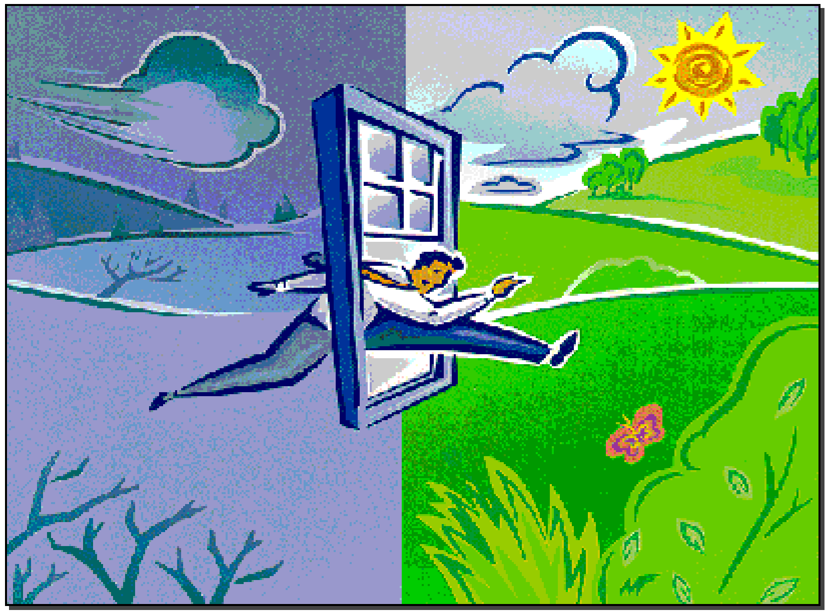
Pyris Software for Windows



Version 3.7

Release Information

File No. 0000-0032
Release: 3.7
Release Date: September 1999

Copyright Information

Trademarks

Notice

The information contained in this document is subject to change without notice.

Perkin Elmer LLC makes no warranty of any kind with regard to this material, including, but not limited to, the implied warranties of merchantability and fitness for a particular purpose. Perkin Elmer LLC shall not be liable for errors contained herein or for incidental consequential damages in connection with the furnishing, performance, or use of this material.

Table of Contents

Chapter 1: Pyris 1 DSC

Chapter 2: DSC 7

Chapter 3: Pyris 6 DSC

Chapter 4: Pyris 6 TGA

Chapter 5: Pyris 1 TGA

Chapter 6: TGA 7

Chapter 7: DMA 7/7e

Chapter 8: TMA 7

Chapter 9: DTA 7

Index

Chapter 1
Pyris 1 DSC

Overview

The Pyris 1 DSC is a power-compensated differential scanning calorimeter. In power compensation DSC, the sample and reference material are each confined to a separate, self-contained calorimeter. In addition to the traditional benefits of the power compensation design, the Pyris 1 DSC includes improved baseline and subambient performance, lower baseline noise, and higher measurement sensitivity. The Pyris 1 DSC is connected directly to the computer containing Pyris Software for Windows which controls the analyzer via temperature control programs. Using Pyris Software for Windows, you can program the Pyris 1 DSC to run from an initial to a final temperature through transitions in the sample material such as melting, glass transitions, solid-state transitions, or crystallization. The temperature range is scanned by changing the temperature at a linear rate in order to study these endothermic and exothermic reactions of samples.

Pyris Software for Windows supports the Pyris 1 DSC autosampler accessory. The autosampler allows testing of up to 48 samples. The autosampler is useful in laboratories that analyze many samples of the same or different types. Combined with the Pyris Player software, the runs are entirely automated. You can create “play lists” that contain multiple methods for analyzing one sample; multiple samples to be analyzed by one method, and many ways to perform postrun data analysis.

Other features of the Pyris 1 DSC are

- Platinum resistance thermometers, not thermocouples, are used for linear temperature measurement.
- ThermalGuard – a unique combination of trapped air, high-tech epoxies, and aluminized coatings that provides superior baseline reproducibility and unequaled day-to-day instrument precision.
- Cryogenic temperatures for subambient operation (as low as –170°C) are achieved with a small, internal liquid nitrogen dewar that rests on a thermal island of epoxy insulating materials. Liquid nitrogen consumption is minimized thus decreasing daily operating costs. The internal dewar is automatically filled from Perkin Elmer’s new CryoFill Liquid Nitrogen Cooling System. The CryoFill control unit ensures a very stable liquid level in the internal LN2 dewar. The liquid level is held to within a few millimeters regardless of the pressure or flow rate from the LN2 supply tank. You fill the internal dewar by connecting it to the LN2 supply tank via the liquid nitrogen transfer line. This line provides a safe connection to the tank while allowing easy separation from the tank during refilling operations.

- Pyris 1 DSC is shipped with a cold finger and a water/ice reservoir already in place. This allows you to begin experiments at temperatures as low as 25°C. It is also shipped with a turbulent chamber for liquid circulation. With the turbulent chamber, convenient operation above ambient is possible.
- The AirShield prevents moist air from settling into the sample holder because a thin curtain of air flows over the cold region. This protective device uses laminar flow techniques to keep the sample holder region dry and frost-free. The air curtain turns on automatically whenever you open the sliding sample holder enclosure cover. The honeycomb-like arrangement of the AirShield operates at low velocities effectively forming the equivalent of multiple air blankets over the cold sample holder surface. This protection is accomplished without the use of a dry box.
- The adjustable control panel allows constant visual contact with the analyzer status and the ability to stop the DSC scan.

The features of the Pyris 1 DSC and autosampler are covered in the following topics:

- **Safety Precautions**
- **Baseline Adjustment**
- **Calibration**
- **Operating Variables and Sample Handling**
- **Subambient Operation**
- **Maintenance**
- **Accessories**
- **Part Numbers**
- **Pyris 1 DSC Autosampler**

Safety Precautions

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

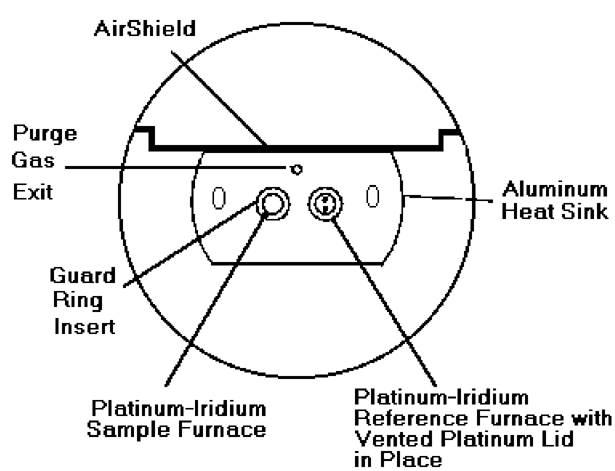
The following precautions must be observed when using the Pyris 1 DSC:

- Never press the Reset button on the computer if the software appears to malfunction. Press the **Ctrl–Alt–Del** keys simultaneously and select Task Manager. End the Pyris Software for Windows task.
- Never remove the outer instrument cover or inside electrical cabinet panels of the Pyris 1 DSC without turning off the power and disconnecting the power cord from the power source.
- Always use helium as the purge gas when liquid nitrogen is used in the subambient reservoir of the Pyris 1 DSC. At other times, use nitrogen, argon, oxygen, or air as the purge gas.
- Do not immerse the purge gas exit line in a liquid since the liquid may be drawn back into the sample holder.
- Always observe the precautions indicated for Intracoolers 1P and 2P cooling devices.

- Always observe the precautions indicated for operating the Pyris 1 DSC with the CryoFill Liquid Nitrogen Cooling System.
- When operating the Pyris 1 DSC in ambient mode, make sure that the Cover Heater is OFF.
- When operating the Pyris 1 DSC in subambient mode, make sure that the Cover Heater is ON.
- Only high-quality purge gases should be used with Pyris 1 DSC. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for the removal of any moisture from the purge gases.
- Always observe the startup or shutdown procedures for Pyris 1 DSC and all related instruments.
- Do **NOT** use aluminum sample pans above 600°C. Since aluminum melts at 660°C, the pans will alloy with and destroy the sample holders. Entering a temperature above 600°C will cause the computer to display a dialog box with a cautionary message. Always make sure that you are not using aluminum pans in either the sample of reference furnace before extending the Pyris 1 DSC temperature range above 600°C.
- Always encapsulate indium, tin, lead, and zinc standards in aluminum or graphite pans. These metals will alloy with gold, copper, or platinum pans.
- Always have the guard ring inserts in place when removing or inserting sample pans or sample holder covers into the Pyris 1 DSC sample holder. This will protect the sample holder leads.
- It is important that nothing fall down into the cavity surrounding the sample holders. If this happens, turn off the power **IMMEDIATELY** and call your Service Representative for instructions.
- Do not force the platinum sample holder covers to fit the sample holders and do not deform the covers in any way. The sample holder covers should fit easily and loosely into the holders. When required, reform the covers with the reforming tool.

Pyris 1 DSC Sample Holder

The Pyris 1 DSC sample holder is on the top of the instrument and sits underneath the sliding sample holder enclosure cover.



Platinum - Iridium Sample Furnace

There are two low-mass platinum – iridium sample cells (furnaces) embedded in a large aluminum heatsink. The left furnace is used for encapsulated sample materials; the right furnace is used for reference materials. For example, the right furnace is typically an empty sample pan and lid or an empty sample container of the type used in the sample cell.

Platinum Sample Holder Lid with Vent

Vented platinum lids are used to cover both the sample and the reference furnaces. These lids should be similarly oriented when they are placed in their respective furnace. The lids should fit snugly into the furnace but should not be forced. If a lid is bent or deformed, use the Sample Holder Cover Reforming Tool (P/N 0319-0030) to reform the lids. Whenever sample lids or sample materials are removed or placed into the instrument, make sure that the guard ring inserts are in place.

Sample Holder Cover Reforming Tool (P/N 0319-0030)

DSC performance is enhanced with sample holder covers that are formed properly and are not degraded by extended usage. This tool allows the analyst to reform the platinum covers to their original dimensions.

AirShield

The AirShield prevents moist air from settling into the sample holder because a thin curtain of air flows over the cold region. If you turned AirShield on by using the adjustable control panel menu, then the AirShield will turn on as soon as the sliding sample holder cover is pushed back and exposes the sample holder. This protective device uses laminar flow techniques to keep the sample holder region dry and frost-free. The honeycomb-like arrangement operates at low velocities effectively forming the equivalent of multiple air blankets over the cold sample holder surface.

Aluminum Heat Sink

The aluminum heat sink provides a constant temperature environment for the sample and reference calorimeters. A coolant must always be used during operation of the Pyris 1 DSC so the aluminum heat sink is maintained at a constant temperature.

