 1887 thermal analysis was introduced by the French scientist H. Le Chatelier during his study of the decomposition of clays using heat. The early attempts to characterize materials by Le Chatelier involved making photographic records of the heat emitted by the clays, then classifying the clays according to their heating patterns. It was thus that thermal analysis came to be used to identify materials such as minerals, clays, and metallic alloys.

General Applications

Thermal analysis is now used as an analytical tool to measure the following physical and reactive properties of substances based on temperature:

- Thermal decomposition of solids and liquids
- Solid-solid and solid-gas chemical reactions
- Material specification, purity, and identification
- Inorganic solid material adsorption
- Phase transitions.

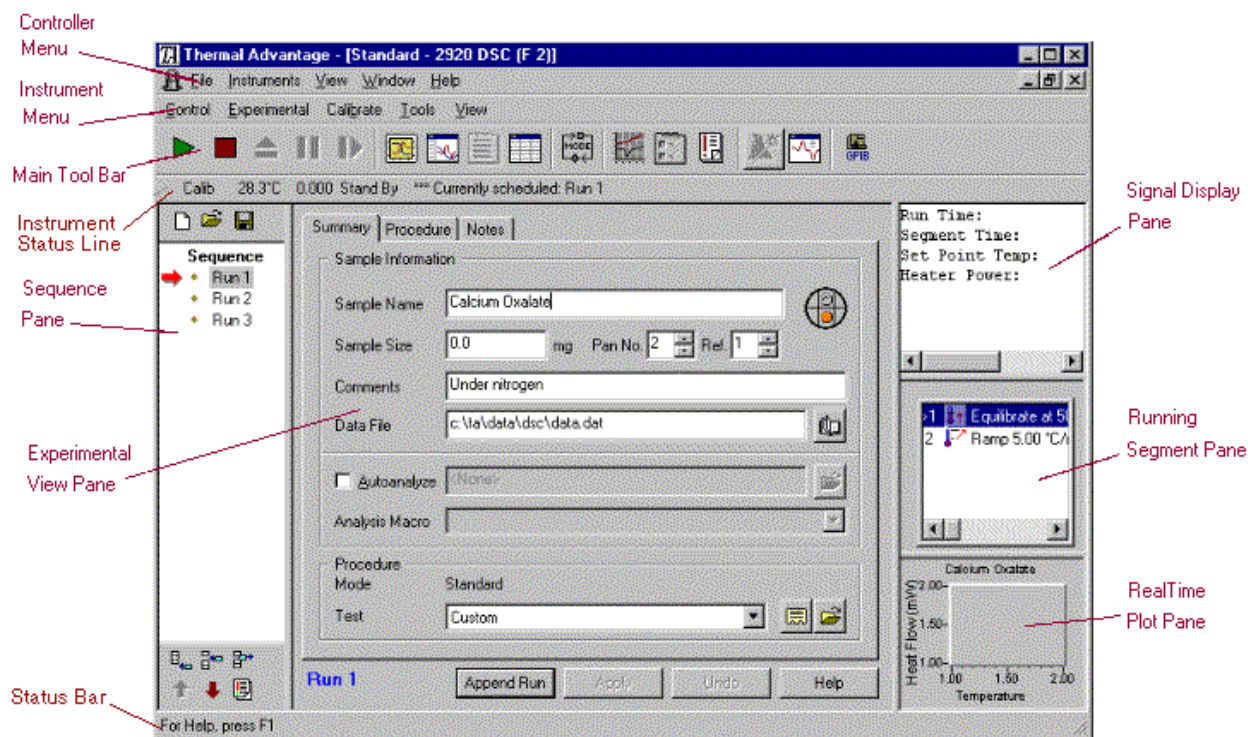
Using this information, the scientist can characterize polymers, organic or inorganic chemicals, metals, or other common classes of materials.

Introducing *Advantage*™


Thermal Advantage is a thermal analysis instrument control program that operates the following TA Instruments units:

- DSC (Differential Scanning Calorimeter)
- High-Temperature DTA (Differential Thermal Analyzer)
- SDT (Simultaneous Differential Technique) Analyzer
- TGA (Thermogravimetric Analyzer)
- DMA (Dynamic Mechanical Analyzer)
- TMA (Thermomechanical Analyzer)
- DEA (Dielectric Analyzer)

The first thing that you may notice when you open the *Advantage* program for Windows NT is that you can see more information displayed on your monitor (as seen in the figure below). TA Instruments has designed the program's opening window to allow you to view the progress of your experiment as the data is plotted by the RealTime Plot Pane,



even while you are setting up a new experiment using the Experiment View. You can also track the working of the instrument by the Signal Display Pane and determine which segment is in progress by looking at the Running Segment Pane. A tool bar is available to help you quickly perform certain functions.

At first all of this information may seem complicated, but you will soon become familiar with the displayed panes and be able to effectively use the many options that are available with *Advantage*TM. You can even bypass some of the more detailed testing options and use the instrument's Wizard  to easily and accurately set up and run experiments.

The opening window for the *Advantage*TM software (shown on the left-hand page) displays the main controller window with cascaded windows for each instrument that is online with the controller inside the main window. Each instrument window is basically divided into 3 sections:

- The left pane displays the sequence (or run) list. The middle pane is used to display either the Experimental, Table, or RealTime Plot views.
- The top right side pane contains the signal display information and the middle right pane contains the running method information.
- The lower right pane shows a thumbnail view of the realtime plot information when either Experimental or Table View is displayed.

NOTE: User Preference options may alter this basic layout.

You can right click with the cursor inside a pane to display a pop-up menu specific to that pane, if one is available.

The basic function of each part of the window is described as follows:



- The **Controller menu** is used for overall control of the instruments online (e.g., reconfiguring, overview status, etc.)
- Each instrument window contains an Instrument menu that is specific to that



instrument and a **Main Tool Bar** (DSC shown here) to control and set up experiments.

- The **Sequence Pane**, shown to the right, (available in multi-run mode only) displays the list of runs that are contained in the current sequence. Up to ten runs may be preprogrammed (up to 65 for autosampler instruments). The run with the arrow next to it is the scheduled run (*i.e.*, the run that will begin when Start is selected). Buttons are available in this pane to make changes to the run order in the sequence.



- When you use the **Experimental View Pane** (shown to the right), you can set up and edit sample information and the procedure that will be used for each run contained in the sequence. This pane includes the ability to set up your own custom experiments or use the available preprogrammed tests.

Summary A | Summary B | Procedure | Notes

Sample A Information

Sample Name: Calcium Oxalate

Sample Size: 0.0000 mg Pan No. 2 Ref. 1

Comments: Under nitrogen

Data File: c:\ta\data\dsc\data.dbj

☒ Autoanalyze C:\Program Files\TA Instruments\Thermal Solutions\Wi

Analysis Macro: Autqueue 1

Procedure Mode: Standard

Test: Isothermal

Run 1 Append Run Apply Undo Help

- To view the instrument's output signals, use the **Signal Display Pane** (left figure below). A pop-up menu is available by right-clicking inside this pane. This menu is used to zero or offset the signals.

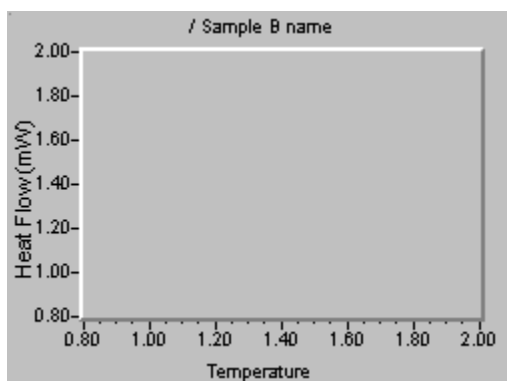
Run Time:	0.00	min
Segment Time:	0.00	min
Set Point Temp:	0.00	°C
Heater Power:	0.00	watts

- When a run is in progress, you can see which step the

1	Equilibrate at 400.00 °C
2	Ramp 20.00 °C/min to 1300.00 °C
3	Mark end of cycle 0
4	Ramp 10.00 °C/min to 400.00 °C
5	Mark end of cycle 0
6	Repeat segment 2 for 4 times

instrument is processing in the procedure by viewing the **Running Segment Pane** (above). A pop-up menu is available by right-clicking inside this pane. This menu allows you to modify the running procedure.

- You can watch the data points as they are collected from the instrument during a run by looking at the **RealTime Plot Pane** (shown to the left). A pop-up menu is available by right-clicking inside this pane. This menu allows you to customize the RealTime Plot display.



Reconfiguring Instruments Online

Use the **Instruments/Reconfigure** menu item to bring online all of the instruments that are powered up and connected to the controller.

If you want to install new instrument software, you will need to use the **Reconfigure** function to perform this function. See “Loading Instrument Software” below for information.

Loading Instrument Software

To properly configure the instruments with the controller, you will need to load new instrument software now and periodically thereafter, as new updates are received. Use the SCROLL key on the instrument keypad, if available, to access and view the software version currently loaded in your instrument. Check this against the latest version. If you need to update the software, follow the directions below.

NOTE: Instruments that do not have the proper version of the software will not configure with the controller.

This procedure will return the instruments to their default settings (for Experimental, Advanced, and Post-Test Parameters). The method(s) stored in the instrument will be lost, along with the following calibration parameters:

- Temperature Calibration Table* (exception: DSC 2920)
- Cell Calibration* for DSC 2910/2010 and TMA 2940
- Tare for TGA 2950/2050 and SDT 2960
- DTA Baseline Calibration* for SDT 2960
- Probe Calibration for TMA 2940
- Clamp Calibration for DMA 2980
- Sensor Calibration* for DEA 2970

* You can preserve these calibrations by writing them down before downloading the new instrument software, then reentering the values.

1. Check that the instruments are connected to the controller properly, and that their POWER switches are turned on.
2. Press and hold the STOP key on the instrument keypad while you press the instrument Reset button or turn the instrument on. (See the instrument operator’s manual for their location.)

3. Continue holding the STOP key until the instrument display reads “Module loader Vx.x” or the Ready light comes on. The instrument will go offline with the controller, if it was previously online.
4. Start the Instrument Control program. (If the Instrument Control window is already open, select **Instruments/Reconfigure.**)
5. Select **Start**. Downloading of the instrument software will begin as a window is displayed tracking the progress. When the downloading has been completed, the instrument will go offline and a message will appear.
6. Select OK to clear the message.
7. Repeat steps 1 through 6 for each instrument and then reconfigure the instruments online with the controller.

NOTE: It is normal to see an Error 15 on the instrument after new instrument software has been loaded. Press the STOP key on the instrument keypad to clear the error.

Setting Up Program Preferences

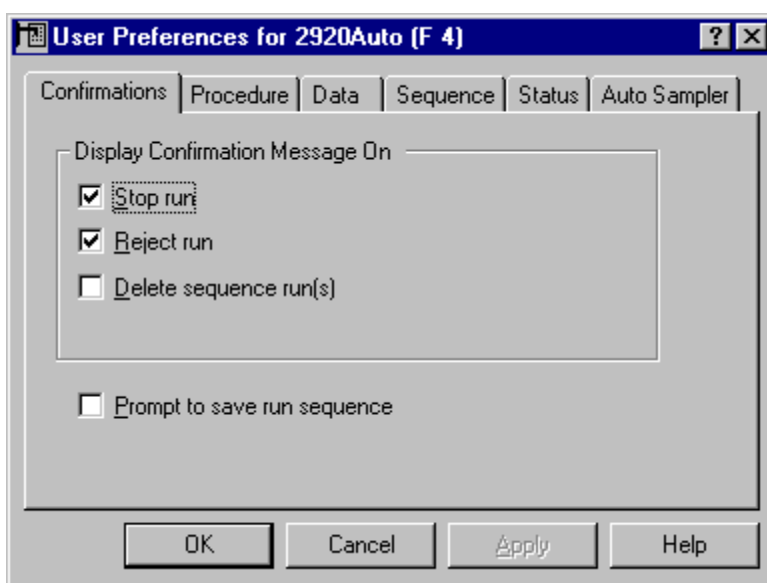
There are several programming options that you can use to set up the way the *Advantage* program looks and operates during experiments. The topics in this section will give you the information that you need to set up the program and customize its appearance.

Selecting Confirmation Message Display

Use the **Tools/User Preferences/Confirmations Page** (shown here) to turn on or off a confirmation message that is displayed each time one of the actions shown is performed. To switch to a different page, click the tab at the top of the page.

Choose from the following options:

- **Confirm on Stop Run:** Select this option to have a confirmation message displayed each time Stop is chosen from the Control menu or Tool bar.
- **Confirm on Reject Run:** Select this option to have a confirmation message displayed each time Reject is chosen from the Control menu or Tool bar.
- **Confirm on Delete Sequence Run(s):** Select this option to have a confirmation message displayed each time you delete a run from a sequence (either in Table View or from the Sequence Pane).
- **Prompt to Save Run Sequence:** Select this option to have a prompt message displayed each time you create a new run sequence or open a previously saved run sequence to allow you to save the run sequence. When this message is displayed, you can save the run sequence by selecting Yes in response to the prompt. A Save Sequence window is displayed to allow you to name and save the sequence file.

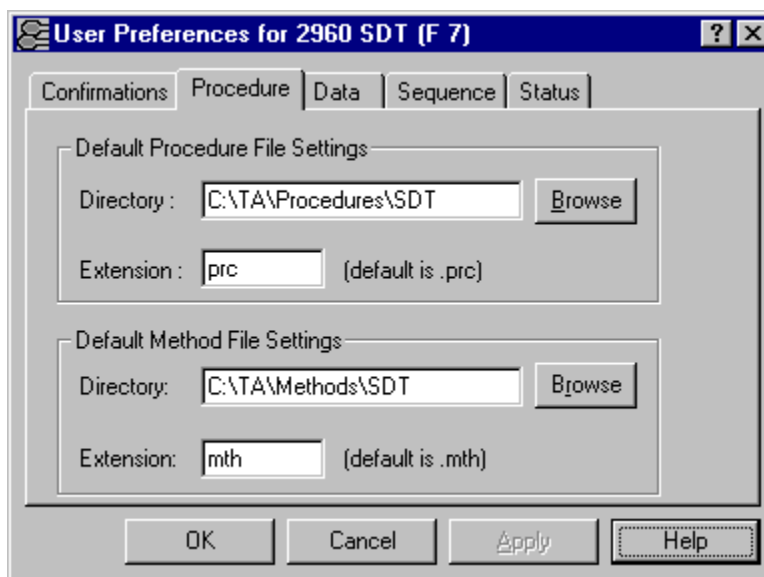


Choosing a Procedure File Location

Use the **Tools/User Preferences/Procedure Page** (SDT shown here) to choose default settings that will be applied to the handling of procedures and method files. To switch to a different page, click the tab at the top of the page.

To use this page follow these steps for both procedure and method files:

1. Enter or use the Browse button to locate a default directory that you want to use to save your files. If the directory that you enter does not exist, you will be prompted to create a new directory.
2. Enter an alphabetic or numeric extension to be used as the default extension for the procedure files saved (up to three characters may be used). The default extension is .prc for procedure and .mth for method files.
3. Click the Apply or OK button when finished.

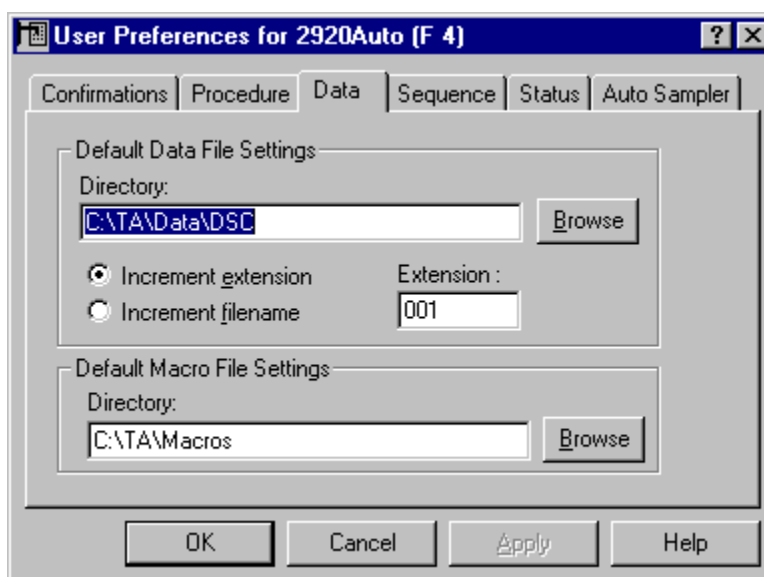


Choosing Data and Macro File Locations

Use the **Tools/User Preferences/Data Page** (shown here) to customize program settings related to data files. To switch to a different page, click the tab at the top of the page.

To use this page follow these steps:

1. Use the Browse button to locate a directory. If the directory that you enter does not exist, you will be prompted to create a new



directory. This directory will be used when the program is first started. Once the program has started, any data file path that you select after that will be recalled and used. Example: C:\TA\Data\DSC

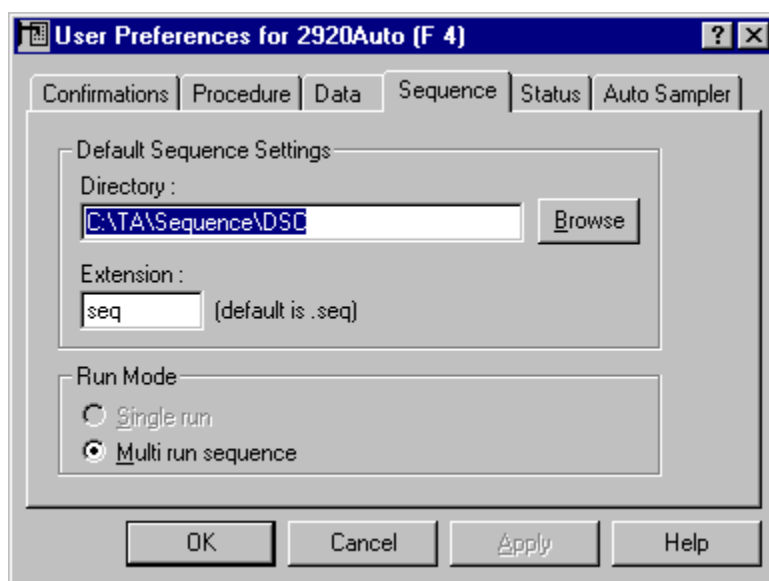
2. Select a radio button to increment the filename (e.g., data1.dat, data2.dat, etc.) or extension (e.g., data.001, data.002, etc). The data filename or extension is automatically incremented when **Append Run** is used or when an existing filename is entered, to limit the chance of a duplicate filename or extension. The characters of the selected choice are changed numerically or alphabetically depending on character type.
3. Enter a default extension that will be used if you do not specify an extension when saving data files. If an extension is not specified for a data filename, the default extension will be used. The default extension is .001.
4. Use the Browse button to locate a default directory that you want to use for your macro files that will be used for Autoanalysis and Universal Analysis. Macros that are used for autoanalysis can be selected from the macro file directory chosen here. Example: C:\TA\Macros
5. Select Apply or OK to save settings.

Selecting the Default Sequence Settings

Use the **Tools/User Preferences/Sequence Page** (shown here) to customize program settings related to sequence files. To switch to a different page, click the tab at the top of the page.

To use this page follow these steps:

1. Enter or use the Browse button to locate a default directory that you want to use to save your sequence files. If the directory that you enter does not exist, you will be prompted to create a new directory. The default sequence path is TA\Sequence\DSC.
2. Enter an alphabetic or numeric extension to be used as the default extension for the sequence files saved (up to three characters may be used). The default extension is .seq.



3. *Non-autosampler instruments only:* (Autosamplers always default to multiple run mode.) Click on one of the following Run Mode radio buttons to determine the instrument control program's operating mode:

- **Single Run Mode:** When you select the Single run mode, the instrument control program for the current instrument will operate handling one run (experiment) at a time. The Sequence pane is not displayed and only one run can be created and started at a time. When the run is completed, another run can be created and started. The Table View option is not available in this mode.
- **Multi-Run Mode:** When you select the Multiple run mode, the instrument control program for the current instrument will handle up to ten runs in a sequence file at a time. The Sequence pane is displayed on the left side of the window. More than one run can be created and placed in the list with other runs. You can set up a maximum of ten runs (all of them must have the same instrument mode) for execution. The run arrow displayed next to the run number indicates the scheduled run, which will be executed when Start is selected.

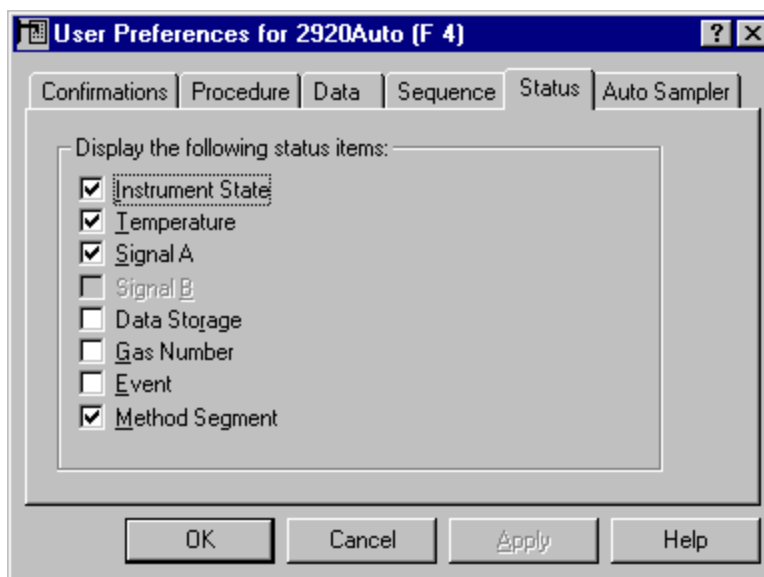
4. Click the Apply or OK button when finished.

Customizing the Instrument Status Line

Use the **Tools/User Preferences/Status Page** (shown here) to customize the status line display. The parameters selected will be displayed on the status line on the main instrument control window. The status line is the text that appears under the tool bar (or under instrument menu, if the tool bar is not displayed). To switch to a different page, click the tab at the top of the page.

To set up the status line, follow these instructions:

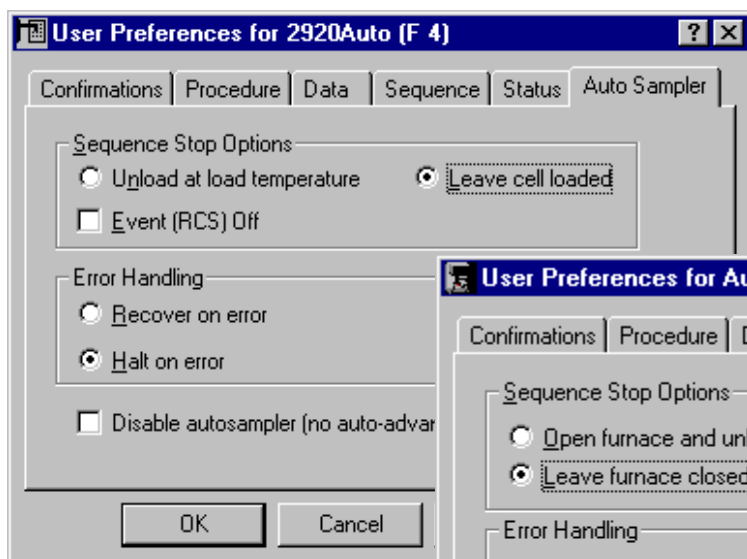
1. Select the check box next to each desired Display Option.
2. Uncheck any status parameter that you do not want displayed.
3. Select Apply or OK to save the settings.



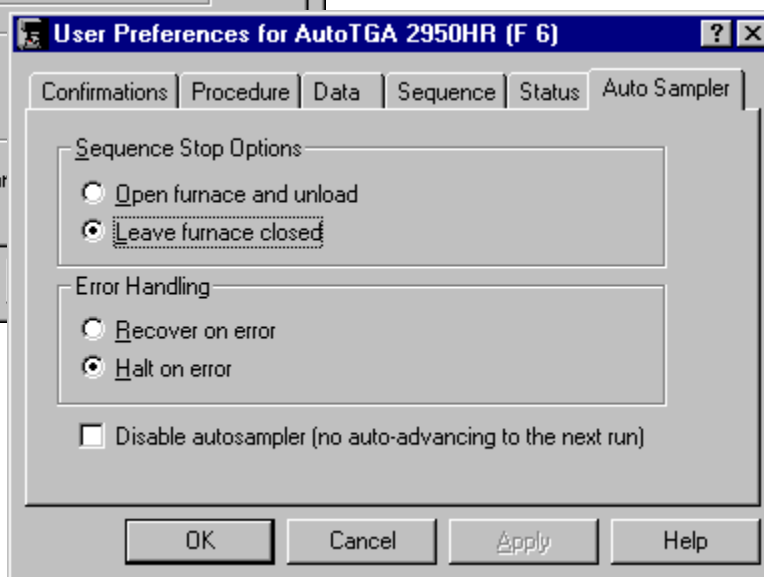
Status Item	Description
Instrument State Status	Displays the current state of the instrument on the Status Line. Example: "Heating," "Cooling," and "Temp."
Temperature Status	Displays the current temperature of the instrument on the Status Line. Example: 156.34°C
NOTE: An asterisk (*) will appear next to the temperature, if a temperature calibration table is active.	
Signal A Status	Signal A refers to the primary (first stored) signal in the data file. This option displays the Signal A value on the Status Line.
Signal B Status	Signal B refers to the secondary (second stored) signal in the data, where applicable. If there is no Signal B, this selection is grayed out. This option displays the Signal B value on the Status Line (when available).
Data Storage Flag Status	Displays the state of data storage, "store off" or "store on," on the Status Line.
Gas Number Status	Displays the current gas, "Gas 1" or "Gas 2" on the Status Line. Automatic gas switching between Gas 1 and 2 is activated through the Gas Switching Accessory (GSA). The default gas number is 1.
Event Status	Displays the status of the event relay (off=open, on=closed) connected to the instrument through the Event connector, on the Status Line.
Method Segment Status	Displays the method segment that is currently running on the Status Line. Example: 5: Ramp 5°C/min

Configuring the Autosampler Items (DSC and TGA Autosamplers only)

Use the **Tools/User Preferences/Autosampler Page(s)** to set up the actions of the DSC or TGA Autosampler. The stop options instruct the autosampler on the action to take when the run sequence has been stopped or runs to completion. The error handling options determine whether the autosampler will keep going or halt when a nonfatal error is encountered. Use the last checkbox on this page to use the instrument as an autosampler or as a single sample unit. To switch to a different page, click the tab at the top of the page.



DSC Autosampler Page



TGA Autosampler Page

Follow the instructions on the next several pages to configure the autosampler settings. When finished select **Apply** or **OK** to save the settings.

DSC Autosampler Sequence Stop Options

The Sequence Stop Options offers these actions that can occur when the DSC Autosampler run sequence is stopped or runs to completion:

1. Select the desired radio button to determine the actions of the autosampler:
 - **Unload at load temperature:** This option will cause the DSC Autosampler to unload the pans and remove the lids at the end of the successful run sequence, once the cell is within the specified load temperature range. In the event of an error condition, where the sequence is prematurely stopped, the cell will be left in that current state.
 - **Leave cell loaded:** If you choose this option, the DSC Autosampler will not unload the cell or remove the lids when the run sequence stops.
2. Check or uncheck the **Event (RCS) Off** box to control the state of an external device (Refrigerated Cooling System--RCS) connected to the instrument with the event cable at the end of the Autosampler run sequence.
 - Check the box to turn the event device (RCS) off at the end of the last autosampler run in the sequence. This allows you to leave the RCS running during all of the methods in the run sequence, but turn it off at the end of the run sequence.
 - Uncheck the box (the default condition) to leave the event device (RCS) in the state it was in at the end of the run sequence. If the RCS was on at the end of the run sequence, it will remain on, if this box is left unchecked.

NOTE: In the event that the run sequence is stopped due to an error, the RCS will automatically be turned off, regardless of your selection, to prevent frosting.

TGA Autosampler Sequence Stop Options

The Sequence Stop Options offers one of two options that could occur when the TGA Autosampler run sequence is stopped or runs to completion:

Select the desired radio button to determine the actions of the autosampler:

- **Open furnace and unload:** The Open furnace and unload option will cause the TGA Autosampler to open the furnace and unload the sample pan at the end of the run sequence.
- **Leave furnace closed:** If you choose the Leave furnace closed option, the TGA Autosampler will not unload the sample pan when the run sequence stops, and the furnace will remain closed.

Error Handling

Select the radio button next to the desired setting to turn it on:

- **Recover on error:** When you choose this setting (the default setting), the autosampler attempts to continue operation if it encounters a recoverable, nonfatal error condition. It will make several attempts to correct the error and, if it is unsuccessful, the Autosampler will skip the current run and go on to the next one. An entry of the error will be added to the error log. This setting is recommended for unattended runs.
- **Halt on error:** When you choose the Halt on error setting, the autosampler suspends all further operation when it encounters any error condition. When an error occurs, you must first correct the error, then continue operation from that point. An entry of the error is added to the error log.

Disabling the Autosampler

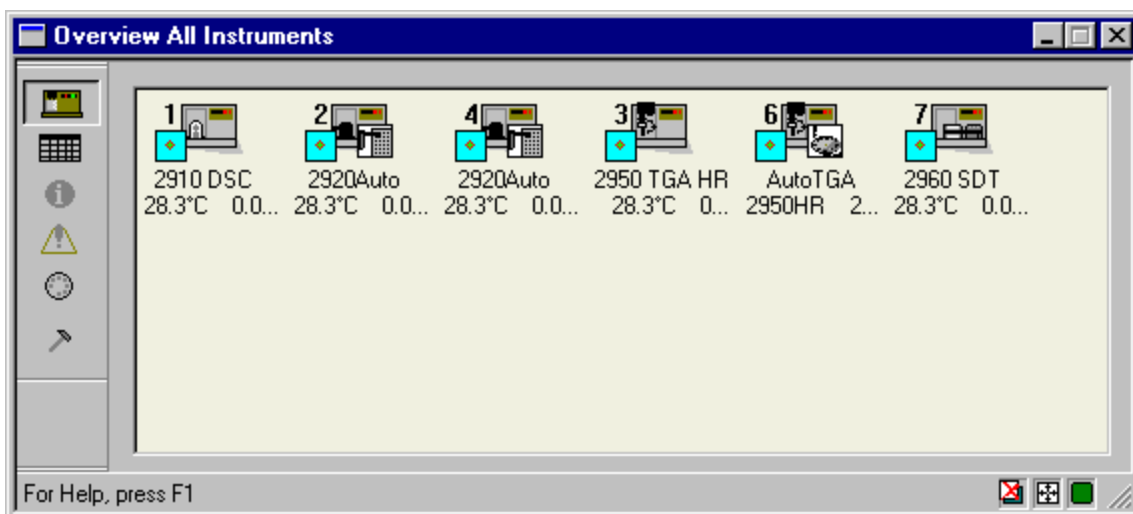
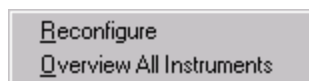
Check **Disable autosampler** on the **User Preferences - Autosampler Page**, if you want the autosampler to work as a single sample instrument. The instrument will not automatically advance to the next run. If this option is unchecked (default condition), the autosampler will operate as usual.

Choosing Your Viewing Options


You can control the appearance of your program by specifically selecting the view options that you want to display. There are several viewing options possible with the *Advantage*™ program. Two **View** menus are available, one in the **Controller** menu and one in the instrument menu. These viewing options are detailed in the following sections.

Observing an Overview of All Instruments

When you have several instruments connected to a controller, you may want to be able to see what is happening with each of the instruments. The *Advantage* program offers a way to observe all of the instruments at once by selecting **Instruments/Overview All Instruments** (shown here) from the **Controller** Menu. This opens a window (shown below) that displays the most current information regarding all of the instruments that are powered up and online with the controller. Use this window to view various types of current information about the instruments that are powered up and online with the controller.



Use the icon buttons on the left side of the Overview window to choose from the following types of information to view:

-  **View Instruments:** This is the default option. If the icon view of the instruments and their temperatures is not displayed, click the button to open the icon view. The icon view displays each instrument online, its GPIB address, instrument type, current temperature, signal value, and instrument state. The instrument state is represented by a small icon.

-  **View Status:** This button displays a table (shown below) of the information such as that listed below:

Instrument: Name of each instrument powered up and online with the controller.

Instrument State: Calib(rating), Standby, Complete, Reject, Setup, Ready, Hold, or Running

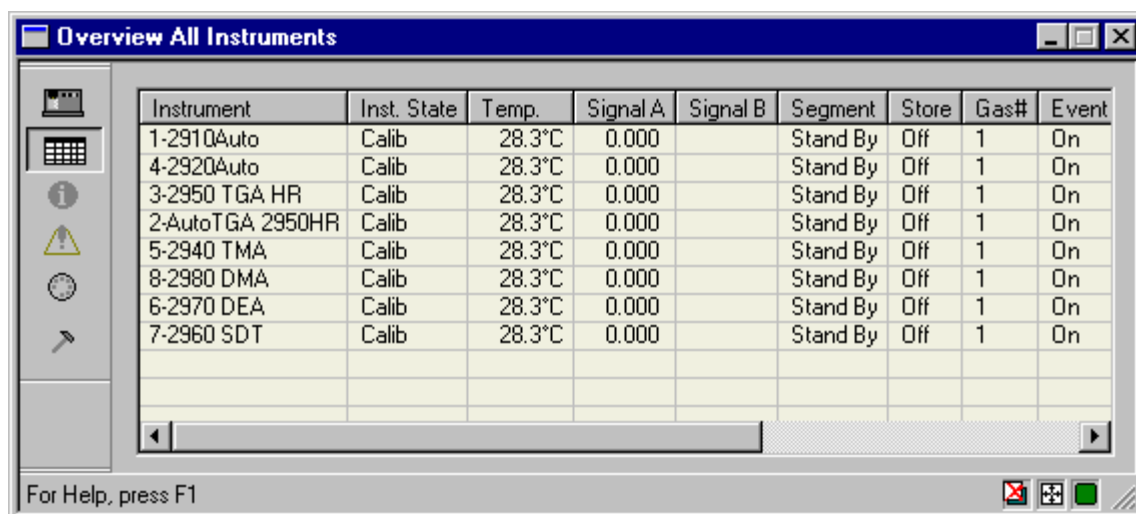
Temperature: Current temperature in degrees C.

Signal A: Current signal A value

Signal B: Current signal B value

Segment: Current method segment being executed.


State of Controls, etc.



The screenshot shows a software window titled "Overview All Instruments". On the left is a vertical toolbar with icons for a printer, a table (selected), an information icon, a warning icon, a clock, and a magnifying glass. The main area contains a table with the following data:

Instrument	Inst. State	Temp.	Signal A	Signal B	Segment	Store	Gas#	Event
1-2910Auto	Calib	28.3°C	0.000		Stand By	Off	1	On
4-2920Auto	Calib	28.3°C	0.000		Stand By	Off	1	On
3-2950 TGA HR	Calib	28.3°C	0.000		Stand By	Off	1	On
2-AutoTGA 2950HR	Calib	28.3°C	0.000		Stand By	Off	1	On
5-2940 TMA	Calib	28.3°C	0.000		Stand By	Off	1	On
8-2980 DMA	Calib	28.3°C	0.000		Stand By	Off	1	On
6-2970 DEA	Calib	28.3°C	0.000		Stand By	Off	1	On
7-2960 SDT	Calib	28.3°C	0.000		Stand By	Off	1	On


At the bottom of the window, it says "For Help, press F1" and has standard window control buttons (minimize, maximize, close).


-  **View Information:** Click this button on the **Overview All Instruments** window to display a table containing the following information:

Instrument: Name of each instrument powered up and online with the controller.

Mode: Current instrument mode. To change the mode, select Experimental/Mode from the menu.

Instrument Version: Current instrument software version.

-  **View Errors:** Click this button on the **Overview All Instruments** window to display a list of current errors associated with each instrument. These errors are any that have occurred since the window was opened, since you started your instrument, or since the time the log was last cleared.

-  **View Autosampler Information:** Click this button on the **Overview All Instruments** window to display a list of information about any autosamplers that are powered up and online with the controller. The following information is provided:

Instrument: Name of each instrument powered up and online with the controller.

Autosampler State: Current state of the autosampler.


Run #: The run number that is currently being executed on the autosampler listed.

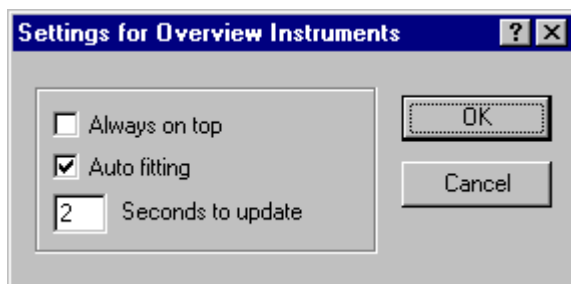
Autosampler Status: Current status of the autosampler.

Sample Name: The name of the sample currently being tested.

Sample B Name: The name of the second sample (for dual sample autosamplers) currently being tested.



-  **View Overview Settings:** Click this button on the Overview All Instruments window to open the **Settings for Overview Instruments** window (shown below). This window lets you choose from several different settings:



- **Always on Top:** Check this box to have the **Overview All Instruments** window displayed on top of any other open windows at all times. If left unchecked, the window will be displayed in the regular fashion.

— **Auto Fitting:** Check this box to have the information displayed on the **Overview All Instruments** window adjusted so that the column width is automatically adjusted to the appropriate size based on context.

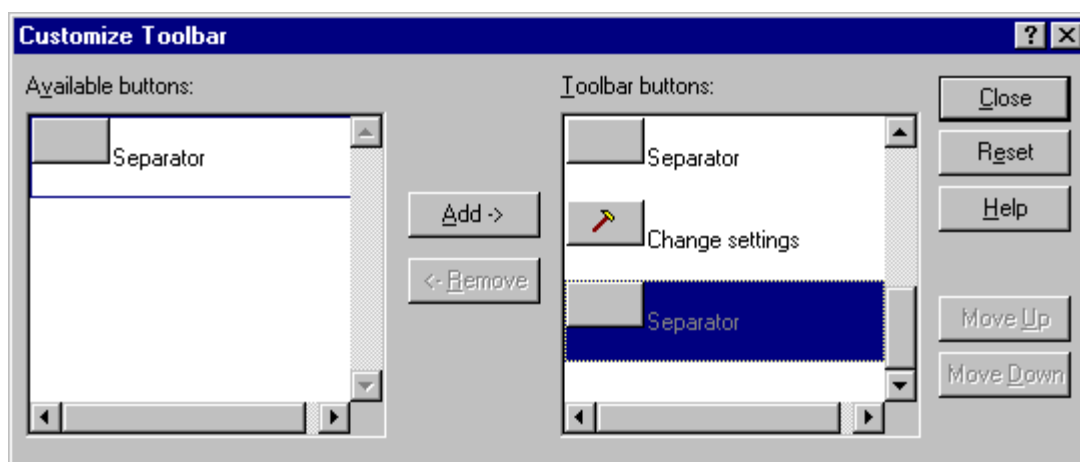
- **Seconds to Update:** Enter the amount of seconds that you want to elapse in between updating of the **Overview All Instruments** window, providing fresh information at the time interval entered.

You can also change these settings using the buttons on the right side of the status line as seen in the figure below. Move the mouse over the button to see the current setting. Double click on the button to change the setting.



Customizing the Overview Instruments Tool Bar

A drop-down menu (shown here) is available by clicking on the icon on the upper right corner of the **Overview All Instruments** window. Included with the standard menu items is the **Customize Tool Bar** function. When you select this option the **Customize Toolbar** window (shown here) is displayed. Use this window to customize the tool bar on the **Overview All Instruments** window.



To add a button to the tool bar:

1. Click on the button that you want to add from the list of "Available buttons."
2. Click the Add button. The button will be added to the list of "Tool bar buttons."
3. Click the Close button when finished.

To remove a button:

1. Click on the button that you want to remove from the list of "Tool bar buttons."
2. Click the Remove button. The button will be deleted from the list of "Tool bar buttons" and displayed in the list of "Available buttons."
3. Click the Close button when finished.

To change the order of the buttons:

1. Click on the button that you want to move in the list of "Tool bar buttons."

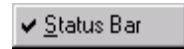
- Click either the Move Up or Move Down button to move the button one position up or down. Repeat this until the button has been moved to the desired position. (You can also drag-and-drop the button at its new location.)
- Click the Close button when finished.

To restore the program default tool bar buttons:

- Click the Reset button.
- Click the Close button when finished.

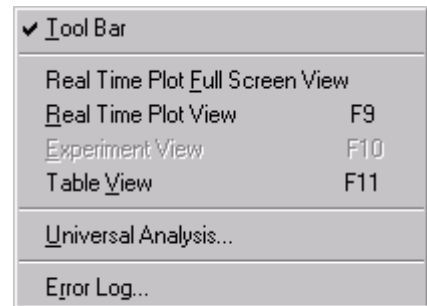
Controller Viewing Options


When you use the controller **View** menu (shown here), you can choose to show or cancel the status bar (at the bottom of the window. See page 1-2 for an illustration of these window parts. The status bar is displayed when you open the *Advantage*™ program for the first time (default condition). When the item is checked, it will be displayed. When the item is not checked, it will not be displayed.




Instrument Window Viewing Options


Each instrument has its own **View** menu (shown here) to control several viewing options. You can check or uncheck the **View/Tool Bar** option to turn the tool bar on or off. The middle four menu items control the four different window views, which are explained below, along with a brief explanation of the last two menu items.




- RealTime Plot Full Screen View:** Use the  button or select **View/Realtime Plot Full Screen View** from the menu to display the **RealTime Plot** window in full screen view. Only RealTime Plot is displayed. This is a good view to use when you have multiple instruments online and you want to tile the windows to view multiple plots at a glance.

- **RealTime Plot Pane View:** Use the  button or select **View/RealTime Plot View** from the menu to display the RealTime Plot window in the middle pane along with the Sequence Pane, Signal Display Pane, and Running Segment Pane.



NOTE: You can double click on the RealTime Plot Pane to automatically switch to the Experimental Pane View.

- **Experimental Pane View:** Use the  button, select **View/Experiment View** from the menu, or press F10 to display the Experimental Pane in the middle pane along with the Sequence Pane, Signal Display Pane, Running Segment Pane, and a small (thumbnail) view of RealTime Plot. When you switch to a different view, after making any changes to your experiment, the information is automatically applied.

NOTE: You can double click on the RealTime Plot thumbnail view to automatically expand it to full view.

- **Table Pane View:** Use the  button, select **View/Table View**, or press F11 to display the Table View Pane in the main pane along the Signal Display Pane, Running Segment Pane, and a small (thumbnail) view of RealTime Plot.

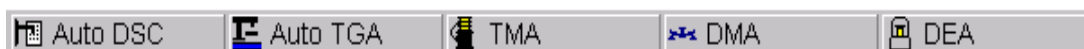
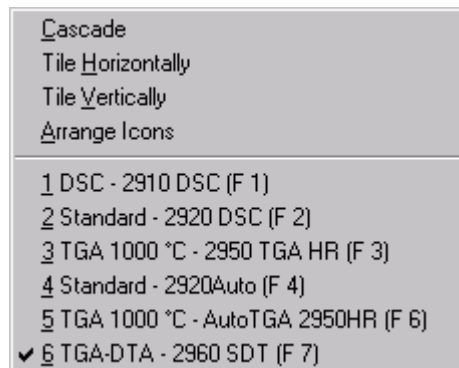
NOTE: You can resize the panes just like you resize windows, by pulling the sides or corners using the double-headed arrows. When you close the *Advantage* program, the views and pane sizes are automatically saved for future recall.

- **Universal Analysis View:** Use the  button or select **View/Universal Analysis** to open the *Thermal Advantage* Universal Analysis program. See the documentation accompanying this program for further information.
- **Error Log View:** Use the  button or select **View/Error Log** to open the Error Log window. See Chapter 6 for further information on the Error Log.

Switching Between Windows

There are basically three ways that you can switch between windows to move a particular instrument window to the forefront, which makes it the active window:

- You can use the **Window** menu to choose the desired numbered instrument submenu selection. Click on the window name desired. An example of the menu is shown here. The active instrument control window will be checked on the menu.
- You can move the cursor to any part of a window that may be showing behind the current window and click the mouse. The window you clicked on will become the active window.
- You can click on the desired taskbar button at the bottom of the window (shown below) to move that window to the forefront and make it the active window.



Chapter 2

Calibrating the Instruments

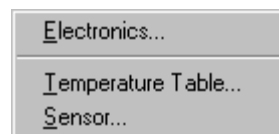
Overview

All thermal analysis instruments need to be calibrated on a certain schedule in order to maintain accurate results. Most of the calibration procedures can be performed through use of the individual instrument's **Calibrate** menu (or through some special calibration tool bar buttons). Since each instrument requires different calibration procedures, we have separated the information here by instrument. Choose the instrument and calibration procedure you are interested in from the list that follows:

- Calibrating the DEA — see pages 2-1 through 2-7
- Calibrating the DMA — see pages 2-8 through 2-22
- Calibrating the DSC and DTA — see pages 2-23 through 2-42
- Calibrating the SDT — see pages 2-43 through 2-64
- Calibrating the TGA — see pages 2-65 through 2-78
- Calibrating the TMA — see pages 2-79 through 2-91

Calibrating the DEA

Use the DEA **Calibrate** menu (shown here) to choose from three main types of calibration: electronics, temperature, and sensor.




Each DEA must be calibrated for accuracy, using the following basic guidelines.

- Perform **Sensor** calibration every time that you install a new ceramic parallel plate, ceramic single surface, or sputter coat sensor to obtain precise geometry and RTD values. When you install the remote single surface sensor, you need to enter the chip ID.
- Use the **Temperature Table** to input or reset temperature calibration data. Temperature calibration is not routinely performed with the DEA.
- Perform instrument **Electronics** calibration when you first receive the instrument, and at least once a month to calibrate the electronics on the analog board.

Refer to the following pages for the instructions needed for each type of DEA calibration.

Electronics Calibration

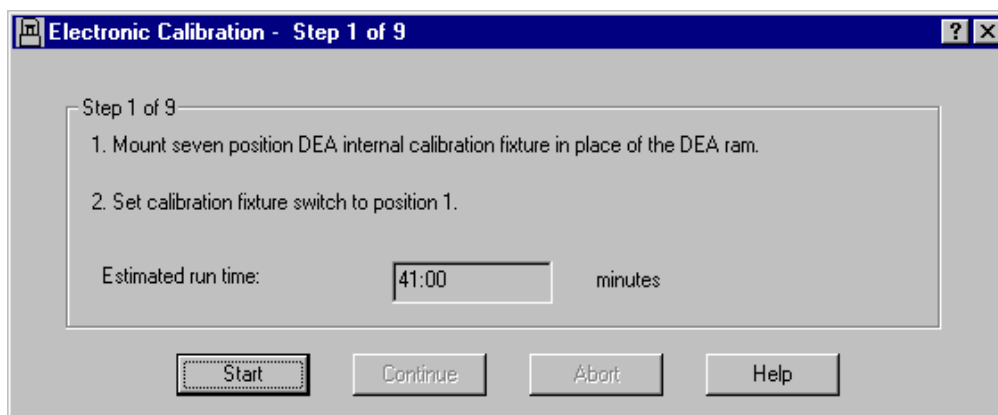
Select **Calibrate/Electronics** from the DEA menu or click the  button on the tool bar to perform the DEA electronics calibration procedures.

Electronics calibration is performed at least once a month to calibrate the electronics on the analog board. This is a nine-step procedure using two calibration fixtures. The approximate time needed to complete the electronics calibration is 2.5 hours.

NOTE: Calibration is particularly important with changing weather and laboratory conditions.

Before you can begin the software portion of the calibration, you will need to perform the following steps:

1. Remove the ram and any sensor(s) from the DEA instrument. Disconnect the remote interface cable from the instrument base, if applicable.
2. Install the seven-position DEA internal calibration fixture in place of the ram. Replace the bell jar and purge the enclosure at 0.5 L/min during each step of the calibration.
3. Select **Calibrate/Electronics** from the DEA menu, the first calibration window (shown here) is displayed.



NOTE: You must advance to each step while performing this calibration.

These steps are performed to calibrate each setting in turn.

4. Make sure that the seven-position DEA internal calibration fixture has been mounted in place of the DEA ram, as directed in the window.
5. Replace the bell jar and purge the enclosure at 0.5 L/min during each step of the calibration.
6. Turn the calibration fixture switch to the appropriate position. The **Estimated run time** for the entire procedure will be displayed. (The run time feature is provided for your convenience so that you can gauge the time needed to run this step and start the next step.)
7. Select Start.
8. When the calibration is finished ("Calibration Complete" is displayed in the title bar and the software will beep), select **Continue** to go on to the next step.
9. Repeat steps 5 through 9 for all seven positions, then remove the seven-position DEA calibration fixture.
10. Mount the two-position calibration fixture in position 8.
11. Select Start.
12. When the calibration is finished ("Calibration Complete" is displayed in the title bar), select **Continue** and mount the two-position calibration fixture in position 9.
13. Select Start.
14. When the calibration is finished ("Calibration Complete" is displayed in the title bar), select **Save Calibration**.



CAUTION: If you Abort (stop) the calibration, without completing all of the steps, the DEA will be uncalibrated and will not be able to function as it should. You must go back and start the electronic calibration from the beginning and complete all of the steps.

Sensor Calibration

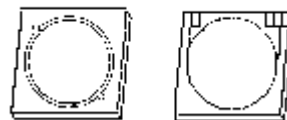
Use the Sensor calibration option to display a window that allows you to verify or enter the calibration values for a DEA sensor.

The sensors are interchangeable parts of the DEA instrument. They are the key to the DEA system, providing precise control in benchtop analysis of bulk sample properties and sample surface properties.

There are four types of sensors, which correspond to the DEA modes of operation:

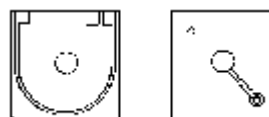
DEA Ceramic Parallel Plate

Used to evaluate bulk dielectric properties in a material.



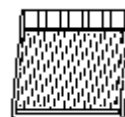
DEA Sputter Coated

Used to evaluate bulk dielectric properties in a thin sample material.



DEA Ceramic Single Surface

Used for surface property evaluations and curing experiments.



DEA Remote Single Surface

Used to evaluate changes in a material that occur during a cure.



For detailed information on the DEA sensors, refer to the instrument operator's manual.

NOTE: The information displayed in the Sensor Calibration window varies according to the instrument mode (sensor) selected. See "Using the Mode Functions" in Chapter 3 for instructions on how to change the sensor selection.

To perform sensor calibration, turn to the section that applies to the type of sensor you have installed on your instrument.

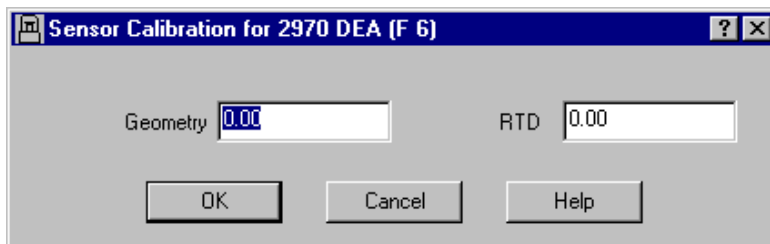
Sensor Calibration for Sputter Coated, Ceramic Single Surface, and Parallel Plate Modes

Use this procedure to calibrate the sensor in either the sputter coated, ceramic single surface, or parallel plate modes. Follow the instructions below.

1. Install the appropriate ram and sensor(s).
2. Place the bell jar on the instrument. (NOTE: Sensor calibration cannot be completed unless the bell jar is in place.)
3. Turn on the nitrogen purge and allow the DEA to purge for five minutes at a flow rate of 0.5 liters/min.
4. Select Sensor Calibrate on the DEA keypad. After approximately one minute, sensor calibration will be complete and the ram will open.

To display or edit the calibration values, follow these instructions:

1. Select **Calibrate/Sensor** on the DEA menu. The **Sensor Calibration** window (shown here) is displayed.
2. Verify (ceramic single surface or parallel plate modes) or enter (sputter coated mode) the Geometry.



NOTE: The geometry calibration value requires manual entry for the sputter coated sensor.

For the Sputter Coated Mode: Enter the sputter coated area on the sample (mask area). To obtain the value for your particular mask, measure the inside diameter of the mask to +/-0.01 mm. This value is used to calculate the geometry. For the sputter coat mask supplied, the geometry value is 340 sq.mm +/- 1 sq.mm.

For the Parallel Plate and Ceramic Single Surface Modes: The geometry is automatically calculated during sensor calibration.

3. Verify the RTD value, which is automatically calculated during sensor calibration.
4. Select OK.
5. Record the geometry and RTD values if you plan to reanalyze a sample after external pretreatment while the sample is on the sensor (e.g., ceramic single surface sensor).

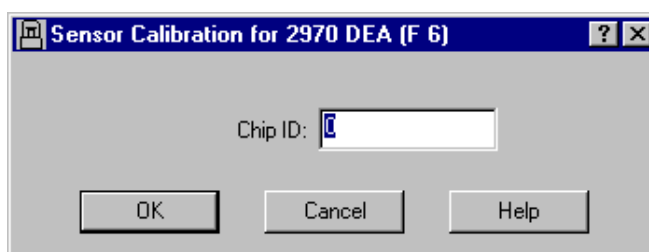
NOTE: The first time you calibrate the sensor, the plunger may not be correctly lined up with the furnace. If this happens, call TA Instruments for service.

NOTE: The calibration procedure is complete when the ram opens and the instrument status changes back to standby. Do not stop the calibration procedure before it is complete, or your experiment will run without the needed sensor calibration data.

Sensor Calibration for the Remote Single Surface Mode

Use this procedure to enter the sensor chip ID in the remote single surface mode. Follow the instructions below.

1. Select **Parameters/Sensor** on the main menu. The **Sensor Calibration** window is displayed.
2. Enter the **Chip ID** number, which is printed on the connector end of the ribbon cable of the remote single surface sensor (the numbers before the decimal point).
3. Select OK.

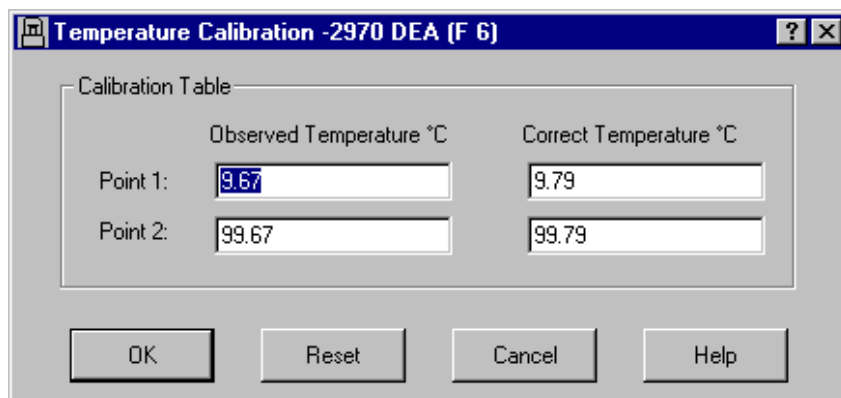


NOTE: Disconnect the remote interface cable from the instrument when it is not in use.

Using the DEA Temperature Table

Select **Calibrate/Temperature Table** to display the temperature table that the instrument applies to the collected data. Use this window (shown here) to enter from one to two temperature calibration points (pairs of observed and correct temperature points).

The observed and correct temperature corresponds to the experimental and theoretical transition temperature (e.g., melting point) of the calibrant respectively.



The image shows a software dialog box titled "Temperature Calibration -2970 DEA (F 6)". It contains a "Calibration Table" with two columns: "Observed Temperature °C" and "Correct Temperature °C". There are two rows for calibration points. Point 1 has values 9.67 and 9.79. Point 2 has values 99.67 and 99.79. At the bottom are four buttons: OK, Reset, Cancel, and Help.

	Observed Temperature °C	Correct Temperature °C
Point 1:	9.67	9.79
Point 2:	99.67	99.79

Temperature calibration is not routinely performed with the DEA.

NOTE: The Temperature Table is not available when the instrument is in the calibration mode.

NOTE:

A single-point calibration shifts the sample temperature by a constant amount.

A two-point calibration shifts the temperature with a linear correction (straight line) and projects this correction to temperatures above and below the two calibration points.

The Platinum RTDs used on the ceramic sensors are highly accurate and should not need any further calibration after the sensor has been calibrated.

- If these values are correct, select OK or Cancel.
- If new values are to be entered manually:
 1. Select the **Reset** button to remove all previously stored values.
 2. Determine the temperature calibration points.
 3. Enter the **Observed** and **Correct** temperature points in the table.
 4. Select OK when all points have been entered to save the settings to the instrument.

NOTE: When temperature calibration is in effect, Temp in °C appears in the status line. When it is not in effect, Temp in °F appears in the status line.

Calibrating the DMA

Use the **Calibrate** menu for the DMA (shown here) to choose from several types of calibration. Two of the types shown in the menu have submenus to choose from. This section provides step-by-step information on how to perform all of these calibrations.



Each DMA must be calibrated for accuracy, using the following basic guidelines.

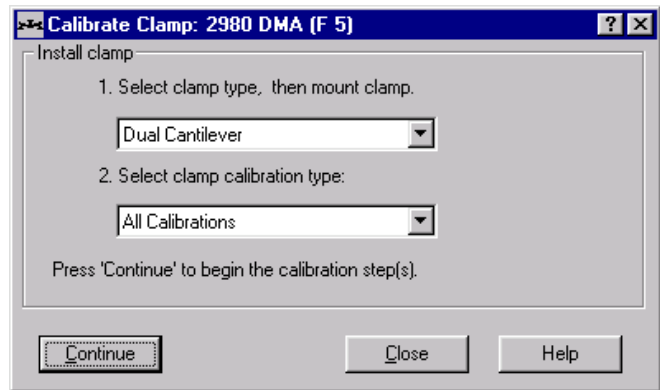
- **Clamp Calibration:** You need to perform the various Clamp Calibrations when you change from one clamp type to another.
- **Position Calibration:** You need to perform the Position Calibration when the DMA is moved or turned off, or at least once a month. The purpose of this calibration is to calibrate the absolute position of the drive shaft (and slide) as read by the optical encoder.
- **Instrument Calibrations:** You need to perform the following Instrument Calibrations under the following circumstances—when the instrument is moved, if the feed hose for the air cool or GCA is either removed or installed, or on a regular (monthly) basis.
 - Electronics calibration
 - Force calibration (balance and weight)
 - Dynamic calibration (mass, compliance, and analyze)
- **Calibration Reports:** You can obtain a report on clamp and instrument types of calibrations after you have performed the steps needed.
- **Temperature Calibration:** Temperature calibration should be done when you first receive the instrument, when the DMA is moved or turned off, or at least once a month. After you perform the calibration, the data is updated in the Temperature Table.

Refer to the following pages for the instructions needed for each type of DMA calibration.

Clamp Calibration

Select **Calibrate/Clamp** from the DMA instrument menu when you install a clamp for the first time or change from one clamp to another on the DMA 2980. When you change the clamp selection, you will be prompted to perform a clamp calibration. Follow these steps:

1. Select **Calibrate/Clamp** from the DMA menu. The introduction window (shown here) is displayed.
2. Select the clamp type from the drop-down list. Install the desired clamp (do not mount a sample).
3. Select the type of clamp calibration to be performed.

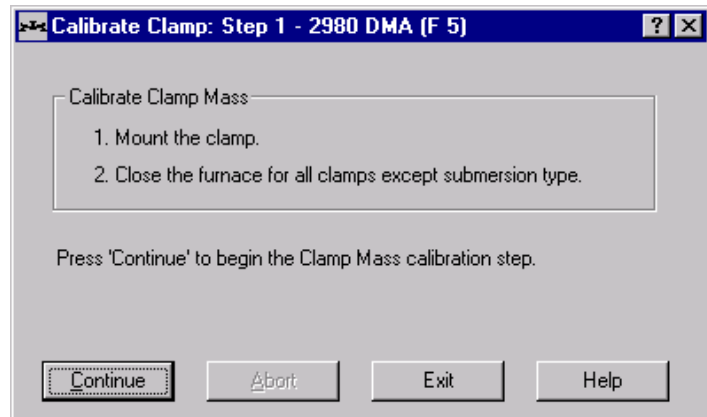


4. Click the Continue button to proceed. You will then perform the Clamp calibration, following the instructions displayed. The steps will lead you through the clamp mass, clamp zero, or compliance calibration procedures, depending on the clamp and calibration type(s) you have selected.

Clamp Calibration - Step 1 (Clamp Mass)

Clamp Mass calibration is performed to allow the instrument to compensate for the mass of a specific clamp. It is the first type of calibration to be performed when you either install a clamp for the very first time, or change from one clamp to another.

You can run a check to see if your clamp mass needs calibration by performing a small test. Press the FLOAT key to release (float) the clamp. Manually position the clamp in the middle of its range of travel and release it. The clamp should maintain its position or slowly drift up or down. If the clamp rapidly sinks or rises, the clamp mass requires calibration as described below:



1. Clear the clamp area on the instrument. If you are using a submersion type clamp, skip to step 3.

2. Press the FURNACE key to close the DMA furnace. (Do not close the furnace when submersion type clamp is installed.)
3. Press the **Continue** button on the Step 1 window to begin the clamp mass calibration step.

The furnace will open when the clamp mass calibration has completed. (It normally takes approximately seven minutes to complete the clamp mass calibration.) The next step that is displayed depends upon the type of clamp being calibrated—follow the steps appropriate to the window displayed.

NOTE: If the clamp continues to rapidly sink or rise instead of floating, after the clamp mass has been calibrated, you will need to perform a position calibration.

Clamp Calibration - Step 2

Clamp Zero calibration is needed to determine the point of zero sample length for automated sample length (tension) or thickness (compression and penetration) measurement. This step is performed when you install a film or fiber tension clamp, a penetration clamp, or a compression clamp. It is not performed when you install a single or dual cantilever clamp, a shear sandwich clamp, or a 3-point bending clamp.

NOTE: You can also perform the clamp zero calibration by pressing the ZERO CLAMP key on the instrument keypad.

For Film/Fiber Tension Clamps:

To zero the clamp for a film or fiber tension type of clamp, follow these steps:

1. Measure the offset gauge, found in the accessory kit, with calipers to determine its length.
2. Mount the offset gauge in the fixed clamp. If you are using a submersion type clamp, skip to step 4.
3. Press the FURNACE key to close the DMA furnace. (Do not close the furnace when a submersion type clamp is installed.)
4. Enter the length of the offset gauge (range of 5 to 30 mm) in the appropriate field.
5. Select **Continue**. The zero length point for the sample is determined to be the underside of the fixed clamp. The furnace will open when the clamp zero calibration has been completed.
6. Remove the offset gauge when the furnace opens.

For Compression or Penetration Clamps:

1. If your clamp needs a gauge block, enter its size in the gauge block length field. (These clamps normally do not use a gauge block, so this entry can be left unchanged at 0.0.) If you are using a submersion type clamp, skip to step 3.
2. Press the FURNACE key to close the DMA furnace. (Do not close the furnace when a submersion type clamp is installed.)
3. Select **Continue**. The clamp faces will be pressed together to determine the zero length point. The furnace will open when the clamp zero calibration has completed.
4. Press **Continue** to proceed to the next window.

Calibrate Clamp Compliance - Step 3

Compliance calibration is used to measure the flexibility of a clamp and calibrates the instrument to that flexibility. Compliance calibration is performed when you install a single or dual cantilever clamp, 3-point bending clamp, or a film tension clamp using a rigid precision steel sample with known dimensions. It is also performed without a sample when you install a penetration clamp or compression clamp. Compliance calibration is not performed when you install a shear sandwich or fiber tension clamp (e.g., step 3 of clamp calibration is not displayed).

The procedure used for compliance calibration is slightly different depending on the type of clamp installed. Choose the instructions that are applicable to your clamp.

For Cantilever, Film Tension, or 3-Point Bending Clamps:

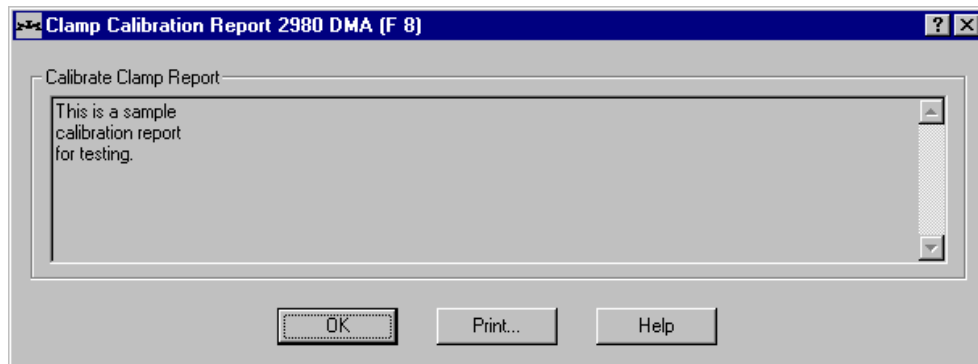
1. Measure the precision steel compliance sample, provided in the accessory kit, with calipers to determine its thickness and width.
2. Mount the compliance sample and measure the sample length. If you are using a submersion type clamp, skip to step 4.
3. Press the FURNACE key to close the DMA furnace. (Do not close the furnace when a submersion type clamp is installed.)
4. Enter the compliance sample dimensions (in this order - length, width, and thickness).
5. Select **Continue**. The furnace will open when the compliance calibration has completed.
6. Press **Continue** to proceed to the next window.

For Compression and Penetration Clamps:

1. If you are not using a submersion type clamp, press the FURNACE key to close the furnace. When a submersion type clamp is installed, skip to step 2.
2. Select **Continue** to calibrate the clamp compliance.
3. Press **Continue** to proceed to the next window.

Clamp Calibration Report

After you have completed the clamp calibration procedures, the results are put into a report, which is displayed in the window shown below. Use the Clamp Calibration Report option to view or print this report.



Select OK when you have finished reviewing the report.

Select Print to send the report to a printer.

Position Calibration

Use the **Position** calibration function to calibrate the DMA when it is moved or turned off, or at least once a month. The purpose of this calibration is to calibrate the absolute position of the drive shaft (and slide) as read by the optical encoder.

The instrument stores the clamp position in memory until the next calibration. In the event that the instrument loses the stored position, you will see one of these errors: Error 255, 274, 300 or 322.

NOTE: To retain the Position calibration, make sure that you press the STOP key and wait several seconds before you reset or turn off the instrument.

Clamp Check

You can run a check to see if your clamp position needs calibration by performing a small test. Press the FLOAT key to release (float) the clamp.

For all clamps except penetration and compression: Manually move the clamp to the top of the travel. The position signal on the instrument display should read 0 ± 0.5 mm. Move the clamp to the bottom of the travel. The position signal should read 25.0 ± 0.5 mm. If this is not the case, recalibrate the position as directed in this section.

For penetration and compression clamps: Manually position the clamp in the middle of its range of travel and release it. The clamp should maintain its position or slowly drift up or down. If the clamp rapidly sinks or rises, and the clamp mass has already been calibrated, the clamp position requires calibration.

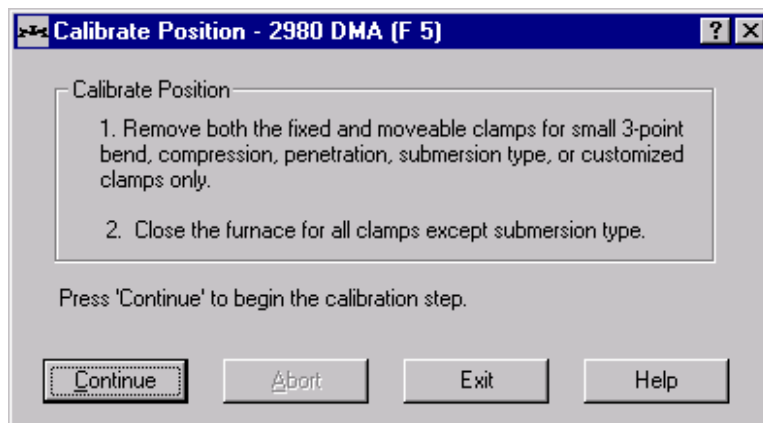
Position Calibration Procedure

If your clamp position needs calibration after you have performed the clamp check above, follow these steps:

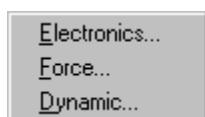
1. Remove both the fixed and moveable clamps, unless you have the large 3-point bending, film tension, or fiber tension clamp installed. (Those clamps can be left on for this calibration because they do not interfere with the instrument's movements.) Skip to step 3 if you are using a submersion type clamp.
2. Press the FURNACE key to close the DMA furnace. (Do not close the furnace when a submersion type clamp is installed.)
3. Select **Calibrate/Position** from the DMA menu. The window shown on the next page is displayed. Select **Continue**. The position will be calibrated automatically.

The furnace will open when the calibration has completed. (It normally takes approximately five minutes to complete the position calibration.)

4. Select **Continue** again to complete the procedure.



Instrument Calibration



Instrument calibration needs to be done when the DMA is moved, the feed hose for the air cool or GCA is removed or installed, or at least once a month. The menu shown on the left is displayed when you select **Calibrate/Instrument** from the DMA menu. For further information on each of the three main types of instrument calibration, see the sections

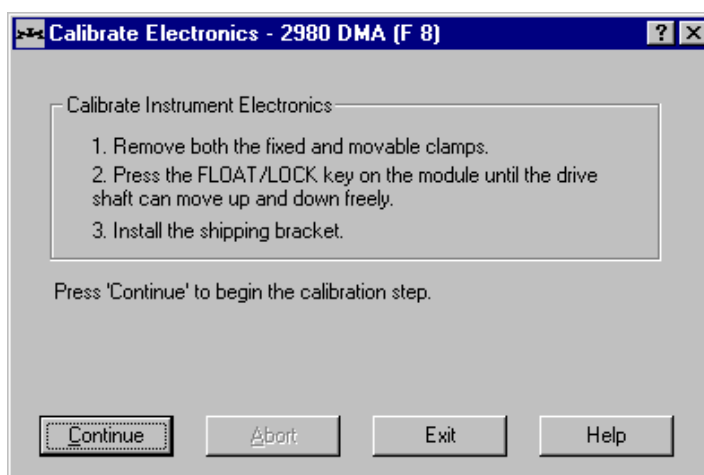
on the following pages.

Electronics Calibration

Electronics calibration is performed at least once a month to calibrate the electronics on the analog board. This calibration automatically removes the air to lock the slide (drive shaft) and applies a static force to the motor as a calibration reference. The instrument then uses a series of frequencies to perform the calibration.

Follow the steps below:

1. Remove both the fixed and moveable clamps.
2. Press the FLOAT/LOCK key until the drive shaft can move up and down freely.
3. Install the shipping bracket (see Chapter 2 of the instrument manual for the instructions).
4. Select **Calibrate/Instrument/ Electronics** from the DMA menu. The window shown here is displayed.



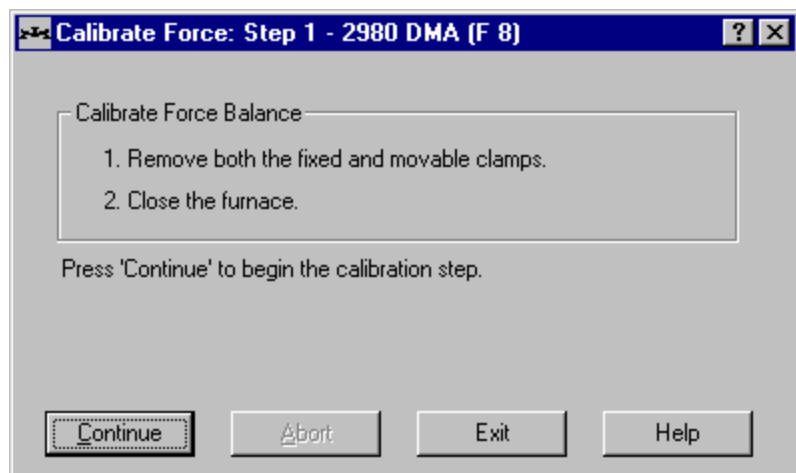
5. Select the **Continue** button.
6. Remove the shipping bracket when "Cal Done" status appears on the instrument's display.
7. Perform the following force calibration procedure after the electronics calibration has been completed.

Force Calibration

Force calibration is used to adjust the force exerted by the clamp on the sample and the force registered by the instrument as the experiment proceeds. There are two types of force calibrations that should be performed regularly—balance and weight calibration. Both of these calibrations must be done in the following situations:

When the DMA is moved,
or
At least once a month.

1. Select **Calibrate/Instrument/Force** from the DMA instrument menu. The first step is displayed in the window shown here. The first step is Force/Balance calibration, which measures the amount of force needed to hold the position of the drive shaft.

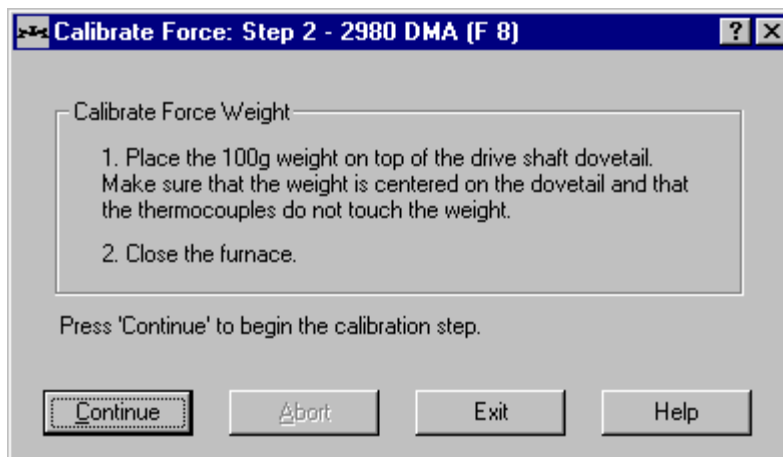


2. Remove both the fixed and moveable clamps.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue**. The instrument will be calibrated automatically. The furnace will open when the calibration has completed. (It normally takes approximately seven minutes to complete the force/balance calibration.) Press **Continue** again to proceed.

The next step in the force calibration procedure is Force/Weight calibration, which measures the amount of force needed to hold the position of the drive shaft when weight has been added.

Follow the steps below to perform the force/weight calibration:

1. After completing step 1 the step 2 window (shown here) is displayed.



3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue**. The instrument will be calibrated automatically. The furnace will open when the calibration has completed. (It normally takes approximately seven minutes to complete the force/weight calibration.)
5. Remove the weight.
6. Perform the **Dynamic** calibration after the Force/Weight calibration has been completed.

Dynamic Calibration

Dynamic calibration is used to measure several samples of known stiffness and loss to characterize the dynamic performance of the instrument. Dynamic calibration must be done in the following situations:

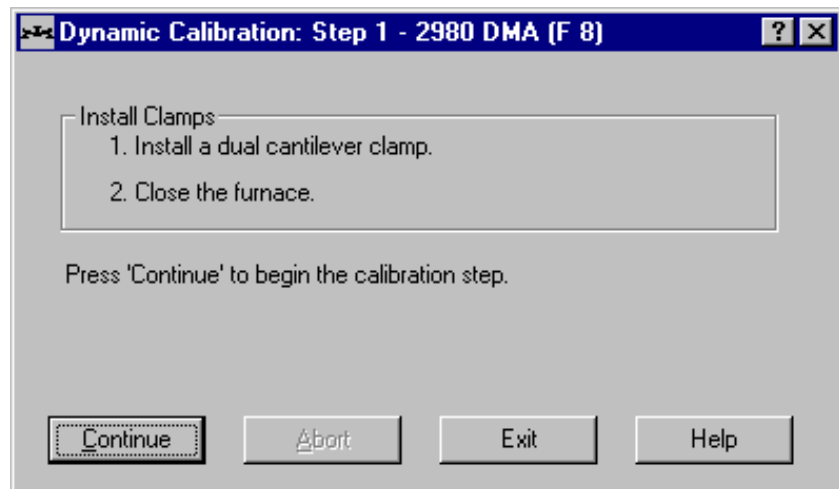
- When the DMA is moved or turned off, or
- At least once a month.

NOTE: Depending on the amount of compliance samples that you use, Dynamic calibration can take from 15 minutes up to two hours for completion.

Select **Instrument/Dynamic** from the DMA Calibrate menu and follow the steps provided on the series of windows displayed. The first step in the series of dynamic calibration windows is shown below.

Step 1: Install Clamps

1. Install the 35-mm dual cantilever clamp on the DMA.
2. Close the furnace.
3. Select **Continue** to begin calibration. Select **Continue** again to proceed to the next step (compliance calibration) in the dynamic calibration procedure.

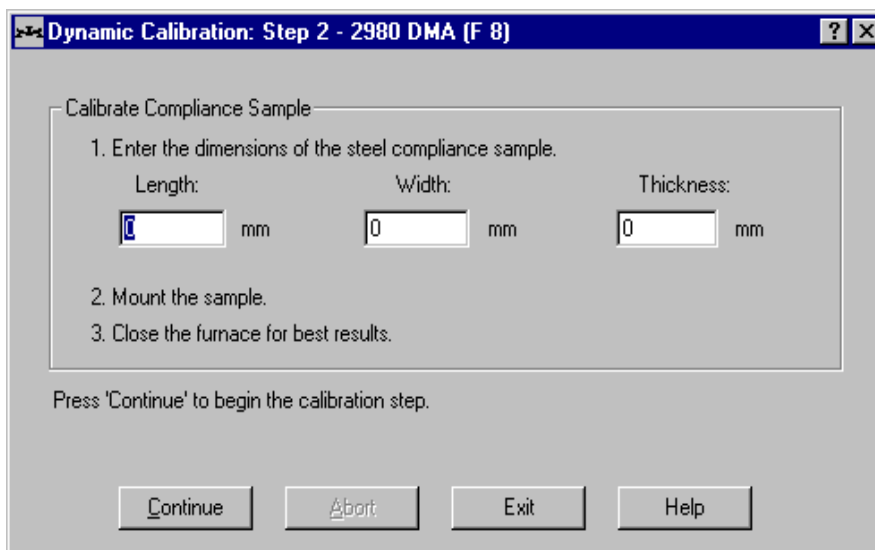


(continued on next page)

Step 2: Calibrate Compliance Sample

Compliance calibration is performed using a rigid precision steel sample with known dimensions. It is used to measure the flexibility of a sample and calibrates the instrument to that flexibility.

1. Measure the precision steel compliance sample, provided in the accessory kit, with calipers to determine its thickness and width.



2. Enter the measured width and thickness of the precision steel compliance sample in the appropriate fields and enter 35 mm as the length. Then select **Continue**. The furnace will open when the clamp mass calibration has been completed.

3. Mount the compliance sample in both the fixed and floating clamps.
4. Press the FURNACE key to close the DMA furnace.
5. Select **Continue**. The furnace will open when the clamp mass calibration has completed. Select **Continue** again to proceed to the next step.

The next step requires calibration of the 0.005-inch steel sample.

Dynamic Calibration - Step 3 Calibrate 0.005 Steel

1. Remove the compliance sample, which was calibrated in step 2, from the clamp.
2. Mount the 0.005-inch (0.12-mm) steel sample carefully. Make sure that the sample is not bent or otherwise deformed.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue** to begin calibrating this sample. The furnace will open when the clamp mass calibration has completed. Select **Continue** again to proceed to the next step.

The next step requires calibration of the 0.010-inch (0.25-mm) steel sample.

Dynamic Calibration - Step 4

Calibrate 0.010 Steel

1. Remove the 0.005-inch steel sample, which was calibrated in step 3, from the clamp.
2. Mount the 0.010-inch (0.25-mm) steel sample. Make sure that the sample is not bent or otherwise deformed.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue** to begin calibrating this sample. The furnace will open when the clamp mass calibration has completed. Select **Continue** again to proceed to the next step.

The next step requires calibration of the 0.020-inch (0.50-mm) steel sample.

Dynamic Calibration - Step 5

Calibrate 0.020 Steel

1. Remove the 0.010-inch steel sample, which was calibrated in step 4, from the clamp.
2. Mount the 0.020-inch (0.50-mm) steel sample. Make sure that the sample is not bent or otherwise deformed.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue** to begin calibrating this sample. The furnace will open when the clamp mass calibration has completed. Select **Continue** again to proceed to the next step.

The next step requires calibration of the 0.030-inch (0.76-mm) steel sample.

Dynamic Calibration - Step 6

Calibrate 0.030 Steel

1. Remove the 0.020-inch steel sample, which was calibrated in step 5, from the clamp.
2. Mount the 0.030-inch (0.76-mm) steel sample. Make sure that the sample is not bent or otherwise deformed.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue** to begin calibrating this sample. The furnace will open when the clamp mass calibration has completed. Select **Continue** again to proceed to the next step.

Dynamic Calibration - Step 7

Analyze Calibration or Continue

If you completed the first six steps of the dynamic calibration procedure, you have completed calibrating all of the steel samples. At this point, you can decide to choose one of the following options:

- If you are planning to run low stiffness samples frequently, you may wish to calibrate additional steel samples. Select **Continue** to calibrate up to 5 additional steel samples and go on to step 8 (see below).
- If you feel that calibration of the first 5 steel samples is sufficient, you can select **Analyze** to analyze the current calibration data. The program automatically analyzes and evaluates the results from all of the compliance samples that you have already run to determine the instrument calibration constants. This process can be viewed on the **Signal Display Pane**. See the Dynamic Calibration Report for the results of the calibration.

NOTE: Depending on the number of compliance samples that you use, Dynamic calibration can take from 15 minutes up to two hours for completion.

NOTE: If this calibration is not successful, repeat the calibration using 0.015-inch and 0.025-inch steel samples. If the calibration is still not successful, repeat the calibration again using 0.020-inch and 0.050-inch steel samples. If the calibration fails for a third time, call TA Instruments for service.

Optional: Dynamic Calibration - Step 8

Calibrate Additional Steel Samples

1. Remove the mounted steel sample from the clamp.
2. Mount a new steel sample. Make sure that the sample is not bent or otherwise deformed.
3. Press the FURNACE key to close the DMA furnace.
4. Select **Continue** to begin calibrating this sample. The furnace will open when the clamp mass calibration has completed.

At this point, you can decide to choose one of the following options:

- You may wish to calibrate additional steel samples. Select **Continue** to calibrate additional steel samples (total of five additional samples).

- If you feel that calibration of the samples already done is sufficient, you can select Analyze to analyze the current calibration data. The program automatically analyzes and evaluates the results from all of the compliance samples that you have already run to determine the instrument calibration constants. This process can be viewed on the Signal Display Pane. See the Dynamic Calibration Report for the results of the calibration.

Using the DMA Temperature Table

There are two types of temperature calibration that can be performed for the DMA 2980 instrument. See the following sections for a description of the two temperature calibration types and instructions on how to perform the procedures.

Absolute Temperature Calibration

Perform these steps for absolute temperature calibration of the DMA sample thermocouple:

1. Select **Calibrate/ Temperature Table** to open the **Temperature Table** (shown here). Click the **Reset** button to clear any previous calibrations from the instrument and select **OK**.

	Observed Temperature °C	Correct Temperature °C
Point 1:	<input type="text"/>	<input type="text"/>
Point 2:	<input type="text"/>	<input type="text"/>
Point 3:	<input type="text"/>	<input type="text"/>
Point 4:	<input type="text"/>	<input type="text"/>
Point 5:	<input type="text"/>	<input type="text"/>

Buttons: OK, Reset, Cancel, Help

2. Install the desired clamp. If the clamp was just installed, select **Calibrate/ Clamp**. Perform clamp calibration, then position the sample thermocouple so that the tip of the thermocouple is near the sample, but does not interfere with the motion of the clamps or slide.
3. Make sure that the DMA 2980 is held at ambient temperature, with the furnace open, for at least one hour before beginning the routine.
4. Measure and record the ambient temperature in the general area of the DMA 2980. You can do this using a reliable external thermometer.
5. Note the ambient temperature reading of the DMA 2980.

6. Select **Calibrate/Temperature Table** to open the **Temperature Table** again. Under Point 1, enter the temperature reading from the external thermometer as the correct temperature, and the reading from the DMA as the observed temperature. Then select OK.

Dynamic Temperature Calibration Procedure

If you are going to perform temperature ramp experiments on the DMA 2980, with the single/dual cantilever or 3-point bending clamps, you can use the following steps to compensate for temperature lag. However, similar temperature calibration standards are not currently available for the other DMA clamps.

NOTE: ASTM Standard Test Method E1867-97*, "Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzer" describes another method for dynamic temperature calibration based on a polymer encapsulated metal standard.

1. Perform the steps above to correct for absolute temperature.
2. Mount the thin polycarbonate test bar (provided in the accessory kit) on the clamp.
3. Measure the glass transition (T_g) of the polycarbonate sample under stepwise-isothermal conditions.
4. Set up the same instrument parameters you intend to use in your experiment.
5. Measure the T_g of the polycarbonate sample at the underlying heating (or cooling) rate you intend to use in your experiment.
6. Using the Universal Analysis program, analyze both data files, and note the temperature of the glass transition. (See the Universal Analysis online help or online manual for information on using the program.)
7. Select **Calibrate/Temperature Table** to open the **Temperature Table**. Under Point 1, enter the T_g measured by the stepwise-isothermal method as the correct temperature, and the T_g measured from the dynamic experiment as the observed temperature. Then select OK.

The instrument is now compensated for thermal lag at the specific underlying heating rate chosen. If a different heating rate is required, the dynamic run should be repeated at the required rate. The compensation for temperature lag can be significant. After completion, the ambient temperature reading may be unusually high or low, which is normal, and should be expected.

*You may obtain ASTM E1867-97 from: ASTM, 100 Bar Harbor Drive, West Conshohocken, PA 19428-2959, (610) 832-9500.

Calibrating the DSC and DTA

To obtain accurate experimental results you should calibrate each standard DSC, dual sample DSC (2920 only), pressure DSC, and 1600°C DTA when you first install it. For the DSC 2920 cells, the calibration will be stored in the cell's memory and automatically entered when the cell is installed. Therefore, you can easily change cells on the 2920 without recalibrating each time you do. For the best results, however, you should recalibrate periodically.

Perform calibration runs that encompass the temperature range you plan to use in your experiments. If you change the general temperature range of your experiments later, you may wish to recalibrate within the new range.