Guard Ring Insert

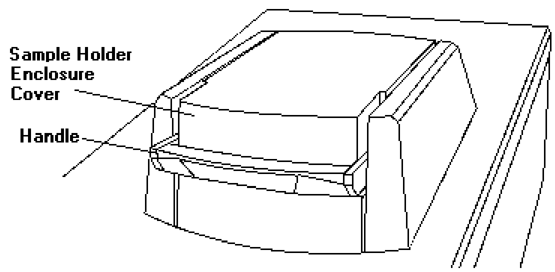
The guard ring insert (P/N N537-3126) for the Pyris 1 DSC is positioned around the outside of the furnace. It prevents samples, pans, and other foreign objects from falling into the cavity around the furnace. Note the slit. The slit must be aligned with the purge gas exit hole. Once you are sure that there is proper alignment, with the end of a tweezers gently press down on the guard ring until it is flush with the top of the heat sink.

Purge Gas Exit

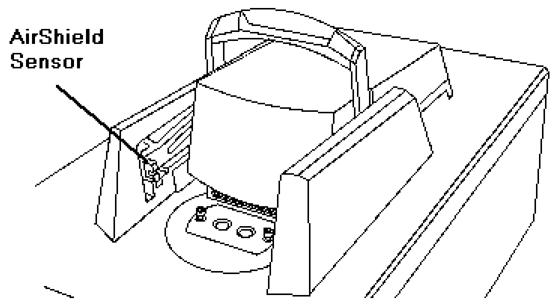
The purge gas exits from the tiny hole on the top of the aluminum heatsink.

Sliding Sample Holder Enclosure Cover

The sliding sample holder enclosure cover resides on top of the analyzer lid and covers the sample holders. It is contained between two rails upon which it slides. It has a handle that, when pushed down, locks the cover in place.



When locked, the AirShield is turned off, as well as the secondary purge gas to the cover. When you lift the handle and push the cover back along the rails, the sensor on the inside of the left rail detects that the sample holders are exposed.



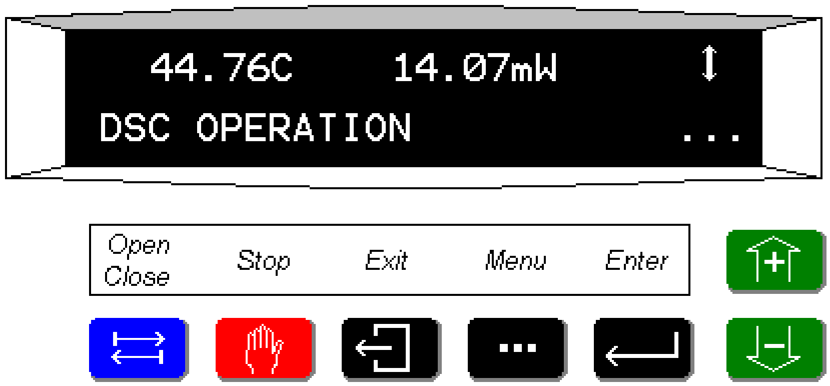
CAUTION: If frost develops inside the sliding sample enclosure cover from opening and closing it, you must clean off the frost using the acid brush provided in the Spares Kit (0401-0109).

It you have turned on the AirShield feature on the adjustable control panel, the AirShield will begin to blow air from the honeycomb outlets across the sample holders. This prevents condensation. The secondary purge gas will also begin to flow through the cover and out the front of the cover.

The cover can be heated if you activate the Turn Cover Heater On/Off feature from the menu on the adjustable control panel.

See **Maintenance** for more information on the Pyris 1 DSC sample holder.

Adjustable Control Panel



The adjustable control panel is divided into two parts:

- **Panel Display**
- **Control Keys**

Panel Display

The adjustable control panel display of the Pyris 1 DSC is a 2-line, 20-character-per-line vacuum fluorescent display.

- The display seen when the instrument is turned on is

  PERKIN–ELMER
  PYRIS 1 DSC

- After boot-up, the nonrun default items are displayed:

  –170.00C –750.00mW
  . . .

- The top line displays real-time temperature in °C and the heat flow or energy signal in mW. The bottom line shows the Menu symbol (**. . .**). This symbol indicates that the Menu button can be pressed to access special menus.

- When a run is in progress, the top line will display the temperature and heat flow values along with the ↕ symbol which represents the scroll arrow buttons:

and the status line changes messages indicating what is going on, e.g.,

–170.00C –750.00mW ↕
01 Heat to –100 . 00C . . .

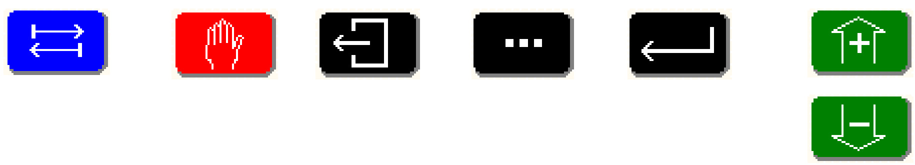
–170.00C –750.00mW ↕
01 Iso for 999 : 59 . . .

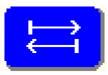
–170.00C –750.00mW ↕
01 5999 : 59 to End . . .

The **Scroll** buttons indicate that you can move between menu items. At the DSC OPERATION display, pressing the **Scroll Down** arrow accesses the **DIAGNOSTICS** item; pressing the **Scroll Up** arrow accesses the **SELECT LANGUAGE** item. The Menu symbol (. . .) indicates that by pressing the **Menu** button (...), you access additional selections.

Control Keys

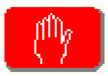
Below the display on the adjustable control panel are the control keys that you can use to interact with the Pyris 1 DSC directly. The major keys are labeled. Labels are provided in six languages for localization.





Open / Close

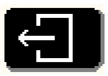
This button is not used.



Stop

When you select this button during a run, the run terminates immediately and a Run Stopped message appears on the bottom line of the display. The final method step number flashes on the default display.

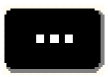
Within a special menu, Stop can be used to terminate a particular action, e.g., stop the furnace cleaning procedure.



Exit

This key is used in four ways:

1. Exit a secondary menu display without performing any action and return to the display from which you came, i.e., the parent user-selected display.
2. Exit a user-selected display without performing any action and return to the default display.
3. Exit a secondary special menu without performing any action and return to the parent special menu.
4. Exit a special menu without performing any action and return to the default display.



Menu

From the default display on the display panel:

–170.00C –750.00mW
. . .

the **Menu** button is used to select special menus. The **Menu** button is functional whenever the Menu symbol (. . .) is displayed on the bottom display line. The action that results from pressing **Menu** depends on what is displayed. With the default items displayed, when you press the **Menu** button, the display changes to

–170.00C –750.00 mW ↕
DSC OPERATION . . .

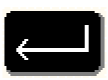
With DSC OPERATION displayed, pressing the **Menu** button here brings up the first option in the DSC OPERATION menu — CLEAN FURNACE. The display appears as follows:

DSC OPERATION ↕
CLEAN FURNACE? . . .

This option is one of six available on the DSC OPERATION menu:

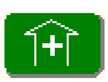
- **Clean Furnace**
- **Adjust Slope**
- **Adjust Balance**
- **Turn CryoFill On/Off**
- **Turn Cover Heater On/Off**
- **Turn AirShield On/Off**

The items Turn AirShield On/Off, Turn Cover Heater On/Off, and Turn CryoFill On/Off are for subambient operation.



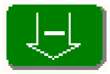
Enter

1. Press **Enter** to move between the default display and user-selected displays.
2. Press **Enter** to move between the default display and special menus.
3. Within secondary menus, **Enter** can be used to accept a condition, start an action, or enter a lower-level submenu.



Scroll Up

1. This button scrolls ahead through a menu's items. For example, at the DSC OPERATION display, pressing the **Scroll Up** button successively scrolls from DSC OPERATION to SELECT LANGUAGE to DIAGNOSTICS and back to DSC OPERATION.
2. In the DSC OPERATION secondary menu, which starts at CLEAN FURNACE, pressing the **Scroll Up** button successively displays the following: Adjust Slope, Adjust Balance, CryoFill LN2 Accy, Cover Heater, AirShield.
3. Within secondary menus, pressing **Scroll Up** increments parameter values.



Scroll Down

1. This button scrolls back through a menu's items. For example, at the DSC OPERATION display, pressing the **Scroll Down** button successively scrolls from DSC OPERATION to DIAGNOSTICS to SELECT LANGUAGE and returns to DSC OPERATION.
2. In the DSC Operation secondary menu, which starts at CLEAN FURNACE, pressing the **Scroll Down** button successively displays the following: AirShield, Cover Heater, CryoFill LN2 Accy, Adjust Balance, Adjust Slope.
3. Within secondary menus, pressing **Scroll Down** decrements parameter values.

Clean Furnace

To automatically clean the Pyris 1 DSC furnace, access the Clean Furnace special menu on the adjustable control panel:

1. At the default display, press the **Menu** button. DSC OPERATION should be displayed on the bottom line.
2. Press the **Menu** button. You see CLEAN FURNACE?
3. To respond Yes, press the **Enter** button. The first of the secondary menu items is displayed: REMOVE SAMPLE PAN.
4. To scroll through the secondary items of the Clean Furnace menu, perform the actions described at each prompt and press the **Enter** button at each new prompt. After pressing the **Enter** button at the REMOVE SAMPLE PAN prompt, the following prompts are seen:

   REMOVE REF PAN ↵
   REPLACE PT LIDS ↵
   READY TO CLEAN? ↵

5. After pressing the **Enter** button at the last prompt, the status “CLEANING” and the time are seen. During the cleaning cycle, the furnace is heated to 600°C in air to combust contaminants present. The furnace is then cooled to the load temperature. The only action you can take while the furnace is being cleaned is terminating the process by pressing the **Stop** button.
6. At the end of the cleaning process, or after pressing **Stop**, the status becomes “CLEAN STOPPED.”
   a. Either press Exit to return to the default display or
   b. Press Enter to go back to the parent menu, CLEAN FURNACE?

Adjust Slope

NOTE: You must be in the DSC Operation... mode with Adjust Slope displayed before you can make adjustments.

You can access the Adjust Slope selection on the Specialty menu one of three ways:

1. If you are at the default display,
   a. press the **Menu** button to obtain the DSC OPERATION display,
   b. press the **Menu** button to display CLEAN FURNACE?,
   c. press the **Scroll Down** button five times or the **Scroll Up** button once.

2. If you have just performed the Clean Furnace procedure, you should see CLEAN FURNACE? on the display. Press the **Scroll Up** button to go to the ADJUST SLOPE display.
3. If you have just performed ADJUST BALANCE, press the **Scroll Down** button to go to the ADJUST SLOPE display.