For precise experimental results you will need to generate a new calibration file whenever you change one of the following parameters:

- Ramp rate (selected in the procedure)
- Purge gas
- Cooling technique (LNCA, RCS, or DSC Cooling Can)
- Pressure (in PDSC experiments)
- First use of the cell.

Calibration is performed in the instrument's calibration mode, which is accessed through the **Mode Parameters** window (see Chapter 3 for details on modes).

Types of Calibration

Calibration consists of several different types of calibrations, which are specific to each cell, each of these will be explained in detail in this chapter. A brief description of each type of calibration begins below.

- Baseline slope and offset (all cells)
- Cell constant calibration (DSC, Dual Sample DSC, and Pressure DSC cells)
- Temperature calibration (all cells)
- Pressure calibration (DSC 2920 Pressure cells only)
- Crosstalk calibration (DSC 2920 Dual Sample cells only).

Baseline Calibration Type

Baseline calibration needs to be performed for each cell. This calibration involves heating the cell through the entire temperature range in which it will be operated and using the same heating rate and purge gas to be used in subsequent experiments.

The baseline calibration measures the amount by which the heat flow curve deviates from zero so that this deviation can be taken into account during calibration.

The baseline calibration is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. The minimum RMS (root mean square) deviation from the data is calculated to determine the best straight-line fit. This adjusts the difference in output between the reference and sample thermocouples.

- DSC 2910 and 2010 defaults are 0.00 (slope) and 50000.00 (offset).
- DSC 2920 defaults are 0.00 (slope) and 0.00 (offset).

Cell Constant Calibration Type

The *cell constant* determination is based on a run in which a known sample (standard) is heated through its transition temperature and data is gathered for analysis. The DSC cell constant and *onset slope* are determined from the data for calibration of the cell.

- *Cell constant* is the ratio of the known heat of fusion to the measured experimental heat of a standard. The experimental heat is calculated using the peak integration area, normalized to the sample weight.
- *Onset slope*, or thermal resistance, is a measure of the temperature drop that occurs in a melting sample in relation to the thermocouple. Theoretically, a standard sample should melt at a constant temperature. As it melts and draws more heat, a temperature difference develops between the sample and the sample thermocouple. The thermal resistance between these two points is calculated as the onset slope of the heat flow versus temperature curve on the front side of the melting peak. The onset slope value is used in most DSC kinetic and purity calculations to correct for this thermal resistance.

Temperature Calibration Type

This calibration type is used to calibrate the instrument for temperature. The information gathered from temperature calibration is entered in the temperature calibration table, which is applied to the collected data.

NOTE: A multiple-point calibration is more accurate than a one-point calibration.

A single-point calibration shifts the sample temperature by a constant amount.

A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and shifts by a constant amount after the last point.

Pressure Calibration

Pressure calibration is an optional calibration procedure for the DSC 2920 Pressure cells only. It is based on comparing the pressure readings at two points—typically one atmosphere and another pressure value selected by you—to the pressure reading on an external pressure gauge. See page 2-37 for more information.

Crosstalk Calibration

Crosstalk calibration is an additional calibration procedure that is optional when the dual sample DSC cell is used. This calibration uses the cell constant data to calculate and eliminate any signal cross-over occurring between the two sample sides of the dual sample DSC cell. Crosstalk between samples A and B is minimal and typically less than 0.20%. The default value is 0.0. See page 2-41 for more information.

General Calibration Instructions

The first three types of calibrations have steps that are common to all three. You will need to perform the following general steps to calibrate the DSC and DTA for baseline, cell constant, and temperature:

1. Put the instrument in Calibration mode: Select **Experimental/Mode** or click the



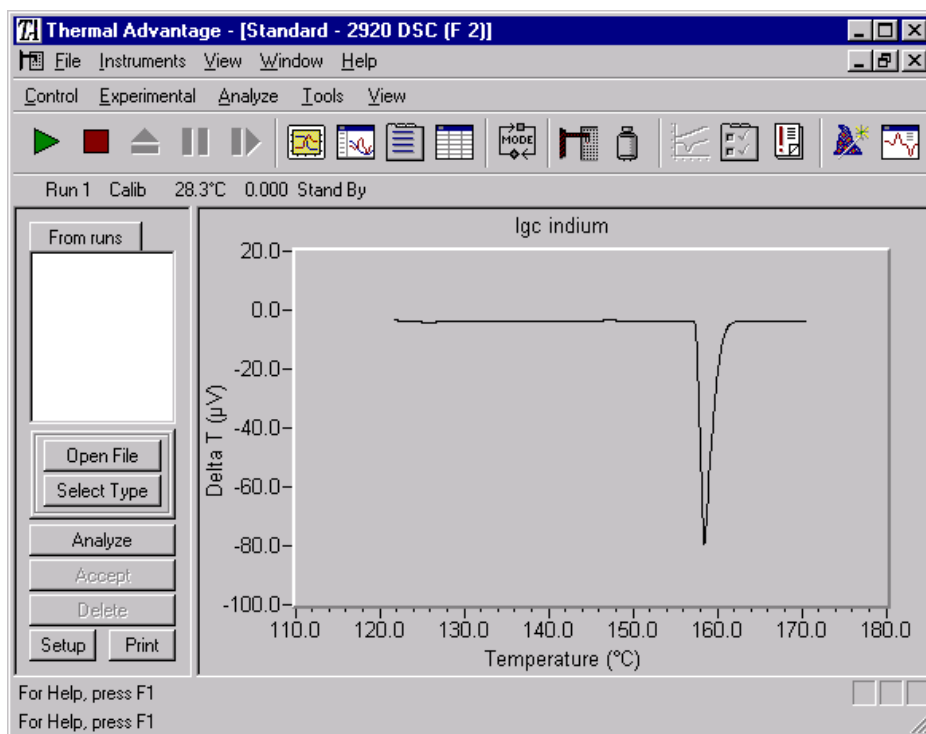
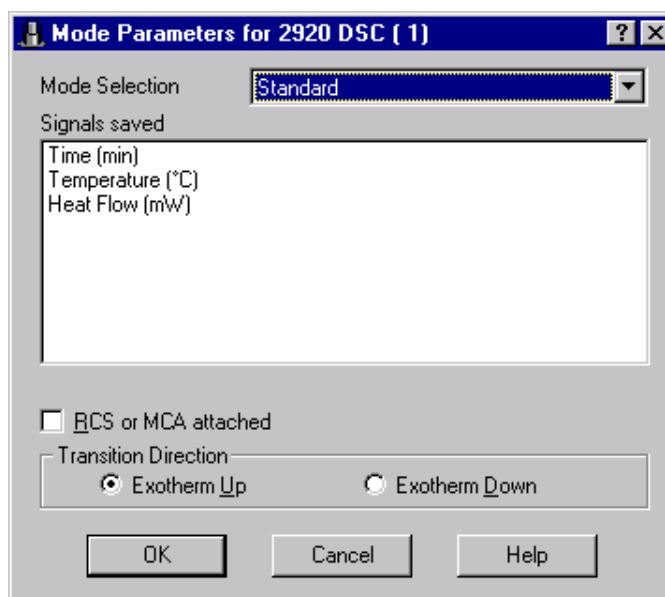
button on the tool bar. This opens the **Mode Parameters** window (shown here). Click the arrow to open the Mode Selection list and choose the calibration mode.

2. Conduct the appropriate calibration experiments as directed in the next section "Gathering Calibration Data."

3. Select **Analysis** from the **DSC Calibrate** menu or click on the



button on the tool bar to open the calibration analysis window (shown below).



4. Analyze the calibration files (for baseline, cell constant, and temperature calibration) following the instructions in the section called "Analyzing Calibration Data" on page 2-32.

Gathering Calibration Data


To calibrate the DSC for baseline, cell constant, and temperature, you need to first collect calibration data files that will be analyzed. All calibration runs must be performed in the calibration mode. After the files have been analyzed, the resulting calibration information is stored in the instrument for use when you run experiments.

To gather calibration data, follow the instructions found on the next several pages, based on the type of calibration desired.

Obtaining Baseline Calibration Data

Baseline calibration involves heating an empty cell through the entire temperature range expected in subsequent experiments. The calibration program is used to calculate the slope and offset values needed to flatten the baseline and zero the heat flow signal.

A general procedure for baseline calibration is given below:

1. Remove any pans from the cell and replace the lids (for a DTA cell, load empty sample cups in the cell and replace the furnace tube and furnace).
2. Check that your purge gas is connected and set to the desired flow rate.
3. Select **Experimental/Mode** from the main menu. Set the instrument to calibration mode using the Mode Selection list.
4. Click the  button on the tool bar to select the **Experiment View**. Select Baseline from the sample name list. Enter the requested sample information, including zero as the sample size on the **Summary Page**. Select the Baseline test from the Test list.
5. Click on the Procedure tab. Enter the requested test parameters that will heat the DSC cell over the same temperature range, at the same heating rate that will be used for subsequent experiments.
6. Click on the Notes tab. Enter/verify the requested information.
7. Click on the Apply button to save the experimental and sample parameters entered for this run.
8. If more than one run is in the sequence list, schedule this run. (An arrow will appear next to the run number in the **Sequence Pane** for the scheduled run.)
9. Select Start to begin the calibration run.

10. Wait until the run is complete, then access the calibration program using **Calibrate/Analysis** on the DSC menu.

For DSC Cells: Once the run is complete (indicated by a checkmark next to the run in the sequence list), the calibration program will automatically bring the file into the calibration window. (You could also accomplish the same steps by selecting **Open File** on the main **Calibration** window and then select the DSC temperature data file created above.)

11. Select the appropriate calibration type (Baseline) as specified by the test procedure and analyze the data. See "Analyzing Calibration Files" for details.
12. Save the calibration results by clicking the **Accept** button (or by selecting **Analyze/Accept results** from the calibration menu). This will update the current temperature parameters with this calibration data.

You can verify this result by viewing the **Cell/Temperature Table** window (selected from the **Calibrate** menu). See "Using the Cell Calibration Page" for details.



Obtaining Cell Constant Calibration Data

Cell constant is a calibration factor used to adjust the calorimetric response of a DSC cell. The cell constant is the ratio of the known value to the measured experimental variable. Typically, the heat of fusion of indium (DSC) is used. The calibration is based on a run made in the calibration mode using the experimental conditions to be used in subsequent measurements.

The temperature calibration data from this experiment is automatically stored when the cell constant calibration results are saved.

A general procedure for cell constant calibration is given below:

1. Choose an appropriate calibration material with a known heat (*e.g.*, indium).
2. Prepare approximately 10 mg of the calibration material in a sample pan. Crimp the pan, then position it on the sample platform.
3. Prepare a reference pan using the same pan type used for the sample. Crimp the pan, then position it on the reference platform.
4. Replace the cell lids.
5. Check that your purge gas is connected and set to the desired flow rate.
6. Select **Experimental/Mode** from the main menu. Set the instrument to calibration mode using the Mode Selection list.

7. Click the  button to select the **Experiment View**. Select the calibration material from the sample name list. Click on the  button and verify the calibration information for this standard.
8. Enter the requested sample information, including the exact sample mass on the **Summary Page**. Select the Cell Constant test from the Test list.
9. Click on the **Procedure Page** tab. Enter the requested test parameters that will program the DSC to: (a) equilibrate to 100°C below the onset of the literature melting temperature of the material, and (b) heat the material, at the same heating rate that you will use in your subsequent experiments, to 50°C above the literature melting temperature.
10. Click on the **Notes Page** tab. Enter/verify the requested information.
11. Click on the **Apply** button to save the experimental and sample parameters entered for this run.
12. If more than one run is in the sequence list, schedule this run. (An arrow will appear next to the run number in the **Sequence Pane** for the scheduled run.)
13. Select **Start** to begin the calibration run.
14. Wait until the run is complete, then access the calibration program using **Calibrate/Analysis** on the DSC menu. Once the run is complete (indicated by a checkmark next to the run in the sequence list), the calibration program will automatically bring the file into the calibration window. (You could also accomplish the same steps by selecting **Open File** on the calibration window and then select the DSC cell constant data file created above.)
15. Select the appropriate calibration type as specified by the test procedure and analyze the data. See “Analyzing Calibration Files” for details.
16. Save the calibration results by clicking the **Accept** button (or by selecting **Analyze/Accept results** from the calibration menu). This will update the current cell calibration parameters (cell constant and onset slope) and update the temperature table.


You can verify this result by viewing the **Cell/Temperature Table** window (selected from the **Calibrate** menu). See “Using the Cell Calibration Page” for details.

Obtaining Temperature Calibration Data

Temperature calibration is based on a run in which a temperature standard (e.g., indium) is heated through its melting point using the same conditions to be used in subsequent measures (e.g., heating rate and purge gas). The recorded melting point of this standard is compared to the known melting point and the difference is calculated for temperature calibration. Up to five standards may be used for temperature calibration. A single-point calibration shifts the sample temperature by a constant amount. A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points, and shifts by a constant amount after the last point.

During cell constant calibration, the temperature calibration data from the cell constant calibration experiment is automatically stored when the cell constant calibration results are saved.

A general procedure for temperature calibration is given below:

1. Choose an appropriate calibration material with a known melting point.
2. Prepare approximately 10 mg of the calibration material in a sample pan. Crimp the pan, then position it on the sample platform.
3. Prepare a reference pan using the same pan type used for the sample. Crimp the pan, then position it on the reference platform.
4. Replace the cell lids.
5. Check that your purge gas is connected and set to the desired flow rate.
6. Select **Experimental/Mode** from the main menu. Set the instrument to calibration mode using the Mode Selection list.
7. Select the **Experiment View - Summary Page**. Select the calibration material from the sample name list. Click on the  button and verify the calibration information for this standard. Enter the requested sample information, including the exact sample mass on the **Summary Page**. Select the Temperature test from the Test list.
8. Click on the **Procedure Page** tab. Enter the requested test parameters that will program the DSC to: (a) equilibrate to 100°C below the onset of the literature melting temperature of the material, and (b) heat the material, at the same heating rate that you will use in your subsequent experiments, to 50°C above the literature melting temperature.
9. Click on the **Notes Page** tab. Enter/verify the requested information.
10. Click on the Apply button to save the experimental and sample parameters entered for this run.


11. If more than one run is in the sequence list, schedule this run. (An arrow will appear next to the run number in the **Sequence Pane** for the scheduled run.)
12. Select Start to begin the calibration run.
13. Wait until the run is complete, then access the calibration program using **Calibrate/Analysis** on the DSC menu. Once the run is complete (indicated by a checkmark next to the run in the sequence list), the calibration program will automatically bring the file into the calibration window. (You could also accomplish the same steps by selecting **Open File** on the main **Calibration** window and then select the DSC temperature data file created above.) See “Analyzing Calibration Files” for details.
14. Select the appropriate calibration type as specified by the test procedure and analyze the data.
15. Save the calibration results by clicking the Accept button (or by selecting **Analyze/Accept results** from the calibration menu). This will update the current temperature parameters with this calibration data.

These calibration points are automatically updated in the **Cell/Temperature Table** window after temperature calibration has been performed, accepted, and saved using the **Calibrate/Analysis** function. Temperature calibration experiments are conducted with the instrument in the calibration mode, which ignores previous calibration factors. See “Using the Temperature Calibration Page” for details.

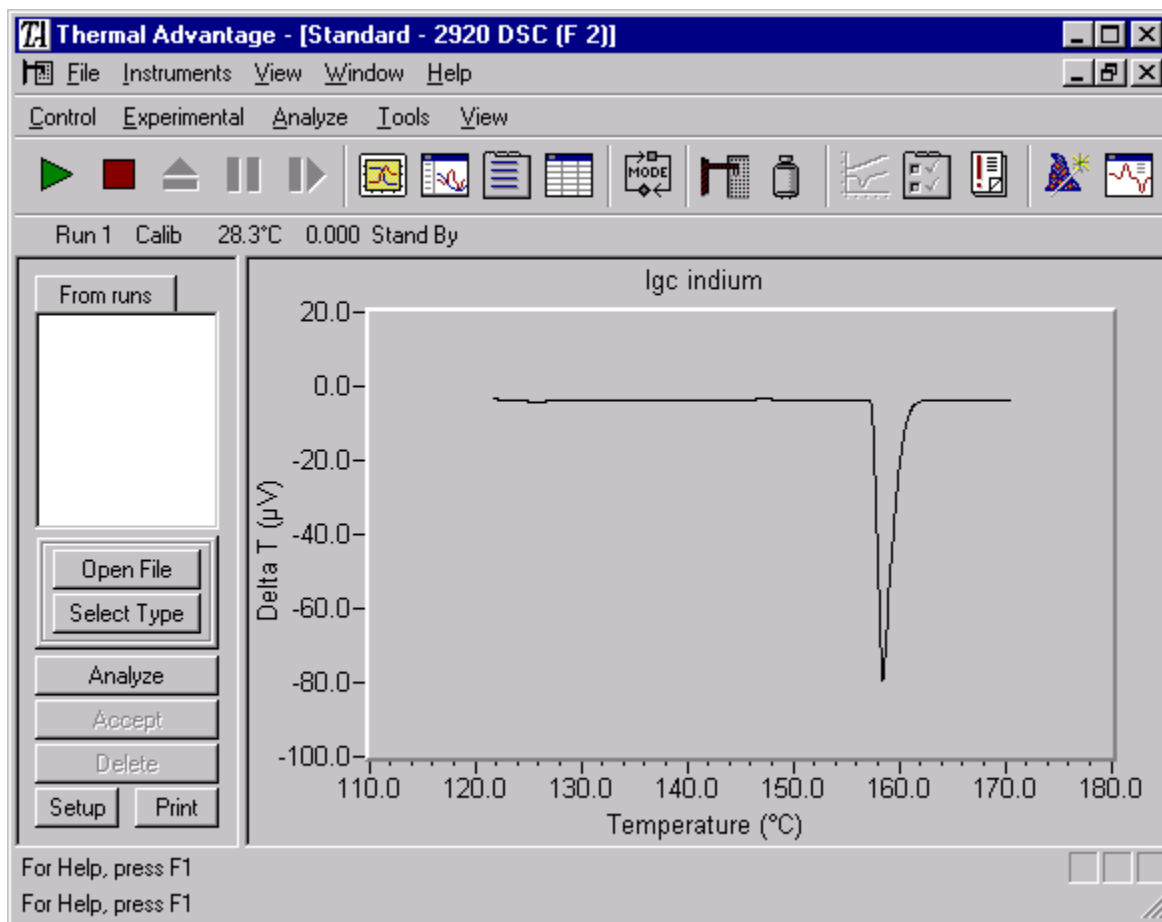
NOTE: A multiple-point calibration is more accurate than a one-point calibration. A single-point calibration shifts the sample temperature by a constant amount. A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and maintains a constant temperature shift after the last point.

Analyzing Calibration Files

Once the DSC calibration experiments are complete, as indicated by a check mark next to the run number(s) on the sequence list, you will need to analyze them. To do this, select

Calibrate/Analysis from the DSC menu or click the  button on the DSC tool bar.

The main calibration window (shown below) is displayed.



This window displays the data from your calibration experiment(s) and is used to determine and save the requested calibration parameters.

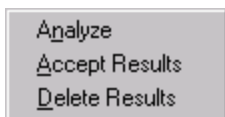
Calibration results may be analyzed and saved after each DSC calibration experiment or all together (e.g., after all the experiments have been completed).

1. Two options are available for displaying calibration files: **From Runs** or **From File**.
 - The **From Runs** option is only valid in the calibration mode and when there are completed files (as indicated by a check mark next to the run) in the sequence list. When you access the calibration window these runs will be automatically retrieved from the sequence list (From Runs option). A list of completed runs will be displayed in the file list.

- The **From Files** option requires that you manually specify the file. To use this option click the Open File button (or choose Open a File from the pop-up menu). A standard Open File window is displayed. Select the appropriate calibration file(s) and click OK. (NOTE: Multiple calibration files may be chosen at one time.)
2. The calibration program will automatically try to determine the calibration type (baseline, cell constant, or temperature) and the standard used (if required) from the information in the data file. A B, C, or T will appear next to each run indicating the calibration type-baseline, cell constant, or temperature respectively. The program will then display the data and analyze the curve according to the default calibration settings. Baseline calibrations will automatically display the analysis limits markers, but wait for you to specify the desired temperature range and then select **Limits OK**.
 3. If the correct calibration type is not selected, click the **Select Type** button (or choose **Select Type** from the pop-up menu). The DSC Calibration Analysis Specification window (shown here) is displayed. Choose the type of calibration desired and, if applicable, the standard to be used. To edit this standard, see "Editing DSC Calibration Standard Settings" on the page 2-35. (A standard is needed for DSC cell constant or temperature calibration.) Then click OK. The file is plotted on the plot area of the calibration window.

Standard Name	Melt Point (°C)	Standard Heat (J/g)
Mercury	-38.83	11.44
Water	0.01	335.00
Indium	156.60	28.71
Tin	231.93	60.60
Lead	327.50	23.10
Zinc	419.53	108.00
Aluminum	660.33	400.10
Baseline	0.00	0.00

4. If the results are acceptable, all you need to do is save the results for each calibration by clicking on the **Accept** button or by choosing **Analyze/Accept results** from the pop-up menu (shown on the left). These results will be automatically sent to the instrument. Once the data has been saved a check mark will appear next to the file within this window.



If the results are not acceptable, continue with the following steps.

5. Rescale the plot, if desired. This can be done using the mouse to expand part of the curve.

6. Select the **Analyze** button or select **Analyze** from the Calibration pop-up menu. The current limit points will be indicated with markers on the graph. **NOTE:** The active marker is indicated by a larger red cross-hair. Other markers are indicated by smaller red cross-hairs.
7. Move the active marker to the desired position on the curve using either the mouse or keyboard (see the directions below).

Moving the Markers Using the Mouse:

1. Position the pointer at the desired location.
 2. Double-click the left mouse button to move the active marker to this point. This action causes the next marker to become active.
- or
1. Position the pointer over the active marker.
 2. Press and hold down the left mouse button while you drag the marker to the desired location.
 3. Release the mouse button. At this point you may use the arrow keys to position the marker more closely (see the directions below).

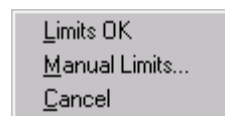
Moving the Markers Using the Keyboard:

Press the arrow keys to move the active marker to the desired position.

NOTE: The marker will move in the following increments:

- Arrow key = smallest step
- Alt/arrow key = second smallest step
- Shift/arrow key = second largest step
- Control/arrow key = largest step

8. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB [Shift-TAB] will activate the previous marker.)
9. Repeat steps 7 and 8 until you have selected all the necessary points for analysis.
10. Click the **Limits OK** button or select **Limit OK** from the pop-up menu to accept the points. When calculated, the calibration results will be displayed.
11. Accept the calibration results by clicking the **Accept** button or selecting **Analyze/Accept results**. (Delete results by clicking the **Delete** button or choosing **Analyze/Delete results** from the popup menu.)
12. Print the results, if desired, by clicking on the **Print** button (or by choosing **Print** from the pop-up menu).
13. If more than one listing appears in the file list, click on each file (run) to view, modify (if necessary) and save the data.
14. To switch between the files and runs list, click on the **From Runs/From Files** button located at the top of the file list.



After the calibration results have been saved, and the analysis program is closed, the calibration parameters are automatically updated to the DSC. (If you are calibrating the DSC 2920, the calibration parameters are stored in the cell's memory, allowing cells to be changed without the need for recalibration.) The data files resulting from calibration may be reused when needed.

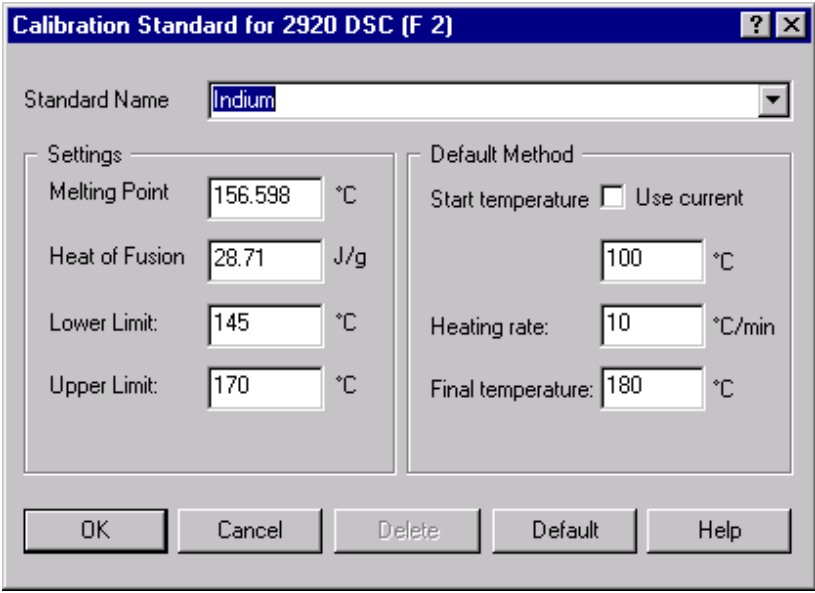
Editing DSC Calibration Standard Settings

To set up the parameters that will be used to automatically analyze DSC calibration files for baseline, cell constant, or temperature, follow these steps:

1. Click the  button on the tool bar to open the main **Calibration** window.
2. Click the **Setup** button. The window shown here is displayed.

You can temporarily change the theoretical values for the specified standard that will be used during calibration or to enter a new standard.

1. Enter the name of the material that you are using as your standard calibration material (e.g., aluminum oxide or indium).
2. Enter the literature melting point value, in degrees C, and the literature standard heat of fusion value, in Joules/gram, for the standard chosen. Consult your Chemistry handbook for the values.
3. Enter the default analysis limits.
4. Edit the default method conditions to be displayed when this standard is chosen.
4. Select OK to save the changes.



Calibration Standard for 2920 DSC (F 2)	
Standard Name: Indium	
Settings	
Melting Point	156.598 °C
Heat of Fusion	28.71 J/g
Lower Limit:	145 °C
Upper Limit:	170 °C
Default Method	
Start temperature	<input type="checkbox"/> Use current 100 °C
Heating rate:	10 °C/min
Final temperature:	180 °C
OK Cancel Delete Default Help	

NOTE: You can select the Default button to set all of the values displayed to their factory defaults.

After you have opened a file and selected your calibration standard, the DSC main **Calibration** window is displayed again to allow you to analyze the data. See "Analyzing Calibration Files" for information.

Rescaling/Customizing the Plot

To zoom in on a particular portion of the analysis curve, displayed on the main **Calibration** window, follow these steps:

1. Position the pointer in the general area that you wish to enlarge and hold down the mouse button. As you move the mouse, a dotted line box will be drawn from the original pointer position.
2. Move the mouse to draw a dotted line around the portion of the curve to be enlarged.
3. Release the mouse button. The curve will be rescaled immediately. This process can be used repeatedly to expand a smaller portion of the curve.

To reverse the expansion process, right click the mouse button to display the popup menu (shown to the right) and select **Previous Scale**. (Shortcut: **Ctrl-B**) You can repeatedly perform this action, until you have stepped backwards through the expansion increments to the desired point.

If you want to return the curve to its original full scale state, right click the mouse button to display the popup menu and select **Full Scale**.



NOTE: You can not expand the curve when the markers are displayed. Remove the markers by clicking on the Cancel button or choosing Cancel from the pop-up menu.

After the curve has been rescaled, you can perform the analysis functions.

You can also change the font used on the plot by selecting **Font/Axis** to change the fonts used on the axis and labels or by selecting **Font/Analyze** to change the fonts used for the calibration results.

Printing the Calibration Results

Once you have completed analyzing the calibration data file on the main calibration window, you can print it easily by clicking the **Print** button or selecting **Print** from the Calibration Analysis pop-up menu. The results, along with the data plot, will be printed.

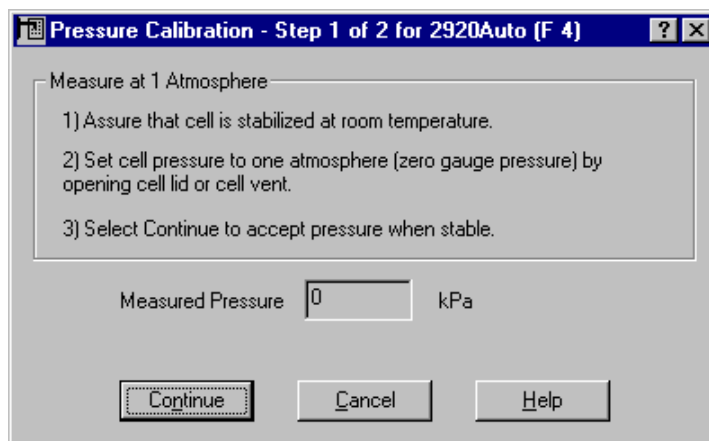
Performing Pressure Calibration

Pressure calibration is an optional calibration procedure for the DSC 2920 Pressure Cells only. It is based on comparing the pressure readings at two points—typically one atmosphere and another pressure value selected by you—to the pressure reading on an external pressure gauge.

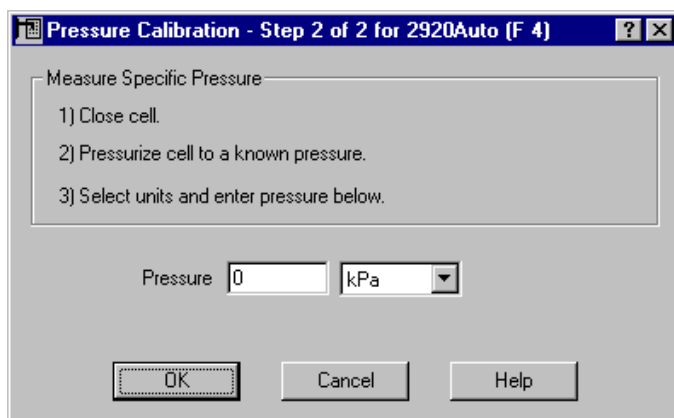
Pressure Calibration - Step 1 of 2

When you chose **Calibrate/Pressure** from the DSC menu, the first window displayed is the **Pressure Calibration Step 1** window. The first step in pressure calibration is to measure the pressure reading from the cell at the normal room pressure of one atmosphere:

1. Make sure that the cell has returned to room temperature before calibrating.
2. Open the cell lid or cell vent to allow the cell to stabilize at one atmosphere. The pressure gauge will indicate zero.
3. Watch the displayed pressure reading. When it stops fluctuating, accept the measured pressure by selecting **Continue**. To stop the calibration process select **Cancel**.



Pressure Calibration - Step 2 of 2



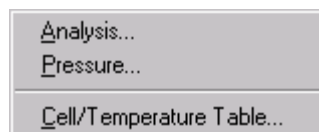
The second step in pressure calibration, as displayed on the **Pressure Calibration Step 2** window, is to measure a specific pressure reading from the cell using a pressure gauge.

1. Close the vent valve.
2. Connect the external pressure gauge to the outlet port and open the outlet valve on the Pressure DSC (PDSC) cell.
3. Close the inlet valve and connect the pressure source to the inlet valve on the PDSC cell.

4. Gradually pressurize the PDSC cell to a known pressure (refer to the operator's manual for PDSC).
5. Allow the cell to stabilize and then read the pressure gauge. Enter that reading in the Pressure field.
6. Click on the arrow to the right of the Pressure field and select the appropriate units.
7. Select OK to accept the pressure. To stop the calibration process select Cancel.

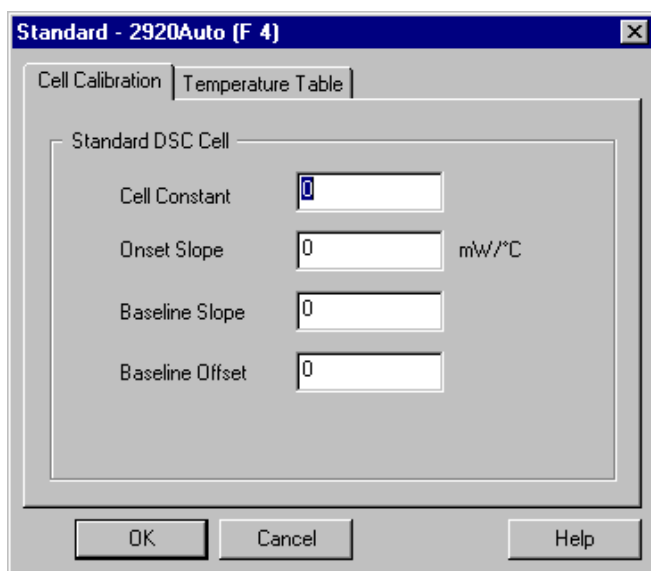
Using the Cell Calibration Page

Select **Calibrate/Cell Temperature Table** from the DSC menu to access either the **Cell Calibration Page**, which is used to verify/input calibration constants, or the **Temperature Table Page**, which is used to verify, input, or reset temperature calibration data.



NOTE: The Cell/Temperature Table is not available when the instrument is in the calibration mode.

Click on the **Cell Calibration Page** to set the following calibration parameters that the instrument will apply to the corrected data. These calibration parameters are automatically updated in this window after cell constant calibration and baseline calibration have been performed, accepted, and saved.



- **Cell Constant:** Cell Constant is a calibration factor used to adjust the calorimetric response of a DSC cell. The cell constant is the ratio of the known value to the measured experimental variable. Typically, the heat of fusion of indium (DSC) is used. The calibration is based on a run made in the calibration mode using the experimental conditions to be used in subsequent measurements.

- **Onset Slope:** This parameter is the slope of the onset of the melting curve for a standard. The onset slope, or thermal resistance, is a measure of the temperature drop that occurs in a melting sample in relation to the thermocouple. Theoretically, a standard sample should melt at a constant temperature. As it melts and draws more heat, a temperature difference develops between the sample and the sample

thermocouple. The thermal resistance between these two points is calculated as the onset slope of the heat flow versus temperature curve on the front side of the melting peak. This is always a negative term. Default is 0.00 mW/°C.

- **Baseline Slope:** This parameter is a calibration constant that accounts for the baseline slope of an empty cell. The baseline slope is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. The RMS (root mean square) average of the data is calculated to determine the best straight-line fit. From this line the baseline slope is calculated to account for the difference in output between the reference and sample thermocouples. Default is 0.00.
- **Baseline Offset:** Baseline Offset is a calibration factor that shifts the curve up or down along the y-axis to balance the heat flow at the sample and reference thermocouple positions. The baseline offset is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. The RMS (root mean square) average of the data is calculated to determine the best straight-line fit. From this line the baseline offset is calculated to return the curve to the ideal reference (zero).
- **Crosstalk (For Dual Sample DSC):** Crosstalk calibration is an additional calibration procedure that is optional when the dual sample DSC cell is used. This calibration uses the cell constant data to calculate and eliminate any signal cross-over occurring between the two sample sides of the dual sample DSC cell. Crosstalk between samples A and B is minimal and typically less than 0.20%. The default value is 0.0. See “Performing Crosstalk Calibration” for details.

NOTE: Since all DSC cells do not operate exactly the same, there are different cell calibration parameters available depending upon the cell installed on your instrument. For example, Dual Sample DSC cells require crosstalk calibration, along with the other calibration parameters.

Using the Temperature Table Page

Temperature calibration is useful for experiments in which precise transition temperatures are essential.

Select **Calibrate/Cell/Temperature Table** from the DSC menu, then click the Temperature Table tab. The **Temperature Table Page** displayed shows the temperature calibration table that the instrument applies to the collected data. One to five temperature calibration points (pairs of observed and correct temperature points) may be used. Temperature calibration is based on a run that heats a high purity temperature standard, such as indium, through its melting point. The observed and correct temperatures correspond to the experimental and theoretical transition temperature (*e.g.*, melting point) of the calibrant respectively.

These calibration points are automatically updated in this window after temperature calibration has been performed, accepted, and saved using the **Calibrate/Analysis** function. Temperature calibration experiments are conducted with the instrument in the calibration mode, which ignores previous calibration factors.

	Observed Temperature °C	Correct Temperature °C
Point 1:	9.67	9.79
Point 2:	99.67	99.79
Point 3:		
Point 4:		
Point 5:		

NOTE: A multiple-point calibration is more accurate than a one-point calibration. A single-point calibration shifts the sample temperature by a constant amount. A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and maintains a constant temperature shift after the last point.

- If these values are correct, select OK.
- To manually edit these values:
 1. Select the **Reset** button to remove all previously stored values.
 2. Enter the Observed and Correct temperature points in the table.
 3. Select OK when all points have been entered to save the settings to the instrument.

NOTE: When temperature calibration is in effect, Temp in *C appears in the status line. When it is not in effect, Temp in °C appears in the status line.

Performing Crosstalk Calibration (for Dual Sample DSC Cells)

If you wish to perform crosstalk calibration, follow the instructions outlined below:

1. Select **Calibrate/Cell/Temperature Table** from the DSC menu to open the **Cell Calibration Page**. Set the crosstalk parameters to zero. Select OK.
2. Create a thermal method that heats the sample from ambient to 180°C. For accurate sample analysis, use the heating rate you plan to use most often in your experiments. Perform two experiments using this method under the following circumstances:

Run 1

Side A: 10 mg indium sample sealed in the type of sample pan you plan to use in subsequent experiments

Side B: empty (no pans)

Reference: empty (no pans)

Run 2

Side A: empty (no pans)

Side B: 10 mg indium sample sealed in the type of sample pan you plan to use in subsequent experiments

Reference: empty (no pans)

NOTE: These runs are performed in the standard mode. NOT the calibration mode.

3. For each run, analyze each data set (side A and B) using Universal Analysis program. Determine the peak area of the Indium melt by performing a peak integration analysis (standard area). Perform the same analysis on the corresponding empty side file using the same analysis limits (baseline area).

NOTE: Before conducting this analysis, access the Analysis Options within Universal Analysis. Uncheck 'Normalized Area' on the Onset/Peak Integration page to obtain the correct units. If you do not have this program you will need to manually convert the result to non-normalized units.

4. Calculate the crosstalk value for each side using the following equation:

$$\text{crosstalk (\% to Side A)} = \frac{\text{baseline area (mJ)}}{\text{standard area (mJ)}} \times 100\% \quad [\text{data from run 2}]$$

$$\text{crosstalk (\% to Side B)} = \frac{\text{baseline area (mJ)}}{\text{standard area (mJ)}} \times 100\% \quad [\text{data from run 1}]$$

5. Enter the resulting crosstalk values into the Dual Sample DSC **Cell Calibration Page**.
Select OK.

Calibrating the SDT

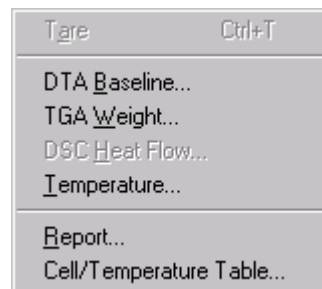
The SDT 2960 is capable of providing several pieces of valuable information about thermal events in materials. All of the information provided (*e.g.*, heat flow, weight change, transition temperature) is quantitative, if proper calibration is done prior to running sample materials. To obtain accurate experimental results, you should calibrate the SDT when you first install it, and periodically thereafter.


The SDT has two standard modes of operation: **DSC-TGA*** and **TGA-DTA**. The calibrations that you will need to perform depend on the mode you are planning to use for your experiments. Either DSC Heat Flow or DTA Baseline calibration must be performed. The other calibrations (TGA Weight and Temperature) are generic calibrations, but must be conducted in the proper calibration mode.

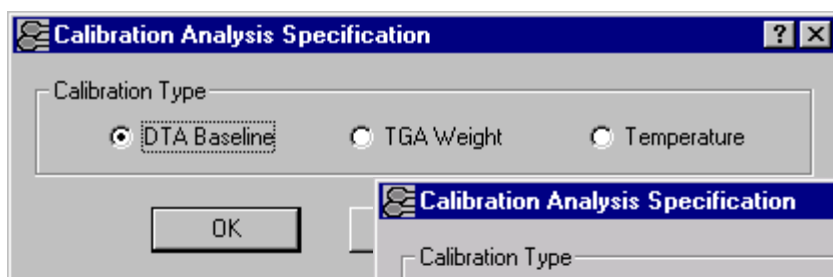
Types of Calibration

The types of SDT calibration procedures that will be performed depend upon the mode that the instrument is currently using. Several types of calibration require gathering calibration data files and then analyzing the files using the calibration analysis functions. Refer to the instructions for the type of calibration desired to find out what is needed.

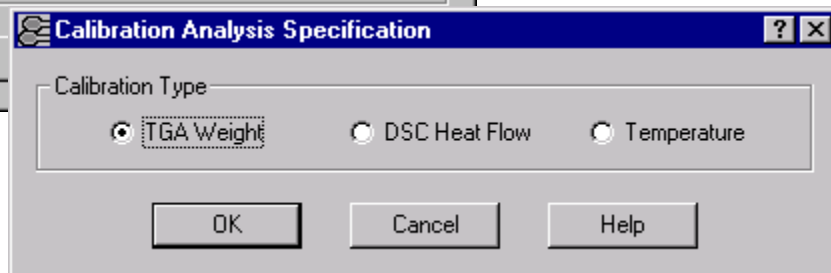
You can access the calibration functions by choosing the desired type of calibration from the **Calibrate** menu (shown here)



or by clicking on the  button on the tool bar to display the applicable **Calibration Analysis Specification** window (both modes shown below). (NOTE: The items listed on this window will vary based on instrument mode.)



TGA-DTA Mode



DSC-TGA Mode

*The DSC-TGA mode is not available on SDT instruments with the 2960 TGA-DTA name on the overlay. However, those instruments can be upgraded with this capability. Contact your local TA Instruments representative for details.

When you calibrate the DTA Baseline or DSC Heat Flow, or perform temperature calibration, you will need to gather and analyze calibration files for the SDT instruments, using the following basic steps:

1. Conduct the calibration experiments.
2. Select a calibration data file.
3. Choose an SDT calibration standard, if applicable.
4. Rescale the plot.
5. Perform the analysis.
6. Print the calibration results.


First this section will discuss taring the SDT, followed by instructions on how to conduct SDT calibration experiments to gather calibration data, then it will cover instructions on performing the remaining types of calibration.

Taring the SDT

The Tare function ensures that the weight measured by the balance reflects the weight of the sample only. You should tare the sample pan before each experiment, even if you use the same pan in consecutive experiments.

When a pan is tared, the instrument reads the weight of the empty sample pan and then stores the weight as an offset, which is subtracted from subsequent weight measurements. For optimum accuracy, the weight reading must be stable before it is accepted as an offset. The SDT determines when the weight reading is sufficiently stable.

Tare procedure:

1. Place the empty sample pan on the sample platform.
2. Close the furnace.
3. Select **Tare** (either on the instrument keypad or by using the *Advantage*TM program to select **Calibrate/Tare** or click the  button on the tool bar). After taring the SDT cups, you can load your reference material (such as aluminum oxide), if desired.

NOTE: A reference material (such as aluminum oxide) is recommended for TGA-DTA experiments as a way to minimize the difference in heat capacity between the reference and sample pans; thereby improving the baseline. Reference material should not be used for DSC-TGA experiments. Rather, calibration and subsequent sample experiments should be performed with an empty alumina cup as the reference.

NOTE: Alumina absorbs moisture. Before using alumina, you will need to dry it. Store alumina powder in a dessicator.

Conducting SDT Calibration Experiments

To calibrate the SDT for DTA Baseline, TGA Weight, DSC Heat Flow, and Temperature, you need to first collect calibration data files that will be analyzed. After the files have been analyzed, the resulting calibration information is stored in the instrument for use when you run experiments. Before you begin performing the experiments to gather calibration data files, there are several issues that you may want to take into consideration. See the section below for details.

SDT Experimental Considerations:

In addition to calibration, there are several experimental considerations that must be considered for the best results:

- **Sample Size and Sample Cups:** Optimum sample size will vary depending upon the magnitude of the transition being investigated. Typically 2 to 20 mg are used. The 90 μ L alumina sample cups should be used for DSC-TGA evaluations.
- **Sample Cup Placement:** The accuracy of the TGA weight signal, which is used to normalize DSC heat flow, depends on consistent placement of the sample cup on the beam's platinum sensor. Position the sample and reference cups as close as possible to the beam side of the sensor. Mark each cup and make sure that you position the cup so that the mark is rotationally in the same position each time you place the cup on the sensor.
- **Temperature Range:** Using the SDT calibration software procedures provide optimum heat flow accuracy and precision in the temperature range of 200 to 1500°C. Outside this range, results can be improved using an additional calibration constant based on evaluation of a heat of fusion standard.

Now you should be ready to gather your data. Refer to the section on the next several pages that is applicable to the type of calibration data desired.

Obtaining DTA Baseline Calibration Data

DTA Baseline calibration is a calibration, which is applied when experiments are conducted in the TGA-DTA mode. Follow these steps when gathering and analyzing DTA Baseline calibration data. If you have already performed TGA weight calibration, you can utilize the first calibration run (without weights) for DTA Baseline calibration. Skip to step 11.

1. Check that your purge gas is connected and set to the desired flow rate (Typically 100 ml/min.)
2. Remove all SDT cups, sample, and weights from the sample and reference beams.
3. Close the furnace.
4. Select **Experimental/Mode** from the SDT menu. Set the instrument to the TGA-DTA Calibration mode using the Mode Selection list.
5. Select the **Experimental View**. On the **Summary Page**, enter the requested sample information, entering zero for both weights. Specify a data file name using the browse button.
6. Select the Ramp test procedure. Click on the **Procedure Page** and select "Use current" as the starting temperature. Enter your upper temperature (or 1500°C) as the Final Temperature. Enter the heating rate that will be used in subsequent experiments (e.g., 20°C/min).
7. Click on the **Notes Page**. Enter the requested information.
8. Click on **Apply** to store the changes made to these pages.
9. Schedule this run, if more than one run is in the sequence list.
10. Select **Start** to begin the calibration run.

Turn to page 2-53 for instructions on using the DTA baseline calibration data you have just obtained.

Obtaining TGA Weight Calibration Data

TGA Weight calibration is a calibration, which is applied when experiments are conducted in either the DSC-TGA or TGA-DTA modes. Follow these steps when gathering and analyzing TGA Weight calibration data. If you have already performed the DTA baseline experiment, you can utilize that same file as the first run (without weights) for TGA Weight Calibration. Skip to step 10.

1. Check that your purge gas is connected and set to the desired flow rate (Typically 100 ml/min.)
2. Remove all SDT cups, samples, and weights from the sample and reference beams.
3. Close the furnace.
4. Select **Experimental/Mode** from the SDT menu. Set the instrument to the TGA-DTA Calibration mode using the **Mode Parameters** window.
5. Select the **Experimental View**. On the Summary Page enter the requested sample information, entering zero for both weights. Specify a data file name using the browse button.
6. Select the Ramp test procedure. Click on the **Procedure Page**. Select "Use current" as the starting temperature. Enter your upper temperature (or 1500°C) as the Final Temperature. Enter the heating rate that will be used in subsequent experiments (e.g., 20°C/min).
7. Click on the **Notes Page**. Enter the requested information.
8. Click on **Apply** to store the changes made to these pages.
9. Schedule this run, if more than one run is in the sequence list.
10. Select **Start** to begin the calibration run. Wait for the experiment to finish and allow the SDT to cool (<50°C).
11. Press the Furnace key on the SDT keypad to open the furnace and place the calibration weights on the beams as follows: the weight marked with an "S" should be placed on the sample (front) platform and the unmarked weight should be placed on the reference (back) platform. Close the furnace.

NOTE: Do not touch the calibration weights with your fingers; use tweezers.

12. From the sequence list, double click on the complete icon (check mark) next to re-schedule the run. This will copy the existing information from the previous run and automatically increment the filename. Alternatively, you could just change the filename or append a new run.
13. Select the **Experimental View**. On the **Summary Page** enter the requested sample information, entering the weights of the sample and reference calibration weights found on the bottom of the box. The filename will automatically increment. Use the same procedure values as found in step 6.
14. Click on the **Notes Page**. Enter the requested information.
15. Click on **Apply** to store the changes made to these pages.
16. Schedule this run, if more than one run is in the sequence list.
17. Select **Start** to begin the calibration run. Wait for the experiment to finish.

Turn to page 2-56 for instructions on using the TGA weight calibration data you have just obtained.

Obtaining SDT Temperature Calibration Data

Temperature calibration is useful for experiments in which precise transition temperatures are essential. To temperature calibrate the SDT, you need to analyze a high-purity metal for its melting temperature, and then enter the observed and correct values in the temperature calibration table. The temperature calibration parameters (observed and correct values) are automatically updated in the **Cell/Temperature Table** window after Temperature Calibration has been performed, accepted, and saved. They may also be manually entered. See "Using the SDT Cell/Temperature Table" in this chapter for details.

A general procedure for SDT temperature calibration is given below.

1. Choose standards with known melting points that encompass your experimental range. Multiple standards may be needed for this purpose. Up to five temperature calibration points may be used. Choose standards whose melting points differ substantially; each pair of melting points must differ from all other pairs by at least 10°C.
2. Create and save a method that will program the SDT to: (a) specify a data sampling interval of 0.5 sec/pt, (b) equilibrate to 200°C above the literature melting temperature of your material (step to premelt the sample prior to analysis), then (c) equilibrate to 200°C below the melting temperature of the material, and (d) heat the material to about 100°C above the literature melting temperature.
3. Place the instrument in calibration mode by selecting **Experimental/Mode**, then choosing TGA-DTA Calibration from the Mode Selection list.

4. Select **Experiment View**, if needed. Click on the **Summary Page**, then enter the requested sample information, entering zero for the sample and reference calibration weights.
5. Prepare approximately 3 to 10 mg of the calibration material in a sample pan, and then position it on the sample platform (front).
6. Prepare a reference pan using the same pan type as that used for the sample, and position it on the reference platform.
7. Close the furnace.
8. Check that your purge gas is connected and set to the desired flow rate. (Typically 100 mL/min.)
9. Select **Start** to begin the calibration run and wait until it has been completed.
10. Repeat steps 1 through 9 for each temperature standard used.

Turn to page 2-61 for instructions on using the temperature calibration data you have just obtained.

Obtaining DSC Heat Flow Calibration Data

DSC Heat Flow calibration is a calibration, which is applied when experiments are conducted in the DSC-TGA mode. Follow these steps when gathering and analyzing DSC Heat Flow calibration data.

1. Check that your purge gas is connected and set to the desired flow rate. (Typically 100 mL/min.) Remove all SDT cups, samples, and weights.
2. Place two clean and dry, empty alumina (ceramic) cups on the sample and reference beams. Adjust the position of the cups on the platinum sensor to be as close to the beams as possible. Use the same cup position in all subsequent experiments.

NOTE: Obtaining the best heat flow reproducibility and accuracy in the DSC-TGA mode requires careful attention to details like cup positioning. The cups should be placed on the sensor in the same position for both calibration and regular experiments. The "same position" in this context refers, not only to lateral position, but also rotational position.

3. Close the furnace.
4. Select **Experimental/Mode**. Set the instrument to the DSC Calibration mode using the **Mode Parameters** window.

5. Once the balance has stabilized, press the TARE key on the instrument keypad.
6. Select the **Experimental View Pane - Summary Page**. Enter the requested sample information. Specify a data file name using the Browse button.
7. Select the Ramp test procedure. Click on the **Procedure Page** and select "Use current" as the starting temperature. Enter your upper temperature (or 1500°C) as the Final Temperature. Enter the heating rate that will be used in subsequent experiments (e.g., 20°C/min).
8. Click on the **Notes Page** and enter the requested information.
9. Click **Apply** to store the changes made to these pages.
10. Schedule this run, if more than one run is in the sequence list.
11. Select **Start** to begin the calibration run. Wait for the experiment to finish, and allow the SDT to cool (<50°C).
12. Once the balance has stabilized, press the TARE key on the instrument keypad.
13. Press the FURNACE key on the SDT keypad to open the furnace, and place the sapphire standard (PN 915079.903) into the sample (front) cups. Adjust the cup positions, if needed, as described in step 2.
14. Close the furnace.
15. On the list of runs in the **Sequence Pane**, double click on the complete icon (check mark) next to reschedule the run. This will copy the existing information from the previous run and automatically increment the filename. Alternatively, you could just change the filename or append a new run.
16. Select the **Experimental View - Summary Page**. Alter the sample information to specify sapphire as the sample. The filename will automatically be incremented. Repeat the same experiment, using the same procedures as above with the sapphire standard.
17. Click on **Apply** to store the changes made to these pages.
18. Schedule this run, if more than one run is in the sequence list.
19. Select **Start** to begin the next calibration run. Wait for the experiment to finish.


Turn to page 2-58 for instructions on using the DSC heat flow calibration data you have just obtained.

Obtaining Cell Constant Calibration Data

Cell constant is a calibration factor used to adjust the calorimetric response. The cell constant is the ratio of the known value to the measured experimental variable. Typically, the heat of fusion of zinc is used. The calibration is based on a run made in the DSC-TGA mode using the experimental conditions (*i.e.*, purge gas, heating rate) to be used in subsequent measurements.

NOTE: The cell constant information obtained with the resulting data file must be manually entered. See page 2-60 for information.

A general procedure for cell constant calibration is given below:


1. Check that your purge gas is connected and set to the desired flow rate.
2. Select **Experimental/Mode** from the main menu. Set the instrument to DSC-TGA mode using the Mode Selection list.
3. Place two clean and dry, empty alumina (ceramic) cups on the sample and reference beams. Adjust the position of the cups on the platinum sensor to be as close to the beams as possible. Use the same cup position in all subsequent experiments.
4. Close the furnace.
5. Select **Calibrate/Cell/Temperature Table** from the SDT menu. Enter 1.00 into the Cell Constant field (see page 2-63).
6. Select **Tare** (either on the instrument keypad or by using the *Advantage*TM program to select **Calibrate/Tare** or click the  button on the tool bar).
7. Prepare approximately 5 mg of the zinc (supplied in the accessory kit) in a sample cup.
8. Click on the **Summary Page** tab in the Experimental View. Select a Custom test procedure.
9. Click on the **Procedure Page** tab. Click the Editor button and create the following method:
 - 1 Sample interval: 0.5 sec/pt
 - 2 Data storage: Off
 - 3 Equilibrate at 550.00°C
 - 4 Equilibrate at 250.00°C
 - 5 Data Storage: On
 - 6 Ramp 20.00°C/min to 50.00°C

10. Click on the **Notes Page** tab. Enter/verify the requested information.
11. Click on the **Apply** button to save the experimental and sample parameters entered for this run.
12. If more than one run is in the sequence list, schedule this run. (An arrow will appear next to the run number in the **Sequence Pane** for the scheduled run.)
13. Select **Start** to begin the calibration run.

After you have obtained the calibration file, turn to page 2-60 for instructions on how to analyze the file.

Analyzing SDT Calibration Files

After gathering the necessary calibration files as directed on the previous pages, you will need to analyze the data and save the calibration results. To do this you will need to access the calibration functions by choosing the desired type of calibration from the

Calibrate menu or by clicking on the  button on the tool bar to display the applicable **Calibration Analysis Specification** window. Follow the instructions in the appropriate section found on the next several pages to analyze your calibration files.

Performing the DTA Baseline Calibration (for SDT)


DTA Baseline Calibration is based on analyzing data collected from a baseline run conducted over the temperature range expected in subsequent TGA-DTA mode experiments. (This experiment usually utilizes the same baseline run obtained for TGA Weight Calibration.)

The DTA baseline is corrected by a linear (slope and offset) function of the sample temperature. Data is collected from a baseline run either with or without pans. This results in shifting and rotating the baseline so that the calibrated portion is near 0°C. This calibration is applied when using the TGA-DTA mode.

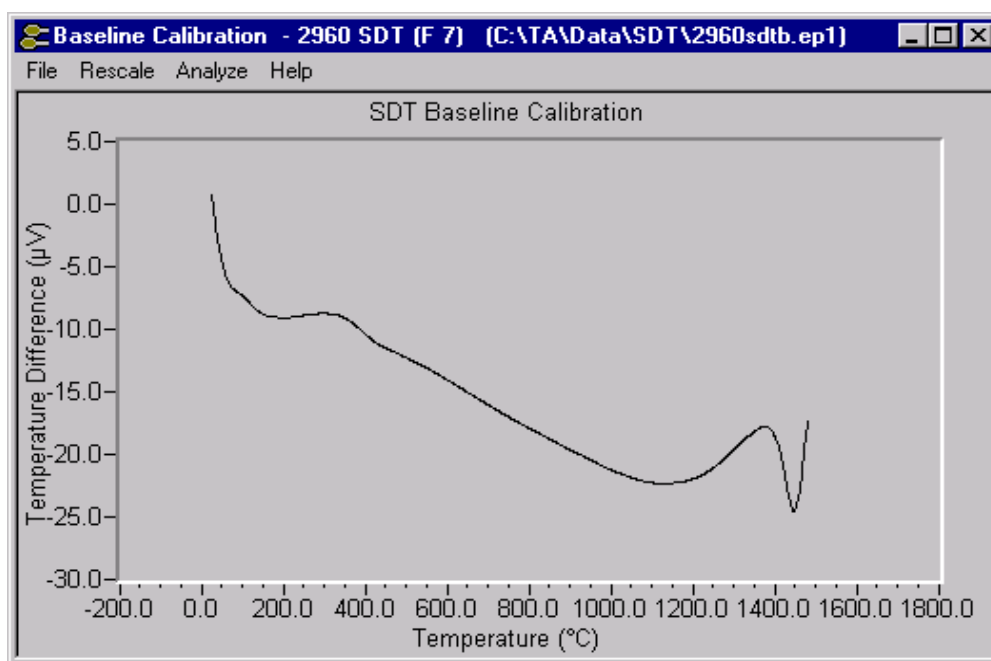
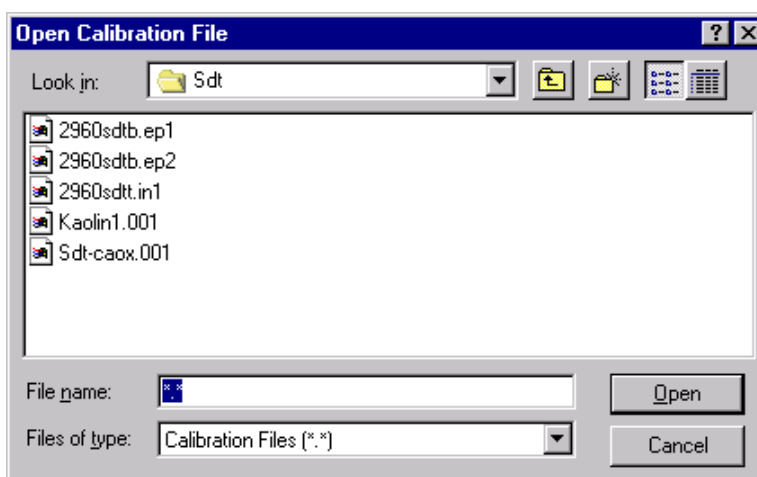
NOTE: The calibration data should be obtained with the instrument in TGA-DTA Calibration mode (selected on the Mode Parameters window).

NOTE: The DTA baseline calibration is not required when using the SDT as a DSC-TGA. This is because the DSC Heat Flow Calibration includes a baseline subtraction. However, the shape of the DTA baseline is valuable as a quick verification that the SDT beams are correctly positioned and that DSC heat flow calibration can be successfully completed.

Follow these steps to perform the calibration:

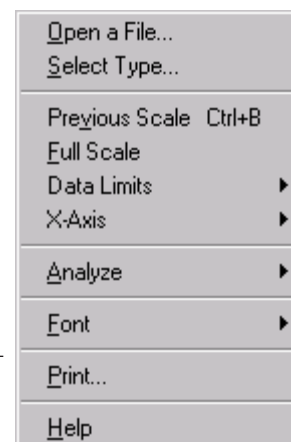
1. Put the instrument in TGA-DTA Calibration mode (selected on the **Mode Parameters** window).
2. Conduct the calibration experiments.
3. Select **DTA Baseline** either from the SDT Calibrate menu or by clicking on the  button on the tool bar to display the **Calibration Analysis Specification** window.

4. After you choose **DTA Baseline Calibration**, an **Open Calibration File** window (shown here) is displayed allowing you to specify the appropriate file for the selected calibration.
5. Select the DTA baseline calibration file you have already created. The main **SDT Calibration** window is displayed.



6. Select **Analyze/Analyze** from the menu (or select **Analyze** from the Calibration pop-up menu shown to the right). The current limit points will be indicated with markers on the graph.

NOTE: The active marker is indicated by a full-length vertical red line. Other markers are indicated by smaller red cross-hairs.



7. Move the active marker to the desired position on the curve using either the mouse or keyboard (see the directions below).

Moving the Markers Using the Mouse:

1. Position the pointer at the desired location.
2. Double-click mouse button 1 to move the active marker to this point. This action causes the next marker to become active.

or

1. Position the pointer over the active marker.
2. Press and hold down the left mouse button while you drag the marker to the desired location.
3. Release the mouse button. At this point you may use the arrow keys to position the marker more closely (see the directions below).

Moving the Markers Using the Keyboard:

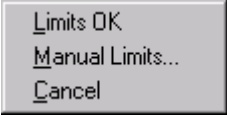
Press the arrow keys to move the active marker to the desired position.

NOTE: The marker will move in the following increments:

- Arrow key = smallest step
- Alt/arrow key = second smallest step
- Shift/arrow key = second largest step
- Control/arrow key = largest step

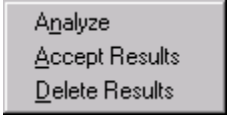
8. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB (Shift-TAB) will activate the previous marker)
9. Repeat steps 7 and 8 until you have selected all the necessary points for analysis.

10. Select **Analyze/Limits OK** or select **Limits OK** from the pop-up menu (shown here) to accept the points. When calculated, the calibration results will be displayed.



Limits OK
Manual Limits...
Cancel

11. Accept the calibration results by selecting **Analyze/Accept results** or selecting **Accept Results** from the pop-up menu (shown here). You can delete results by selecting **Analyze/Delete results** or **Delete Results** from the pop-up menu.




Analyze
Accept Results
Delete Results

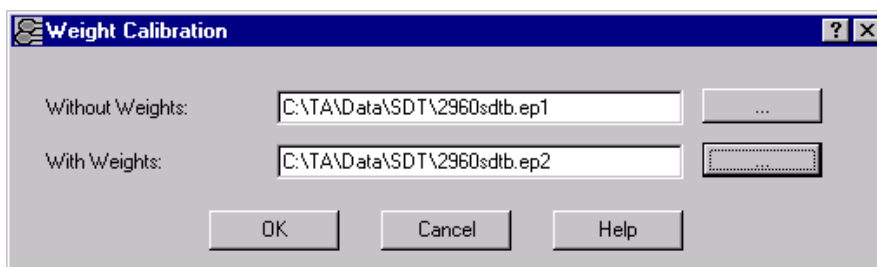
Performing TGA Weight Calibration (for SDT)

Weight calibration should be performed on the SDT at least once a month. TGA weight calibration is based on two runs conducted in the TGA-DTA calibration mode—one using calibration weights and one using no weights (empty beams). The data from both runs is analyzed and beam and weight correction factors are calibrated. This calibration is applied when using either the DSC-TGA or TGA-DTA mode.

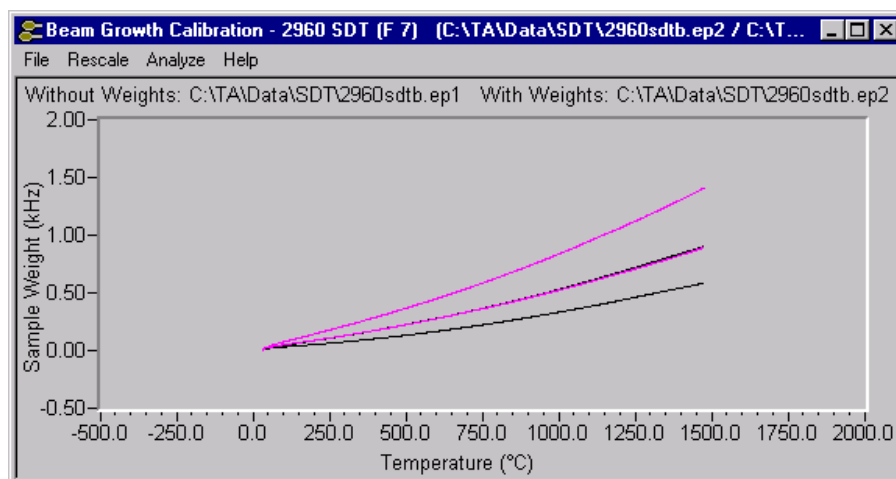
Follow these steps to perform the calibration:

1. Put the instrument in TGA-DTA Calibration mode (selected on the **Mode Parameters** window).
2. Conduct the calibration experiments.
3. Select **TGA Weight** from the SDT Calibrate menu or by clicking on the  button on the tool bar to display the **Calibration Analysis Specification** window.

4. After you select **TGA Weight calibration**, the **Weight Calibration** window (shown here) is displayed.



5. Enter the filename (including path) for the first experiment (empty beams) or click on the Browse button to choose the file. Enter the filename for the second experiment (with weights) or click on the Browse button to choose the file.
6. Select **OK** when both the calibration files have been entered. The Calibration program will display a series of four curves. (NOTE: In the example shown below, two of the middle curves are overlapping and appear as one).



7. Select **Analyze/Analyze** from the menu (or select **Analyze** from the Calibration pop-up menu). The current limit points will be indicated with markers on the graph. When selecting your TGA limits, the default limits of 200 and 1400°C are the optimum limits to use when calibrating the SDT up to 1500°C. If you are only interested in a particular portion of the curve, such as running methods up to 1000°C, you may decide to choose more appropriate limits.

NOTE: The active marker is indicated by a full-length vertical red line. Other markers are indicated by smaller red cross-hairs.

8. Move the active marker to the desired position on the curve using either the mouse or keyboard.
9. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB (Shift-TAB) will activate the previous marker.)
10. Repeat steps 8 and 9 until you have selected all the necessary points for analysis.
11. Select **Analyze/Limits OK** or select **Limits OK** from the pop-up menu to accept the points. When calculated, the calibration results will be displayed.
12. Accept the calibration results by selecting **Analyze/Accept results** or selecting **Accept Results** from the pop-up menu. (TGA weight calibration values are not displayed, only the resultant curve.) The weight signals should track each other in a smooth continuous upwards sweep (see the SDT Operators manual for details). Likewise, the corrected baseline signal should be flat across the screen using the default scaling. If any of these curves are not as described, rerun your baseline calibration. Pay careful attention to make sure your instrument is not physically jarred, that the calibration weights are seated properly on the platform, and that the purge flow is steady. If the curves still do not look proper after rerunning the calibration, contact your service representative.

You can delete results by selecting **Analyze/Delete results** or **Delete Results** from the pop-up menu.

13. Close the Calibration window.

The Calibration Report menu can be used to view the date this calibration was last conducted.

Performing DSC Heat Flow Calibration (for SDT)


*This calibration is available only for DSC-TGA mode** and is required when running in this mode. SDT heat flow calibration is based on analyzing the heat capacity curve for sapphire over the range 200 to 1500°C and the heat of fusion of high purity zinc metal. Three experimental runs are required: one using an empty alumina cup (90µL) for both the reference and sample (baseline run) and one using a sapphire standard (supplied in the SDT Accessory Kit) as the sample. The measured heat capacity for sapphire is compared with the literature value at multiple temperatures across the range and mathematically fitted to generate an E-curve.

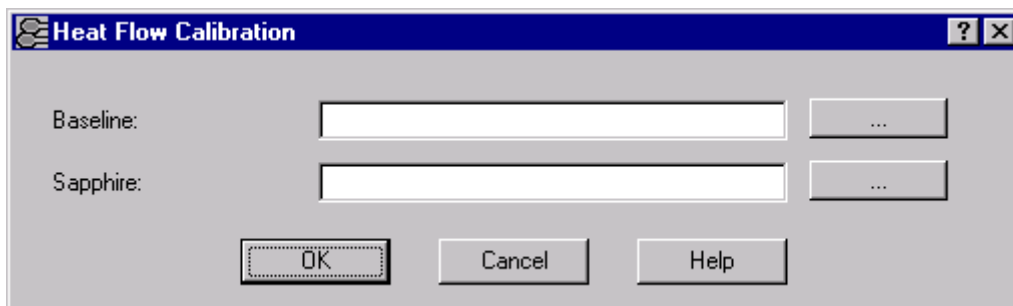
This E-curve may be further refined by a third experiment that measures the heat of fusion of high purity zinc wire (supplied in the accessory kit). The heat of fusion is measured and the cell constant is calculated using the known value of the heat of fusion of zinc (108.7 J/g) and the equation “Cell Constant = Known Value/Measured Value.” The calculated value of the cell constant is then entered into the Cell Constant field in the **Calibration/Cell/Temperature Table** from the SDT menu.

*This calibration is only available when the optional DSC-TGA capability is enabled for the SDT.

NOTE: The calibration data should be obtained with the instrument in DSC Calibration mode (selected on the Mode Parameters window).

Follow these steps to perform the calibration:

1. Put the instrument in DSC Calibration mode (selected on the **Mode Parameters** window).
2. Conduct the calibration experiments.
3. Select **DSC Heat Flow** from the SDT Calibrate menu or by clicking on the  button on the tool bar to display the **Calibration Analysis Specification** window. The **DSC Heat Flow Calibration** window (shown below) is displayed.



4. Enter the filename (including path) for the first experiment (empty alumina cups), or click on the Browse button to choose the file. Enter the filename for the second experiment (with sapphire), or click on the Browse button to locate the file.
5. Select OK when both the calibration files have been entered. The Calibration program will display both files.
6. Select **Analyze/Analyze** from the menu (or select **Analyze** from the Calibration pop-up menu). The current limit points will be indicated with markers on the graph.

NOTE: When you are selecting your analysis limits, the default limits of 200 and 1400°C are recommended as the optimum limits to use when calibrating the SDT up to 1500°C.

7. Move the active marker to the desired position on the curve using either the mouse or keyboard.
8. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB (Shift-TAB) will activate the previous marker).
10. Select **Analyze/Limits OK** or select **Limits OK** from the pop-up menu to accept the points. When calculated, the calibration results will be displayed.
11. Accept the calibration results by selecting **Analyze/Accept results** or selecting **Accept Results** from the pop-up menu. (DSC Heat Flow calibration values are not displayed, only the resultant curve.)

NOTE: The baseline file data must be greater than the sapphire data to complete the analysis.

If results are not satisfactory, rerun your calibration experiments. Pay careful attention to make sure that the beams are positioned within the center of the furnace, that the ceramic cups are seated properly on the platform, and that the purge flow is steady. If the results are still not satisfactory after rerunning the calibration, contact your service representative.

12. Close the Calibration window.

The Calibration Report menu can be used to view the date this calibration was last conducted.

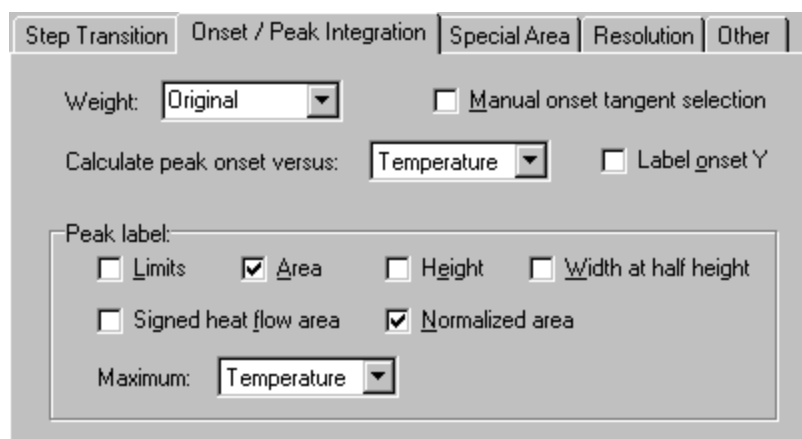
Performing the Cell Constant Analysis for SDT

This analysis involves the use of the Advantage Universal Analysis program. For details on the use of that program, see the online help available when the program is opened.

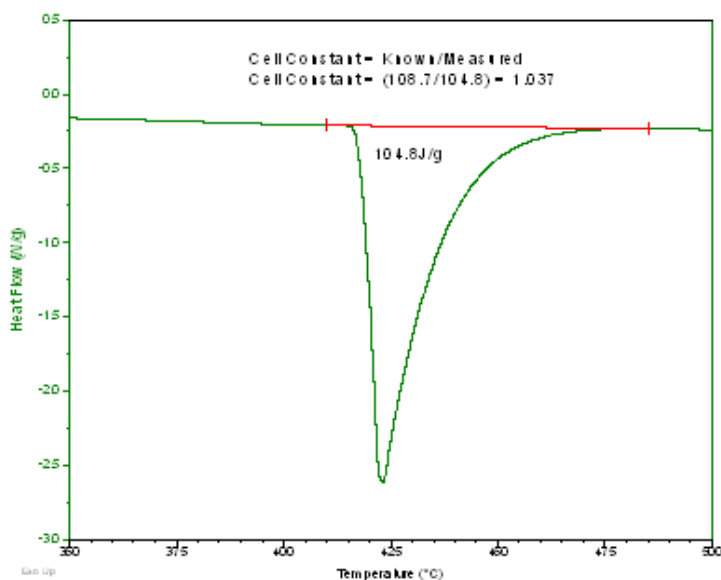
1. Double click the Universal Analysis icon to start the program.

2. Open the zinc data file gathered for the SDT calibration.

3. Select **Analyze/Options** from the menu. Then, click on the **Onset/Peak Integration Page** and set up the options as shown in the figure to the right.



4. Integrate the melting endotherm using the **Analyze/Peak Integration/Linear Baseline** function. The normalized area of the melting endotherm is then used to calculate the cell constant. See the example below.




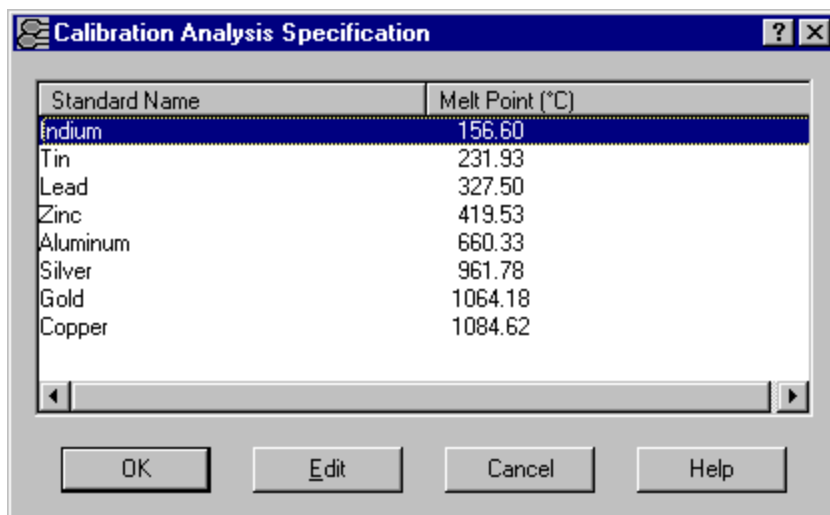
5. Return to the Advantage Instrument Control program and select **Calibrate/Cell/ Temperature Table** from the SDT menu. Enter the calculated value of the cell constant into the Cell Constant field (see page 2-63).

Performing Temperature Calibration for SDT

Temperature calibration is useful for experiments in which precise transition temperatures are essential. To temperature calibrate the SDT, you need to analyze a high-purity metal for its melting temperature, and then enter the observed and correct values in the temperature calibration table. The temperature calibration parameters (observed and correct values) are automatically updated in the **Cell/Temperature Table** window after Temperature Calibration has been performed, accepted, and saved. They may also be manually entered.

Follow these steps to perform the calibration:

1. Put the instrument in TGA-DTA Calibration mode (selected on the **Mode Parameters** window).
2. Conduct the calibration experiments.
3. Select **Temperature** from the SDT Calibrate menu or by clicking on the  button on the tool bar to display the **Calibration Analysis Specification** window.
4. After you choose **Temperature** calibration, a window is displayed to allow you to open the file to be used for analysis. Select the calibration data file created previously and select OK.
5. The **Calibration Analysis Specification Standard** window is displayed. This window (shown here) displays the most common standards used to temperature calibrate the SDT, along with their theoretical melting point temperature.
6. Select the standard to be used from the list. (If you want to edit the standard or its melting point, highlight the standard and click the Edit button.)
7. Select OK. The Calibration program will plot file.



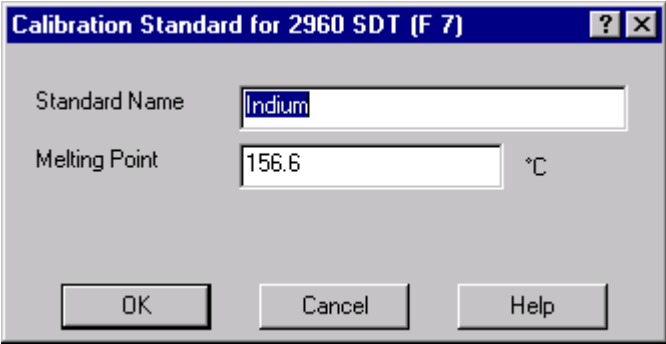
8. Select **Analyze/Analyze** from the menu (or select **Analyze** from the Calibration pop-up menu). The current limit points will be indicated with markers on the graph. Integrate the melting endotherm to determine onset to melting temperature.
9. Move the active marker to the desired position on the curve using either the mouse or keyboard.
10. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB (Shift-TAB) will activate the previous marker.)
11. Select **Analyze/Limits OK** or select **Limits OK** from the pop-up menu to accept the points. When calculated, the calibration results will be displayed.
12. Accept the calibration results by selecting **Analyze/Accept results** or selecting **Accept Results** from the pop-up menu.
13. Close the Calibration window.

The temperature calibration parameters (observed and correct values) are automatically updated in the **Cell/Temperature Table** window after Temperature Calibration has been performed, accepted, and saved. See “Using the Cell/Temperature Calibration Table” on the next page for details.

Editing an SDT Standard

To edit an SDT standard value or enter a new standard, follow steps 1 through 5 on the previous page to open the **Calibration Analysis Specification Standard** window. Double-click on the standard name or click **Edit**. The **Calibration Standard** window (shown here) appears.

Enter the desired values and click OK when finished.



NOTE: The standards list will return to the default values each time the Temperature Calibration Standard Specification window is reopened.

NOTE: The melting points for indium, tin, zinc, aluminum, silver, gold, and copper are defined by the International Temperature Scale of 1990 (ITS-90).

Using the SDT Cell/Temperature Calibration Table

When you select **Calibrate/Cell/Temperature Table** from the SDT menu, the **Cell/Temperature Table** (shown here) is displayed. The items shown on this window depend on the instrument mode. This window is to edit the cell calibration parameters or the temperature calibration table.

Cell Calibration Parameters

Use this table to set the cell constant, baseline slope, or baseline offset that the instrument will apply to the corrected data. Each cell calibration parameter is briefly described as follows:

- **Cell Constant for SDT:** Cell Constant is a calibration factor used to adjust the instrument's response. The default value is 1.00.
- **Baseline Slope for SDT:** This parameter is a calibration constant that accounts for the DTA baseline slope of an empty cell. The baseline slope is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. The RMS (root mean square) average of the data is calculated to determine the best straight-line fit. From this line the baseline slope is calculated to account for the difference in output between the reference and sample thermocouples. Default is 0.00.

Cell Calibration	
Cell Constant	0
Baseline Slope	0
Baseline Offset	0

Temperature Calibration Table		
	Observed Temperature °C	Correct Temperature °C
Point 1:	9.67	9.79
Point 2:	99.67	99.79
Point 3:		
Point 4:		
Point 5:		

Reset Table

OK Cancel Help

NOTE: This calibration only applies when the instrument is in the TGA-DTA mode.

- **Baseline Offset for SDT:** Baseline Offset is a calibration factor that shifts the curve up or down along the y-axis to balance the heat flow at the sample and reference thermocouple positions. The baseline offset is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. The RMS (root mean square) average of the data is calculated to determine the best straight-line fit. From this line the DTA baseline offset is calculated to return the curve to the ideal reference (zero).

NOTE: This calibration only applies when the instrument is in the TGA-DTA mode.

To verify/edit the cell calibration parameters, follow these steps:

1. If the value is correct, select OK.
2. If a change is desired, enter the correct value in the appropriate field and select OK.

The baseline calibration parameters are automatically updated in this window after DTA baseline calibration has been performed, accepted, and saved. Baseline slope and offset are not available in the DSC-TGA mode.

Temperature Calibration Table

The lower part of the **Cell/Temperature Calibration Table** shows the temperature calibration table that the instrument applies to the collected data. You can use this area to observe the values that were automatically entered after you performed temperature calibration, or to manually enter from one to five temperature calibration points (pairs of observed and correct temperature points). The observed and correct temperature corresponds to the experimental and theoretical transition temperature (e.g., melting point) of the calibrant respectively.

NOTE: A multiple-point calibration is more accurate than a one-point calibration. A single-point calibration shifts the sample temperature by a constant amount. A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and maintains a constant temperature shift after the last point.

- If these values are correct, select OK.
- If new values are to be entered:
 1. Select the Reset button to remove all previously stored values.
 2. Determine the temperature points.
 3. Enter the Observed and Correct temperature points in the table.
 4. Select OK when all points have been entered to save the settings to the instrument.

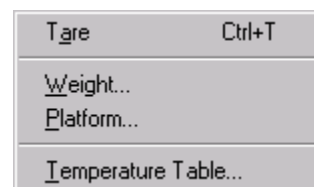
NOTE: When temperature calibration is in effect, Temp in *C appears in the status line. When it is not in effect, Temp in °C appears in the status line.

Calibrating the TGA

The TGA 2950 is capable of providing several pieces of valuable information about thermal events in materials. All of the information provided (*e.g.*, weight change) is quantitative, if proper calibration is done prior to running sample materials. To obtain accurate experimental results, you should calibrate the TGA when you first install it, and periodically thereafter.

Types of Calibration

You can access the calibration functions by choosing the desired type of calibration from the TGA **Calibrate** menu (shown here). Refer to the instructions for the type of calibration desired on the next several pages.



Taring the TGA


The **Tare** function ensures that the weight measured by the balance reflects the weight of the TGA sample only.

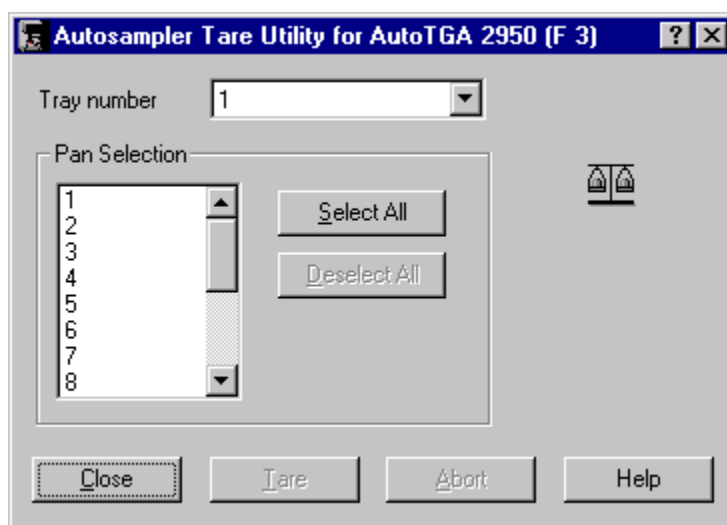
When a pan is tared, the instrument reads the weight of the empty sample pan and then stores the weight as an offset, which is subtracted from subsequent weight measurements. For optimum accuracy, the weight reading must be stable before it is accepted as an offset. The TGA determines when the weight reading is sufficiently stable.

You should tare the sample pan before each experiment, even if you use the same pan in consecutive experiments. Taring is done for both TGA weight ranges.

Tare procedure:

1. Place the empty sample pan(s) on the sample platform.

2. Select **Tare** either on the instrument keypad, by selecting **Calibrate/Tare** from the TGA menu, or by clicking on the  button on the tool bar. If you are using an autosampler (AutoTGA), the **Tare Utility** window will be displayed. This allows you to specify whether you will be taring the entire platform or specific pan number(s).



Weight Calibration for TGA

Weight calibration should be performed on the TGA at least once a month. Because the TGA has two weight ranges, taring is done for both ranges. The tare weight is stored by the instrument for the appropriate weight range.

The weight calibration functions guide you through the calibration procedure step-by-step. You will need to obtain the following items for this procedure:

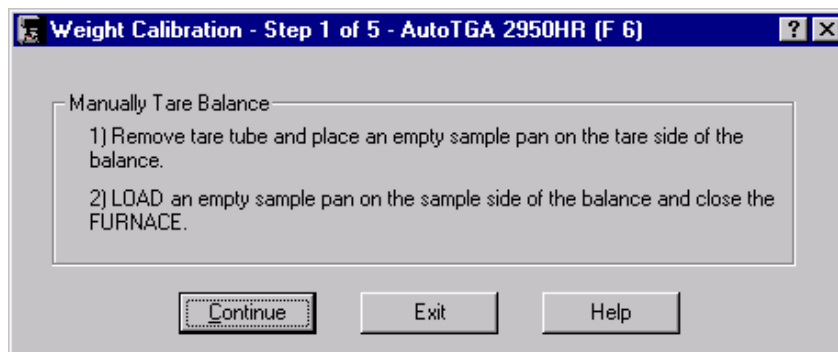
- two (2) empty sample pans
- calibration weights
- brass tweezers.

NOTE: Always handle the calibration weights with brass tweezers, not with your fingers. The oils and salt from your skin can change the calibration weight.

The next several pages provide the steps needed to perform TGA weight calibration. The instructions can also be found on the windows displayed as you step through the procedure using the *Advantage™* program.

Step 1 of 5: Manually Tare the Balance

When you choose **Calibrate/Weight** from the TGA menu, the first window that is displayed is the **Weight Calibration - Step 1 of 5** window (shown here). Follow these steps:

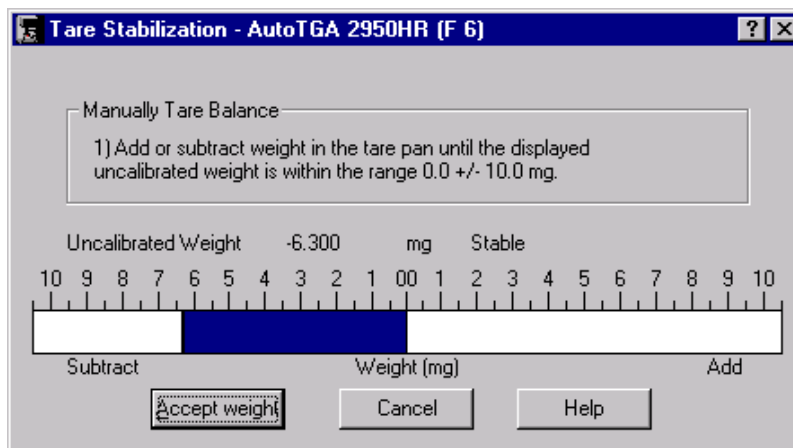


1. Unscrew the tare tube in a clockwise direction, then remove it.
2. Hang an empty sample pan of the same type and size as your experimental sample pan on the tare hook. This is your tare pan.
3. Place the sample pan that you plan to use in your experiment on the sample platform.
4. Press the LOAD key on the instrument keypad to load the pan onto the sample hook.
5. Press the FURNACE key on the instrument keypad to close the furnace. Closing the furnace prevent air currents from affecting the weight reading.