With ADJUST SLOPE on the display, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go back to the CLEAN FURNACE? display.
3. Press the **Scroll Up** button to go to the ADJUST BALANCE screen.
4. Press the **Menu** button. The display will now show “ADJUST SLOPE” on the top line and the value on the second line.
    a. Press the **Scroll Down** button to decrease the Slope value or the **Scroll Up** button to increase the value.
    b. Return to the default display by pressing the **Exit** button.
    c. Press the **Enter** button to accept the displayed value and return to the parent display.

Adjust Balance

> **NOTE:** You must be in the DSC Operation... mode with Adjust Balance displayed before you can make adjustments.

You can access the Adjust Balance selection on the Specialty menu one of three ways:

1. If you are at the default display,
    a. press the Menu button to obtain the DSC OPERATION display,
    b. press the Menu button to display CLEAN FURNACE?,
    c. press the Scroll Down button four times or the Scroll Up button twice.
2. If you have just performed the Adjust Slope procedure, you should see Adjust Slope on the display. Press the **Scroll Up** button to go to the Adjust Balance display.
3. If you have just turned the CryoFill either on or off, you should see CryoFill LN2 ACCY on the display. Press the **Scroll Down** button to go to the Adjust Balance display.

With Adjust Balance on the display, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go back to the Adjust Slope display.
3. Press the **Scroll Up** button to go to the CryoFill LN2 ACCY screen.
4. Press the **Menu** button The display will now show “Adjust Balance” on the top line and “Coarse” on the second line.
    a. Press the **Exit** button to return to the default display.
    b. Press the **Menu** button to obtain the “Coarse Balance” display. The current Coarse Balance value is seen on the second line.
        i. Press the **Exit** button to return to the default display.
        ii. Adjust the Coarse Balance value to 0 by pressing the **Scroll Up** or the **Scroll Down** button.

iii. Press the **Enter** button to accept the Coarse Balance value and return to the parent display.

c. Press the Scroll Down button to obtain the "Adjust Balance Fine" display.

i. Press the **Exit** button to return to the default display.

ii. Press **Scroll Up** or **Scroll Down** to return to the "Adjust Balance Coarse" display.

iii. Press the **Menu** button to obtain the "Fine Balance" display. The current Fine Balance value is seen on the second line.

1. Press the **Exit** button to return to the default display.
2. Adjust the Fine Balance value to 50 by pressing the **Scroll Up** or the **Scroll Down** button.
3. Press the **Enter** button to accept the Fine Balance value and return to the parent display.

d. Press the **Scroll Up** button to return to the Adjust Balance display.

After adjusting the balance, perform a run without any sample in place. The curve should be displayed with Heat Flow as the Y axis and Temperature as the X axis. Repeat adjustment of the Coarse and the Fine balances until the curve is horizontal.

Turn CryoFill On/Off

You can access the CryoFill LN2 ACCY selection on the Specialty menu one of three ways:

1. If you are at the default display,

   a. press the Menu button to obtain the DSC OPERATION display,

   b. press the Menu button to display CLEAN FURNACE?,

   c. press the Scroll Downbutton three times or the Scroll Up button three times.

2. If you have just performed the Adjust Balance procedure, you should see ADJUST BALANCE on the display. Press the **Scroll Up** button to go to the CryoFill LN2 ACCY display.

3. If you have just performed the Cover Heater procedure, you should see COVER HEATER on the display. Press the **Scroll Down** button to go to the CryoFill LN2 ACCY display.

With CryoFill LN2 ACCY on the display, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go back to the ADJUST BALANCE display.
3. Press the **Scroll Up** button to return to the COVER HEATER screen.
4. Press the **Menu** button This brings up the prompt "TURN CryoFill OFF?" if it is on or "TURN CryoFill ON?" if it is off.

   a. Press the **Enter** button to either of these prompts to respond Yes and also return to the parent display.

   b. Move on from the CryoFill LN2 ACCY item by pressing **Exit**, **Scroll Down,** or **Scroll Up**.

Turn Cover Heater On/Off

Caution: Do NOT turn the cover heater on when operating the Pyris 1 DSC in ambient mode. When operating in subambient mode, make sure that the cover heater is on.

You can access the Cover Heater selection on the Specialty menu one of three ways:

1. If you are at the default display,
   a. press the Menu button to obtain the DSC OPERATION display,
   b. press the Menu button to display CLEAN FURNACE?,
   c. press the Scroll Down button twice or the Scroll Up button four times.
2. If you have just performed the AirShield procedure, you should see AirShield on the display. Press the **Scroll Down** button to go to the COVER HEATER display.
3. If you have just performed the CryoFill procedure, you should see CryoFill LN2 ACCY on the display. Press the **Scroll Up** button to go to the COVER HEATER display.

With COVER HEATER on the display, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go back to the CryoFill LN2 ACCY display.
3. Press the **Scroll Up** button to go to the AirShield screen.
4. Press the **Menu** button. This brings up the prompt “TURN HEATER OFF?” if it is on or “TURN HEATER ON?” if it is off.
   a. Press the **Enter** button to either of these prompts to respond Yes and also return to the parent display.
   b. Move on from the Cover Heater item by pressing **Exit**, **Scroll Down**, or **Scroll Up**.

Turn AirShield On/Off

Caution: The AirShield should be ON whenever the Pyris 1 DSC is operating below ambient.

You can access the AirShield selection on the Specialty menu one of three ways:

1. If you are at the default display,
   a. press the Menu button to obtain the DSC OPERATION display,
   b. press the Menu button to display CLEAN FURNACE?,
   c. press the Scroll Down button to go to the AirShield display.
2. If you have just performed the Clean Furnace procedure, you should see CLEAN FURNACE? on the display. Press the **Scroll Down** button to go to the AirShield display.
3. If you have just performed the Cover Heater procedure, you should see COVER HEATER on the display. Press the **Scroll Up** button to go to the AirShield display.

With AirShield displayed on the bottom line, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go back to the COVER HEATER display.
3. Press the **Scroll Up** button to return to the CLEAN FURNACE? screen.
4. Press the **Menu** button. This brings up the prompt “TURN AirShield OFF?” if it is on or “TURN AirShield ON?” if it is off.
   a. Press the **Enter** button at either of these prompts to respond Yes and also return to the parent display.

b. Move on from the AirShield item by pressing **Exit**, **Scroll Down,** or **Scroll Up**.

Select Language

To access the SELECT LANGUAGE option on the Pyris 1 DSC menu,

1. Display the default display:

–170.00C –750.00mW
. . .

2. Press the **Menu** button to display

–170.00C –750.00mW ↕
DSC OPERATION . . .

3. Press the **Scroll Up** button to display the following:

–170.00C –750.00mW ↕
SELECT LANGUAGE . . .

With SELECT LANGUAGE on the display, you can do one of the following:

1. Press the **Exit** button to return to the default display.
2. Press the **Scroll Down** button to go to the CLEAN FURNACE? display.
3. Press the **Scroll Up** button to go to Diagnostics.
4. Press the **Menu** button to display the first of the language selections: English. At this screen, you can do one of the following:
   a. Press **Scroll Down** to go to the next language.
   b. Press **Scroll Up** to go to the preceding language.
   c. Return to the default display by pressing **Exit**.
   d. Press **Enter** to change the language and return to the parent display.

There are six languages to choose from:

- English
- French
- German
- Italian
- Japanese
- Spanish

Diagnostics

This item in the DSC OPERATION menu is accessed from the screen

47.27C 13.53mW ↕
DSC OPERATION . . .

by pressing the **Scroll Down** button. You can also access it by first pressing the **Scroll Up** button at this display to display SELECT LANGUAGE and then select **Scroll Up** again. The DIAGNOSTICS display appears:

47.27C 13.53m W↕
DIAGNOSTICS . . .

Press the **Menu** button to display:

FIRMWARE VERSION
N218-8888 REV 1_2 ↵

Press the **Enter** button to return to the DIAGNOSTICS display.

Baseline Adjustment

Optimizing the baseline of the Pyris 1 DSC involves adjusting the Balance and Slope controls.

NOTE: If the Balance control is adjusted, you may have to recalibrate the temperature axis. Thus, always optimize the baseline before the instrument is calibrated. Ensure that the analyzer has stabilized before optimizing the baseline.

To optimize the Pyris 1 DSC baseline, perform the following three procedures:

- **Manual Baseline Optimization**
- **Baseline Curvature Correction**
- **Baseline Slope Adjustment**

Manual Baseline Optimization

1. Select the temperature range over which the baseline is to be optimized. It is not necessary to optimize the baseline over the entire range if most of your experiments will be performed over only a smaller portion of the range.

   For example, if your work involves the analysis of the melting profiles of thermoplastic materials, you will generally be working in the range of 50°C – 250°C. Thus, you need to optimize the baseline between only those temperatures.

2. Select the coolant or cooling accessory that will be used for your experiment. An ice water bath, liquid nitrogen, Intracoolers, or a CryoFill LN2 Cooling System can be used. Allow enough time for the system to equilibrate after selecting the desired coolant or cooling accessory.

3. Set the purge gas to a flow rate of approximately 20 – 30 cc/min (20 – 30 psi at the regulator).

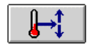
4. The Balance and Slope controls are set at the factory; if the settings have been changed, set them to a reading of 50 before proceeding.

   See **Adjust Balance** and **Adjust Slope** on how to set Balance and Slope to 50.

5. Make sure that the sample and reference holders are empty. Place the vented platinum covers into the sample and reference holders. Close and secure the sample holder enclosure cover.

6. In the Pyris software, click on the **Pyris 1 DSC** button in the Pyris Manager to load the Instrument Application. The Pyris 1 DSC Control Panel and the Method Editor are displayed on the screen.

7. From the Method Editor, select **File** followed by **Open Method**. Find the pyris\methods directory on your system. A list of method filenames is displayed in the Open Method dialog box.

8. Double click on the filename **baseline.dcm.** This method is displayed on the screen. It can be used for obtaining routine baselines. If necessary, change any of the values to accommodate your situation.
9. Make sure that the Sample Weight value is 0.00.
10. Check the current Go To Temperature value in the Pyris 1 DSC Control Panel. Make sure it is set to 50.00°C. If it is not, then click inside the Go To Temperature box and type 50°C. Click on the **Go To Temperature** button  in the Control Panel. The analyzer will now program to 50°C and hold there. The Y value signal will stabilize.
11. Click on the **Start** button  located in the Control Panel to start the run. The message **Heating to XXX.xx °C** is displayed in the analyzer's adjustable control panel and the analyzer starts heating to that temperature.