- Click Continue to proceed. The **Tare Stabilization** window is displayed. See “Using the Tare Stabilization Window” below for instructions.

Using the Tare Stabilization Window

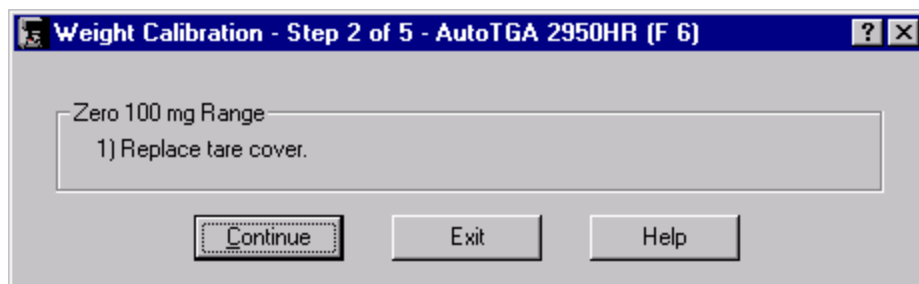
The **Tare Stabilization** window displays the uncalibrated weight of the sample pan and the sample pan weight reading. Using this information you can manually tare the balance. Observing the scale, you will need to add or subtract calibration weights until the displayed Uncalibrated Weight is within the range 0.0 +/-10.0 mg as follows:



- If the scale shows a reading that is on the positive side, you will need to add calibration weights (found in the TGA Accessory kit) to the tare pan.
- If the scale shows a reading that is on the negative side, you will need to remove calibration weights from the tare pan.
- Continue to add or remove weight until the weight has stabilized to within the acceptable range, then select Accept weight.

Step 2 of 5: Zero the 100 mg Range

The **Weight Calibration - Step 2 of 5** window (shown here) should now be displayed. The instrument is now measuring the weight in the 100 mg range. The tare weight is stored by the instrument for the 100 mg range.



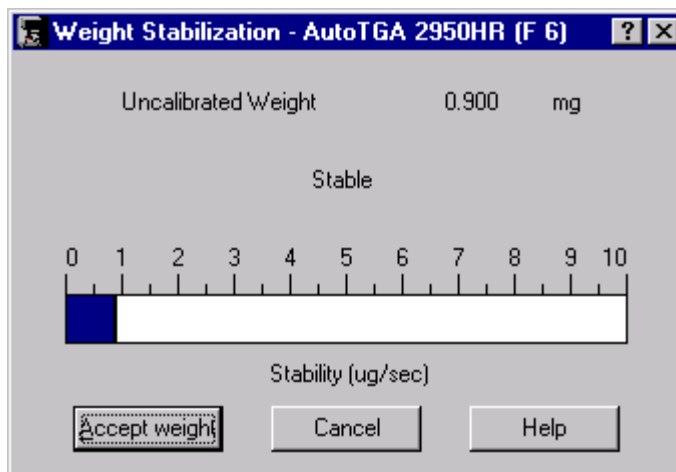
- Replace the tare tube, by turning it in a counterclockwise direction to screw it back into the instrument. This will prevent air currents from affecting the weight measurement.

2. Select **Continue** to go on with the calibration procedure. The **Weight Stabilization** window is displayed. See "Using the Weight Stabilization Window" below for instructions.

Using the Weight Stabilization Window

The **Weight Stabilization** window shows you the progress and status of the weight on the TGA. Follow these instructions:

1. Wait for the weight to stabilize. You will see the display read "Settling" as the balance reaches equilibrium. The weight may not equal zero, but it must be stable when you accept it, as indicated by the displayed word "Stable."
2. Select **Accept weight** to go on with the calibration procedure.



Step 3 of 5: Zero the 1000 mg Range

The instrument is measuring the weight in the 1000 mg range. The tare weight is stored by the instrument for the 1000 mg range.

Select **Continue** to go on with the calibration procedure. The **Weight Stabilization** window is displayed. See "Using the Weight Stabilization Window" above for instructions.

Step 4 of 5: Calibrate the 100 mg Range

This step in the procedure calibrates the 100 mg weight range for the TGA instrument.

1. Obtain the 100 mg class M standard weight from the TGA Accessory Kit.
2. Place the 100 mg weight in the sample pan.
3. Select LOAD to load the pan onto the sample hook.
4. Press the FURNACE key to close the furnace. Closing the furnace prevent air currents from affecting the weight reading.

5. Enter the exact mass of the standard as seen on the TGA display (default value = 100.0 mg), then select **Continue**. The system will begin measuring the combined weight of the standard and the pan. The **Weight Stabilization** window is displayed. See "Using the Weight Stabilization Window" on the previous page for instructions.

Step 5 of 5: Calibrate the 1000 mg Range

This step in the procedure calibrates the 1000 mg weight range for the TGA instrument.

1. Obtain the 1000 mg class M standard weight from the TGA Accessory Kit.
2. Select UNLOAD to unload the sample pan and remove the 100 mg weight.
3. Place the 1000 mg weight in the sample pan.
4. Select LOAD to load the pan onto the sample hook.
5. Enter the exact mass of the standard as seen on the TGA display (default value = 1000.0 mg), then select **Continue**. The system will begin measuring the combined weight of the standard and the pan. The **Weight Stabilization** window is displayed. Once the weight reading is close to 1000 mg (± 50 mg), then press the FURNACE key to close the furnace and stabilize the weight. See "Using the Weight Stabilization Window" on the previous page for instructions. After you accept the weight, the calibration is complete.
6. Unload the sample pan.

Adjusting the TGA Sample Platform

The Sample Platform Adjust procedure is used if the sample hang-down wire fails to pick up a sample pan during an automatic loading procedure. The sample platform must be adjusted so that the instrument can properly load and unload the sample pans.

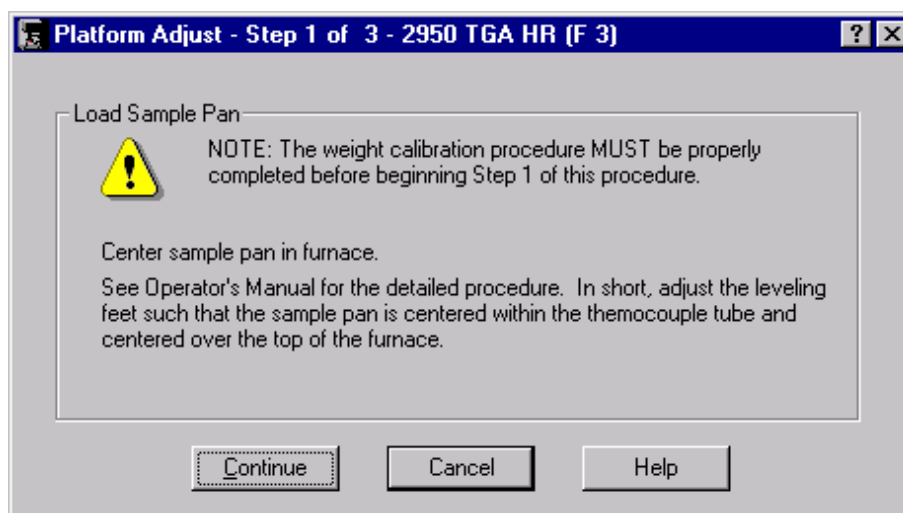
To avoid weight signal noise, the TGA must be level so that the sample pan and hang-down wire hang inside the furnace and thermocouple tube without touching them. The first step of the sample platform calibration procedure adjusts and levels the instrument.

Select **Calibrate/Platform** from the TGA menu. The first window in a step-by-step series of instructions is displayed. The next several pages provide the steps needed to perform TGA sample platform calibration. The instructions can also be found on the windows displayed as you step through the procedure using the *Advantage*TM program.

NOTE: The sample platform adjustment procedure is slightly different for the TGA Autosampler, turn to page 2-70 for those instructions.

Step 1: Center the Sample Pan

In order to center the sample pan, the top and bottom of the sample hang-down wire must be adjusted and the instrument leveled. When you choose **Calibrate/Platform** from the TGA menu, the **Platform Adjust Step 1** window (shown here) is displayed.

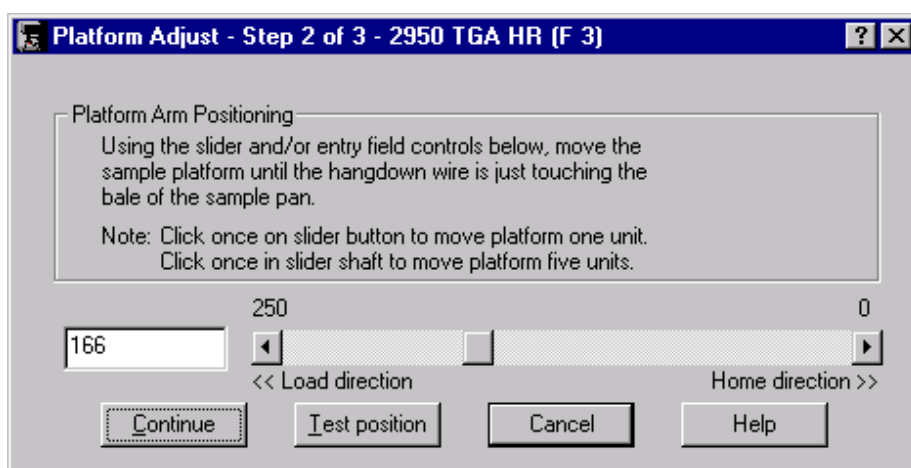


1. Load an empty sample pan on the instrument balance (it can be done automatically or manually).
2. Check to see whether the top end of the sample hang-down wire is hanging freely and roughly centered within the top of the thermocouple tube inside the balance chamber. If the wire is not roughly centered, turn the balance adjustment screw (see your instrument manual for details) until the wire is centered.

3. Raise the furnace just to the bottom of the sample pan.
4. Check the alignment of the sample pan within the furnace. It should hang freely, roughly centered, and should not be touching the sides of the furnace or the hang-down tube. If the pan is not centered, adjust the feet on the bottom of the instrument until the pan hangs correctly. Turn the feet clockwise to lengthen or counterclockwise to shorten the instrument leveling feet.
5. Select **Continue** to go on with the calibration procedure.

Step 2: Platform Arm Positioning

The **Platform Adjust - Step 2** window (shown here) should now be displayed. Once the sample pan has been centered over the furnace, you will need to adjust the position of the platform arm so that the pans can load and unload correctly.



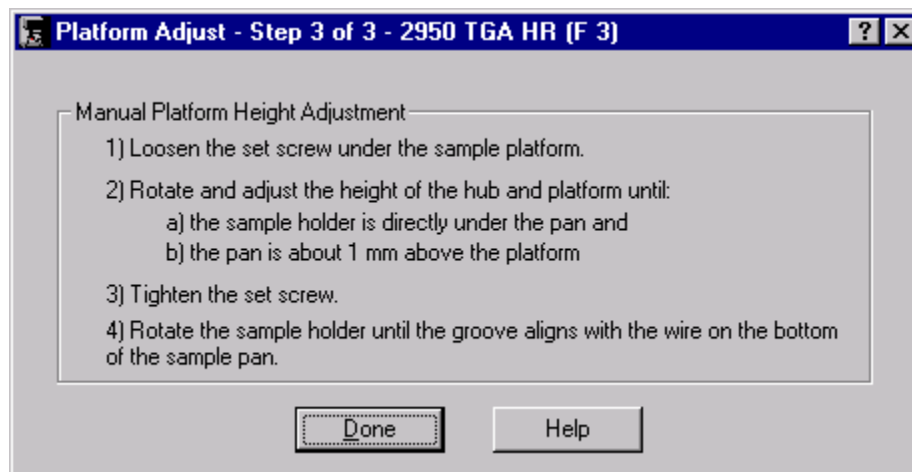
- You can enter the number of units that you want to move the platform in the box on the left side of the window,
- or
- You can use the slide bar to change the platform position by: (a) clicking on the appropriate load direction button to move the platform one unit at a time, (b) clicking in the slider shaft to move the platform five units for each click, or (c) placing the cursor on the slider and holding the mouse button down as you slide it in the desired direction.

Select the **Test Position** button to move the platform arm to the selected position, then select **Continue** to go on with the calibration procedure.

Step 3: Manual Platform Adjustment

The **Platform Adjust - Step 3** window should now be displayed.

1. Use a screwdriver to loosen the setscrew located under the sample platform.
2. Move the sample platform: (a) rotate it until the sample holder is directly under the pan, then (b) adjust the height of the platform until the bottom of the pan is located approximately 1 mm above the platform.
3. Use the screwdriver to tighten the setscrew again (do not overtighten as you may strip the threads).
4. Rotate the sample pan holder until the groove in the pan hole aligns with the wire on the bottom of the sample pan.
5. Select **Done** when these steps have been accomplished. The Sample Platform Adjustment has been completed.



Adjusting the AutoTGA Sample Platform

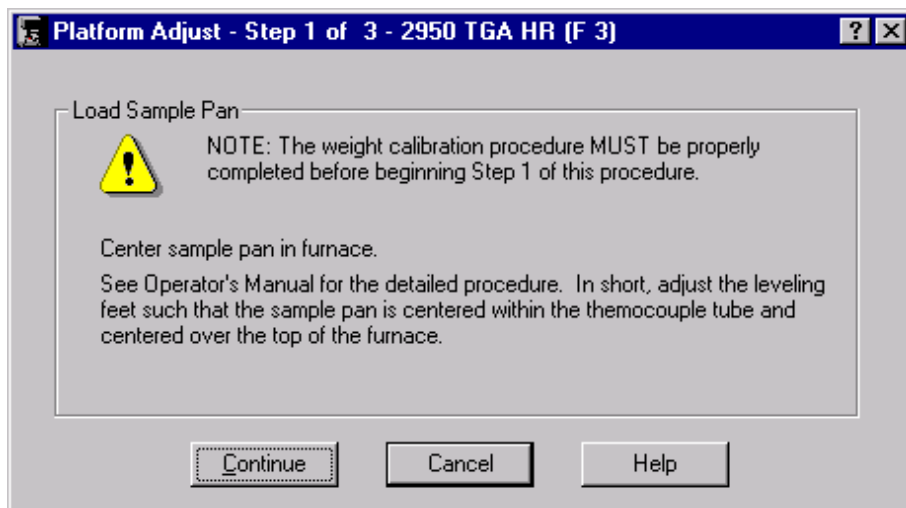
The Sample Platform Adjust procedure is used if the sample hang-down wire fails to pick up a sample pan during an automatic loading procedure. The Autosampler sample platform must be adjusted so that the instrument can properly load and unload the sample pans.

To avoid weight signal noise, the TGA must be level so that the sample pan and hang-down wire hang inside the furnace and thermocouple tube without touching them. The first step of the sample platform calibration procedure adjusts and levels the instrument.

Select **Calibrate/Platform** from the AutoTGA menu. The first window in a step-by-step series of instructions is displayed. The next several pages provide the steps needed to perform the AutoTGA sample platform calibration. The instructions can also be found on the windows displayed as you step through the procedure using the *Advantage* program.

Step 1: Center the Sample Pan

In order to center the sample pan, the top and bottom of the sample hang-down wire must be adjusted and the instrument leveled. When you choose **Calibrate/Platform** from the TGA menu, the **Platform Adjust Step 1** window (shown here) is displayed.



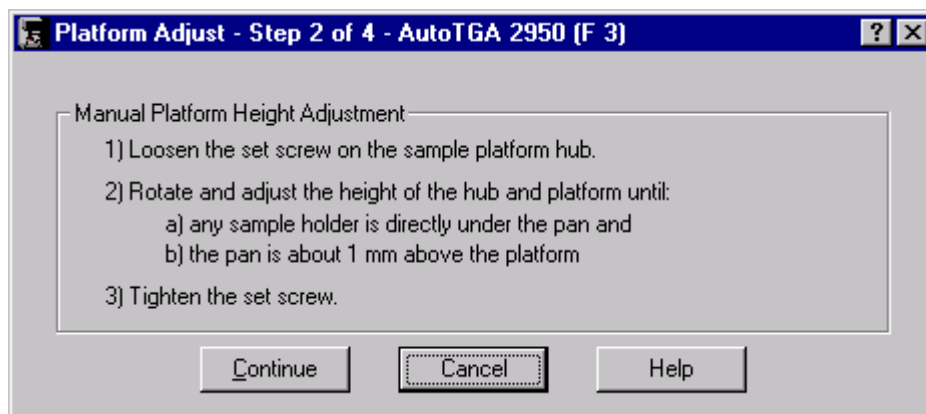
1. Load an empty sample pan on the instrument balance (it can be done automatically or manually).
2. Check to see whether the top end of the sample hang-down wire is hanging freely and roughly centered within the top of the thermocouple tube inside the balance chamber. If the wire is not roughly centered, turn the balance adjustment screw (see your instrument manual for details) until the wire is centered.
3. Raise the furnace just to the bottom of the sample pan.
4. Check the alignment of the sample pan within the furnace. It should hang freely, roughly centered, and should not be touching the sides of the furnace or the hang-

down tube. If the pan is not centered, adjust the feet on the bottom of the instrument until the pan hangs correctly. Turn the feet clockwise to lengthen or counterclockwise to shorten the instrument leveling feet.

5. Select **Continue** to go on with the calibration procedure.

Step 2: Manual Platform Height Adjustment

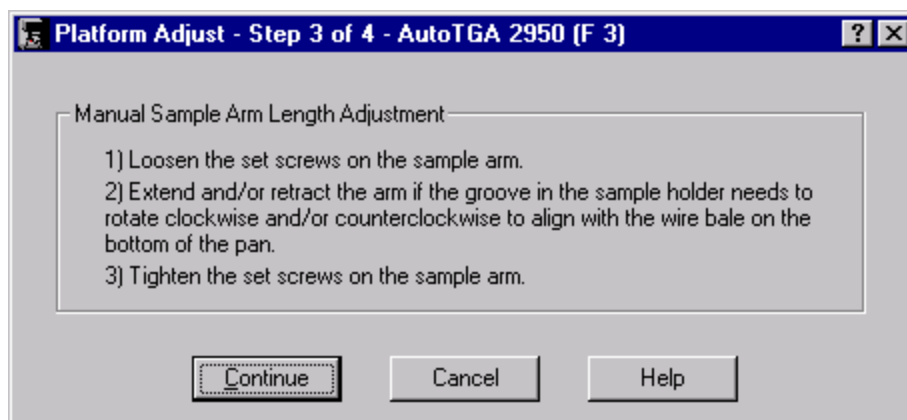
The **Platform Adjust - Step 2** window (shown here) should now be displayed. After the sample pan has been centered in step 1, the height of the platform must be manually adjusted as follows:



1. Use a screwdriver to loosen the setscrew located in the platform hub.
2. Move the sample platform: (a) rotate the hub and platform until the sample holder is directly under the pan, then (b) adjust the height of the hub and platform until the bottom of the pan is located approximately 1 mm above the platform.
3. Use the screwdriver to tighten the setscrew again (do not overtighten as you may strip the threads).
4. Select **Continue** to go on with the calibration procedure.

Step 3: Manual Sample Arm Length Adjustment

The **Platform Adjust - Step 3** window (shown here) should now be displayed. The length of the autosampler arm must now be adjusted to allow proper pickup of the samples.



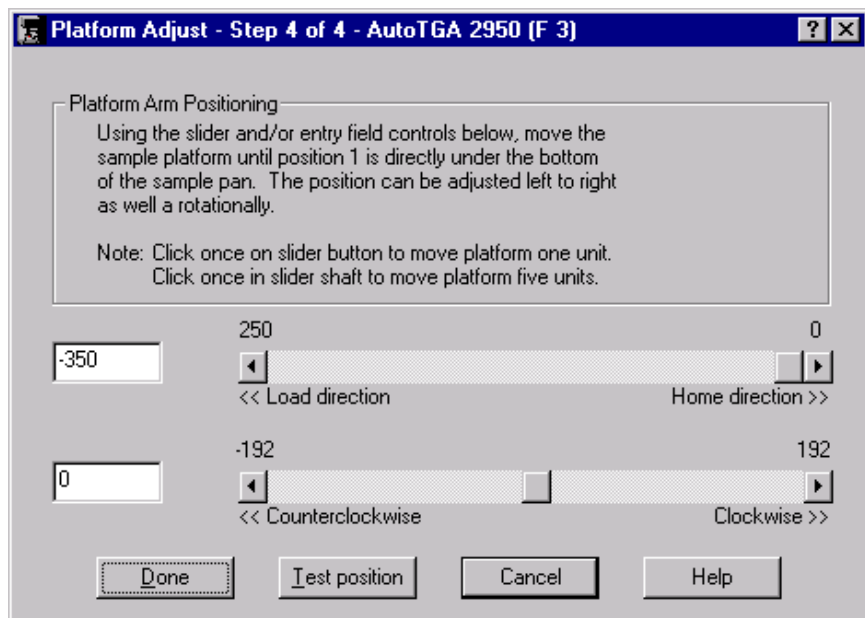
1. Use a screwdriver to loosen the two setscrews located on the sample arm.
2. Move the sample arm to adjust the position of the groove: (a) move the sample arm OUT to make the sample holder groove rotate clockwise, or (b) move the sample arm IN to make the sample holder groove rotate counterclockwise. When the groove aligns with the wire bale on the bottom of the pan, go on to the next step.
3. Use the screwdriver to tighten the two setscrews on the sample arm.
4. Select **Continue** to go on with the calibration procedure.

Step 4: Platform Arm Positioning

NOTE: There may be a slight delay while the instrument initializes to prepare for this step. The window controls will be disabled until this operation has been completed.

The **Platform Adjust - Step 4** window (shown here) should now be displayed. Once the sample pan has been centered over the furnace, you will need to adjust the position and rotation of the platform arm so that the pans can load and unload correctly.

- You can enter the number of units that you want to move the platform, in the box on the left side of the position or rotation slide bar.



or

- You can use the slide bar to change the platform position and rotation by: (a) clicking on the appropriate direction button to move the platform one unit at a time, (b) clicking in the slider shaft to move the platform five units for each click, or (c) placing the cursor on the slider and holding the mouse button down as you slide it in the desired direction.

Select the **Test Position** button to move the platform arm to the selected position and rotation, then select **Done** when you are satisfied that the platform arm is in the correct position.

Temperature Calibration for the TGA

Temperature calibration is useful for experiments in which precise transition temperatures are essential. To temperature calibrate the TGA, first you need to analyze a high-purity metal for its curie temperature, and then enter the observed and correct values in the temperature calibration table.

NOTE: The Temperature Calibration Table is not available when the instrument is in the calibration mode.

Before you can perform the temperature calibration procedure for the TGA, you must first reset the **Temperature Calibration Table** as follows:

1. Select **Calibrate/ Temperature** from the TGA menu. The **Temperature Table** (shown here) is displayed.
2. Click the **Reset** button to reset all temperature calibration data before performing any new calibration experiments.

	Observed Temperature °C	Correct Temperature °C
Point 1:	9.67	9.79
Point 2:	99.67	99.79
Point 3:		
Point 4:		
Point 5:		

Buttons: OK, Reset, Cancel, Help

3. Click OK.
4. Gather the calibration data by following the steps on the next page to determine the curie temperature.
5. Perform the temperature calibration as directed on the next page.

Determining Curie Temperature

A general procedure for curie temperature calibration is given below.

1. Choose standards that encompass your experimental range. Multiple standards may be needed for this purpose. Choose standards whose curie temperature differs substantially; each pair of curie temperatures must differ from all other pairs by at least 10°C.
2. Place the curie temperature sample in a tared TGA sample pan and position the pan on the sample platform.
3. Select the **Experiment View**. Enter the requested sample information, including name on the **Summary Page**. Select the Ramp test from the Test list.
4. Click on the **Procedure Page**. Enter the requested test parameters that will program the TGA to: (a) equilibrate to 100°C below the onset of the literature curie temperature of your material, and (b) heat the material, at the same heating rate that you will use in your experiments, to above the literature curie temperature.
5. Click on the **Notes Page**. Enter/verify the requested information.
6. Click on the **Apply** button to save the experimental and sample parameters entered for this run. If more than one run is in the sequence list, schedule this run. (An arrow will appear next to the run number in the **Sequence Pane** for the scheduled run.)
7. Start the method and observe Signal A (weight loss %) on the **Signal Display Pane**.
8. Slowly raise the magnet under the furnace until a weight gain (< 2%) is detected. Secure the magnet in this position for the duration of the experiment.
9. Determine the curie temperature by analyzing the extrapolated end point of the S-shaped curve using the data analysis program.
10. Enter the Observed and Correct temperatures in the Temperature table when all the experiments are complete as directed in the next section.

Enter the Temperature Calibration

1. Select **Calibrate/Temperature** from the TGA menu. The **Temperature Table** is displayed. This window shows the temperature calibration table that the instrument applies to the collected data. Use this window to enter from one to five temperature calibration points (pairs of observed and correct temperature points). The observed and correct temperature corresponds to the experimental and theoretical transition temperature (*e.g.*, melting point) of the calibrant respectively.
2. If these values are correct, select OK.

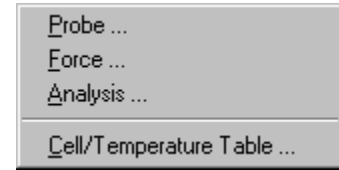
If new values are to be entered:

- a. Enter the Observed and Correct temperature points in the table.
- b. Select OK when all points have been entered to save the settings to the instrument.

NOTE: When temperature calibration is in effect, Temp in *C appears in the status line. When it is not in effect, Temp in °C appears in the status line.

Calibrating the TMA

Use the TMA **Calibration** menu to choose from three types of calibration: probe, force, and analysis. These calibrations should be performed when you first receive the instrument, when software is changed, and periodically (approximately once a month) thereafter.



- **Probe calibration** calibrates the LVDT and adjusts the force calibration factor to compensate for any differences in the different TMA probe types
- **Force calibration** is done with any probe in place on the instrument. It calibrates the force exerted by the probe on the sample during experiments.
- The **Analysis** menu item is used to access the calibration program used to conduct cell constant and temperature calibration. Cell constant calibration is used to adjust the dimension data through the calibration of thermal expansion. Temperature calibration is used to adjust the sample thermocouple.
- The **Cell/Temperature Table** option is used to enter the cell parameters and auto measure offset (for film/fiber calibration). The cell parameters are determined using the analysis options above.

Calibrating the TMA Probe

You will need to perform the TMA probe calibration every time you change the instrument mode or when you change the probe on the TMA. This procedure is sometimes called “initializing.” The following actions are performed whenever you calibrate a probe:

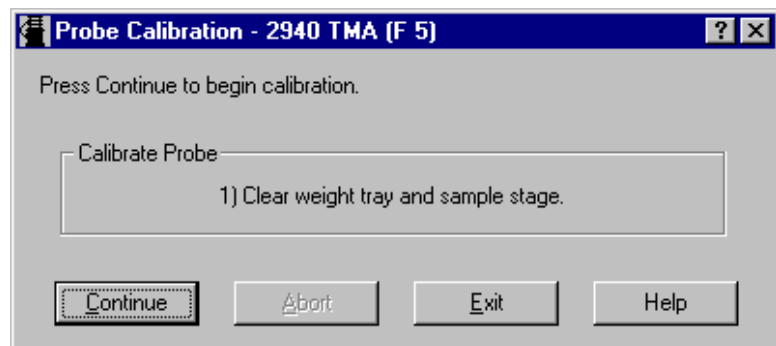
- Calibrate the LVDT.
- Calibrate the probe's mass.
- Calibrate the probe's compliance.

To calibrate the probe installed on the instrument, follow the directions displayed.

1. Select **Calibrate/Probe** from the TMA menu or



located on the tool bar. The **Probe Calibration** window (shown here) is displayed.



2. Take all weights off the weight tray and clear all samples off the sample stage.
3. Click the **Continue** button on the window. The calibration will be performed and a message is displayed when the procedure has been completed.

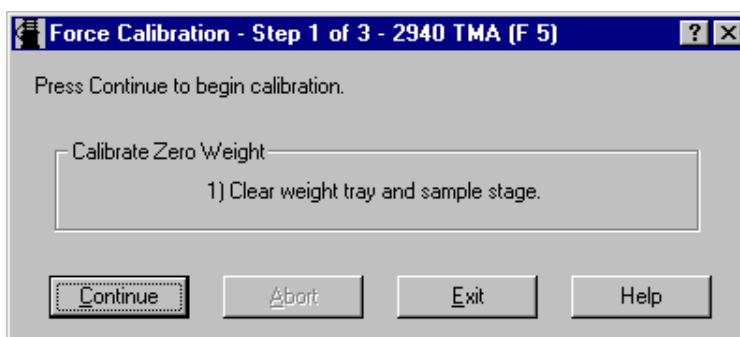
Performing the TMA Force Calibration

The TMA force calibration procedure is used to calibrate the force exerted by the probe on the sample during experiments. It can be done with any probe in place on the instrument. You will need to perform the force calibration on a regular basis to maintain the accuracy of the instrument.

Step 1: Calibrate Zero Weight

The first step is performed to provide a zero reference point for calibration.

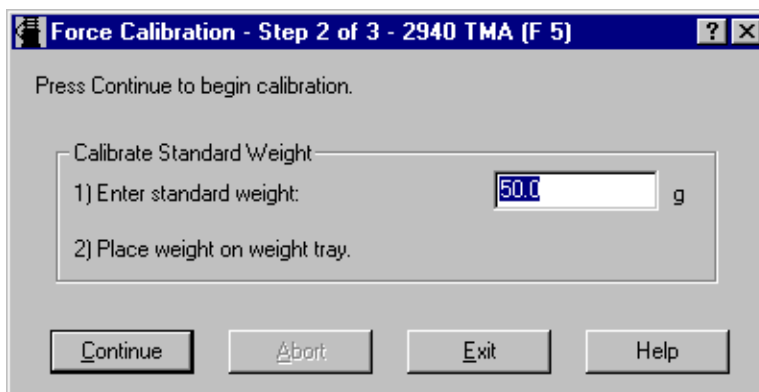
1. Select **Calibrate/Force** from the TMA menu. The **Force Calibration - Step 1 of 3** window (shown here) is displayed.



2. Install the desired probe and its stage on the TMA. (Any probe type may be used.)
3. Clear the weight tray and the sample stage of any items.
4. Select Continue to go on with the calibration procedure.

Step 2: Calibrate Standard Weight

After performing step 1, the **Force Calibration - Step 2 of 3** window (shown here) is displayed. The second step in the force calibration procedure is performed to provide a standard calibration reference point. The recommended weight amount is 50 grams, although from 10 to 100 grams is allowable.

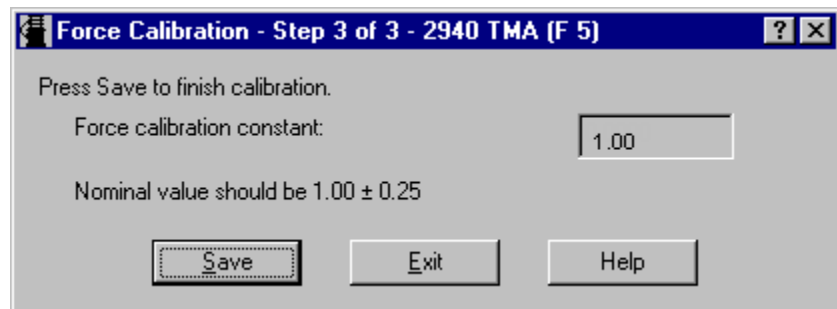


1. Enter the weight, in grams, of the standard you are using. The standard weight value entered must be between 10.0 and 100.0 grams.
2. Place the standard weight on the weight tray.
3. Select Continue to go on with the calibration procedure.

NOTE: If you choose Abort, you must restart from step 1 to complete the force calibration or select Exit to cancel the calibration.

Step 3: Observe results

The force calibration constant is calculated and displayed on the **Force Calibration - Step 3 of 3** window (shown here). The value should be in the range of 1.00 ± 0.25 .



- If the force calibration constant does fall within this range, select Save.
- If the force calibration constant does not fall within this range, select Exit and repeat the calibration procedure.

Using the TMA Analysis Function

The TMA calibration analysis functions are used to perform two types of calibrations—cell constant and temperature calibration.

Cell constant is calculated by dividing the literature value for the coefficient of linear thermal expansion (CLTE) of aluminum by the measured value. If a standard material other than aluminum is used, consult the ASTM Standard Test Method E-381 for the procedure to calculate the cell constant based upon the expansion of that material.

The cell constant parameter is automatically updated in the TMA Calibration Parameters window after cell constant calibration has been performed, accepted, and saved.


The basic steps needed to perform a calibration analysis are:

- Gathering calibration data.
- Selecting a calibration data file.
- Choosing the calibration type and standard, if applicable.
- Rescaling the plot.
- Choosing analysis limits.
- Analyzing and saving the data.
- Printing the calibration results.

Follow the instructions in the sections that follow to perform these steps.

Conducting TMA Calibration Experiments

To calibrate the TMA for cell constant and temperature, you need to first collect calibration data files that will be analyzed. All calibration runs must be performed in the appropriate calibration mode, which depends on the type of probe installed. After the files have been analyzed, the resulting calibration information is stored in the instrument for use when you run experiments.

Select **Experimental/Mode** from the TMA menu or click the  button on the tool bar to open the **Mode Parameters** window. Select one of the following calibration modes:


- **TMA Calibration Mode:** The mode to be used to generate uncorrected data, which is needed to perform temperature and cell constant calibration runs used to calibrate the instrument.
- **TMA Film/Fiber Calibration:** The mode to be used to generate uncorrected data, which is needed to perform temperature and cell constant calibration runs to calibrate the instrument when the film/fiber probe is installed.

To gather calibration data, follow the instructions found on the next several pages, based on the type of calibration desired.

Obtaining TMA Cell Constant Data

Cell constant calibration is based on a run in which a known sample (standard) is heated through its transition temperature, and data is gathered for analysis. The cell constant is calculated by dividing the actual coefficient of expansion of the standard by the measured coefficient of expansion.

Follow these steps when gathering and analyzing TMA cell constant calibration data:

1. Select and install the desired probe on the TMA.
2. Select **Experimental/Mode** from the TMA menu. Set the instrument to appropriate calibration mode using the Mode Selection list (TMA Calibration or TMA Film/Fiber Calibration).
3. If you are switching between the standard and film/fiber modes, you will need to perform probe calibration. Select **Calibrate/Probe** or click the  button on the tool bar and follow the directions needed to calibrate the probe.
4. Select the **Experimental View**. Enter the requested sample information on the **Summary Page**. Select the Ramp test template.
5. Click on the Procedure tab. Enter the requested test parameters that will program the TMA to: (a) Start the experiment at least 20°C below the desired start temperature limit, and (b) Heat the material, at the same heating rate that you will use in your subsequent experiments, to 20°C above the desired upper temperature limit. Use a preload and applied force of 0.01 to 0.05 N.
6. Click on the Notes tab. Enter/verify the requested information.
7. Click on the **Apply** button to save the experimental and sample parameters entered for this run.
8. Zero the probe using the ZERO LENGTH on the TMA keypad.
9. Load the aluminum standard found in the TMA Accessory Kit or use aluminum foil for the film/fiber probe.
10. Verify the thermocouple position. For all probes, except film/fiber accessory, position the tip of the thermocouple so that it is against the platform. The tip should be angled towards the sample location. For the film/fiber accessory, position the thermocouple tip so that it is at the middle of the sample length.
11. Press MEASURE LENGTH on the TMA keypad. The size field on the **Experimental View** will be updated automatically once this process is complete.
12. Check that your purge gas is connected and set at the desired flow rate.

13. If more than one run is in the run sequence list, schedule this run. (An arrow will appear next to the run number in the run sequence list for the scheduled run.) Select Start to begin the calibration run.
14. Wait until the run is complete, then access the calibration program using Calibrate/Analysis on the TMA Menu. Select the TMA cell constant calibration data file created above.
15. Analyze the calibration data file using the procedures found in the section “Analyzing TMA Calibration Data.”

Obtaining TMA Temperature Calibration Data

Temperature calibration is based on a run in which a temperature standard (*e.g.*, indium) is heated through its melting point. The recorded melting point of this standard is compared to the known melting point, and the difference is calculated for temperature calibration.


In addition, you can use up to four other standards to calibrate temperature. If you use one pair of known and observed points, the entire curve is offset, or shifted, to the actual melting point. If you use multiple standards, the temperature is corrected by a cubic spline fit.

NOTE: A multiple-point temperature calibration is more accurate than a one-point calibration.

A single-point calibration shifts the sample temperature by a constant amount.

A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and shifts by a constant amount after the last point.


This calibration should be performed when the sample thermocouple is changed, periodically (approximately once a month), and when you are changing the temperature range of interest.

1. Select and install the desired probe on the TMA.
2. Select **Experimental/Mode** from the TMA Menu. Set the instrument to appropriate calibration mode using the Mode Selection list (TMA Calibration or TMA Film/Fiber Calibration).
3. If you are switching between the standard and film/fiber modes, you will need to perform probe calibration. Select **Calibrate/Probe** or click the  button on the tool bar and follow the directions needed to calibrate the probe.

4. Select the **Experimental View**. Enter the requested sample information on the Summary Page. Select the Ramp test template.
5. Click on the Procedure tab. Enter the requested test parameters that will program the TMA to: (a) Start the experiment at least 20°C below the desired start temperature limit, and (b) Heat the material, at the same heating rate that you will use in your subsequent experiments, to 20°C above the desired upper temperature limit. Use a preload and applied force of 0.01 to 0.05 N.
6. Click on the Notes tab. Enter/verify the requested information.
7. Click on the **Apply** button to save the experimental and sample parameters entered for this run.
8. Zero the probe using the ZERO LENGTH on the TMA keypad.
9. Load the melting standard on the stage. For all probe types, except the film/fiber accessory, small flattened pieces of standard metals are placed on the stage. To protect the stage from amalgamation with the metal, it is recommended that aluminum or platinum be placed between the stage and the metal standard. The end of the probe can also be wrapped with foil for added protection. For the film/fiber probe, metal wires can be crimped into the aluminum balls and used for calibration.
10. Verify the thermocouple position. For all probes, except film/fiber accessory, position the tip of the thermocouple so that it is against the platform. The tip should be angled toward the sample location. For the film/fiber accessory, position the thermocouple tip so that it is at the middle of the sample length.
11. Press MEASURE LENGTH on the TMA keypad. The size field on the Experimental View will be updated automatically once this process is complete.
12. Check that your purge gas is connected and set at the desired flow rate.
13. If more than one run is in the run sequence list, schedule this run. (An arrow will appear next to the run number in the run sequence list for the scheduled run.) Select Start to begin the calibration run.
14. Wait until the run is complete, then access the calibration program using Calibrate/Analysis on the TMA Menu. Select the TMA temperature calibration data file created above.
15. Analyze the calibration data file using the procedures found in the section “Analyzing TMA Calibration Data.”

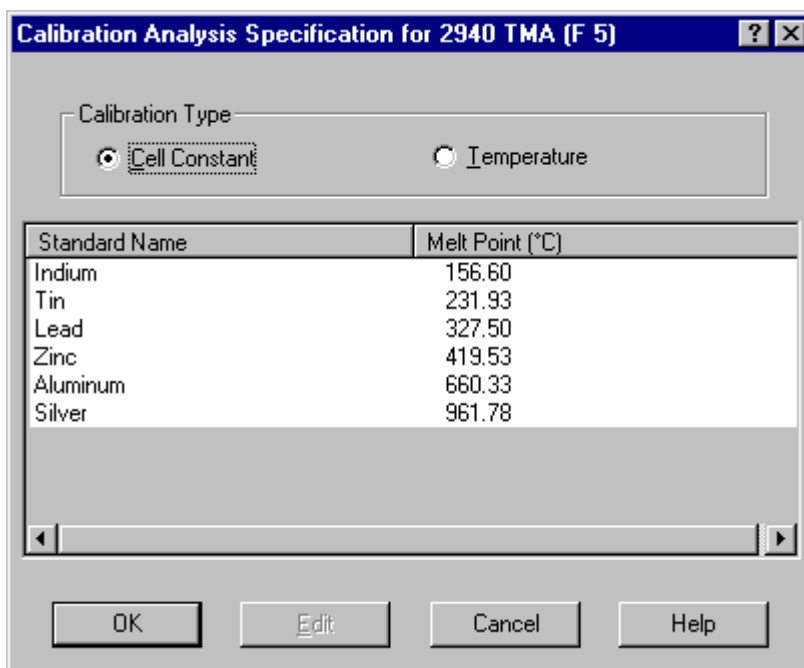
Analyzing TMA Calibration Data

After you have gathered the calibration data using the procedures on the previous page, you will need to analyze the data.

1. Select the **Calibrate/Analysis** menu item on the TMA menu or click on  located

on the tool bar. An open file window is displayed to allow you to select the calibration file you have already created.

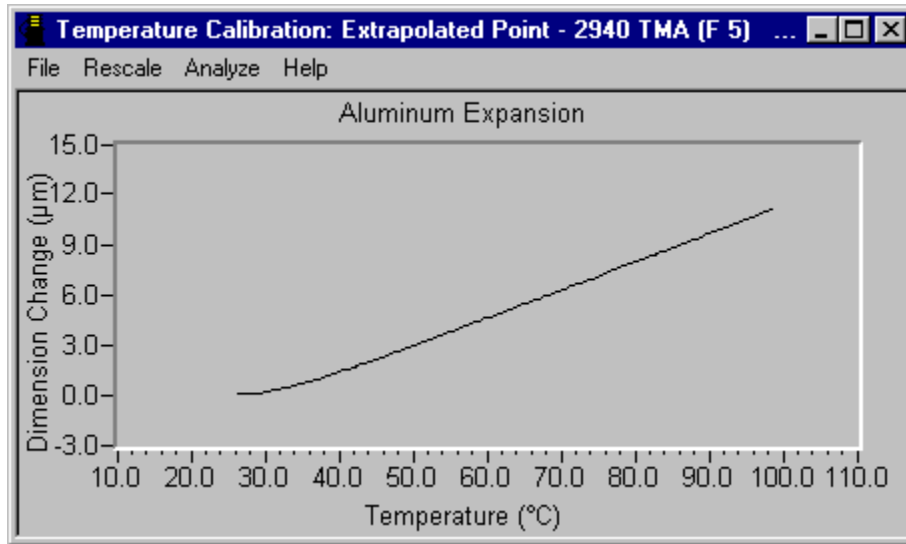
2. Select the appropriate file and click OK. The **Calibration Analysis Specification** window (shown here) is displayed. This window is used to select the type of calibration to be performed.



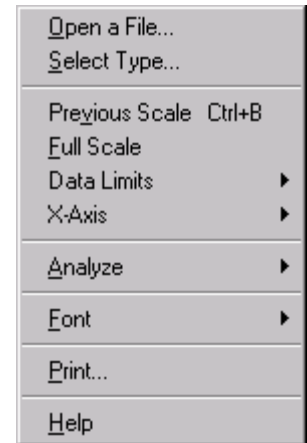
The dialog box titled "Calibration Analysis Specification for 2940 TMA (F 5)" contains a "Calibration Type" section with two radio buttons: "Cell Constant" (selected) and "Temperature". Below this is a table with two columns: "Standard Name" and "Melt Point (°C)". The table lists Indium (156.60), Tin (231.93), Lead (327.50), Zinc (419.53), Aluminum (660.33), and Silver (961.78). At the bottom are four buttons: OK, Edit, Cancel, and Help.

Standard Name	Melt Point (°C)
Indium	156.60
Tin	231.93
Lead	327.50
Zinc	419.53
Aluminum	660.33
Silver	961.78

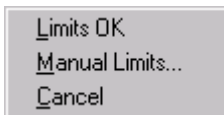
- **Cell Constant** is a calibration factor used to adjust the calorimetric response of a DSC cell or the thermal expansion of a TMA. The cell constant is the ratio of the known value to the measured experimental variable. Typically, either the heat of fusion of indium (DSC) or the expansion coefficients of aluminum or copper (TMA) are used. The calibration is based on a run made in the calibration mode using the experimental conditions to be used in subsequent measurements.
 - **Temperature calibration** is used to calibrate the instrument for temperature. The information gathered from temperature calibration is entered in the temperature calibration table, which is applied to the collected data. If you choose to perform temperature calibration, you will also need to select a standard to be used.
3. Select the desired calibration type.
 4. Select the standard to be used from the list (if applicable). You can use one of the standards listed on the window or you can edit the standard by following the directions in the section called "Editing a TMA Standard."
 5. Select OK. The Calibration program will open and plot the file (as seen on the next page).



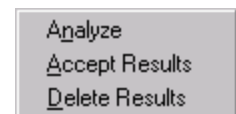
6. Rescale the plot, if desired.
7. Select **Analyze/Analyze** from the menu or select **Analyze** from the Calibration pop-up menu (shown here). The current limit points will be indicated with markers on the graph.
8. Move the active marker to the desired position on the curve using either the mouse or keyboard.
9. Select (activate) the next marker by: (a) Pressing the TAB key or (b) Positioning the pointer over a marker and clicking the left mouse button. (Pressing back TAB (Shift-TAB) will activate the previous marker.)



10. Select **Analyze/Limits OK** or select **Limits OK** from the pop-up menu to accept the points. When calculated, the calibration results will be displayed.



11. Accept the calibration results by selecting **Analyze/Accept results** or selecting **Accept Results** from the pop-up menu.



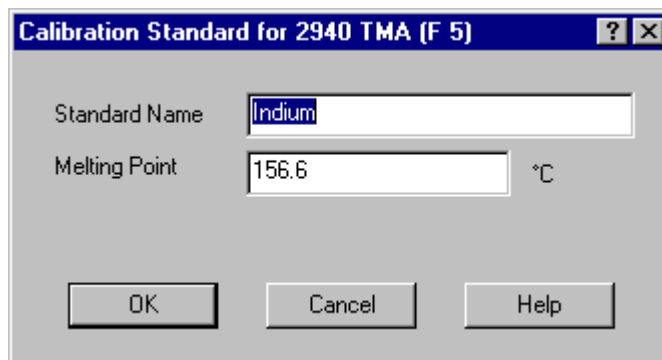
12. Close the Calibration window.

The cell constant and temperature calibration parameters (observed and correct values) are automatically updated in the **Cell/Temperature Table** window after the calibration has been performed, accepted, and saved. See "Using the Cell/Temperature Calibration Table" for details.

Editing a TMA Calibration Standard

To edit a TMA standard value or enter a new standard, follow steps 1 through 4 to open the **Calibration Analysis Specification Standard** window. Double-click on the standard name or click **Edit**. The **Calibration Standard** window (shown here) appears.

Use this window to temporarily change the theoretical values for the specified standard that will be used during calibration or to enter a new standard.



1. Enter the standard name.
2. Enter the literature melting point in °C.
3. Select OK.

NOTE: The standards list will return to the default values each time the Calibration Analysis Specification window is reopened or if the Default button is clicked.

NOTE: The melting points for indium, tin, zinc, aluminum, silver, gold, and copper are defined by the International Temperature Scale of 1990 (ITS-90).

Using the Cell/Temperature Table

When you select **Calibrate/Cell/Temperature Table** from the TMA menu, the **Cell/Temperature Table** (shown here) is displayed. The items shown on this window depend on the instrument mode. This window is used to verify/edit cell calibration information, such as cell constant and auto measure offset, and to input or reset temperature calibration data.

	Observed Temperature °C	Correct Temperature °C
Point 1:	9.67	9.79
Point 2:	99.67	99.79
Point 3:		
Point 4:		
Point 5:		

NOTE: This function is not available when the instrument is in the calibration mode.

Cell Calibration Parameters

Use the Cell Calibration Parameters to set calibration parameters that the instrument will apply to the corrected data.

- **Cell Constant:** The cell constant parameter is automatically updated in this window after cell constant calibration has been performed, accepted, and saved under Calibrate/Analysis.
- **Auto Measure Offset:** This calibration parameter is available in TMA Film/Fiber mode only.

Calculating the Auto Measure Offset

The Auto Measure Offset compensates for the added thickness of the film/fiber probe and stage. To calculate the value of the offset, follow these steps:

1. Install the film/fiber probe and stage as directed in the instrument manual.
2. Start *Advantage*TM and configure the TMA online with the controller.
3. Select **Experimental/Mode** and select the TMA Film/Fiber mode.
4. Select **Cell/Temperature Table** from the **Calibrate** menu.
5. Take note of the value displayed on the window under Auto Measure Offset and record it on a piece of paper as the Current Offset.
6. Use calipers to measure the gap in the Auto Measure Offset Gauge and record this value as the Actual Size.
7. Open the probe and insert the Auto Measure Gauge, which is found in the TMA accessory kit. (See the TMA instrument manual for details.)
8. Press the Measure Length key on the instrument keypad and record this value as the "Displayed Size."
9. Calculate the new offset using this equation:

$$\text{New Offset} = (\text{Actual Size} - \text{Displayed Size}) + \text{Current Offset}$$

10. Enter the new offset as the "Auto Measure Offset" on the TMA **Cell/Temperature Table** window.

Temperature Calibration Table

The lower part of the **Cell/Temperature Calibration Table** shows the temperature calibration table that the instrument applies to the collected data. You can use this area to observe the values that were automatically entered after you performed temperature calibration, or to manually enter from one to five temperature calibration points (pairs of observed and correct temperature points). The observed and correct temperature corresponds to the experimental and theoretical transition temperature (*e.g.*, melting point) of the calibrant respectively.

Temperature calibration is useful for experiments in which precise transition temperatures are essential.

To temperature calibrate the TMA, you need to analyze a high-purity metal for its melting temperature by running the calibration experiment as directed previously. After the data has been analyzed the calibration values are automatically entered in this table.

- If these values are correct, select OK.
- If new values are to be entered:
 1. Select the Reset button to remove all previously stored values.
 2. Determine the temperature points.
 3. Enter the Observed and Correct temperature points in the table.
 4. Select OK when all points have been entered to save the settings to the instrument.

NOTE: When temperature calibration is in effect, Temp in *C appears in the status line. When it is not in effect, Temp in °C appears in the status line.

Chapter 3

Setting Up Experiments

Overview

Each instrument requires slightly different set-up procedures. This chapter provides basic information that you will need to set up an experiment using the *Advantage*[™] program. For more detailed information on a specific instrument or specialized items found on a window, access the online help for that instrument.

This chapter assumes that you have read Chapter 1 and are generally familiar with the way the program operates. More detailed information about the panes and other parts of the program are provided in this chapter and the remainder of this manual.

The first step in preparing for an experiment is generally the selection of a sample and the holder for that sample, whether it is a pan, cup, clamp, or probe. The type of holder can be required information that is entered as part of your experiment setup in the *Advantage*[™] program. Another piece of information that is required for an experiment is the mode and signals that the instrument will be using during the experiment. This mode selection tells the instrument to perform a certain way. The first section in this chapter deals with setting up the instrument mode.



Each time you do an experiment you will need to enter sample information and notes, as well as define the experimental procedure, which can contain the method, data sampling information, method-end conditions, frequency or amplitude table, etc. Two basic options for setting up experiments are available—using the **Experimental View** or using the guided **Wizard**. This chapter will tell you how to use these options.

NOTE: If you are just starting out with the *Advantage*[™] program, you may want to begin using the Wizard.

Basic Experimental Steps

All experiments, regardless of the technique used to create them, will follow these basic steps:

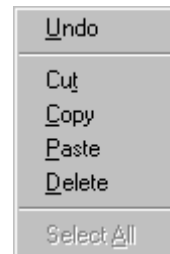
1. If you are running more than one instrument on your system, select the desired instrument control window using the **Window** menu or by clicking on the desired task bar button to bring the window to the front.

2. Select the instrument mode. See the next section for details.
3. Select the desired option to set up your experiment—click the  button to access the **Experimental View** or click the  button to start the **Wizard** functions.
4. Set up the sample and procedure information.
5. Prepare the instrument (prepare samples, load pans or cups, tare the instrument, etc.),
6. Schedule the run.
7. Start the experiment.
8. Monitor the real-time signals, plot, and method progress.

Editing Fields

When you have your cursor in an entry field that can be edited, you can click the right mouse button to display the menu shown here. Then select from one of the following options.

- **Undo:** Reverses previous editing operation.
- **Cut:** Deletes data from the field and moves it to the clipboard.
- **Copy:** Copies data from the field to the clipboard.
- **Paste:** Pastes data from the clipboard into the location of the cursor.
- **Delete:** Erases the data from the field.
- **Select All:** Selects all of the data available in the indicated field.



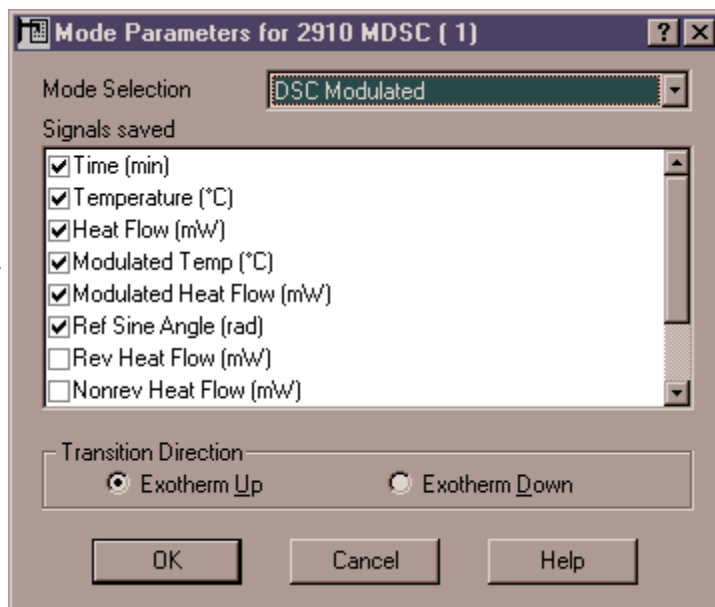
Using the Mode Functions

The instrument mode defines the type of experiments and signals saved during an experiment. For example, if you want to calibrate the DSC instrument, choose the Calibration mode from the Mode Selection list. Each instrument mode has a set of signals associated with it. In certain modes (such as MDSC and SDT), the signals that are saved from the list shown on the window can be selected by you (checkboxes will appear next to selectable signals).

You can access the **Mode Parameters** window (DSC shown here) by selecting **Experimental/Mode** from the instrument menu or by

clicking on the  button.

NOTE: Mode selection should be the first step when setting up your experiment because other parameters may be affected, depending on the mode chosen.



Selecting a Mode and Signals

The mode selection field lists the available instrument modes. The type of experiment to be run determines the instrument mode.

1. Access the **Mode Parameters** window as directed above to select the instrument mode.
2. Click on the down arrow next to the Mode selection field to display the list of modes. This list varies depending on the instrument. See the online help for the available instrument modes.
3. Select the mode desired for your instrument. The type of experiment determines the instrument mode. A list of signals for the selected mode is displayed in the Signals saved box. Check any signals you want to save and click to remove the check next to any signals you do not wish to save.
4. Select OK.

NOTE: When you set up your experiment using the Wizard, the mode is automatically set for you based on your experiment selection.

Some instruments can have additional fields that apply to specific instruments and modes, such as those listed below:

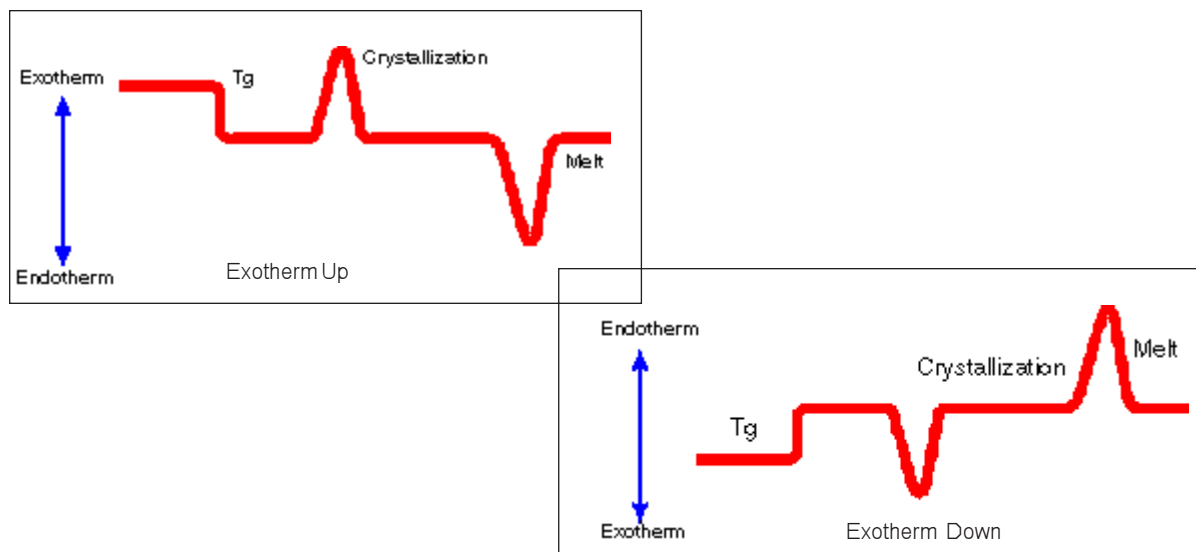
- Exotherm Up/Exotherm Down (DSC, SDT)
- Clamp type (DMA)
- RCS/MCA attached (DSC, TMA).

The next three sections deal with these additional items.

Setting the Transition Direction

The Transition direction for the exotherm graph is applicable only to DSC and SDT instruments. It is used to select the direction for display of transitions within the sample.

Exotherm Up saves the data in the traditional TA Instruments format, while **Exotherm Down** inverts the signal.



1. Select the direction that you want for your exothermic transition curve.
2. Click on OK to save the mode parameters settings.

Choosing a DMA Clamp Type

TA Instruments offers various types of DMA clamps for use in your experiments. The clamps are generally classified as tensioning or non-tensioning. See the instrument manual for details on the clamp types.

- When you use a tensioning clamp, force is constantly applied to the sample so that the sample does not buckle and so that the clamp does not lift off the sample during oscillation.
 - When you use a nontensioning clamp, the moveable clamp moves around the "zero" point, requiring no additional tensioning force.
1. Click on the down arrow next to the Clamp field on either the **Mode Parameters** window or the **Experimental View - Summary Page** to display the list of clamps.
 2. Select the clamp desired for your experiment. (For more information on the clamps, refer to the *DMA Operator's Manual*.) The clamp types are as follows:

Tensioning Clamps:

3-Point Bending

Tension: Film

Tension: Fiber Compression

Penetration Submersion Film Tension

Submersion Compression

Non-Tensioning Clamps:

Dual Cantilever Single Cantilever

Shear Sandwich


Using an MCA or RCS Accessory

Check the box next to RCS or MCA attached, if you are using either a Refrigerated Cooling System or Mechanical Cooling Accessory with your DSC 2920 or TMA 2940 instruments. (The MCA is only available with the TMA.) This parameter limits the temperature that the instrument reaches during an experiment to 400°C for DSC or 350°C for TMA. This is the maximum temperature allowable for the RCS and MCA.

Programming the Experiment


TA Instruments has designed the *Advantage*TM program with the idea that running our instruments should be easy. All of the information needed to set up the experiment (*e.g.*, sample information and procedure) is accessed through the **Experimental View Pane**.

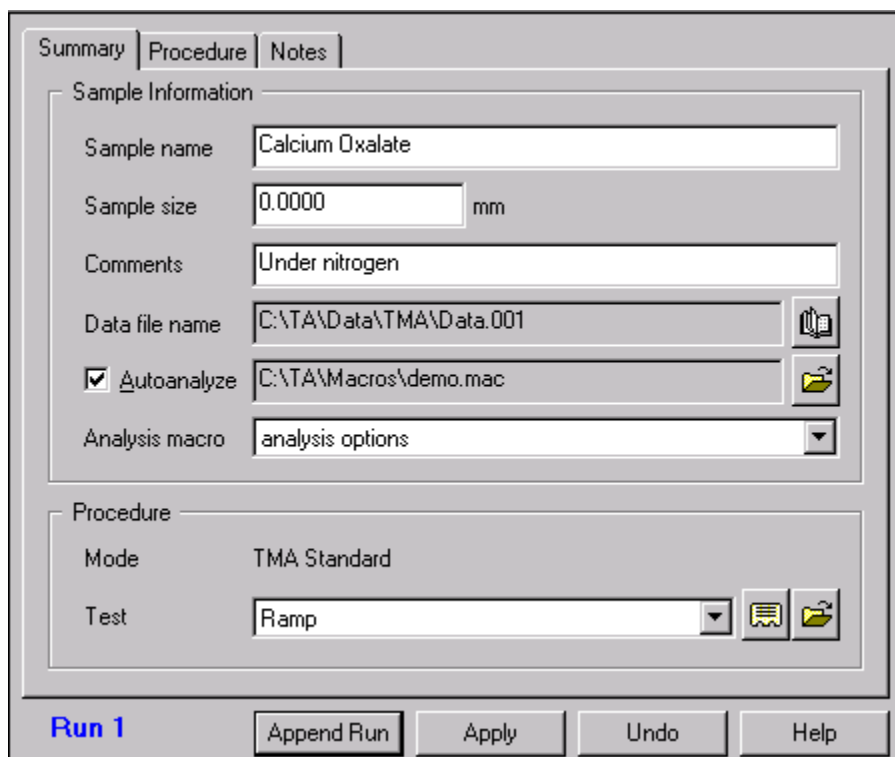
To begin setting up your experiment,

click on the  button, or choose **View/Experimental View** from the instrument menu, or press F10. As you can see, there are three different pages available in this view. These pages are used as follows:

- **Summary Page:** Contains the sample information, autoanalysis selection, procedure and test selection.
- **Procedure Page:** Contains detailed test definitions including the Advanced and Post Test conditions.
- **Notes Page:** Contains additional experimental comments.

The information on these pages should be verified or updated with the correct information before you start the experiment. Once the information is correct, you can either click on the **Apply** button to save the changes or on the **Append** button to save the changes

and add another run to the end of the sequence. (NOTE: The  button on the tool bar will be grayed if the **Apply** button is active for the scheduled run.) If more than one run is contained in the sequence list, the information displayed on the **Experimental View** pages refers to the highlighted run number. Each of the **Experimental View** pages is explained in further detail on the following pages.




The screenshot shows the 'Summary' tab of the 'Experimental View' dialog box. It has three tabs: 'Summary', 'Procedure', and 'Notes'. The 'Sample Information' section includes fields for 'Sample name' (Calcium Oxalate), 'Sample size' (0.0000 mm), 'Comments' (Under nitrogen), 'Data file name' (C:\TA\Data\TMA\Data.001), a checked 'Autoanalyze' checkbox with file path C:\TA\Macros\demo.mac, and an 'Analysis macro' dropdown menu set to 'analysis options'. The 'Procedure' section shows 'Mode' as 'TMA Standard' and 'Test' as 'Ramp'. At the bottom, there are buttons for 'Run 1', 'Append Run', 'Apply', 'Undo', and 'Help'.



Using the Summary Page

The **Summary Page** (shown on the previous page) is used to enter the basic information needed to create a run. You can set up the sample name, pan number (if applicable), and data file; enter comments about the run; turn on the Autoanalyze feature; and choose the test to be used.

For the DSC instrument, this summary also applies to the cell depicted by the icon in the upper right corner of the page. (To change the cell used in the experiment, change the cell on the instrument and then select the appropriate mode on the **Mode Parameters** window.)

To use the **Summary Page**, follow these directions:

1. Before setting up your experiment verify that the mode displayed is the one you want the instrument to use. If the mode is not correct, select **Experimental/Mode** from the instrument menu or click the  button on the tool bar. Then you can select the mode you want to use.
2. Enter the Sample Name.
3. Enter the Sample Size, if applicable to your instrument.
4. *Autosampler instruments only:* Specify the Pan No.(s).
5. Enter any Comments.
6. Enter a data filename by clicking on the Browse button to the right of Data File, then select the desired storage location and enter in a filename. Click OK when finished.
7. If you want to automatically analyze the resulting data file using a macro (created with the Universal Analysis Autoanalysis software option), check the Autoanalyze box. Then locate the desired macro filename using the Browse button. Click on the Analysis Macro list and select a macro from those displayed. For more information on Autoanalysis, see the Universal Analysis online help or manual.
8. *DMA instruments only:* Select the clamp to be used from the drop-down list of available clamps. (If you change clamps you will be prompted to perform clamp calibration.)
9. *DMA instruments only:* Select the sample shape from the list and enter the sample's dimensions in the specified units.
10. Select the type of test you want to use from the drop-down Test list available. The list contains both preprogrammed tests (test templates) and a custom test. The option you choose will depend on the specific method you wish to perform.

11. Check the available template options to see if any of those meet your needs. You can view a summary of the chosen procedure by selecting the  button to display the **Procedure Summary** window. If you want to use your own procedure, rather than a preprogrammed test, select the Custom test. This allows you to create a customized method or to load an existing method file (*.mth). (To change any of the information, except for the mode, select the **Procedure Page**.) To open a saved procedure file, click the  button. The procedure file will be added to the test list once you have selected it.
12. When you have finished setting up this run summary, you should verify the information contained on the **Procedure Page** and the **Notes Page**. These pages are explained in detail later in this chapter.
13. Click the **Apply** button to apply the information on this page to the current run.

NOTE: In the case of a Dual Sample DSC, there are two Summary Pages, one for each sample.

Using the Procedure Page

Once the test has been chosen on the **Summary Page**, the **Procedure Page** will be adjusted for the type of test selected.

- If you choose a Custom test, you can then use the **Editor** button on the **Procedure Page** to make changes to the method listed. See “Creating a Custom Test” in this chapter for further information.
- *Advantage™* also supplies a list of common tests that can be chosen on the **Summary Page**. When one of the template tests are selected, the **Procedure Page** (DMA shown here) reflects the information that you will need to enter when you set up your run using that test.

The screenshot shows the 'Procedure' tab of a software interface. At the top are three tabs: 'Summary', 'Procedure' (selected), and 'Notes'. Below the tabs is a 'Procedure Information' section with a 'Test' dropdown set to 'Temperature Ramp /Frequency Sweep' and a 'Notes' text area containing 'Material is heated at a constant rate and deformed (oscillated) at a constant amplitude (strain) over a range of frequencies. Provides'. Below this is a 'Method' section with various input fields: 'Amplitude' (15.0 μm), 'Preload force' (0.010 N), 'Force track' (checked, 125.0 %), 'Start temperature' (35.00 °C, with a 'Use current' checkbox), 'Soak time' (5.00 min), 'Final temperature' (150.00 °C), 'Ramp rate' (5.00 °C/min), and 'Hold time at final temperature' (30.00 min, with an unchecked checkbox). To the right of these fields are 'Advanced...' and 'Post Test...' buttons. At the bottom of the 'Method' section are two tabs: 'Method' (selected) and 'Frequency Table'. Below the tabs are four buttons: 'Run 1' (in blue text), 'Append Run', 'Apply', 'Undo', and 'Help'.

Advantage™ Test Templates



TA Instruments has set up a collection of the tests that are commonly run on our thermal analysis instruments. The experimental conditions used with each of these tests has been put into a set of preprogrammed test templates that can be selected and used to run with your samples. The following is a table of the instruments and their associated test templates that are available through the *Advantage™* program.

Instrument	Test Name
DEA	Remote Single Surface/Frequency Sweep Isothermal Temperature/Frequency Sweep Temperature Ramp/Frequency Sweep Temperature Ramp/Single Frequency Temperature Step/Frequency Sweep
DMA	Creep Stress/Strain Temperature Ramp/Controlled Force Isostrain Stress Relaxation Strain Sweep <u>Multifrequency Tests:</u> Isothermal Temperature/Frequency Sweep Temperature Ramp/Frequency Sweep Temperature Ramp/Single Frequency Temperature Step/Frequency Sweep
DSC/DTA	DSC/DTA Ramp DSC/DTA Heat/Cool/Heat DSC/DTA Cyclic DSC/DTA Isothermal Oxygen-Induction Time Conventional MDSC MDSC Heat Only MDSC Quasi-Isothermal Baseline Calibration Cell Constant Calibration Temperature Calibration

(table continued)

Instrument	Test Name
SDT	Temperature Ramp Cyclic Isothermal Stepwise Isothermal
TGA	Ramp Heat and Hold HiRes Dynamic HiRes Constant Reaction Rate HiRes Stepwise Isothermal Modulated Standard Modulated Survey
TMA	Ramp Isostrain Stress/Relaxation Stress/Stain Creep

To use the **Procedure Page**, follow these directions:



1. Before setting up your experiment: Verify that the mode displayed is the one you want the instrument to use. If the mode is not correct, select **Experimental/Mode** from the instrument menu or click  on the tool bar. Then you can select the mode you want to use.
2. Select **View/Experimental** or click the  on the tool bar to open the **Experimental View**, if needed.
3. Set up the **Summary Page**, then click on the tab to open the **Procedure Page**.
4. View the current test displayed (you can select a new test on the **Summary Page**).
5. Type any notes that you want to write about the test you will be running, if Custom test was selected. A preprogrammed test template will display notes specific to the test chosen.

6. Enter the requested method parameters, if you choose one of the test templates. However, if you chose a Custom test, click the **Editor** button to display the **Method Editor** window. This allows you to create a customized method or load an existing method file (*.mth). See “Creating a Custom Method” on page 3-13 for more details.
7. *DEA or DMA Only* If you are using a DEA or DMA, there may be two tabs at the bottom of the **Procedure Page**. The **Method Page** displays the experimental parameters associated with the chosen test. Make any changes desired. Click on the **Frequency/Amplitude Page** to set up the desired table. See “Setting Up the Frequency/Amplitude Table” on page 3-23 for further details.
8. Click the **Advanced** button to display the **Advanced Parameters** window applicable to the current instrument. If desired, you can change the parameters that the instrument will use to perform the experimental run. See “Entering Advanced Parameters” for further information.
9. Click the **Post Test** button to display the current **Post Test** window. If desired, you can change the conditions that will apply at the end of the run (method-end conditions). See “Entering Post-Test Conditions” for further information.
10. When you have finished setting up this procedure you should verify the information contained on the **Summary** and the **Notes Pages**.
11. Click the **Apply** button when finished to save these changes or click the **Append Run** button to add another run to the end of the sequence.

If you want to create your own procedure, see “Creating a Custom Test” on the next page. To find more about runs and sequence files, turn to Chapter 4.

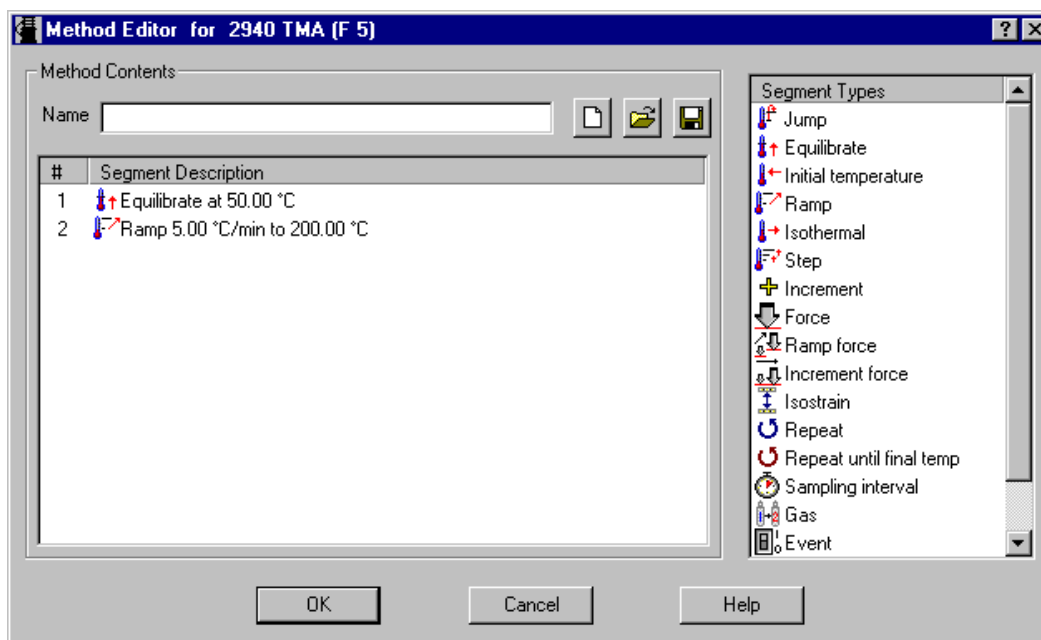
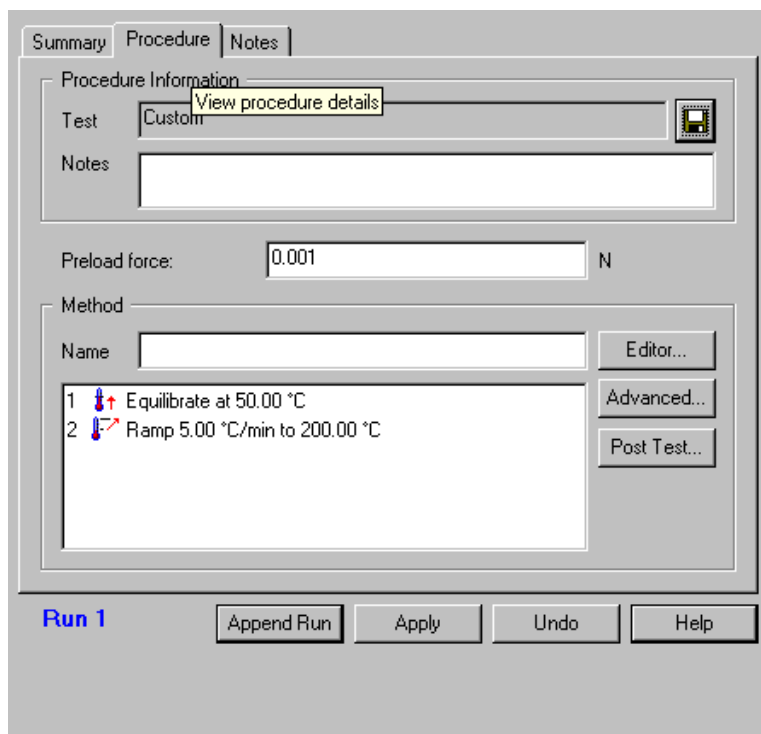
Creating a Custom Test



If you would prefer to use a test of your own design, you can create a custom-made test using the following instructions:


1. Before setting up your experiment: Verify that the mode displayed is the one you want the instrument to use. If the mode is not correct, select **Experimental/Mode** from the instrument menu or click  on the tool bar. Then you can select the mode you want to use.
2. Select **View/Experimental** or click the  on the tool bar to open the **Experimental View**, if needed. Click the **Summary** tab.
3. Enter the Sample Name.
4. Enter the Sample Size, if applicable to your instrument. *For the DSC Autosampler Only:* The sample size entered here should be the sample contained in the selected Pan Number and using the selected Reference Number.
5. Select the Pan No. from the list, if applicable to your instrument.
6. Enter any Comments.
7. Enter a data filename by clicking on the Browse button to the right of Data File, then select the desired storage location and enter in a filename. Click OK when finished.
8. If you want to automatically analyze the resulting data file using a macro (created with the Universal Analysis Autoanalysis software option), check the **Autoanalyze** box. Then locate the desired macro filename using the Browse (...) button. Click on the **Analysis Macro** list and select a macro from those displayed. For more information on Autoanalysis, see the Universal Analysis online help or manual.
9. Select Custom from the drop-down **Test** list available. The list contains both preprogrammed tests (test templates) and a custom test.
10. Click the **Procedure** tab to display the **Procedure Page**, which reflects the fact that you have selected a Custom test.

11. Click the **Editor** button on the **Procedure Page** (shown here) to display the **Method Editor** window.

The **Method Editor** window (shown below) is used to create a custom method by building or modifying a preprogrammed series of instructions, called segments, to be executed by the instrument. The segments are added, moved, or deleted from the method using the drag-and-drop technique. Collectively this group of segments makes up a method. Up to 60 segments can be contained in a method.



- **Import/Export:** You may use this window to import (using the  button) an existing method file to use, or export (using the  button) just the method portion of the procedure to file. These last two options provide compatibility with the previous generation TA Instruments Windows NT and OS/2 software. (Using the *Advantage*™ software, procedure files replace method files.)

- **Create New Methods:** To clear the current information displayed in the **Method Editor** window for creation of a new method, click the New button, .
- **Add Segments:** To add a segment to any method:
 1. Click on the desired segment to be added from the Segment Types list to highlight it.
 2. Use the drag-and-drop method to move the segment: Hold down the mouse button while you move the cursor to the desired position in the list of numbered segments (Segment Description List). Then release the button.
 3. Edit the entry field(s) as desired, using the Tab key to move between fields.

To append a segment to the end of a method:

— Double click on the desired segment to be added from the Segment Types list.

or

— Click on the desired segment to be added, then press Ctrl + E to append it to the method.


or

— Follow these steps:

1. Click on the desired segment to be added from the Segment Types list to highlight it.
 2. Use the drag-and-drop method to move the segment: Hold down the mouse button while you move the cursor to the empty area under the list of numbered segments (Segment Description List). Then release the button.
 3. Edit the entry field(s) as desired, using the Tab key to move between fields.
- **Remove Segment:** To remove a segment, just highlight the segment in the list and press the Delete key on the keyboard (or use Ctrl + X).
 - **Edit Segment:** To edit a segment, double click on the segment and edit its parameters (or select the segment and press Ctrl+E). Use the TAB key to move between the parameters.
 - **Reorder Segments:** To reorder the segments, just drag-and-drop a segment in a new position within the segment description list.
 - **Selecting Multiple Segments:** Within the segment description list you can select multiple segments for deletion or copying by holding down the Ctrl key while clicking on the desired segments.

To obtain more detailed information on handling methods and segments, refer to the online help.

Saving a Procedure to File

Once you have customized your procedure, you may save it to file so that you can use it again. To save a test template to a procedure file, click on the  button on the **Procedure Page**. A standard file save window is displayed to allow you to choose a filename and location.


A procedure file (.prc) contains the all of the information contained on the **Experimental View - Procedure Page**, including the following:

- Instrument
- Mode
- Test (either preprogrammed or custom-made)
- Notes (information about the experiment)
- Method Name (name given to the set of segments used)
- Method Segments (steps that are performed during the run)
- Advanced Parameters (parameters needed by the instrument)
- Post Test Parameters (conditions applied after the test is complete)
- Amplitude/Frequency Table Values (used by the DEA and DMA).

NOTE: The procedure file used must match the current instrument mode or you will receive an error message. To use a procedure with a different instrument mode you must change the mode first, then open the procedure file.

Using Existing Method Files

If you have method files from experiments that were done before the *Advantage*[™] software became available, you can still use these files by following these instructions:

1. Copy the method files onto your hard drive.
2. Open the *Advantage*[™] program and switch to **Experiment View**.
3. Select the Custom test on the **Summary Page**.
4. Click on the **Procedure Page**, then click on the **Editor** button. The **Method Editor** window is displayed.
5. Click on the Import Method, , button. The **Import Method** window is displayed.
6. Locate the method file desired and click the Import button. The method file will be imported and the segments displayed on the **Method Editor** window.
7. Click OK. The method is now inserted into the current procedure. You can now set up the rest of the procedure (which is mode dependent) and save it to a file, if desired. You could also continue to use the methods without saving them to a procedure by repeating steps 1 through 7 each time you want to use an old method file.

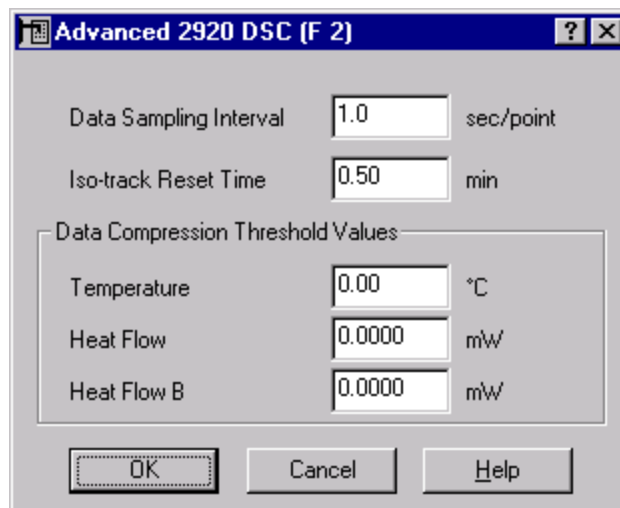
Entering Advanced Parameters

Advanced instrument parameters are those settings that may not normally be changed for experiments, once they are set. The type of parameters applicable to each instrument may vary slightly and may be dependent on the instrument mode.

To access the **Advanced Parameters** window (DSC example shown here), click the **Advanced** button on the **Experimental View - Procedure Page**.

To enter advanced parameters, follow these steps:

1. Enter the values desired for each parameter.
2. Click the OK button when finished.



List of Advanced Parameters

For more information on the parameters that you need to enter for your instrument, select the desired name from the following list.

- **Data Sampling Interval:** This instrument parameter defines how often the system reads and stores a data point per second. For example, if 2.0 is entered, the system stores a data point every two seconds, provided the data compression threshold values are reached. Range is between 0.5 and 1000.0. The value must be divisible by 0.5 and the units are in seconds/point.
- **Iso-Track Reset Time (DSC):** This instrument parameter defines how frequently the heater control monitors the sample temperature and makes appropriate corrections to the heater power so that the sample temperature reaches the set point temperature during an iso-track segment. Enter the desired length of time (in minutes) between heater control monitoring.
- **Data Compression Thresholds:** Enter the value by which a new data value, such as temperature, must differ in linearity from the previous value in order to be stored as a new data point. This option helps reduce the number of data points stored during non-transitional regions.

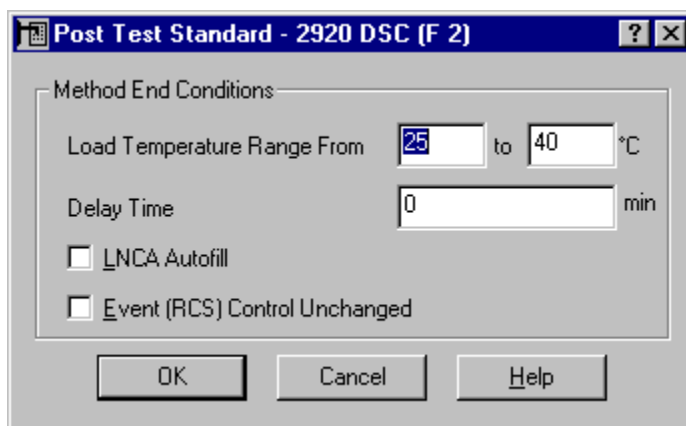
NOTE: If values are entered for all fields, only one condition needs to be met in order for a new data point to be collected. A zero in any field will disable data compression.

- **Additional Weight on Tray (TMA):** This instrument parameter is used to enter the weight, in grams, of the items placed on the weight tray. Up to 100 grams may be added. The static weight is added to the programmed force in the data file.
- **Auto Zero Signal A:** Method Start Conditions are functions that can be automatically executed before a method begins. Selecting Yes automatically sets the signal A value to zero microns (μm) before the method begins. Selecting No maintains the current signal A value at method start.
- **Equilibration Criteria - Rate (DMA):** Equilibration criteria are used at the beginning of a displace/recover method to allow the sample to mechanically and thermally equilibrate before the sample is displaced. Rate defines an equilibration step that will last until the sample deformation rate is below the specific value. Recommended values are 0.1 to 1.0 $\mu\text{m}/\text{min}$, depending upon the displacement expected during the displacement step. You may need to adjust the rate parameter by trial-and-error at the initial temperature in order to keep the experiment reasonably short, while maintaining the quality of your data at the lowest temperatures.
- **Equilibration Criteria - Time (DMA):** Equilibration criteria are used at the beginning of a displace/recover method to allow the sample to mechanically and thermally equilibrate before the sample is displaced.

Entering Post Test Conditions

Post Test Conditions are functions that will occur at the end of a run. Method End Conditions are functions that can be automatically executed at the end of a run to increase turn-around time between experiments. The type of conditions applicable to each instrument varies slightly.

To access the **Post Test** window (AutoDSC shown here), click on the **Post Test** button found on the **Experimental View - Procedure Page**.



To enter post-test conditions, follow these steps:

1. Enter or select the desired conditions.
2. Click the OK button when finished.

List of Post Test Conditions

For more information on the conditions that you need to enter for your instrument, select the desired name from the following list.

- **Return to Temperature (DSC):** When Return to Temperature Range is selected, the instrument will use the cell heater and the air cool (if connected) to bring the cell's temperature to within the specified temperature limits. The cell's temperature will be kept within this range until the STOP key is pressed twice or the next run is started.
- **Load Temperature Range (DSC Autosamplers):** Enter the temperature range that the instrument should reach before unloading the current sample and loading the next sample. The suggested range is from zero to 100°C.



CAUTION: Do not enter temperatures above 100°C unless the Autosampler has been modified for "hot lids." The material under the lids in the lid storage area may begin to soften or melt at high temperatures. (The upper limit with "hot lids" capability is 400°C.) Do not enter temperatures below 0°C unless the Autosampler/DSC system is equipped with a liquid nitrogen cooling accessory (LNCA), as well as some type of purged "environmental enclosure" around the cell/heat exchanger and sample area. Otherwise, frost buildup may prevent normal operation and/or damage the Autosampler grippers.

- **Delay Time (DSC Autosampler):** Enter the time (in minutes) that the instrument will delay, after reaching the load temperature range, before loading the sample.

NOTE: To avoid a start-up time delay after the file validity check, enter a value of zero (0) for the first run in the Autosampler sequence.

- **LNCA Autofill:** LNCA Autofill is a method-end condition that can be automatically activated at the end of a method, if an Autofill LNCA is connected to the instrument. For more detailed information consult the online help.
- **Event (RCS) Control Unchanged (DSC):** The Event (RCS) Control Unchanged option controls the state of an external device (Refrigerated Cooling System--RCS) connected to the instrument with the event cable.
- **Furnace Open/Closed (SDT):** Furnace Open will automatically open the furnace at the end of the method. Furnace Closed allows the furnace to be positioned above the fan when in the closed position, providing the quickest way to cool the furnace.
- **Furnace Open and Unload/Closed (TGA):** Furnace Open and Unload will automatically open the furnace at the end of the method and unload the sample pan on the platform. If an EGA furnace is attached, the furnace will not open until the

temperature is below 500°C. Furnace Closed leaves the furnace shut at the end of the method. If a standard furnace is attached, Furnace Closed will override the Air Cool option.