Baseline Curvature Correction

After running the baseline, you now must determine the amount of curvature correction that must be made to the Balance control by performing the following steps:

1. Recall the data file created when you performed manual baseline optimization if it is not displayed on in the Data Analysis window on the screen. From the **Calc** menu select **Peak Area**.
2. Enter a Left Limit value and a Right Limit value in the dialog box, selecting points near the beginning and the end of the curve. Check the **Include Peak Height** option box.
3. Click on the **Calculate** button. A peak calculation is performed and displayed in the Data Analysis page. Note the peak height value.
4. Invert the peak height value (i.e., if the height is 11.0 mW, make it –11.0 mW) and add this value to the current value displayed in the Balance Control menu.
5. Adjust the setting of the Balance control to the newly calculated value by pressing the appropriate Scroll button on the adjustable control panel until the value calculated above is displayed.

NOTE: Changes made to the Balance control setting affect the temperature calibration. If you change the setting, check the temperature calibration.

6. Select and then perform another baseline run as described above. Most of the baseline curvature should be gone.

Baseline Slope Adjustment

After removing the baseline curvature, baseline slope can be removed. To correct the slope, perform the following steps:

1. Display the Slope menu on the adjustable control panel.
2. Set up and start a run using the **baseline.dcm** method. This method will be running while you are adjusting the Slope control.
3. Slowly adjust the Slope value on the adjustable control panel by pressing the **Scroll Up** or the **Scroll Down** button until the baseline is straight and parallel with the X axis.
4. When there is no more slope in the display, press the **Enter** button on the adjustable control panel to accept the new slope setting.

The baseline optimization and adjustment procedures are now complete.

Operating Variables and Sample Handling

In differential scanning calorimetry, the object of an experiment is to record the heat flow to or from a sample over a linearly changing temperature range or at a single isothermal temperature. The goal is to obtain accurate data in a minimum period of time. The following topics illustrate some of the techniques that can be used to obtain the most accurate DSC data in a minimum amount of time.

- **Sample Preparation**
- **Sample Pans**
- **Sample Encapsulation**
- **Sample Size**
- **Sample Atmosphere**
- **Temperature Range**
- **Scanning Rate**
- **Sample Loading**
- **Specific Heat Analysis**

Sample Preparation

The differential scanning calorimeter can analyze solid or liquid samples. Solid samples can be in film, powder, crystal, or granular form. Although quantitative accuracy will remain the same regardless of sample shape, the qualitative appearance of a run may be affected by the sample configuration. Proper sample preparation that maximizes the contact surface between the pan and the sample will reduce the resistance of the sample to heat flow through the DSC temperature sensors and will result in maximum peak sharpness and resolution.

The best sample shapes for optimum performance are thin disks or films or fine granules spread in a thin layer on the bottom of the pan. Materials such as polymer films can be conveniently prepared by cutting out sections of the film with a standard paper punch or cork borer. Solid materials can be sliced with a razor or knife.

See topics **Prepare Solid Sample**, **Prepare Liquid Sample**, and **Prepare Powdered Sample** in Pyris Multimedia Presentations Help for more information.

Sample Pans

After preparing the sample, it must be placed into a sample pan which will then be placed in the sample holder for analysis. The following types of sample pans are available:

- **Standard Sample Pans**
- **Specialty Sample Pans**
- **Volatile Sample Pans**
- **Large-Volume O-Ring-Sealed Stainless-Steel Pans**
- **High-Pressure Capsules**
- **Robotic System Sample Pans**

DSC Sample Pan Table

This table lists sample pans available for the Pyris 1 DSC for most thermal analysis applications. Associated with each pan, where applicable, is the crimper to be used with that particular pan.

Sample Pan	Operating Range	Vol. (µL)	Pressure	Crimper/Sealer	Qty.	Part No.
Std. aluminum pans *	–170°C to 600°C		1 atm, 100 kPa	0219-0048	400	0219-0041
Std. gold pans *	–170°C to 725°C		1 atm, 100 kPa	0219-0048	10	0219-0040
Std. copper pans*	–170°C to 725°C		1 atm, 100 kPa	0219-0048	200	0319-0026
Alumina pans	–170°C to 725°C		1 atm, 100 kPa	Not required	6	N519-0180
Platinum pans	–170°C to 725°C		1 atm, 100 kPa	Not required	4	0319-0024
Graphite pans	–170°C to 725°C		1 atm, 100 kPa	Not required	4	0319-0026
Volatile aluminum pans *	–170°C to 600°C	20	2 atm, 200 kPa	0219-0061	400	0219-0062
Volatile aluminum pans (pierced)*	–170°C to 600°C	20	1 atm, 100 kPa	0219-0061	100	N519-0788
Volatile gold pans*	–170°C to 725°C	20	2 atm, 200 kPa	0219-0061	10	0219-0080
Stainless-steel/O-ring*	–40°C to 300°C	60	24 atm, 2400 kPa	0990-8467	20 1000	0319-1047 0319-0029
Original high-pressure stainless steel	–170°C to 400°C	30	150 atm, 15,000 kPa	Not available		0419-1761
Original high-pressure gold-plated	–170°C to 400°C	30	150 atm, 15,000 kPa	Not available		0419-1760
High-pressure stainless steel	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2901
High-pressure gold-plated	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2902
High-pressure titanium	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2903
DPA 7	–170°C to 600°C	12.5	1 atm, 100 kPa	Not Required	100	B018-6859
Robotic aluminum	–170°C to 600°C	10	1 atm, 100 kPa	B013-9005	400	B014-3015
Robotic aluminum	–170°C to 600°C	30	1 atm, 100 kPa	B013-9005	400	B014-3016
Robotic aluminum	–170°C to 600°C	50	1 atm, 100 kPa	B013-9005	400	B014-3017

Sample Pan	Operating Range	Vol. (µL)	Pressure	Crimper/Sealer	Qty.	Part No.
Robotic aluminum	–170°C to 600°C	40	1 atm, 100 kPa	B013-9005	400	B014-3021
Robotic aluminum (vented)	–170°C to 600°C	30	1 atm, 100 kPa	B013-9005	400	B014-3018
Robotic aluminum (vented)	–170°C to 600°C	50	1 atm, 100 kPa	B013-9005	400	B014-3019
Robotic gold	–170°C to 725°C	50	1 atm, 100 kPa	B013-9005	10	B014-3024
Robotic aluminum	–170°C to 600°C	10	2 atm, 200 kPa	B013-9005	400	B016-9319
Robotic aluminum	–170°C to 600°C	25	1 atm, 100 kPa	B013-9005	400	B017-4937
Robotic aluminum	–170°C to 600°C	30	2 atm, 200 kPa	B013-9005	400	B014-3020
Robotic aluminum	–170°C to 600°C	50	2 atm, 200 kPa	B013-9005	400	B016-9320
Aluminum*	–170°C to 600°C	12.5	2 atm, 200 kPa	B013-9005	400	B016-9321

* These pans may also be crimped using the Universal Crimper Press with the appropriate sealing insert.

Standard Sample Pans

Standard sample pans come in three different types:

- **Aluminum Sample Pans**
- **Copper Sample Pans**
- **Gold Sample Pans**

In the standard sample pan, the sample is completely enclosed in a highly conductive capsule and distributed in a thin layer such that the internal resistance in the sample itself is very small. The sample pan material and the sample holder design have very small thermal resistance. This contributes to superior thermal conductivity and temperature control between the sample and the sample pan. Because of the high thermal conductivity of aluminum, copper, and gold, heat transfer is maximized. This means that the sample size can be minimized. Because of their high temperature capabilities, platinum and gold can be used to 725°C.

CAUTION: Never exceed 600°C when using aluminum sample pans. Since aluminum melts at 660°C, an aluminum sample pan will alloy with and destroy the sample holder.

The metal standards (indium, tin, lead, and zinc) must be encapsulated in aluminum pans because they will alloy with gold, copper, or platinum pans.

Aluminum Sample Pans (P/N 0219-0041)

Aluminum sample pans are used with nonvolatile solid samples such as plastics and polymers. They are used routinely for applications such as polymer melting, crystallization, glass transitions of polymers, thermoplastics, and thermosets. The pans are crimped but not hermetically sealed. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Gold Sample Pans (P/N 0219-0042)

Gold sample pans are used for any nonmetal, i.e., ceramics, coal, soils, or minerals. They can also be used for any material that reacts with aluminum or which must be heated over 600°C. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Copper Sample Pans (P/N 0319-0026)

Copper sample pans can be used for samples that have transitions that occur above the temperature range of the aluminum pans. Typical applications for the use of copper sample pans include antioxidant testing, thermoset and thermoplastic glass transition, and melting point determinations. These pans are crimped with the Standard Sample Pan Crimper Press (P/N 0219-0048) and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

See the topics **Crimp the Sample Pan by Hand** or **Using the Standard Sample Pan Crimper** in Pyris Multimedia Presentations Help.

Specialty Sample Pans

There are three specialty sample pans for use with a DSC:

Alumina Sample Pans (P/N N519-0180)

Alumina sample pans are used for oxidation studies of metals, minerals, ceramics, clays, and soils. They can also be used for melting and eutectic diagrams in the metals industry. The associated covers are pierced. A crimper is not required.

Graphite Sample Pans (P/N 0319-0025)

Graphite sample pans should be used in an inert atmosphere. They are used primarily with metals, but not for oxidation studies. They have extremely good heat transfer properties that yield sharp transition curves. A crimper is not required.

Platinum Sample Pans (P/N 0319-0024)

Use platinum sample pans and covers when gold and graphite pans and covers are not suitable. These pans are used to analyze mineral, soil, and ceramic samples. They are also used to allow a chemical reaction where platinum acts as a catalyst. Platinum sample pans are easy to clean and are reusable. A crimper is not required.

Volatile Sample Pans

Volatile sample pans are used with volatile solid or liquid samples which exert significant vapor pressure at the temperature of interest. They are available in both aluminum (P/N 0219-0062) and gold (P/N 0219-0080). The benefits of the volatile sample pan are as follows:

- They can withstand an internal pressure of 2 atm (30 psi).
- Aqueous solutions can be scanned up to and through 100°C to observe solute behavior.

- The heats of fusion of materials that sublime can be determined can be determined accurately.
- The effect of an enclosed atmosphere on thermal behavior of a sample can be observed.
- Capsules have an effective volume of 20 µL.
- Gold pans are available for samples requiring temperatures above 600°C.
- Covers with 50-µL-pinhole lids are available [Vapor Pressure Sample Pan Kit (P/N N519-0788)] for measurement of boiling points, heats of vaporization, and sublimation temperatures.

The major applications for this type of pan are purity analyses (i.e., pharmaceuticals, melting of lipids or liquid crystals) and phase transitions, heat of vaporization, and boiling points.

See the topic on how to **Seal a Volatile Sample Pan Using a Volatile Sample Sealer** in Pyris Multimedia Presentations Help.