- **Air Cool To X°C (TGA and SDT):** This Post Test Condition applies to both TGA and SDT instruments. To find out how it will operate for your instrument, see the explanations below.

TGA 2950/2050 Instruments—Check the Air Cool option to open the TGA furnace and allow it to air cool for a specified amount of time (in minutes). When the instrument reaches this time, air cooling will stop.

SDT 2960 Instrument—If you check the Air Cool option for the SDT, it will automatically begin to cool the furnace using a fan that blows air around the outside of the furnace at method end. Select the air cool option to further speed cool-down time by introducing compressed air until a specified temperature (°C). This option will activate when the furnace is below 600°C.

- **Furnace Open/Closed (TMA):** Choosing to open the furnace at the end of the method programs the TMA to raise the furnace, allowing it to air cool. Choosing to leave the furnace closed at the end of the method retains the heat longer.
- **Probe Open/Closed (TMA):** Choosing to open the probe at the end of the method will lift the probe off the stage automatically. Choosing to leave the probe closed at the end of the method will keep the probe in its current position.
- **Return to Temperature Range (TMA and DEA):** When Return to Temperature Range is selected, the instrument will use the furnace and the air cool (if connected) to bring the cell's temperature to within the specified temperature limits. The temperature will be kept within this range until the STOP key is pressed or the next run is started.
- **Auto End on Modulus (DMA):** Select the Auto End check box, then enter the modulus value, to automatically end the method when the measured modulus of the sample drops below or rises above the specified amounts.
- **Return to Temperature Range (DMA):** When Return to Temperature Range is selected, the instrument will use the furnace and the GCA or air cool (if connected) to bring the temperature to within the specified temperature limits. The temperature will be kept within this range until the STOP key is pressed, or the furnace is opened, or the next run is started.
- **Furnace Open/Closed (DMA):** Choosing to leave the furnace closed at the end of the method will keep the furnace in the temperature range selected by the Return to Temperature option until it is opened. Choosing to open the furnace at the end of the method programs the DMA to raise the furnace after it has reached the specified temperature limits.

- **Clamp Up/Closed/Float (DMA):** Select the Clamp Up button to raise, float, or close the clamp at the end of the method.
- **GCA Autofill (DMA):** Check the GCA Autofill box to have the Gas Cooling Accessory automatically refilled with liquid nitrogen at the end of a method.

Setting Up the Frequency/Amplitude Table

DMA Frequency/Amplitude Tables

Frequency tables are needed for DMA Multifrequency mode experiments and amplitude tables are needed for DMA Multistrain mode experiments. These contain a listing of one or more entries (up to 28) over the allowable range. The allowable frequency range is 0.01 to 200 Hz and the allowable amplitude range is 0.5 to 10,000.0 μm .

DEA Frequency Table

Use the Frequency Table to create and edit a DEA frequency table. The frequencies will be scanned by the instrument to measure dielectric properties. Up to 28 frequencies from 0.003 Hz to 100,000 Hz can be scanned in each experiment. The table is ended when a value of zero is encountered.

To create a frequency or amplitude table, follow these guidelines:

1. Click the Frequency or Amplitude tab at the bottom of the **Procedure Page**. The **Frequency/Amplitude** table (shown to the right) is displayed in the lower area of the window.
2. Select one of the radio buttons: **Single**, **Log**, **Linear**, or **Discrete** entries. (NOTE: **Discrete** is only available when setting up a Custom test.) The configuration of the table will vary based on the radio button selected. Follow the directions on the next two pages for the chosen type of entry.
3. *For DEA Only:* The **Estimated Time to Scan** displays the estimated time (in minutes) required to complete one scan of the frequency table, using the current stored data sampling interval and based on the selected table. This number is used to determine the proper heating rate in order to give at least two data points at a given frequency over a 2.5°C interval using the equation:

Selected Heating Rate = 2.5 divided by the Estimated Time per Scan.

Summary Procedure Notes

Procedure Information

Test: Custom

Notes:

Frequency

☐ Single ☒ Log ☐ Linear ☐ Discrete

Frequency: 100.000 to 0.100 Hz

Points Per Decade: 5

Entry	Value
1	100.
2	63.1
3	39.8
4	25.1
5	15.8
6	10.0

Refresh Table

Method: Frequency Table

Run 1 Append Run Apply Undo Help

Setting Up Single Frequency/Amplitude Entries

Single frequency or amplitude is used for screening and comparing materials at a reference value or at an end-use value. 1 or 10 Hz is preferred for single frequency experiments because more complete oscillations are obtained in a shorter time.

1. Click the **Single** radio button.
2. Enter the desired value, then click **Refresh Table** to update the displayed table.

For Multifrequency mode temperature ramp tests, one frequency is normally used. You can use more than one frequency, but they should be higher frequencies (above 1 Hz).

For DMA: To measure the time required to sweep through all of the frequencies in a table, observe the sweeping process using the **Signal Control Pane**, when the drive motor is turned on. Set the heating rate on the **Procedure Page** so that the sweep time occurs in about 2 to 3 degrees.

Setting Up Multiple Frequency/Amplitude Entries

Multifrequency studies (useful for evaluating changes in material response with frequency) should be set up to cover multiple decades of frequency (usually 3-4 decades) and should begin with the highest frequency and end with the lowest.

Multiamplitude studies can include up to 28 amplitudes over the range 0.5 to 10,000 mm. In practice, however, the range of acceptable amplitudes is determined by the stiffness of the sample and the maximum drive force of the DMA 2980 (18N). When sweeping amplitudes, sweep from the lowest to the highest value.

You can choose from several different types of multifrequency/multiamplitude studies, which are described below:

NOTE: When you create a frequency table with more than one frequency, you can begin with the highest frequency and end with the lowest to shorten the sweep time. When you create an amplitude table with more than one amplitude, you can begin with the lowest amplitude and sweep to the highest value.

Log Entries:

1. Click the **Log** radio button to space the entries “logarithmically” (e.g., 10, 5, 2,).
2. Enter the initial and the final entries, then enter the number of **Points Per Decade**. The entries will be calculated automatically.
3. Click the **Refresh Table** button to update the table values displayed.

Linear Entries:


1. Select the **Linear** radio button to space the entries linearly (*e.g.*, 11, 9, 7, 5, 3, 1).
2. Enter the initial and the final entries, then enter the **Number of Points** desired between the range entered. The entries will be calculated automatically.
3. Click the **Refresh Table** button to update the table values displayed.

Discrete Frequency/Amplitude Entries:

If you want to edit the table directly, select the **Discrete** radio button. The Discrete option is only available for Custom tests. The buttons become available for use in editing the table. Place the cursor in the table at the desired point and make your editing changes. The entries will be used in order until the first 0.00 entry is encountered. An entry of 0.00 is recognized as the end of the table.

- Use the **Insert** button to insert a new value at the selected location, shifting all of the other table entries down one position.
- Use the **Delete** button to remove the selected value, shifting all of the table values below the deleted entry up one position.
- Use the **Reset** button to clear all of the table values, leaving only one entry.

Saving a Frequency/Amplitude Table

To save your frequency/amplitude table for future recall, save the entire procedure to a file using the  button located at the top of the **Procedure Page**.

Using the Notes Page

Most instruments have a very similar **Notes Page** (DSC Dual Sample shown here). This page is used to enter information about the experiment as a whole. This type of information probably will not change much between runs, but may change with a different sequence. The information entered on the **Notes Page** will be stored with the data file.

To enter or select the additional information for your experiment, follow these steps:

The screenshot shows the 'Notes' tab selected in the software interface. The 'Notes' section includes a text field for 'Operator' containing 'John Smith', a dropdown menu for 'Pan Type' set to 'Aluminum', and a multi-line text area for 'Extended Text' containing 'Extend text'. Below this is the 'Purge Gas Settings' section, which has two rows: 'Gas #1' with a dropdown set to 'Nitrogen' and a 'Flow Rate' field set to '10mL/min'; and 'Gas #2' with a dropdown set to 'None' and an empty 'Flow Rate' field. At the bottom of the window, there is a 'Run 1' label and four buttons: 'Append Run', 'Apply', 'Undo', and 'Help'.

1. Enter the name of the Operator conducting the thermal analysis experiment. Up to 25 characters may be entered.
2. *For DSC, TGA, and SDT only:* Specify the Sample Pan type used for this experiment from the list of available pans.
3. *For TMA only:* Specify the Probe type used for this experiment from the list of available options.
4. Enter any Extended Text to include additional information about the sample or experiment, such as sample treatment or data sampling parameters. Up to 500 alphanumeric characters may be entered (approximately 12 lines).
5. Specify the primary purge environment (Gas #1) and flow (including units) used in the experiment from the list of available choices. If no purge gas is used, select None from the list. You may also enter in your own purge gas name (up to 9 characters). In DMA, specify the Air Bearing Gas.
6. *DSC, TGA, and SDT only:* Specify the secondary purge environment (Gas #1) and flow (including units) used in the experiment from the list of available choices. If no purge gas is used, select None from the list.


7. Click the **Apply** button to apply the information on this page to the current run.

NOTE: These gas entries do not actually control which gas is currently flowing through the instrument, but merely indicate which gases are plumbed to the instrument. For example, oxidative stability experiments use nitrogen for the primary gas and oxygen for the secondary gas.

Guided Setup Using the Wizard

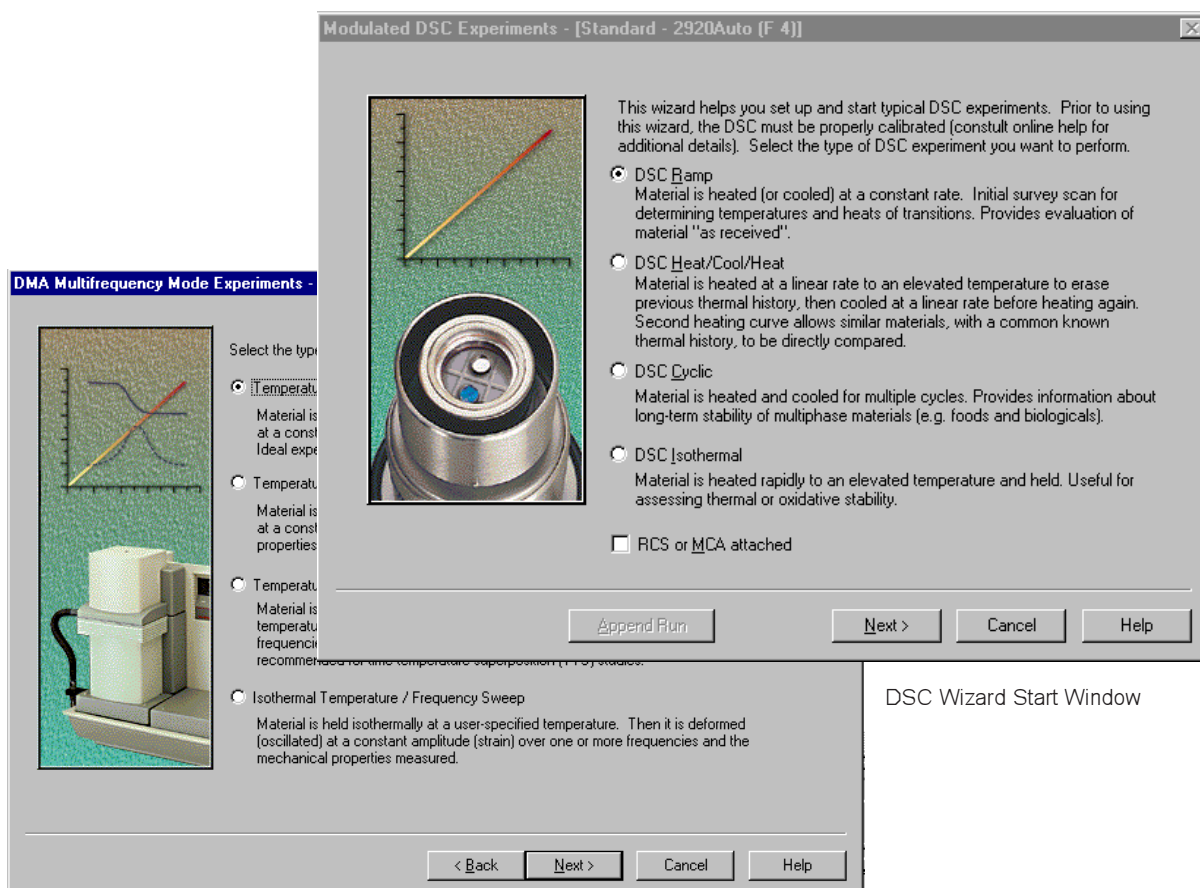
The *Advantage*TM program provides the ability to run experiments by the use of a guided Wizard. The Wizard will present you with options to choose from and provide information to guide you through each choice until you have completely set up and started an experiment.

General Wizard Operation:

To start your guided experiment setup click on the Wizard button, . As you step through the process you will set the mode, define the procedure, prompt for sample information, and display an experimental checklist, and start the run if desired.

The procedures in the Wizard are based on the available test templates for each instrument. Each instrument has a slightly different Wizard since each one operates differently and requires different kinds of tests with somewhat different information. A few examples of Wizard windows are shown below and on the next page. To obtain more detailed information at any time during the Wizard operation, click the **Help** button.

When you have finished selecting the information needed for your experiment, just click the **Start Run** button to begin your experiment.



DMA Wizard Multifrequency Tests Window

Experimental Checklist - [TGA 1000 °C - AutoTGA 2950 (F 3)]

Purge Gas

✓ Be sure that your purge gas(es) are connected and properly regulated. Purge Gas is required for all TGA experiments.

Taring the Sample Pan & Loading the Sample

✓ Before the start of every TGA experiment you will need to tare an empty sample pan. This ensures that the measured weight reflects only the sample. To tare, place a clean, empty sample pan onto the tray platform, and press the TARE button below.

Tare


✓ After taring, load the sample into the pan. Ensure that the pan is properly aligned with the groove in the platform.

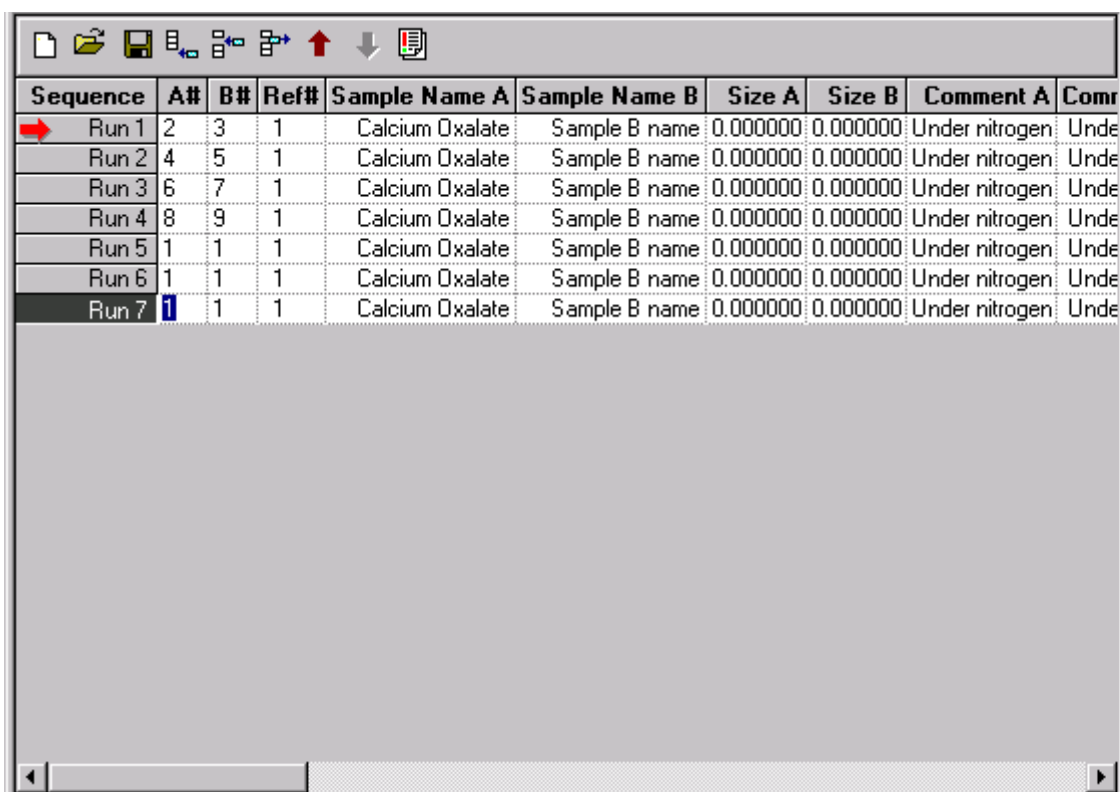
Append Run Start Run < Back Finish Cancel Help

TGA Wizard Checklist Window

Copying and Adapting Procedures

The **Table View** provides an alternative to the **Experiment View** for setting up and editing sample information for a sequence of runs. In this view, key sample information for each run is displayed and can be changed using a spreadsheet format (except for the Mode). **Table View** provides the greatest benefit after you have completely set up one run using the **Experiment View** and want to use the same procedure on other samples. You can just change the sample information. The specifics of the selected procedure cannot be changed in this view (switch to **Experiment View** to change the procedure).

1. Create or open the desired procedure using the **Experimental View** as directed earlier in this chapter.
2. Click the  button, select **View/Table View** from the menu, or press F11 on the keyboard to change to the **Table View Pane** (shown here). A spreadsheet type of



The screenshot shows a software window titled 'Table View' with a toolbar at the top containing icons for file operations and navigation. Below the toolbar is a table with the following data:

Sequence	A#	B#	Ref#	Sample Name A	Sample Name B	Size A	Size B	Comment A	Comment B
Run 1	2	3	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 2	4	5	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 3	6	7	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 4	8	9	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 5	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 6	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 7	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen

display is shown for each run with the column headings representing key experiment information contained in the current sequence. Most of the run information contained in the columns of the **Table View Pane** can be changed from this window, except for the Mode. The Sequence column is a list of the run numbers contained in the current sequence.

3. Make any desired changes to the information contained in the procedure as follows:
 - To change the Mode listed, select Experimental/Mode or click the button on the tool bar. Instrument mode can not be changed for a run sequence containing more than one run.
 - To change Sample Name, Comment:
 1. Click in the desired field.
 2. Type the desired text.
 3. Tab to the next field.
 - To change Sample Number, Reference Number, Pan, Pan Number, Probe, Clamp, Procedure, or Macro Name:
 1. Click in the desired field. A drop-down list becomes available.
 2. Scroll through the drop-down list and select the desired item.
 3. Tab to the next field.
 - To change Data File, Macro File:
 1. Double click in the desired field. A window is displayed to allow you to choose a new file.
 2. Locate the desired folder and select the desired file.
 3. Click the OK button. The filename is inserted into the column.
 4. Tab to the next field.
4. After you have made the desired changes you can schedule the run by positioning the pointer over the desired run in the sequence column of the table. When the pointer changes to an arrow, click the mouse to schedule that run.
5. Switch to the **Experiment View** to start the experiments. Runs cannot be started from the **Table View**.

Chapter 4


Sequencing Runs


Overview

If you have read Chapter 3, you should now be familiar with the basic set up of an experiment and the different options available for entering the required information for a single run (experiment). In this chapter we will describe how these runs can be grouped into a series called a *run sequence*. Run sequences allow you to set up runs for future samples, set up your most common experiments to allow you to easily switch between them, or set up an autosampler sequence. Up to ten runs, all performed using the same instrument mode, may be preprogrammed (up to 65 for an autosampler instrument). Runs are handled using the **Sequence Pane**.

Understanding the Sequence Pane

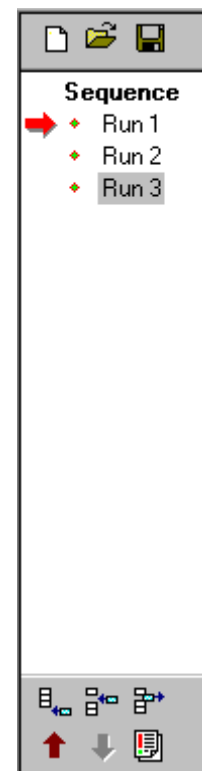
The **Sequence Pane** (shown here) displays the list of runs contained in the current sequence and, at the top and bottom of the pane, displays buttons to allow you to perform certain functions on those runs.

This pane is displayed when you click the  button to show the

Experimental View or when you click the  button to display the **RealTime Plot** in the middle pane. (If you select the **Table View**, the **Sequence Pane** is not shown. In **Table View** a column of the sequences currently available is displayed.)

What is a Run Sequence?

A run sequence is made up of a series of runs. A run is an experiment containing the sample name, procedure, method, post test conditions, and other parameters that are related to the running of an experiment. A sequence can contain up to ten preprogrammed runs that can be sent to the instrument one at a time in any order. The scheduled run is the experiment that will be run when start is selected. At the end of that experiment, you can choose to schedule a new run for execution.



NOTE: The scheduled run will not automatically advance to the next run, unless the instrument is an autosampler.

What is a Sequence File?

A sequence file (default extension is .seq) is made up of one or more run records (maximum of five). Each run record is instrument mode based and contains the sample and procedure information. A sequence file may be saved for future recall and execution. However, a sequence does not need to be saved to be executed. See “Performing Functions on Run Sequences” in this chapter for information.

NOTE: *Thermal Solutions* for NT or OS/2 autosampler sequence files are not compatible with this software.

Using the Sequence Pane

If you take a look at the **Sequence Pane**, you will notice an arrow pointing to one of the run numbers. This is the *scheduled run*. A run status icon (see the next page) will be displayed if the run is in setup, running, or complete state.

You can perform the following tasks using the run arrow:

- You can use the run arrow to schedule the next run. If you move the pointer (cursor) along the left of the run numbers, you should see the pointer change to an arrow. When it becomes an arrow, you can then click the mouse to position the arrow next to the selected run number. This run becomes the scheduled run, which is the experiment that will be run when start is selected.
- You can schedule and start a run using the **Control/Start** function. The arrow will change to green (➡) when the mouse is positioned over the arrow.
- You can double click on a red run arrow (➡) to start the scheduled run.

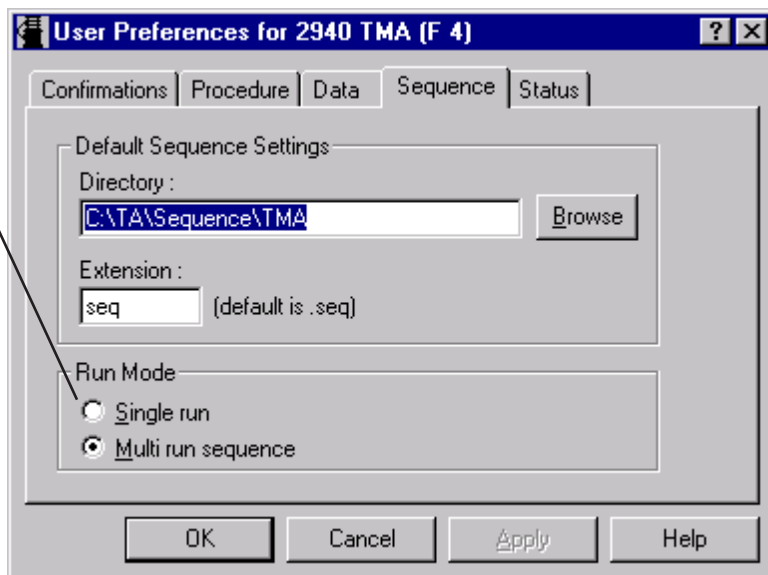
You can perform the following tasks using the run list:

- You can display the sample name by moving the pointer over the run number.
- You can drag-and-drop the runs to reorder the list, if desired.

NOTE: The run number does not follow the run when they are reordered—Run 1 remains Run 1—even though the run has actually been moved to a different position in the sequence list. You can tell that the run has been moved by displaying the sample name. If the run is next in the schedule, the run arrow will move with the run, if you reorder them.

Removing the Sequence Pane

To clear this pane from the Instrument Control window, choose the **Single Run** mode on the **User Preferences - Sequence Page**. This will ensure that the instrument performs one run at a time and does not run sequence files. (Multi-run sequence is the only option available for autosampler instruments.)



Run Status Icons

The status icons (shown below) displayed next to a run number in the **Sequence Pane** indicate the progress of the experiment.

- ◆ Standby
- ✓ Complete
- ✗ Incomplete
- 🏗 Setup
- ⌚ Holding
- 🏃 Running
- ⚖ DSC Autosampler Active (loading/unloading)
- ⚖ TGA Autosampler Active (loading)

Once a run is completed, a checkmark (✓) will appear next to the run number.

- If you click once on this mark, the data filename will reset to the next available name for the increment series.
- If you click twice on this mark, all of the data filenames for the completed runs in the sequence will be reset at once.

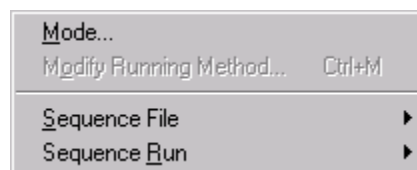
Handling Runs and Sequences

Individual runs can be added, removed, or moved within a run sequence. Run sequences can be created, saved, and opened. All of these functions can be performed using either the buttons or menus described in this section.

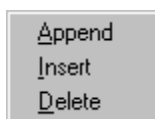


The buttons shown on the **Sequence Pane** are the same buttons that appear in the **Table View** tool bar (shown to the left). These buttons are used to create new sequences, open sequence files, save sequence files, and perform functions on the current sequence.

The **Experimental** menu (shown here) contains two submenus that are used for sequences and runs—**Sequence File** menu and **Sequence Run** menu (see figures below).



Sequence File Menu




Sequence Run Menu

The next several pages tell you how to perform various functions on runs and sequences.


Creating a New Run

See Chapter 3: Setting Up Experiments for instructions on creating a run (experiment).

Appending a Sequence Run

Select **Experimental/Sequence Run/Append** or click the  button on the **Sequence Pane** or **Table View** tool bar to add a run to the end of a run sequence. Append will copy the current run and add it to the end of the sequence, incrementing the filename so that a new sequence can be saved.


Inserting a Run in a Sequence

Select **Experimental/Sequence Run/Insert** or click the  button on the **Sequence Pane** or **Table View** tool bar to add a new run between existing runs in the run sequences. The new run will be added in the selected run position. Up to ten runs can be contained in one sequence. Up to 65 runs can be contained in an autosampler instrument's run sequence.


Information for the newly inserted run is copied from the selected run and the filename is automatically incremented.

NOTE: The sample pan number will not be automatically incremented, you will need to type in a unique sample pan number.

Deleting a Sequence Run


Select **Experimental/Sequence Run/Delete**, press the Delete key, or click the  button on the **Sequence Pane** or **Table View** tool bar to remove the selected run from a run sequence. The run numbers will be automatically renumbered in sequence after a run has been removed.

Moving a Run Up

Click the  button on the **Sequence Pane** or **Table View** tool bar to move the selected run up to a position one number up from its current position. You can click the button repeatedly until you have moved the selected run to the desired position.


You can also use the drag-and-drop method to change the order of the runs in the sequence.

Moving a Run Down


Click the  button on the **Sequence Pane** or **Table View** tool bar to move the selected run down to a position one number down from its current position. You can click the button repeatedly until you have moved the selected run to the desired position.

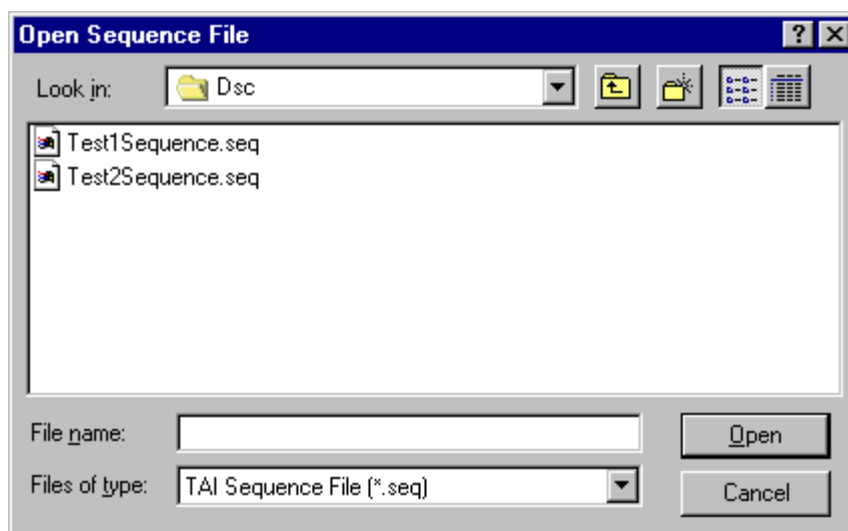
You can also use the drag-and-drop method to change the order of the runs in the sequence.

Creating a New Sequence

Select **Sequence File/New** from the **Experimental** menu (or click the  button on the **Sequence Pane** or **Table View** tool bar) to create a new sequence. When you choose this menu item you are prompted to save the current sequence file (if this confirmation option has been selected on the **User Preferences - Confirmation Page**). After saving the current sequence file, the old runs are erased and a single run record remains in the sequence.

Opening a Sequence File


Select **Sequence File/Open** from the **Experimental** menu (or click the  button on the **Sequence Pane** or **Table View** tool bar) to open an existing sequence file. When you choose this menu item, the **Open Sequence File** window, a standard file open window, is displayed to allow you to locate and select a sequence file to open. The sequence file selected must match the current instrument mode to open and recall the run records stored in the file.



The data files originally saved to this file are only restored if they do not already exist. Otherwise, the current file-naming scheme is used when you switch to **Experimental View**.

NOTE: *Thermal Solutions* for NT or OS/2 autosampler sequence files are not compatible with this software.

Saving a Sequence to File

Select **Sequence File/Save** from the **Experimental** menu (or click the  button on the **Sequence Pane** or **Table View** tool bar) to save a run sequence to file. When you choose this menu item, the **Save Sequence File** window, a standard file save window, is displayed to allow you to locate a directory and enter a new filename for the sequence file.

All the information within the **Experimental View** is saved for each run in the sequence.

Exporting a Sequence File

Select **Sequence File/Export** from the **Experimental** menu to export the contents of the **Table View Pane** in a spread sheet format for importing into other applications for reports and printing. When you choose this menu item, a standard file save window is displayed to allow you to locate a directory and enter a filename.

NOTE: This option is only available when Table View is displayed.

NOTE: The exported file cannot be opened or displayed by the *Advantage™* software. This function is strictly for use by other applications.

Choosing a Procedure File Location

See Chapter 1 for instructions on setting up a default location to save procedure files.

Viewing the Procedure File

You can view a procedure two different ways using the *Advantage*TM program:

- You can view the contents of a procedure file by clicking on the button located on the **Experimental View - Summary Page**. This will display the **Procedure Summary** window.
- You can view and manipulate procedure files by using the **Procedure Utility** window, which is accessed by selecting **Tools/Procedure Utility** from the instrument menu. This window is used to locate, view, print, delete, copy, and rename procedure files.

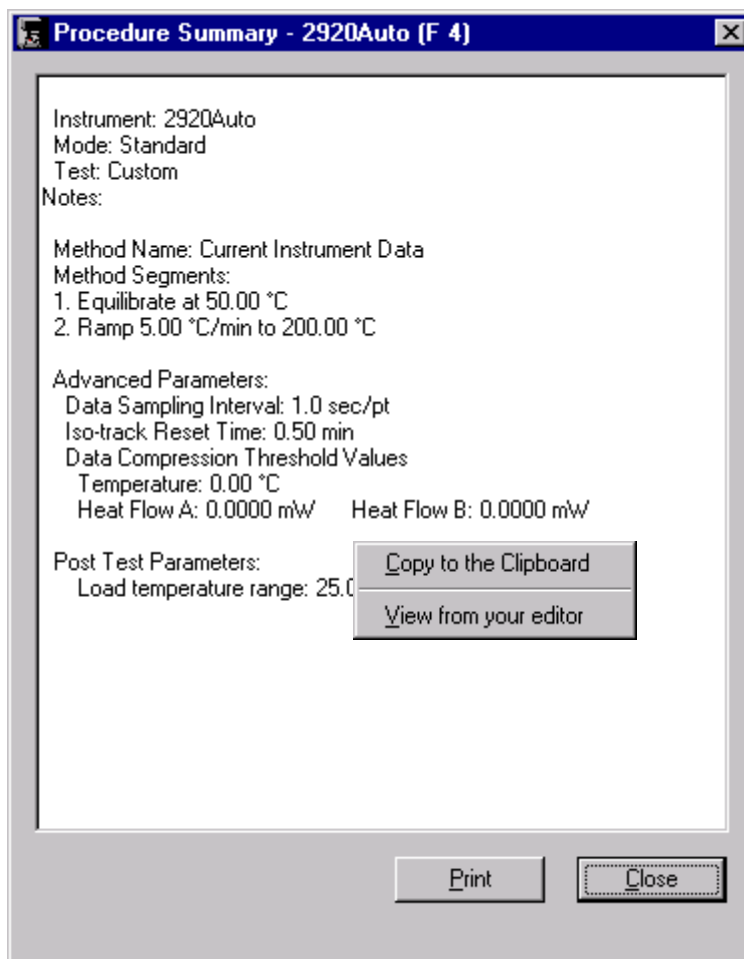
The following sections describe these two options.

Using the Procedure Summary Window

This is a view-only window that displays the contents of the current procedure file. This window contains the following information:

- Instrument name
- Instrument mode
- Test name
- Notes (relating to selected test)
- Method Name
- Method Segments
- Advanced Parameters
- Post Test Parameters

You can right click to display a pop-up menu (shown to the right) that allows you to copy this information to the clipboard or view it from your editor application.



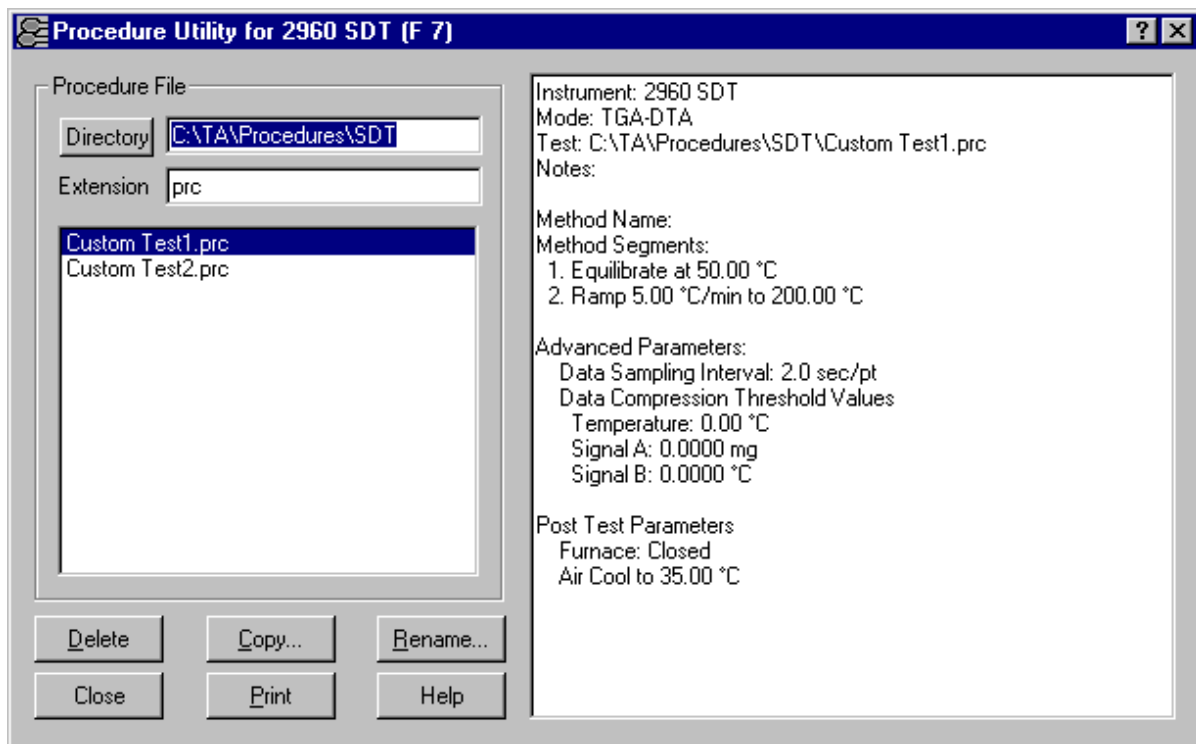
Click the **Print** button to print this information on your default printer.

Click the **Close** button to exit the window without printing the procedure file summary.

Using the Procedure Utility Window

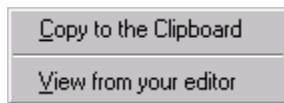
Once you have created procedure files (see Chapter 3 for information), you may need to locate these files for viewing, printing, deleting, copying, or renaming. To perform these functions on procedure files, follow these instructions.

1. Select **Tools/Procedure Utility** from the instrument menu. The **Procedure Utility** window (shown below) is displayed.



2. Enter or use the **Directory** button to locate the folder used for your procedure files.
3. Enter the extension as an alphabetic or numeric extension to be used as the extension for the procedure files (up to three characters may be used) or use the displayed default extension (.prc). A list of procedure files will be displayed below this field.
4. Select (highlight) the desired procedure file. The contents of the file will be displayed in the viewing pane on the right side.

5. Choose the desired function from the buttons on the bottom of the window. (The functions available will vary depending on the number of files highlighted.) For a detailed explanation of each function, see the list below:
- **Print Button:** Use this function to send the selected method file to the default printer.
 - **Delete Button:** Use Delete to remove the selected procedure file(s) from the specified directory.
 - **Copy Button:** Use the Copy button to duplicate the highlighted procedure file. The file may be copied to a diskette or another directory on the hard drive. Enter a new filename as requested.
 - **Rename Button:** Use the Rename button to change the name of the selected procedure file. Enter the new filename as requested on the window displayed.



If you right click in the viewing pane on the right side of the **Procedure Utility** window, a pop-up menu (shown here) is displayed to allow you to copy the entire contents of the procedure to the clipboard for retrieval by another application or display it in your default word editor.

Chapter 5

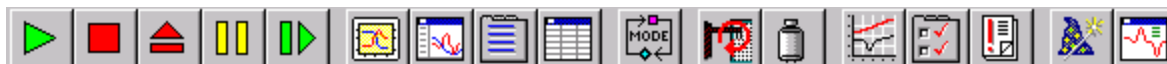
Controlling Your Experiments

Overview

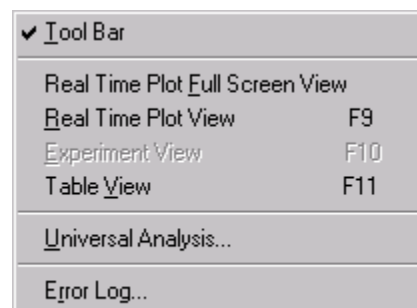
There are various ways to control the operation of the instruments. You can use the keypad on the instrument itself or you can control it through operation of TA Instruments *Advantage*™ program. To find out about instrument keypad operation, refer to the instrument's operators manual. This chapter deals with controlling the instrument through the software program.

Control Through the Tool Bars

Tool bars are a convenient way to operate any software program. The *Advantage*™ program offers a tool bar specific to each instrument control window. This section provides a brief description of all of the buttons found on the various tool bars.



If you decide that you do not want to display the tool bar, you can select the **View** menu (shown here) from the instrument menu and remove the checkmark from the **Tool Bar** menu item by clicking on it. This will remove the tool bar for that instrument from view. (You can check it again any time to display the tool bar once again.)



You can use the tool bar to start, stop, suspend, reject, or resume a running experiment. The items shown on the tool bar will vary based on the instrument. You can also use the tool bar to display selected windows. For an explanation of each button, see the list below.



Start: Use Start to begin an experiment. Start executes the first step in the current run.



Stop: Use Stop to halt an experiment and save the data. Selected Post Test functions will activate after this action.



Reject: Use Reject to halt an experiment and discard the data.



Hold: Use Hold to suspend an experiment, maintaining the current temperature conditions, until you resume or stop the method.



Resume: Use Resume to continue the method that was put on hold, continuing with the suspended segment.



RealTime Plot Full Screen View: Use this button to display the RealTime Plot window in full screen view.



RealTime Plot Pane View: Use this button to display the RealTime Plot window in the middle pane along with the Sequence Pane, Signal Display Pane, and Running Segment Pane.



Experimental Pane View: Use this button to display the Experimental Pane along with the Sequence Pane, Signal Display Pane, Running Segment Pane, and a small (thumbnail) view of RealTime Plot.



Table Pane View: Use this button to display the Table View Pane along the Signal Display Pane, Running Segment Pane, and a small (thumbnail) view of RealTime Plot.



Mode Parameters: Use this button to open the Mode Parameters window.



DSC Autosampler Reset: Use this button to reset the Autosampler.



LNCA/GCA Fill: Use this button to automatically fill the LNCA or GCA.



TGA/SDT Tare: Use this button to automatically tare the instrument.



TGA Weight Calibration: Use this button to begin the weight calibration procedure with Step 1.



DMA Clamp Calibration: Use this button to calibrate the installed clamp.



TMA Probe Calibration: Use this button to begin the probe calibration.



DEA Electronic Calibration: Use this button to calibrate the instrument's electronics.



Calibration Analysis: Use this button to begin the calibration analysis.



User Preferences: Use this button to open the User Preferences window.



Error Log: Use this button to open the Error Log window.



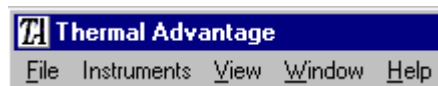
Wizard: Use this button to begin the instrument's Wizard.



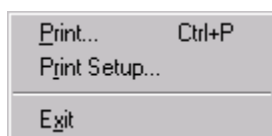
Universal Analysis: Use this button to open or recall the Universal Analysis program.

Controlling the *Advantage*TM Program

The Controller menus across the top of the main controller window perform the same function regardless of the instrument control window that is currently active. This section provides a brief description of these menus and their functions.



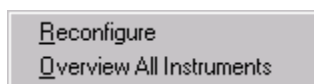
File Menu



The **File** menu (shown here) is used to perform the following functions with the current RealTime Plot data.

- **Print:** Used to print the RealTime Plot of the current instrument. Shortcut: **Ctrl+P**.
- **Print Setup:** Used to select a printer and printer connection.
- **Exit:** Used to exit the program and close the instrument window. If an experiment is in progress, data transfer to the controller will be stopped. Use the Data Transfer function to recover the data.

Instruments Menu



The **Instruments** menu (shown here) is used to bring the instruments online with the Thermal Analyst controller and to observe current information about the instruments that are online.

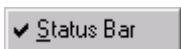
Bringing Instruments Online

Select **Instruments/Reconfigure** from the menu. All of the instruments that are powered up and connected to the controller will be reconfigured and brought online. (Each instrument online must have a unique GPIB address between 1 and 9.) The Reconfigure function is also used to bring an instrument online in the Loader Mode to install new instrument software.

Observing Instrument Information

Select **Instruments/Overview All Instruments** from the menu. The **Overview All Instruments** window is displayed that you can use to observe the temperature and other information about all of the instruments that are online. See Chapter 1 for more information.

View Menu



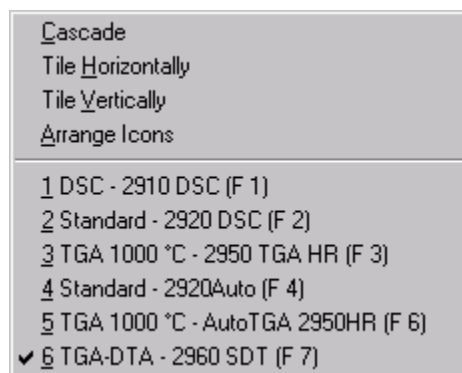
The **View** menu (shown here) for the controller window is used to turn on certain items for display. You can check and uncheck the **View/Status Bar** function. A check mark appears next to the menu item when the Status Bar is displayed.

When checked, the status bar is displayed at the bottom left of the instrument window. The status bar shows actions of menu items as you use the arrow keys to navigate through menus. This area similarly shows messages that describe the actions of tool bar buttons as you move the cursor over them.

Window Menu

The **Window** menu (shown here) offers the following commands, which enable you to select or arrange the views of multiple instruments in the application window:

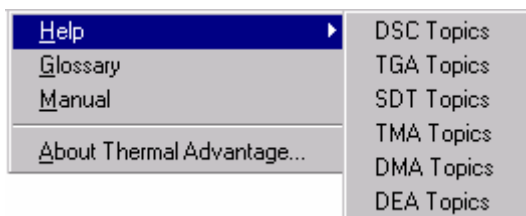
- **Cascade:** Arranges windows in an overlapped fashion.
- **Tile Horizontally:** Arranges windows in non-overlapping horizontal tiles.
- **Tile Vertically:** Arranges windows in non-overlapping vertical tiles.
- **Arrange Icons:** Arranges icons of closed windows.
- **Instruments 1, 2,:** Displays the specified instrument control window. See “Switching Between Windows” in Chapter 1 for more information.



Help Menu

The **Help** menu (shown here) offers access to the following help functions:

- **Help Topics:** Use this menu item to display the help topics available for each instrument. Just click on the instrument that you want to learn about.
- **Glossary:** Use this menu item to display the thermal analysis online glossary of terms. You can locate the definition of a term using the alphabetical listing shown.
- **Manual:** Use this menu item to display this online manual for the Advantage™ software.
- **About Thermal Advantage™:** Use this command to display the copyright notice and version number of your copy of Advantage™.



Controlling Instrument Operation

The **Control** menu is used to start, stop, or control the instrument and its accessories. The various instruments may have slightly different **Control** menus, depending upon the needs of the instrument. A few examples are shown here.

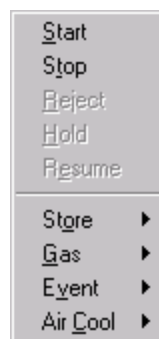
NOTE: Allow the instrument to warm up for at least 30 minutes before starting an experiment.



**TGA & DSC
Autosampler**



DSC



SDT, TGA, & TMA



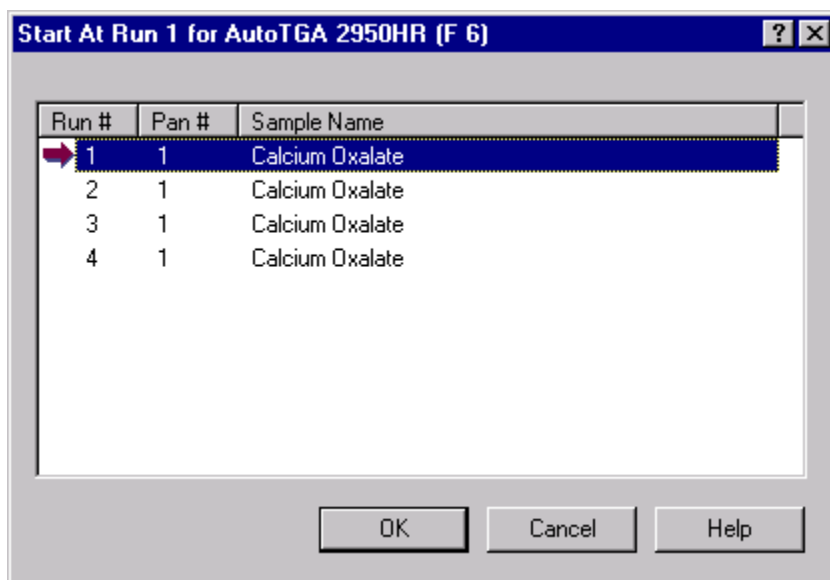
DMA


For more information on the **Control** menus, see the following pages.

Starting an Experiment


Once the sample has been loaded and the experimental parameters set up, you can start your experiment one of four ways:

- Use **Start** on the **Control** menu to begin an experiment. When you select **Start**, and there is more than one run in the sequence list, the **Start At** window is displayed to allow you to choose the run to begin. See the figure here. Click on the desired run to move the run arrow to that run number, then select OK.



- Click  on the tool bar. If more than one run is contained in the run sequence, the scheduled run (run with the arrow pointing to it) will be executed. To schedule a run, prior to starting the experiment, move the pointer (cursor) along the left of the run numbers, you should see the pointer change to an arrow. When it becomes an arrow, you can then click the mouse to position the arrow next to the selected run number. This run becomes the scheduled run, which is the experiment that will be run when Start is selected.
- The scheduled run can also be executed by double clicking on the red run arrow (➡) in the **Sequence Pane**.
- Schedule the desired run using the controller software (as outlined above), then press the START key on the instrument keypad.

Stopping an Experiment

To halt an experiment at the selected time (see list of options below) and *save* the data, you can select **Stop** on the **Control** menu or click the  button on the tool bar. Selected post-test conditions (set on the **Post Test** window) will activate after this action. To open the **Post Test** window, select the **Procedure Page** in **Experimental View**, then click on the **Post Test** button.


A confirmation message will be displayed when **Stop** is chosen, unless this option is turned off on the **Confirmations Page** of the **User Preferences** window accessed under the **Tools/User Preferences**.

Autosampler Stop Options

When an autosampler sequence is in progress, the following stop options are available:

- **Current Run/Action:** Select this autosampler stop option to immediately halt the active run or action (*e.g.*, air cool) only. The next run in the sequence will begin once the post test conditions are executed (*e.g.*, DSC Load Temperature Range or TGA Air Cool Time).
- **Run Sequence Immediately:** Select this autosampler stop option to halt the current run sequence immediately. None of the remaining runs in the sequence will be executed. The autosampler will wait until you select **Start** to begin another sequence.
- **Run Sequence After Current Run:** Select this autosampler stop option to halt the autosampler action after the current run has been completed. The autosampler will wait until you select **Start** to begin another run.

Rejecting a Run

To halt an experiment and *discard* the data, you can use **Reject** on the **Control** menu or click the  button on the tool bar. Selected post-test conditions (set on the **Post Test** window) will activate after this action. To open the **Post Test** window, select the **Procedure Page** in **Experimental View**, then click on the **Post Test** button.

A confirmation message will be displayed when **Reject** is chosen, unless this option is turned off on the **Confirmations Page** of the **User Preferences** accessed under the **Tools/User Preferences**.

Holding a Run

You can suspend an experiment, maintaining the current temperature conditions, until you resume or stop the method by selecting **Hold** on the **Control** menu or clicking the



button on the tool bar. The run will be suspended until you choose to resume it.

Resuming a Run

To continue the run that was put on hold, starting with the suspended segment, select

Resume on the **Control** menu or click the  button on the tool bar. The instrument will continue the run that was suspended.

Storing Data

You can manually turn data storage on or off during a run by selecting **Store/On** or **Store/Off** from the **Control** menu. This control overrides the Data Storage segment in the procedure.

If the selected Data Storage option is currently active, no action will be seen.

NOTE: Data Storage segment is a method segment that turns data collection on and off. In the absence of this segment, data storage is automatically initiated with the first Isothermal, Isotrack, Ramp, or Step segment.

Switching the Gas Purge

To manually select either gas 1 or gas 2, when using the Gas Switching Accessory (GSA), select **Gas/Gas 1** or **Gas/Gas 2** from the **Control** menu. This control overrides the Gas segment in the method.

If the selected gas is currently active, no action will be seen.

NOTE: The Gas segment is a method segment that controls the gas selected by the Gas Switching accessory.

Turning an Event Device On or Off

You can activate (on) or deactivate (off) an external device connected to the instrument with the event cable by selecting **Event/On** or **Event/Off** from the **Control** menu. This control overrides the Event segment in the method.


If the selected Event state is currently active, no action will be seen.

NOTE: The Event segment is a method segment that automatically controls the external event relay through the event jack located on the instrument.

Turning Air Cool On or Off

To open (on) or close (off) the internal solenoid valve connected to the Air Cool connector on the exterior of the instrument, select **Control/Air Cool/On** or **Control/Air Cool/Off**.

Using the LNCA/GCA Fill Option

You can have the Liquid Nitrogen Cooling Accessory or Gas Cooling Accessory automatically refilled from a low-pressure bulk storage tank while an experiment is not active by choosing **Control/LNCA Fill** or **Control/GCA Fill**, or you can click the  button on the tool bar.

Controlling the DMA Drive Motor

To manually turn on or off the DMA drive motor, select **Control/Motor/On** or **Control/Motor/Off** from the DMA menu. When turning on the motor, it will be initialized according to the current operating mode, clamps, instrument parameters, and frequency or amplitude table (as appropriate). This function is useful when setting up the samples in the DMA to determine whether the sample conditions (geometry, mounting, force, frequency, amplitude, etc.) meet your criteria. This function overrides the Motor Drive segment in the method.

If the motor drive is already in the selected state when the option is chosen, no action will be seen.

Raising the DEA Ram

To raise the DEA ram to the fully open position, which allows you to remove the ram, change the sensors, load a sample, or obtain access to the furnace area; select **Control/Ram Up** from the DEA menu.

Beginning the Dielectric Measurement


Dielectric measurement can be started by selecting **Control/Measure** from the DEA menu, without starting the run. This function is used to check the signals when the ram is lowered before the method is started. For example, you can select **Control/Measure**, then view the **Signal Display Pane** to check the dielectric properties, sample thickness, force, etc. before you begin a run.

Controlling the Autosampler

The **Control/Autosampler** menu allows you to choose from several menu items that can be used to control the actions of the Autosampler. The following sections explain these options.

Autosampler/Reset

The **Autosampler/Reset** option in the **Control** menu does different things, depending upon the type of Autosampler you are using.

For DSC Autosampler—Use **Reset** or click the  button on the tool bar to cancel all Autosampler motion, erase the Autosampler memory, and return the Autosampler turret to the home position. If any pans remain in the cell, they will need to be manually removed using tweezers or the motion keys on the Autosampler keypad.

NOTE: All lids should be replaced in their storage positions before resetting the Autosampler or the instrument will report an error.

For AutoTGA—Use **Reset** to open the TGA furnace and return the Autosampler sample platform to the home position. If a pan remains on the hangdown wire, it will need to be manually removed using tweezers or unloaded by pushing UNLOAD on the instrument.

Autosampler/Halt Motion

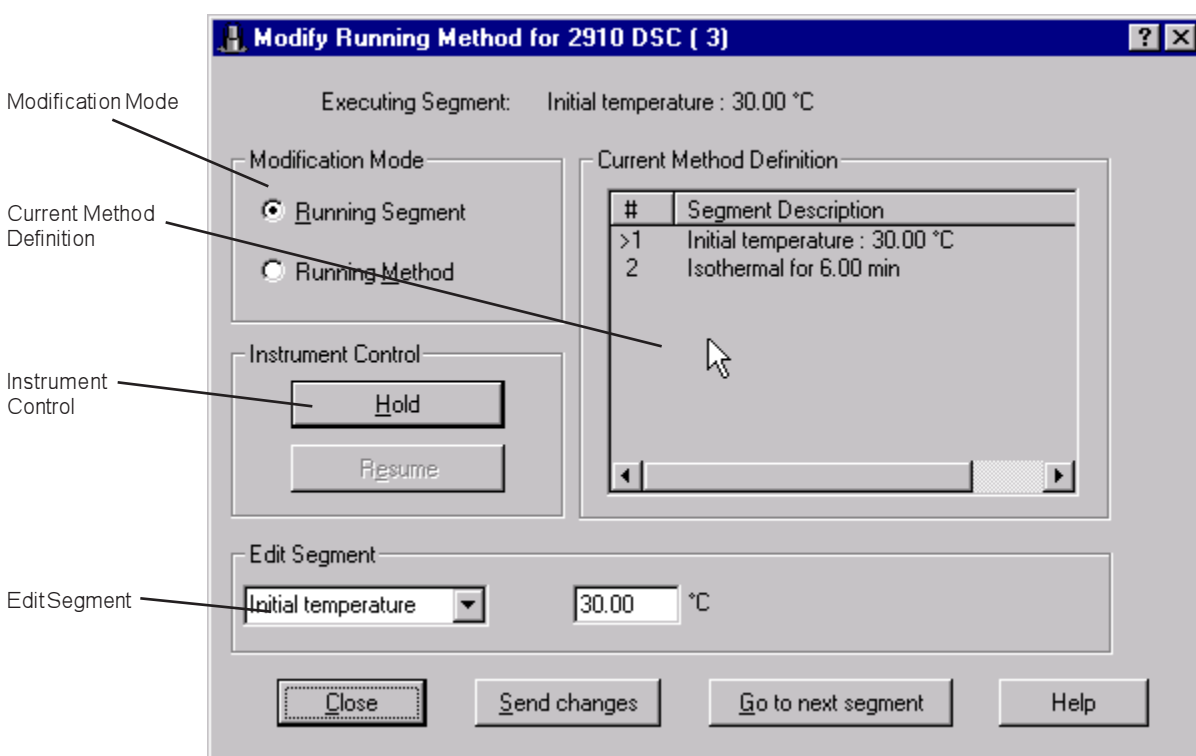
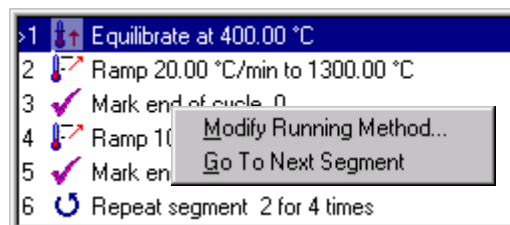
You can also stop the Autosampler motion, without canceling any commands, by choosing **Autosampler/Halt Motion** from the **Control** menu. Select Resume motion to restart the Autosampler motion where it was halted. If the Autosampler is not in motion, this command is ignored.

Autosampler/Resume Motion

Use **Autosampler/Resume Motion** from the **Control** menu to restart the Autosampler motion when the instrument was halted by selecting **Autosampler/Halt Motion**.

Modifying Runs in Progress

You may find that you want to change an experiment while it is in progress. You can do this by either selecting **Experimental/Modify Running Method** or by clicking on the pop-up menu found on the **Running Segment Pane** (shown here). The **Modify Running Method** window is then displayed. This window allows you to edit the segment parameters (values) used in the method. You cannot, however, alter the actual segment types or add additional segments while the experiment is in progress.



This window consists of four main parts:

- **Modification Mode** - choice of Running Segment or Running Method mode using the radio buttons.
- **Current Method Definition** - list of method segments.
- **Instrument Control** - Hold and Resume buttons.
- **Edit Segment** - specification of the segment parameters.

The next two sections deal with your choice of either modifying the segment currently running or modifying the entire method that is currently running.

Modifying a Running Segment

To modify the segment that is currently being executed on the instrument, select the **Running Segment** radio button on the **Modify Running Method** window.

This will have the following effect:

- Make changes to the parameters of only the segment that is currently being executed, if you click the **Send Changes** button.
- Stop the executing segment and proceed to the next segment, if you click **Go to Next Segment**.
- Change the segment definition for the current segment execution without changing the method stored in the instrument.

NOTE: The **Go to Next Segment** button releases a Hold status and resumes the method.

Use this mode to modify the parameters of only the segment that is currently running on the instrument.

To modify the current segment:

1. Select the **Running Segment** radio button as the Modification Mode. The segment currently running will be highlighted in the Current Method Definition field.
2. Make the desired changes in the Edit segment fields. (The segment type may only be changed between Jump, Equilibrate, and Initial Temperature.)
3. Click the **Send Changes** button and the new segment parameters will be sent to the instrument for execution.
4. Select **Close** when you have finished using this window.

You may elect to use the Instrument Control functions—**Hold** to pause the method in progress and **Resume** to continue the method.

NOTE: The modified running segment parameters are not saved to the instrument or to the procedure after the experiment is completed. To make and save changes to a procedure, you must change the test conditions using the Experimental View—Procedure Page.

Modifying a Running Method

To modify the entire method (or run) that is currently being executed on the instrument, select the **Running Method** radio button on the **Modify Running Method** window.

Select this radio button to:

- Make changes to the parameters for any segment in the executing method.
- Change the method definition for the next time the method is executed.
- Use this mode to change the parameters for any segment in the executing method.

NOTE: Changes made here to a running segment will not be executed unless the segment is repeated using the Repeat segment. The Modify Running Segment mode can be used to make changes to a running segment.

To modify the current method:

1. Select the **Running Method** radio button as the Modification Mode.
2. Select the segment that you wish to alter in the Current Method Definition field.
3. Position the cursor in an Edit segment field. (Hint: Double clicking on the highlighted segment will position the cursor in the first Edit segment field.)
4. Make the desired changes.
5. Click **Send Changes** and the method will be altered as seen in the Current Method Definition list.
6. Repeat steps 2, 3, 4, and 5 to modify another segment in the current method.
7. Select **Close** when you have finished using this window.

NOTE: The modifications that you have made to the current method are sent to the instrument, but they are not saved to the procedure. (Therefore, the original procedure remains unchanged.) To make and save changes to a procedure, you must change the test conditions using the Experimental View—Procedure Page.

Transferring Data to the Instrument

The **Data Transfer** function, selected from the **Tools** menu (shown here) enables the manual transfer of collected data from the instrument to a file. The instrument can store data from one experiment at a time; therefore, to retain the data, it must be transferred from the instrument to disk storage on the computer before the start of the next experiment. The data will normally be transferred automatically from the instrument to the controller. Transferring data is needed only in certain situations, listed below.

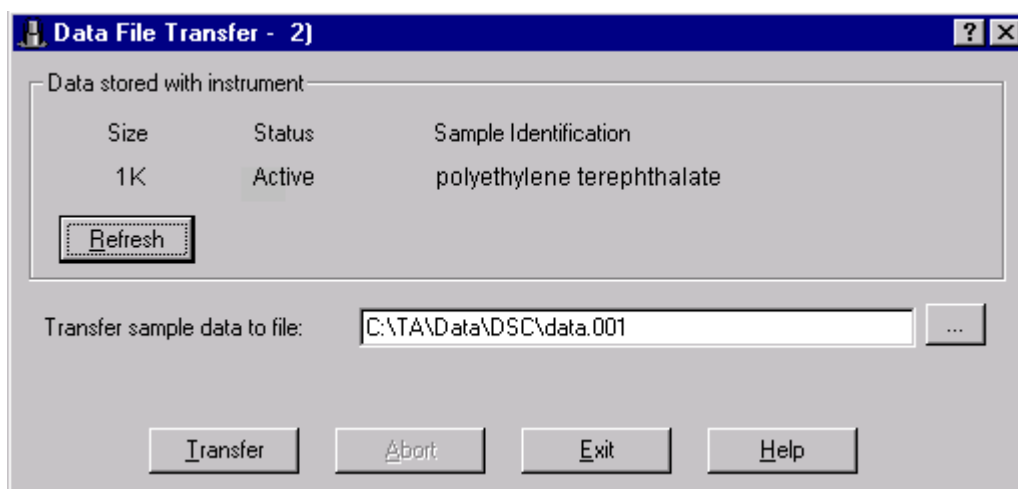


Data file transfer should be used in the following types of situations:

- If the connection between the instrument and the controller is lost during the execution of a method. This may occur if the experiment is in progress and the TGA instrument window is closed or the controller is disconnected, rebooted, or goes offline. The data cannot be automatically transferred when the connection is lost.
- If the target drive for transferred data becomes inaccessible during an experiment. This may occur if the disk becomes full while data is transferring, if a targeted diskette is ejected or the targeted network connection is lost during data transfer.
- If the specified data file already exists, and the situation is not corrected before the experiment begins. Because the existing file will not be overwritten, in this situation the data must then be transferred from the instrument to a new data file.

To transfer a data file:

1. Select **Tools/Data Transfer** from the instrument menu. The **Data File Transfer** window (shown below) is displayed.



2. Enter or use the Browse button to specify the complete path and desired filename to be used for the data file storage. The data filename will always default to <instrument type><GPIB address><.001>.
3. Click the **Transfer** button. A status line will display when the transfer is complete.
4. Select **Exit** when you are finished.

Use **Abort** to stop the data transfer process. The data file that was opened for data transfer is deleted.

NOTE: To get the complete data file, wait until the experiment has run to completion before transferring the data. If you try and transfer a file while an experiment is in progress, the filename displayed is the same as the active filename (the filename is not incremented). You will, in this case, need to specify a different filename to transfer the data. If you wait until the experiment has completed, the filename will increment correctly.

Resetting the Instrument

Select **Reset Instrument** from **Tools** menu (shown here) to return the instrument parameters, weight calibration, sample platform calibration, and temperature calibration to their default conditions and clear all sample information. All sequenced runs are deleted, and any runs stored in the instrument are erased. Calibration routines will need to be performed after this operation.

A message appears to confirm the information above. Select **OK** to reset the instrument or select **Cancel** to abort the operation.

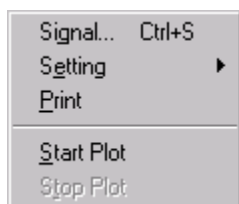


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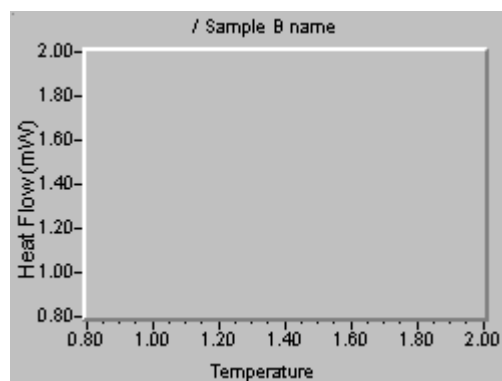
Monitoring and Viewing Your Data

Using the RealTime Plot Pane

The *Advantage*[™] **RealTime Plot Pane** (shown here) provides a continuous display of the instrument status data as it is being generated, as well as access to a right-click pop-up menu (also shown below). The menu enables you to start



and stop the plot, define settings and colors for the plot and plot window, copy the plot to the clipboard, and print the plot. See “Choosing Settings for RT Plot Appearance” for more details.



NOTE: RealTime Plot will stop plotting and automatically default to Signal A when an instrument is reconfigured or when the Mode Parameters window is opened.

NOTE: You can double click on the RealTime Plot Pane to automatically switch to the RealTime Plot Full Screen View.

There are three ways to view the RealTime Plot:

- In the main pane by selecting **View/RealTime Plot View**.
- In full screen view by selecting **View/RealTime Plot Full Screen View**.
- As a small (thumbnail) pane by selecting either **View/Experiment View** or **View/Table View**.

For details on the views listed, see: “Choosing Your Viewing Options” in Chapter 1.

Selecting Signals to Plot

You can set up the plotting of the real-time data by choosing which signals to plot and the line type and grid type to be used for the Y1 and Y2 axes, along with the color and scaling desired for each signal selected. To accomplish this, select **Signal** from the **RealTime Plot** popup menu or from the **Tools/RT Plot** menu. The window shown to the right is displayed.

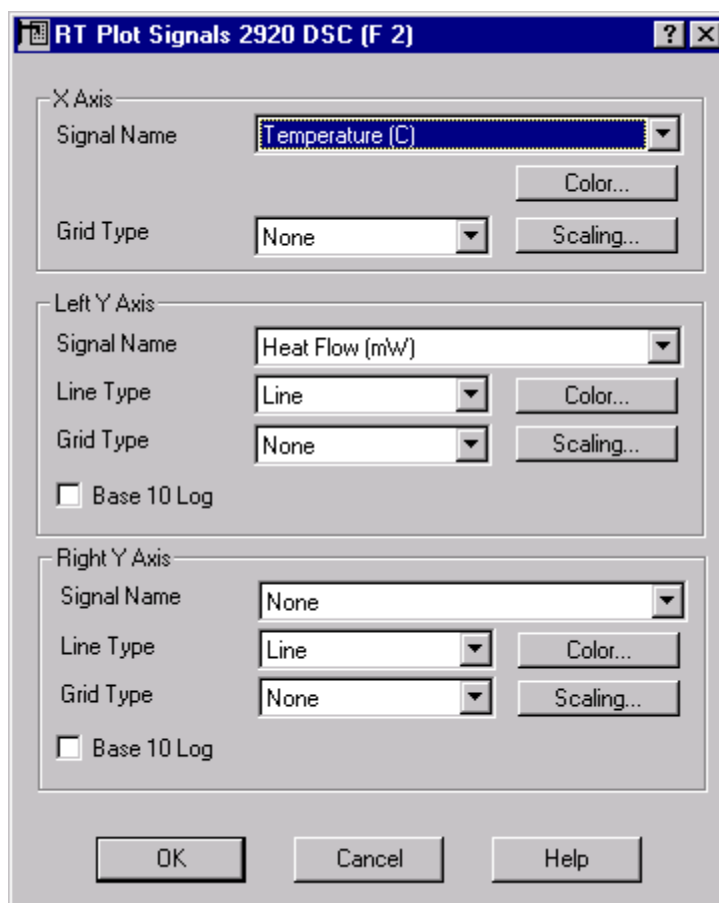
To set up the signals to plot, follow these steps:

1. Click on the arrow next to Signal Name to display the drop-down list of the X-axis signals available (time and temperature) for plotting on the X axis. If an experiment is in progress, the x-axis signal may be changed without losing the previous real-time data.

2. Select the X-axis Grid Type from the drop-down list containing the following types. The grid lines always correspond to the major tick marks only.

- Solid will result in a solid line used for the grid.
- Dotted will result in a dotted line used for the grid.
- Grayed will result in a grid displayed in a gray-out style (generally seen as a background grid).

3. Click on the **Color** button to select the color from the palette to be used for the X axis.
4. Click on the **Scaling** button to open the **Scaling Axis** window (shown here), which is used to select the scaling to be used for the X axis.



5. Set up the lower and upper axis limits for the RealTime plot display area. You can choose to have the limits set automatically, using the data as it is collected, or you can choose to manually enter the axis limits desired.

NOTE: If you choose to manually set the axis limits, data that goes beyond those limits may not be displayed.

- a. Select one of the following radio buttons for the Lower data limit:
 - **Auto**—to have the data limits automatically scaled to fit within the RealTime plot window.
 - **Manual**—to manually choose the data limits to be displayed on the RealTime plot window. If Manual is chosen, you must also enter the desired lower limit in the entry field below the Manual button.
- b. Repeat step a for the Upper data limit. (NOTE: You can have one Auto and one Manual axis limit.)
- c. Click **OK** when finished.

NOTE: When a different axis signal is selected, the axis limits are always reset to Auto.

6. Click on the arrow next to the Left Y-axis Signal Name to display the drop-down list of the signals available for plotting on the left Y axis. This list includes signals available in the current instrument mode. When a change is made to the selected signal while RealTime Plot is active (e.g., during a run), the previously-displayed data is lost. The plot is restarted using the newly selected signals.

NOTE: For dual sample DSC cell users: Displayed in the signal list are two heat flow signals. The first one in the list corresponds to Side A, the second to Side B. This is also true for the temperature signal.

7. Select the Left Y-axis Line Type from the drop-down list containing the following types:
 - **Area**: Select the Area option to create a plot that has the area under the curve filled in with shading. The color used for the area option is defined by the curve color.

- **Line:** Select the Line option to create a plot that is a single line curve. The color used for the line is defined by the curve color.
- **X-Y Line:** Select the X-Y line option to create a plot that has an X placed at the points on the intersection of the x-y axis, and a single line connecting each X point. The color used for the points are defined by the curve color.
- **X-Y Points:** Select the X-Y point option to display the data as points only, with minimal data compression applied. The color used for the points are defined by the curve color. Because data is less likely to be compressed using this option, the maximum number of points displayed will be reached sooner. As the plot progresses, older data points may be lost on the display to make room for incoming data points.

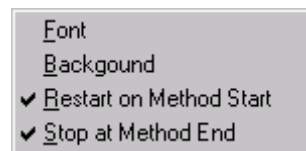
NOTE: The X-Y points option should be selected when plotting multiplex data (i.e., DMA and DEA data).

8. Select the Left Y-axis Grid Type from the drop-down list (see step 2 for a description). The grid lines always correspond to the major tick marks only.
9. Click on the **Color** button to select the color from the palette to be used for the Left Y axis.
10. Click on the **Scaling** button open the **Scaling Axis** window, which is used to select the scaling to be used for the Left Y axis.
11. Check the Base 10 Log box if you want the axis to be plotted as a base ten logarithm. This feature is used most often for DMA and DEA plots.
12. *For SDT and TGA Instruments Only:* Check the Use Normalized Weight Units (%) to use normalized weight units (%) when weight is selected on the left Y axis. When a change is made to this checkbox while RealTime Plot is active (e.g., during a run), the previously-displayed data is lost. The plot is restarted using the newly selected weight units.
13. Repeat steps 6 through 12 for the Right Y axis (or Y2 axis).
14. Click the **OK** button when you have completed your selections.

Choosing Settings for RT Plot Appearance

You can customize the font and background color within the **RealTime Plot Pane** in order to capture more attractive plots for presentations or printing on a color printer.

Click on the arrow next to the **Setting** menu item on either the **Tools/RT Plot** menu or the **RealTime Plot** pop-up menu to display the **Settings** menu shown here.



- **Font:** Use Font to choose the font that will be applied to all of the text in the selected RealTime plot display area. (The RealTime Plot title is associated with the current sample name.) When you select this option, a standard Font window is displayed.
- **Background:** Use the Background option to choose the background color for the selected RealTime Plot area. When you select this option, a standard Color palette window is displayed.

NOTE: To customize the colors used for the axes and their corresponding curves, select Signals from the pop-up RealTime Plot menu to display the Signals window. See "Selecting Signals to Plot."

Starting the RealTime Plot

There are basically two ways to start plotting your real-time data from the instrument:

- You can start it manually by selecting **Start Plot** from the **Tools/RT Plot** menu or from the pop-up **RealTime Plot** menu. This option will refresh the display with new data. If you are plotting data versus time, the time will be reset to zero. Data collected before Start is used will not be displayed.

or

- You can check **Settings/Restart on Method Start** from the **Tools/RT Plot** menu or from the pop-up **RealTime Plot** menu. When you check this option, the graph is cleared and the program will automatically begin plotting the new data on the RealTime Plot window when a method is started and instrument set up is complete.

Stopping the RealTime Plot

There are two ways to stop the plotting of real-time data from the instrument:

- You can manually stop the plotting by selecting **Stop Plot** from the **Tools/RT Plot** menu or from the pop-up **RealTime Plot** menu.

or

- You can check **Settings/Stop at Method End** from the **Tools/RT Plot** menu or from the pop-up **RealTime Plot** menu. When you check this option, the graph is halted on the **RealTime Plot** window between methods. At the end of the method, the data curve will stop being drawn.

NOTE: Do not select this option if you want to continue to display the data after the experiment is complete.

Printing RealTime Plots

You can print the RealTime Plot graph area that appears on your display by selecting **Print** from the **Tools/RT Plot** menu or from the pop-up **RealTime Plot** menu. A standard Windows NT® print window is displayed to allow you to select printer options.

NOTE: Any font options that you select for your RealTime Plot display will not be printed.

Viewing the Signal Display

The **Signal Display Pane** shows the signals that are output from the instrument. These are all of the signals that are currently being displayed by the controller from the instrument online. The signals displayed are dependent on instrument type and mode. (See the instrument online help for a description of each signal.) You can use this window to monitor experiments. A pop-up menu is provided to enable you to zero the signals from the instrument both automatically and manually.

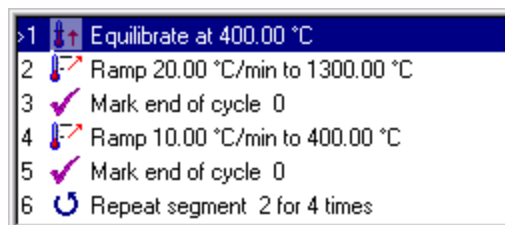
Run Time:	0.00	min
Segment Time:	0.00	min
Set Point Temp:	0.00	°C
Heater Power:	0.0	watts
Data Storage:	0.07	% full
Signal A:	-1.585	Heat Flow (mW)
	-5.83	µV
Offset:	0.00	µV



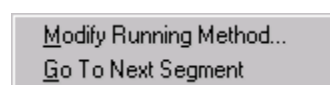
Each instrument can display a **Signal Display** pop-up menu (DSC shown here) that is slightly different depending on the functioning of the instrument. These menus are generally used to zero or offset the signal. Refer to the online help associated with the instrument for more information on these menu items and their functions.

Viewing the Running Method

The **Running Segment Pane** (shown here) is used to view the segments contained in the run that is currently in progress. Each test used in a run contains certain segments, which are displayed in this pane as they are run. You can monitor these segments and change them, if desired, either after the run is finished or during the run. Follow these instructions to change a run.



Changing a Run in Progress




If the experiment is in progress, select either **Modify Running Method** or **Go To Next Segment** from the **Running Segment** pop-up menu (shown here). These options allow you to make the following changes to the run already in progress:

- **Modify Running Method** allows you to edit the segment parameters (values) used in the method. You cannot, however, alter the actual segment types or add additional segments while the experiment is in progress. See “Modifying Runs in Progress” in Chapter 5 for details.
- **Go To Next Segment** advances the method to the next segment at the time of selection.

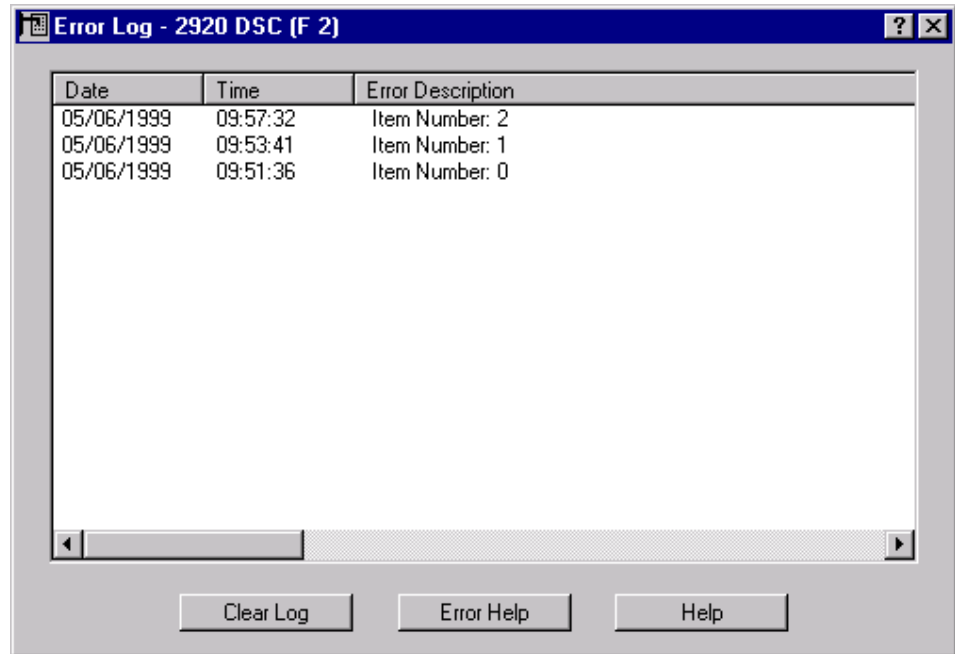
Changing an Inactive Run

To make changes to the segments that are used in a run, you must select a Custom test on the **Summary Page**. Then, to make changes to the Custom test, select the **Procedure Page**, click the **Editor** button to open the **Method Editor** window, then make your changes. These steps are detailed in “Creating a Custom Test” found in Chapter 4.


Viewing the Error Log

You can open the Error Log by clicking on the  button on the tool bar or by selecting **View/Error Log** from the instrument menu. The **Error Log** window displays a list of the error messages that have occurred since you started your instrument or since the time the log was last cleared.

Double-click on the individual error message in the Error List to obtain help for that error or highlight the message and then select **Error Help** either by clicking the button or from the pop-up menu.



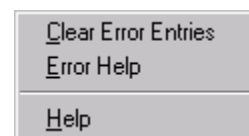
Using the Error Indicator

When an error occurs, an indicator, , is displayed in the status line. The following actions can be used with the error indicator icon:

- Position the pointer over the error indicator to display the full error message (number and description).
- Double click on the error indicator to open the **Error Log** window (above).
- Right click on the error indicator to display the **Error Log** pop-up menu, described on the next page.

Using the Error Log Pop-Up Menu

The **Error Log** window contains both a pop-up menu (shown here) and buttons that can be used to clear the error messages currently displayed on the log, or to obtain Help information.




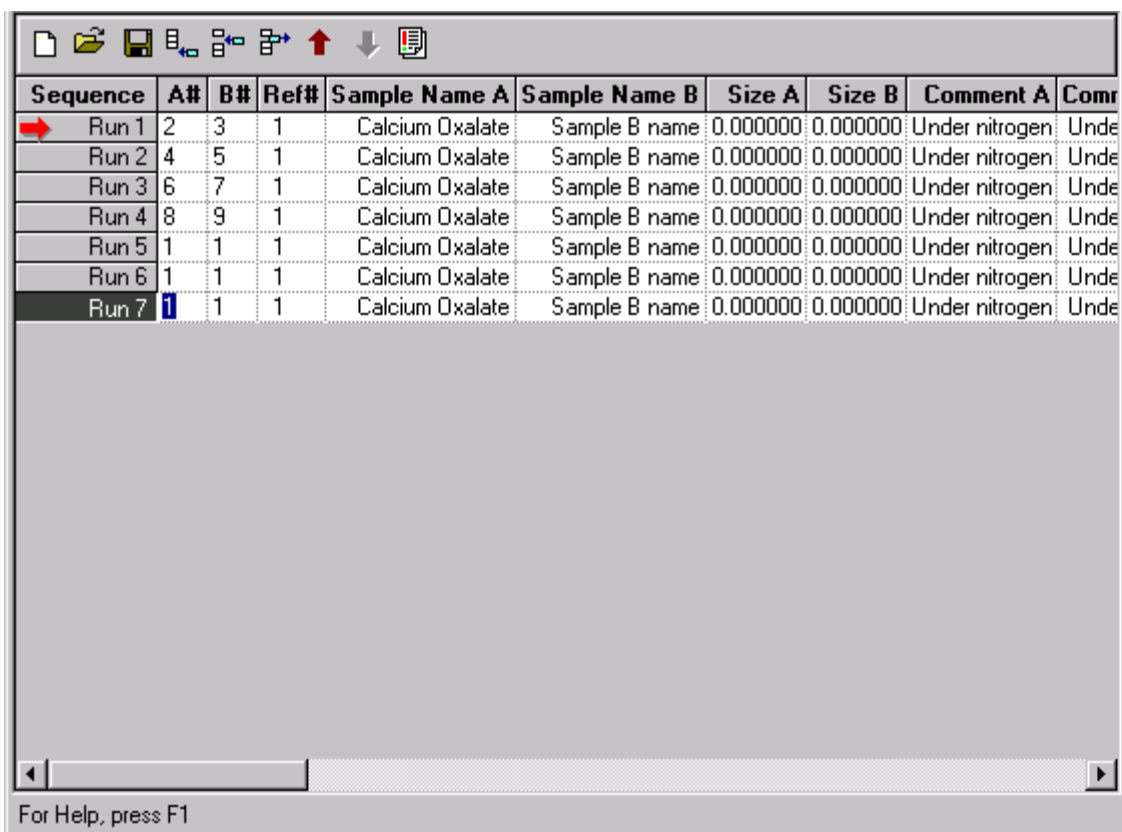
- **Clear Error Entries:** Select this function to erase all of the logged errors.
- **Error Help:** Use Error Help to obtain Help information for the error message currently highlighted in the Error Log.
- **Help:** Use this function to access general online help.

Viewing the Autosampler Log

You can look at a log of the Autosampler's actions and results by selecting **View/Autosampler Log** from the instrument menu.

Using the Table View Options

You can access key run sequence information in a table form by selecting either **View/**
Table View from the instrument menu, by clicking on the  button on the instrument
tool bar, or by pressing F11 on the keyboard. The **Table View Pane** (shown below)
provides an alternative to the **Experiment View Pane** for setting up and editing sample



Sequence	A#	B#	Ref#	Sample Name A	Sample Name B	Size A	Size B	Comment A	Comment B
Run 1	2	3	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 2	4	5	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 3	6	7	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 4	8	9	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 5	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 6	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen
Run 7	1	1	1	Calcium Oxalate	Sample B name	0.000000	0.000000	Under nitrogen	Under nitrogen

For Help, press F1

information for a sequence of runs. In this view, key sample information for each run is displayed and can be changed in a spreadsheet format, except for the Mode.

This view provides the greatest benefit after you have completely set up one run using the **Experiment View** and want to use the same procedure on other samples. You can just change the sample information. See “Copying and Adapting Procedures” in Chapter 3 for details about using the **Table View Pane** to adapt existing procedures for a new experiment.

NOTE: The specifics of the selected procedure cannot be changed in this view (switch to Experiment View to change the procedure).

NOTE: When you make changes in Table View, you must press the TAB key to move out of the field and to save the changes.

Using the Table View Tool Bar

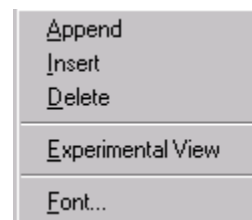
At the top of the **Table View Pane**, just below the instrument status line, is the **Table View** tool bar (shown here). These are the same buttons that are used in the **Sequence Pane**, see page 4-4 “Handling Runs and Sequences” for information on these buttons.



Using the Table View Pop-up Menu

You can make changes to the sequence file contents by right clicking the mouse in the **Table View Pane** to display the menu shown here.

You can use the items in the pop-up menu to make changes to the runs in the sequence, to switch to the **Experimental View**, or to change the font displayed in the table.



Append, **Insert**, and **Delete** work the same as the applicable buttons on the tool bar, see page 4-4 and 4-5 for information.

When you select **Font** from the **Table View** pop-up menu, a standard font selection window is displayed to allow you to choose a font for the table.

Scheduling a Run in Table View

When you have **Table View** opened, you will notice an arrow pointing to one of the run numbers, as shown in the figure on the previous page. This is the scheduled run, which is the experiment that will be run when Start is selected.

To change the scheduled run, position the pointer over the desired run in the sequence column of the table. When the pointer changes to an arrow, click the mouse to schedule that run.

To start the current run, double click on a red (➡) run arrow.

Adjusting Column Widths

The column widths in **Table View** can be edited just like any other Windows® columns. Move the pointer across the column headings until the pointer changes to a two-sided arrow (this occurs when it is positioned at the column dividers). While the two-headed arrow is displayed, hold down the mouse button and drag the column to the desired width, then release the button.

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