Large-Volume O-Ring-Sealed Stainless-Steel Pans

Large-volume O-ring-sealed stainless-steel sample pans (P/N 0319-0218) contain the sample in a sealed environment throughout an experiment. The Viton O-ring allows formation of a seal which suppresses the vaporization of a solvent or contains a volatile reaction product, thereby eliminating the interfering effects of the heat of vaporization. The benefits of this type of sample pan are

- sealed capsules prevent any mass loss
- capsules can withstand an internal pressure of 24 atm
- high internal pressure capability allows water samples to be heated higher than 100°C
- capacity of 60 µL
- large sample capacity yields higher sensitivity
- operating range of –40°C to 300°C unless otherwise limited by sample vapor pressure

The large-volume O-ring-sealed stainless-steel sample pan is used in the study of aqueous biological solutions (such as protein in water) where the dilution requires large sample sizes and water vaporization must be suppressed. They are also used in epoxy and phenolic curing and vulcanization of rubber where the loss of volatiles can otherwise make interpretation difficult.

See the topic on how to **Seal a Large-Volume O-Ring Stainless-Steel Sample Pan** in Pyris Multimedia Presentations Help.

NOTE: A large-volume O-ring-sealed stainless-steel sample pan can be sealed with a **universal crimper press** with optional sealing insert (P/N B050-5340) or with a **quick press** with the quick press spacer (P/N 0319-1047).

High-Pressure Capsules

For DSC runs where high pressure is expected in the capsule, we recommend that you use reusable high-pressure capsules. The reusable high-pressure capsules and sealer are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used. The capsules are safe to handle because of a bursting disk on the cover of the capsule that allows the sample to escape if the pressure should exceed 150 atm.

There are three high-pressure capsules available:

Stainless-Steel High-Pressure Capsules (P/N B018-2901)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number contains 5 capsules and 20 copper seals.

Gold-Plated High-Pressure Capsules (P/N B018-2902)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number consists of 5 capsules and 20 gold-plated copper seals.

Titanium High-Pressure Capsules (P/N B018-2903)

For use in DSC runs where high-pressure is expected in the capsule, e.g., explosive materials. Part number consists of 5 capsules and 20 titanium seals.

Some of the benefits of using the high-pressure capsule are

- permits suppression of the endothermic signal resulting from the volatilization of sample material or from the volatilization or decomposition of reaction by-products
- permits the study of explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used
- availability in stainless steel, gold-plated steel, and titanium
- can withstand an internal pressure of 150 atm maximum
- broad temperature range, subambient to 400°C
- reusable after sealing
- used in hazard testing

The high-pressure capsules are sealed with the high-pressure capsule sealing tool (P/N B018-2864).

See the topic on how to **Use the High-Pressure Capsule Sealing Tool** in Pyris Multimedia Presentations Help.

Robotic System Sample Pans

The robotic system sample pans are used to prepare sample materials for the Pyris 1 DSC autosampler. Pans are available in sealed or vented versions. These pans can also be used for nonautosampler systems. The Universal Crimper Press (P/N B013-9005) provides high-quality sample crimping for the robotic system sample pans.

Below is a list of robotic system pans available:

1-bar (100 kPa) maximum internal pressure

Part No.	Capacity	Thickness	Description
B014-3015	10 µL	0.1 mm	sealed aluminum pans (400)
B014-3016	30 µL	0.1 mm	sealed aluminum pans (400)
B014-3017	50 µL	0.1 mm	sealed aluminum pans (400)
B014-3018	30 µL	0.1 mm	vented aluminum pans (400)
B014-3019	50 µL	0.1 mm	vented aluminum pans (400)
B014-3024	50 µL	0.1 mm	sealed gold pans (10)

2 bar (200 kPa) maximum internal pressure

B014-3020	25 µL	0.15 mm	sealed aluminum pans (400)
B014-3021	50 µL	0.15 mm	sealed aluminum pans (400)

See the topic on how to **Crimp the Robotic System Sample Pan Using the Universal Crimper Press** in Pyris Multimedia Presentations Help.

Robotic System Sample Pan Covers

B014-3003	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (400)
B014-3004	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (400)
B014-3040	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (2000 covers)
B014-3050	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (800)

Starter Kits

B014-6340	Aluminum Sample Pan Starter Kit. Contains 50 of each type of robotic system aluminum sample pan and 350 sample pan covers.
B014-3030	Aluminum Sample Pan Starter Kit. Contains 400 of each type of robotic system aluminum sample pan and 2000 sample pan covers.

Pan and Cover Kit (2 atm, 200 kPa)

B016-9320	Aluminum sample pans and covers, 30-µL internal volume (400 pans and covers)
B016-9321	Aluminum sample pans and covers, 50-µL internal volume (400 pans and covers)
B700-1015	Aluminum sample pan cover with centered 2.5-mm hole. Designed for OIT measurements and for experiments that require an open sample pan for maximum exposure of the sample to the gaseous environment. Can be used with 30- or 50-µL Robotic System aluminum sample pan bottoms (400 lids)
B700-1014	Aluminum sample pan cover with centered 0.05-mm hole. Designed for vapor pressure and boiling point measurements. Can be used with 10-, 30-, or 50-µL Robotic System aluminum sample pans (400 lids)

Sample Encapsulation

The standard aluminum sample pans and covers provided with the DSC are suitable for most thermal analysis applications. The most common method of sample encapsulation is crimping the sample pan cover in place. The normal procedure yields a tightly, but not hermetically, sealed pan. When maximum contact of the sample with the atmosphere is necessary, perforate the sample pan cover with tweezers before crimping or use a cover punched from a 60/100 mesh screen.

For materials that emit volatile byproducts or need to be hermetically sealed, additional sample pans are available.

See the topic **How to Encapsulate Samples** in Pyris Multimedia Presentations Help.

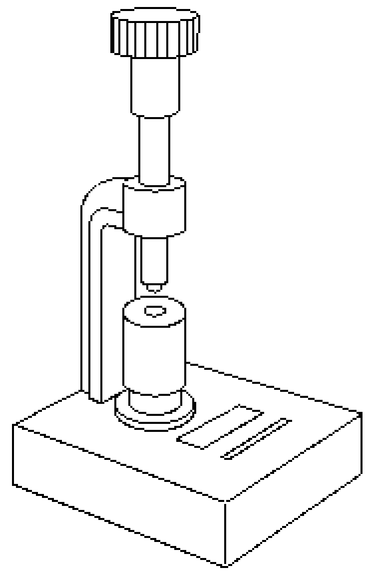
Crimpers

The following table lists the crimpers available for the sample pans used with a DSC for most thermal analysis applications.

Crimper	Use	Part No.
Standard sample pan crimper press	Crimps covers on Standard Sample Pans of aluminum, gold, and copper. Has a replaceable crimper head	0219-0048
Volatile sample sealer	Used with Volatile Sample Pans	0219-0061
High-pressure capsule sealing tool	Used with High-Pressure Capsules	B018-2864
Universal crimper press	Used with the Large-Volume O-Ring-Sealed Stainless-Steel Pans and Volatile Sample Pans. This is accomplished by using appropriate sealing insert.	B013-9005
Quick Press	Used with Large-Volume O-Ring-Sealed Stainless-Steel Pans	0990-8467

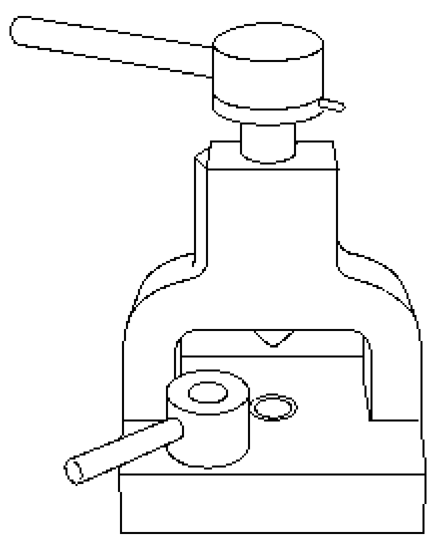
Standard Sample Pan Crimper Press

The Standard Sample Pan Crimper Press (P/N 0219-0048) is used to crimp covers on standard sample pans of aluminum, gold, and copper for use with the Pyris 1 DSC. Samples are crimped in pans but are not hermetically sealed. The design incorporates a replaceable crimper head.



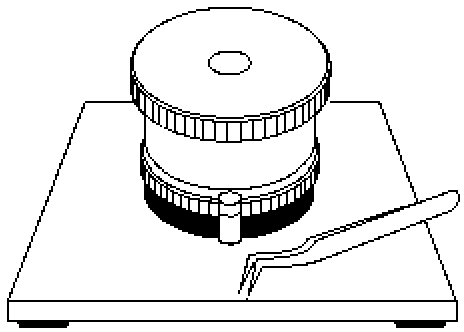
Volatile Sample Sealer

The Volatile Sample Sealer (P/N 0219-0061) is used to seal volatile sample pans. The pans and sealer are used with volatile solid or liquid samples that exert significant vapor pressure in the temperature range of interest. For example, aqueous solutions can be scanned up to and through 100°C to observe solute behavior. The heats of fusion of materials which sublime (e.g., camphor) can be determined accurately using these sample pans. Additionally, the effect of an enclosed atmosphere (e.g., water vapor evolved in dehydration below 100°C) on thermal behavior of a sample can be observed.



High-Pressure Capsule Sealing Tool

The high-pressure capsule sealing tool (P/N B018-2864) is used to seal the reusable high-pressure capsules. Those capsules are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used.



The capsules that can be sealed by this tool are

- **stainless-steel high-pressure capsules**
- **gold-plated stainless-steel high-pressure capsules**
- **titanium high-pressure capsules**

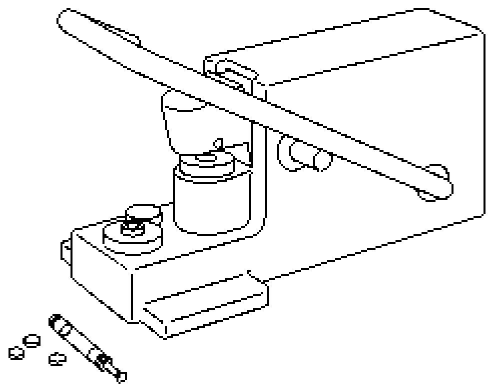
Vapor Pressure Sample Pan Kit (P/N N519-0788)

This kit includes 100 volatile aluminum pans and 100 pierced covers. A 50-µm-diameter hole is centered in the cover. Use this kit for more reproducible measurements of boiling points, heats of vaporization, and sublimation temperatures. Vapor pressure studies can be conducted when using these pans and covers with the high-pressure DSC.

Universal Crimper Press

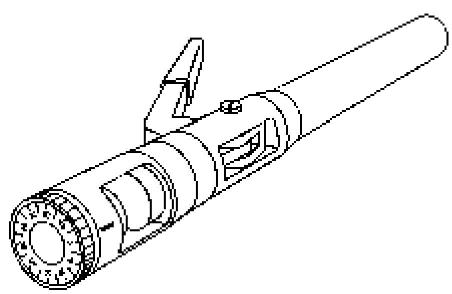
The Universal Crimper Press (P/N B013-9005) is used for sealing all of the Robotic System sample pan types as well as most of the other Perkin Elmer DSC sample pans when used with the appropriate optional sealing insert. Other pans that can be crimped by a universal crimper press are

Part No.	DSC Sample Pan Type
B050-8921	Standard Sample Pans
B050-5340	Large-Volume O-Ring Sealed Stainless-Steel Sample Pans
B014-4637	Volatile Sample Pans
B013-9033	Robotic System Sample Pans (included with the Universal Crimper Press)
B017-4929	12.5-L, 2-atm aluminum sample pans



Quick Press

The Quick Press (P/N 0990-8467) is used to crimp large-volume O-ring sealed stainless-steel sample pans (P/N 0319-0218) which are used with samples that vaporize or contain a volatile reaction product in the temperature range of interest. Application areas include the study of dilute aqueous solutions or curing reactions at temperatures below 300°C (e.g., phenolic or epoxy resins).



Sample Size

The quantity of sample that can be analyzed by the DSC is limited only by the volume of the sample pan that you use. However, the sample size in conjunction with the scanning rate and the sensitivity (Y range) will affect the quality of the results.

With the DSC, running a large sample at a fast scanning rate will improve the usable sensitivity. Running a small sample at a slow scanning rate will improve the resulting peak resolution. Most samples run on the DSC analyzer will be in the 0.5–30-mg range.

NOTE: Use small samples of 1 mg or less for decomposition studies and other analyses in which the sample may tend to contaminate the sample holders or in which the behavior of the sample has not previously been investigated. Also, use a flow through cover in such analyses.

Sample Atmosphere

CAUTION: Always use helium as the purge gas when liquid nitrogen is used for subambient operation. Never leave the purge gas exit line immersed in a liquid.

You can control the atmosphere in which the encapsulated sample is run by using a purge gas to displace or introduce reactive gases into the sample furnaces. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another, always check the temperature and energy calibration. Flow rates of 20 – 30 cc/min of purge gas are recommended. These flow rates will be realized by inlet purge gas rates of 20 – 30 psi.

Argon or nitrogen of 99.9% minimum purity is recommended for purging the sample holders when operating at ambient temperatures and above. **The gas must be dry.** A size 1A cylinder equipped with a suitable regulator is recommended. The regulator should be equipped with a shutoff valve at the outlet. The shutoff valve should have a 1/4-in. NPT male thread on the outlet side for connection to the DSC's purge gas line. A purge gas flow restrictor should not be placed in the line as this is contained in the purge gas system within the instrument.

For degradation studies or for experiments on samples that offgas during transition or reaction, a Pyris 1 DSC Flow Through Cover Kit (P/N N537-0153) is required. The Flow Through Cover permits the purge gas and volatiles to exit directly through the top of the cover. The recommended flow rates of the purge gas are 40 – 50 cc/min. These flow rates will be realized by inlet purge gas pressures of 40 – 50 psi. Air or oxygen may be used as the purge gas.

Helium is the recommended purge gas for the CryoFill LN2 Cooling System. Nitrogen is the purge gas used with Intracoolers 1P and 2P. Recommended flow rates of the purge gas are 20 – 30 cc/min. These flow rates will be realized by inlet purge gas pressures of 20 – 30 psi.

The GSA 7 Gas Selector Accessory is a computer-controlled gas-switching device. The GSA 7 permits the automatic switching of sample purge gases at an operator-selected time or temperature during an analysis. You are allowed two gas changes per temperature program step with the GSA 7.

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

Temperature Range

The temperature range for your analysis depends on the sample and the applications of your results. The Pyris 1 DSC can be used to analyze samples from a temperature of –170°C (when used with a CryoFill LN2 system) to 725°C (in ambient operation). Isothermal analyses also can be performed at any selected temperature in the range of the instrument.

CAUTION: Do not use aluminum sample pans when programming the DSC to run above 600°C. Since aluminum melts at 660°C, the pans will alloy with and destroy the sample holders. Always make sure that you are not using aluminum pans in either the sample or the reference cell when you want the temperature of the DSC to exceed 600°C.

Scanning Rate

The DSC will allow scanning rates of 0.1°C/min to 500°C/min in steps of 0.1°C/min. Proper selection of scanning rate will increase efficiency of your analysis at the desired sensitivity. Generally, slower scanning rates improve the peak resolution while faster scanning rates improve the usable sensitivity.

NOTE: Scanning rates greater than 100°C/min are normally used only for rapidly heating or cooling the DSC to starting temperatures or to selected isothermal temperatures. Typical experimental scanning rates range from 5°C/min to 40°C/min.

Sample Loading

Before loading the sample into the Pyris 1 DSC sample holder, the sample must undergo sample preparation and sample encapsulation. In addition to encapsulating the sample, it is recommended that a reference capsule be used in the reference furnace during the DSC experiment. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated.

Load the sample and reference pans into the Pyris 1 DSC sample holder as follows:

1. Lift up the handle on the sliding sample holder enclosure cover.
2. Slide the cover back until it stops.
3. Note the orientation of the platinum sample holder covers. Remove them from the sample holders with tweezers.
4. Using tweezers, place the encapsulated sample in the left sample holder and put an empty pan and cover (reference) in the right sample holder.
5. Replace the sample holder covers back on the sample holders, with the same orientation as before they were removed. Maintaining the sample holders' orientation improves the baseline repeatability, especially at high temperatures.
6. Pull the sample holder enclosure cover and lock it into place by pushing the handle down.

NOTE: Try to load sample and calibration materials into the Pyris 1 DSC when the furnace temperatures are at room temperature or higher. If you must open the sample holder enclosure cover, make sure that the AirShield is on. Do not keep the cover open for more than a minute since frost will form in front of the cleats and around the AirShield. If this should occur, use the acid brush provided to sweep away any frost, making sure to keep it away from the sample holders.

See the topic on how to **Insert the Guard Rings** in Pyris Multimedia Presentations Help.

Specific Heat Analysis

The determination of specific heat capacity by a power-compensated DSC analyzer is an important tool in thermal analysis. Heat capacity is an intrinsic property of a material. It is the amount of energy needed to increase a unit quantity (e.g., 1 g, 1 mole) of material 1°C. All materials have specific heat. If there is no phase transition or reaction, the specific heat is a positive number that gradually changes with temperature.

Specific heat is fully normalized output of a DSC, i.e., the displacement during a scan is equal to the product of the heat capacity times the sample weight times the scanning rate plus the no-sample baseline. DSC data is sometimes misinterpreted because just one heat flow run was performed. The data from that run may contain information about the instrument itself such as the specific heat of the reference side of the sample holder. This problem can be fixed by performing a baseline run with no sample using the same conditions to be used for the sample. This constitutes the classical two-curve Cp method. This is illustrated here by finding the specific heat of the reference standard sapphire using the Pyris 1 DSC.

The following steps were performed in order to obtain the specific heat of sapphire:

1. Prepare the Pyris 1 DSC.
2. Place an empty standard aluminum sample pan and lid in each sample holder in order to run a no-sample baseline.
3. Enter the parameters into the Preferences pages and the Method Editor. Use a Sample Weight of 0 for the baseline run:

Purge Gas	Nitrogen at 20 cc/min
Cooling Device	Ice bath
Sample Weight	28.12 mg
Initial Temperature	50°C
Y Initial	0 mW
Equilibrate Temp.	0.01°C
Heat Flow	0.01 mW
Wait no longer than	15 min
Method Program	Isothermal – Scan – Isothermal
Heating/Cooling Rate	10°C/min
Temperature Range	50 – 100°C, 100 – 150°C, . . . , 250 – 300°C

4. Click on the **Start Method** button to start the baseline “no sample” run.
5. Find the sapphire standard in the Specific Heat kit (P/N 0219-0136) and encapsulate it in the standard aluminum sample pan used in the baseline run.
6. Enter the sample weight of 28.12 mg into the Method Editor.
7. Click on the **Start Method** button to start the sample run.
8. After data collection is complete, click on the **Data Analysis** button on the toolbar.
9. Using Add Data in the File menu, select the two data files you just collected. The baseline curve should be the lower curve and the sample curve should be the top curve.

10. The Step Select feature should be activated. Make sure that a checkmark appears next to Step Select in the Curves menu. If it does not, click on Step Select.
11. If you have data from multiple scanning steps, the Start Time at Zero feature should not be checked in the Curves menu.
12. The data is now in the form such that you can calculate the specific heat for each iso–scan–iso step.
13. Make sure that the sample curve is the active curve (heavy line display). Click on Heat Flow in the Curves menu. The Step Select dialog box is displayed.
14. From the list of steps, select adjacent iso–scan–iso steps to create a segment. Click on **OK**. If your method had 11 steps, you should be able to define 3 segments.
15. Repeat steps 11 and 12 for the baseline curve but use Baseline Heat Flow from the Curves menu.
16. Click on a segment of the sample curve and choose Multiple Curve from the Specific Heat submenu in the Calc menu.
17. The default selection for the baseline curve should be the segment of the curve that coincides with the segment of the sample curve selected. Click on **OK** or press **Enter** to accept the selection.
18. The specific heat curve and its ordinate scale is added to the display.
19. In the case of multiple-step data, repeat steps 15 and 16 for each scanning step (iso–scan–iso segment).
20. Change the X scale to Temperature using Rescale X in the Display menu or in the toolbar.

Operational Suggestions for Accurate Cp Analysis

To obtain a more accurate specific heat measurement, try to follow the suggestions below:

- **DSC Block Stability:** The DSC analyzer block temperature should be stable. For above-ambient specific heat, use ice water as the coolant. Water alone is less satisfactory because the water slowly warms up over time. An Intracooler is good for subambient work. Allow sufficient equilibration time after changing the heat sink temperature before running a baseline. One hour should be sufficient.
- **Purge Gas:** Use dry nitrogen or argon for the purge gas. Defective or inferior regulators can result in irregular purge flow with resultant baseline wanderation. Avoid using flow restrictors and flow meters on the inlet or outlet purge during data acquisition.
- **Calibration:** When performing a two-curve Cp analysis, it is important that the instrument be carefully calibrated. Single-point calibration is not sufficient. Use high-purity melting standards to bracket the temperature range of interest. Use the melting energy of an accurately weighed specimen of indium for energy calibration.
- **Preconditioning of the DSC:** Before performing the first of several high-sensitivity or high-accuracy DSC analyses, it it good practice to heat the analyzer to the highest temperature that will be achieved during your analysis, hold the temperature briefly, and then return to the Load temperature. Use the **Go To Temp** button on the control panel.
- **Temperature Program:**
    - Isothermal steps must be long enough to allow heat flow equilibration which depends on sample size and pan type. Two to three minutes is usually sufficient for step length.
    - The initial isotherm must be at least 30°C above block temperature.

- No physical or chemical process (e.g., decomposition, moisture loss) should occur during either isotherm.
- Do not try to cover a wide temperature range between isothermal steps. The isothermal steps behave like a reference for heat flow. By aligning the isothermal data, which is done automatically by the software, the effects of a slight baseline shift are minimized. If a wide temperature range must be used, it is preferable to divide the analysis into several steps, selecting the steps so that they bracket regions with transitions.
- During the scanning step, for a small sample, use a rapid heating rate (e.g., 20°C/min); for very large samples, use a low rate (e.g., 5°C/min). This ensures uniform heating of the sample.

- **Sample Pans:** You must use the same sample pan and lid for the sample run that you use for the no-sample baseline run. This assures that there will be no Cp error caused by weight difference. Use crimpable standard aluminum pans. For volatile samples, use a sealed pan that will not burst from the pressure of escaping volatiles. If possible, use a vacuum oven to remove the volatiles prior to the run so that you can use a crimpable sample pan.
- **Sample Size:** The sample size depends on the desired heating rate. A sample of 5 – 25 mg is normal.
- **Reference Side:** Place an empty pan in lid in the reference holder for both the sample run and the baseline run.
- **Platinum Sample Holder Covers:** Always use the platinum sample holder covers (in addition to the sample pan lids). Before beginning specific heat analysis, identify one with a distinguishing mark to help prevent mixing up the two lids. Take care to use the same lid on the same side consistently.

Calibration

Calibration of the Pyris 1 DSC for temperature and energy is accomplished by running high-purity standard and reference materials with known temperature and energy transitions. (Perkin Elmer ships reference materials in the Spares kit with each analyzer.) The data obtained after running these materials are used in the calibration programs to automatically calibrate the DSC. Once the calibration is performed, the analyzer will be calibrated continuously, even when the system is turned off. Unless major analyzer condition changes are made, the DSC should remain calibrated.

Calibration Precautions

The following precautions must be observed when the calibrating a Pyris 1 DSC:

- Do not use aluminum pans above 600°C since they will alloy with and contaminate the sample holders.
- All metal reference materials (indium, tin, lead, zinc) must be encapsulated in aluminum pans because they will alloy with gold or platinum pans.
- All inorganic reference materials (potassium sulfate, potassium chromate) should be encapsulated in gold pans.
- For the best temperature repeatability, the reference material should be flattened and placed in the center of the sample pan and then encapsulated. The sample pan should be placed in the center of the sample holder.

- When using subambient reference materials, it is recommended that they be encapsulated in volatile sample pans for a tight, hermetic seal (P/N 0219-0062 and 0219-0080 and volatile sample sealer 0219-0061).
- When using liquid nitrogen or an Intracooler, always observe the precautions for liquid nitrogen use.

When to Calibrate the DSC

Once you have performed the automatic calibration programs, the temperature and heat flow (energy) calibration of the DSC should remain unchanged for an extended period, provided there are no changes in the instrument operating conditions. (Periodic checks of the calibration typically show variations of <1% after many days.) There are a few conditions that will change either the temperature or the energy calibration and cause the need to recalibrate:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Run a reference material in the new range to determine if the current calibration is still valid.
- If the Balance is adjusted, recalibration may be necessary. Always optimize the baseline before the instrument is calibrated.
- If the purge gas type or purge gas flow rate is changed, the calibration should be checked for highest accuracy.
- If the coolant or cooling accessory used with the DSC is changed, recalibration may be necessary.
- Since temperature calibration depends slightly on scan rate, final calibration should be performed at the scan rate that you will use for your experiments. As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating rates, or heating and cooling rates, it is better to calibrate at the slowest rate to be used.
- If the instrument has been turned off for a long time (i.e., weeks or months), the instrument may appear to need recalibration. Condition the DSC by performing several heating and cooling runs with the sample holder empty and then check the calibration by running reference materials.

DSC Calibration Routines

The following calibration routines are available for the Pyris 1 DSC:

- **Calibrate Temperature**
- **Calibrate Heat Flow**
- **Calibrate Furnace**

NOTE: If you need to **optimize the baseline** using the Balance and Slope controls, do so before calibrating the analyzer.

As part of maintaining proper operating conditions for your instrument, you should calibrate it when it is first installed and when you change the sample holder. The recommended routine to use is the Temperature calibration routine. It is recommended also that the DSC be calibrated every three to six months after installation.

Calibrate Temperature

1. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. Restore the default Temperature calibration by selecting Temperature from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the All command.
3. Select the **Save and Apply** button in the Calibration window to send the default calibration values to the analyzer and save the current calibration file.
4. Select **Close** to close the Calibration window.
5. Complete a scan for each reference material under the same conditions that you run your samples.
6. Perform a Peak Area calculation and include the Onset temperature. Record the ΔH (J/g) and Onset results; you will need the Onset result for Temperature calibration and the ΔH result for Heat Flow calibration.
7. Repeat steps 5 and 6 for each additional reference material to be used.
8. Select the Calibrate command in the **View** menu. The Calibration window appears.
9. Select the Temperature tab. Enter the name of the reference material used, the expected Onset value, and the Onset result just measured for each reference material.
10. Select the check box in the Use column for each reference material to be used in the calibration.
11. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the current calibration file.
12. Go on to the next calibration procedure by clicking on its tab or select **Close** to close the Calibration window and begin using the new calibration values.

Calibrate Heat Flow

1. While using the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. Restore the default Heat Flow calibration by selecting Heat Flow from the Restore menu. If you performed a Temperature calibration just prior to starting a Heat Flow calibration and selected All from the Restore menu, then you do not need to restore the default Heat Flow calibration here.
3. Select **Save and Apply**.
4. Select **Close**.
5. Complete a scan using a reference material or use one that was run for the Temperature calibration.
6. Perform a Peak Area calculation and note the ΔH (J/g) result. You can also use the ΔH result recorded for one of the reference materials used in the Temperature calibration.
7. In the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
8. Select the Heat Flow tab. In the Calibration table, enter the name of the reference material used, the expected ΔH value, the ΔH result just measured, the weight of the reference material used, and the name of the method file used for the run.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration value to the analyzer and save the current calibration file.

10. Go on to the next calibration procedure by clicking its tab or select **Close** to close the Calibration window and begin using the new calibration values.

Calibrate Furnace

NOTE: This calibration is available only if the instrument has DDSC capability.

1. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. If applicable, complete the Temperature calibration.
3. Remove the pans from sample and reference holders.
4. Select the Furnace tab in the Calibration window.
5. In the Minimum field, enter a minimum temperature that is below your normal operating region.
6. In the Maximum field, enter a maximum temperature that is above your normal operating region.
7. Select the **Begin Calibration** button.
8. Wait the designated time for completion of the Furnace calibration.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.
10. Select **Close** to close the Calibration window and begin using the new calibration values.

DSC Calibration Reference Materials

Your company may have to comply with ISO 9000. Perkin Elmer Thermal Analysis calibration materials are ISO 9000 compatible. They are called Reference Materials. ISO has two classifications for calibration materials: Reference Materials and Certified Reference Materials.

Reference Materials (RMs) comply with ISO for instrumentation calibration. They are available from Perkin Elmer and Perkin Elmer provides documentation with each RM that cross references the material's lot code. This declaration should be filed with all other material certificates and documents in your ISO file.

ISO Guide 30 defines a RM as a

> "Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials."

Certified Reference Materials (CRMs) are traceable to national or international standards through an unbroken chain of comparisons. Not every calibration material is a CRM. CRMs can be very expensive and are not available for all thermal analysis techniques.

If you are not sure what material to use, then check with your Quality Assurance Department and discuss the differences between RMs and CRMs. If you find that you need CRMs, you may order them from the National Institute of Standards and Technology (301 975-6776).

Reference Material E is a standard epoxy–glass composite known as FR4. It is bisphenol A-type epoxy supported by four layers of woven E-type glass fibers. This type of material can be assayed by DSC thermal analysis. The supporting glass permits analysis well above the glass transition temperature. The epoxy resin exhibits mechanical relaxations down to liquid nitrogen temperatures. It is highly crosslinked and changes little under normal storage and handling conditions. Reference material E has been analyzed by various techniques and has been found to agree with the manufacturer's specifications. Assay numbers are not indicated on the samples. Recent lots indicate a storage modulus of 1.1e10 to 1.8e10 Pa.

The different reference materials for operation and calibration of your Pyris 1 DSC in the ambient to 725°C temperature range and for subambient operation are outlined the following two topics.

Calibration Reference Materials from Ambient to 725°C

Very-high-purity (>99.9%) metals or inorganic materials are typically used to calibrate the temperature and energy axes of the DSC when operating in the temperature range from ambient to 725°C. Two high-purity metal reference materials [indium (P/N 0319-0033) and zinc (P/N 0319-0036)] are supplied with your instrument for calibration purposes. Other reference materials are also available from Perkin Elmer. The nominal transition temperatures and energies for each of these reference materials recommended for calibration are as follows:

NOTE: Temperature and energy transition values may vary slightly from one lot number to another. Refer to the documentation provided with the reference material to determine the values for that lot number.

Reference Material	Part No.	Transition Point (°C)	Transition Energy (J/g)
Indium	0319-0033	156.60	28.45
Tin	0319-0034	231.88	60.46
Lead	0319-0035	327.47	23.01
Zinc	0319-0036	419.47	108.37
Potassium Sulfate	na	585.0 ± 0.5	33.26
Potassium Chromate	na	670.5 ± 0.5	35.56

Also available from Perkin Elmer are kits of encapsulated reference materials:

0219-0045 includes a large and a small indium, tin, and lead

N519-0762 includes indium and zinc

The selection of the proper reference materials is crucial for the optimal performance of the DSC over the temperature range used for your experiments. Always select a reference material that encompasses that temperature range. For example, if you operate the DSC from 50°C to 200°C, use indium, which melts at 156.60°C.

If you wish to calibrate the energy as well as the temperature, you must accurately weigh the reference material before the run. Typical weights for standards and reference materials range from 1 to 10 mg. The accuracy and precision to which you weigh the sample are directly related to those of the energy measurements made on the DSC.

NOTE: The zinc reference material should not be heated more than twice.

Calibration Reference Materials for Subambient Operation

Very-high-purity (>99.999%) organic materials can be used to calibrate the temperature when operating the DSC in the temperature range from –170°C. See the table below for a list of recommended subambient calibration standards that can be used with your DSC. Normally, indium is the reference material that is recommended for energy calibration for all operating conditions.

Substance	Transition	Transition Temp. (°C)	Transition Energy (J/g)
Cyclopentane	Crystal	–151.16	69.45(a)
Cyclopentane	Crystal	–135.06	4.94(a)
Cyclohexane	Crystal	–87.06	79.58(b)
Cyclohexane	Melt	6.54	31.25(b)
n-Heptane	Melt	–90.56	140.16(c)
n-Octane	Melt	–56.76	182.0(c)
n-Decane	Melt	–29.66	202.09(c)
N-Dodecane	Melt	–9.65	216.73(c)
n-Octadecane	Melt	28.24	241.42(d)
Hexatriacontane	Crystal(*)	72.14	18.74(d)
Hexatriacontane	Crystal	73.84	60.25(d)
Hexatriacontane	Melt	75.94	175.31(d)
P-Nitrotoluene	Melt	51.64	

(*) Shows some thermal history dependence
(a) Aston, J.G. et al., *JAC*, **65**, 341 (1943)
(b) Aston, J.G. et al., *JAC*, **65**, 1035 (1943)
(c) Finke, H.L. et al., *JAC*, **76**, 33 (1954)
(d) Schaerer, A.A. et al., *JAC*, **77**, 2017 (1955)

1. The materials listed here, if used for calibration, must be of 99.999% minimum purity as even small levels of impurity can affect the temperature and/or energy of the transition.
2. These substances must be encapsulated in volatile sample pans. To improve peak resolution, place a small piece of aluminum foil over your sample before sealing the pan.
3. If the peaks are not sharp (as in indium), the sample may be impure and the temperatures you measure may not be correct. Use a higher purity sample or check the purity of the sample by an alternate technique such as gas chromatography.

Subambient Operation

The Pyris 1 DSC can be operated in subambient mode. Perkin Elmer offers subambient accessories for the Pyris 1 DSC to satisfy most temperature requirements. There are three options for Pyris 1 DSC that extend the temperature range down to –30°C, –65°C, and –170°C. These are the Intracooler 1P, the Intracooler 2P, and the CryoFill Liquid Nitrogen Cooling System, respectively. The dry box that is required for subambient operation and an accessory for the DSC 7 is not necessary with the Pyris 1 DSC. The sliding sample holder enclosure cover replaces the dry box on the Pyris 1 DSC. Subambient operation of Pyris 1 DSC is comparable to ambient performance with respect to noise, resolution, baseline linearity, and repeatability.

To determine which subambient configuration you need, you must determine at which temperature you wish to operate.

Subambient operation of Pyris 1 DSC is obtained by using either of the following modes:

- **CryoFill Liquid Nitrogen Cooling System**
- **Intracoolers 1P and 2P**

The CryoFill LN2 Cooling System must be installed initially by a Perkin Elmer Service engineer. Afterward, you can convert back to ambient operation or use an Intracooler (see **From Subambient to Ambient Operation**).

CryoFill Liquid Nitrogen Cooling System

WARNING: **The CryoFill Liquid Nitrogen Cooling System MUST be installed by a Perkin Elmer service engineer. Once the system has been installed, you can switch from this cooling device to another cooling device without the help of a service representative by following the instructions in this manual and the online Installation Help.**

Use of liquid nitrogen in the Pyris 1 DSC is one way to operate in subambient mode. It allows operation down to temperatures as low as –170°C. Operating Pyris 1 DSC with liquid nitrogen is fast, easy, and frost-free.

Cryogenic temperatures are achieved through the use of a small, internal liquid nitrogen dewar. The dewar rests on a thermal island of high-tech epoxy materials that have outstanding insulating properties. The internal dewar is filled automatically from and is part of Perkin Elmer's CryoFill Liquid Nitrogen Cooling System. The CryoFill control unit incorporates a sophisticated mathematical algorithm and a pneumatic technique to ensure a very stable liquid level in the internal LN2 dewar. The algorithm uses a form of "fuzzy logic" allowing the liquid level to be held to within a few millimeters regardless of the pressure or flow rate from the LN2 supply tank. A constant liquid nitrogen level translates into a more reproducible baseline performance.

You connect the liquid nitrogen supply tank to the internal dewar via the liquid nitrogen transfer line. This line provides a safe connection to the tank while allowing easy separation from the tank during refilling operations.

The CryoFill Liquid Nitrogen Cooling System comprises the following parts:

- Cold finger
- Liquid nitrogen dewar that resides in the Pyris 1 DSC

- Control box that regulates the level of liquid nitrogen inside the dewar
- Liquid nitrogen transfer line
- Epoxy plate for subambient operation that has the dewar, liquid nitrogen sensor, and liquid nitrogen filling line attached
- Liquid nitrogen circuit board assembly
- Liquid nitrogen supply tank (CryoFill)

When you order a LN2 system (P/N N537-0540), it includes the LN2 Assembly (P/N N537-0259) and the Dewar Tank (N537-3009).

See the following topics on the CryoFill System and liquid nitrogen with the Pyris 1 DSC:

- **Precautions for Liquid Nitrogen Use in Pyris 1 DSC**
- **Preparing Pyris 1 DSC for Liquid Nitrogen Operation**
- **Operating the Pyris 1 DSC with Liquid Nitrogen**
- **Refilling the Liquid Nitrogen Supply Tank**

Precautions for Liquid Nitrogen Use

Performance of the Pyris 1 DSC in the subambient mode should be similar to performance in the ambient mode with respect to noise, baseline linearity, baseline repeatability, resolution, etc. To achieve this level of performance, however, when using liquid nitrogen as a coolant, the following requirements must be met:

- Helium **must** be used as the purge gas for the sample holder. A gas such as nitrogen or argon should not be used because it has a condensation temperature close to that of the coolant, and, thus, the increased density of that gas may lead to anomalous and nonrepeatable baselines.
- The helium purge gas **must** be exceptionally dry (use of the filter dryer accessory is recommended). Condensation may occur on the walls of the sample holder enclosure and affect baseline performance if the purge gas is not exceptionally dry.
- The sliding sample holder enclosure cover **must** be free of leaks and purged with dry nitrogen.
- The sliding sample holder enclosure cover should not be left open for long periods of time. Close the enclosure cover when not inserting or removing samples.
- The use of liquid nitrogen as a coolant will require recalibration of both temperature and calorimetric sensitivity, which will be affected by a few degrees and a few percentage points, respectively.
- Always use extreme caution when working with liquid nitrogen. It can cause severe burns.
- Use of liquid nitrogen as a coolant requires that the instrument be placed in a well-ventilated area.
- Use protective gloves when removing samples from the sample holder as they get extremely cold.
- When using the Pyris 1 DSC in subambient mode, you must turn on the cover heater.

Preparing Pyris 1 DSC for Liquid Nitrogen Operation

The CryoFill Liquid Nitrogen Cooling System should have been installed on your system by a Perkin Elmer service engineer.

Before operating the Pyris 1 DSC with liquid nitrogen as the coolant, check the following:

- The guard rings are installed.
- The O-ring on the sliding sample holder enclosure cover is in place.
- The liquid nitrogen transfer line is connected properly to the control box and to the analyzer.
- The dry nitrogen gas supply line is connected properly to the control box.
- The sliding sample holder enclosure cover is engaged.

The CryoFill feature should be ON so that the dewar in the analyzer is full and LN2 level sensor is on. You activate this feature either on the adjustable control panel or through the Pyris software.

Operating the Pyris 1 DSC with Liquid Nitrogen

Prior to operating the Pyris 1 DSC with liquid nitrogen as the coolant, see the precautions to be taken and the preparations to be made.

To operate the Pyris 1 DSC in subambient mode using the CryoFill LN2 Cooling System, perform the following steps:

1. Make sure that the sliding sample holder enclosure cover is engaged.
2. Purge the sample holder enclosure cover with dry nitrogen for several minutes.
3. Make sure your helium sample purge gas is flowing at 20 cc/min (regulator pressure of 20 psi).
4. Turn on the CryoFill LN2 System.
5. Check the baseline over the temperature range of interest and optimize the baseline, if necessary.
6. Perform a temperature calibration after the Pyris 1 DSC has equilibrated. Suitable subambient calibration standards are in **Calibration Reference Materials for Subambient Operation**. Volatile sample pans should be used for all liquids and organic solids to prevent vaporization and condensation in the sample holder cavity.

   NOTES: Try to load your sample and reference materials into the Pyris 1 DSC when the furnace temperatures are at room temperature or higher. If you must open the sample holder enclosure cover, make sure that the AirShield is on. Do not keep the cover open for more than a minute since frost will begin to form in front of the cleats and around the AirShield. If this should occur, use the acid brush provided to sweep away any frost, making sure to keep it away from the sample holders.

7. Load your sample and reference materials into the Pyris 1 DSC.
8. Observe the following precautions while performing your experiment:
   - When operating with liquid nitrogen as the coolant, it is recommended that the instrument be left on at all times and the temperature of the sample holders be left at 50°C overnight. Leave the sample holder purge gas flowing.
   - The samples used for temperature calibration must be minimum 99.999% pure as even small levels of impurity can influence the melting point.

Refilling the Liquid Nitrogen Supply Tank

To refill the liquid nitrogen supply tank, perform the steps below:

- **Disconnect the LN2 Supply Tank from Pyris 1 DSC**
- **Fill the LN2 Tank**
- **After Refilling the LN2 Tank**

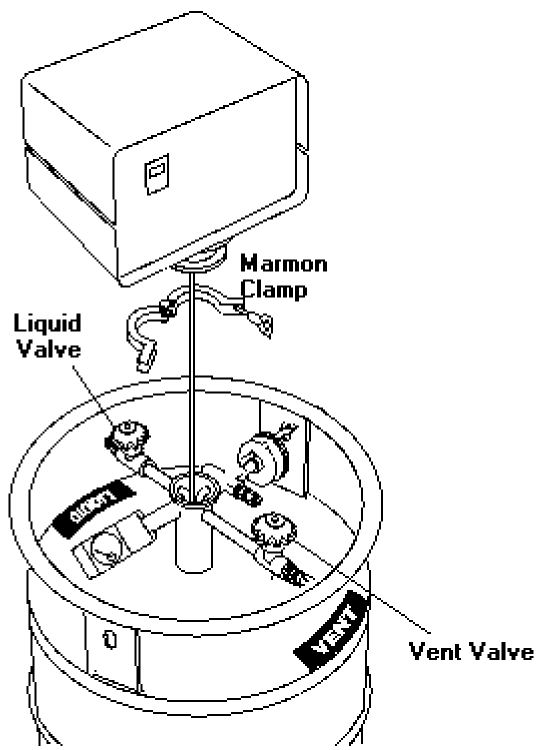
Disconnect the LN2 Supply Tank from Pyris 1 DSC

In order to refill the liquid nitrogen supply tank for the CryoFill Liquid Nitrogen Cooling System, you have to disconnect the supply tank from the Pyris 1 DSC and the dry nitrogen gas supply. Follow the steps below to disconnect the tank.

WARNING: **Do not remove the control box from the liquid nitrogen supply tank.**

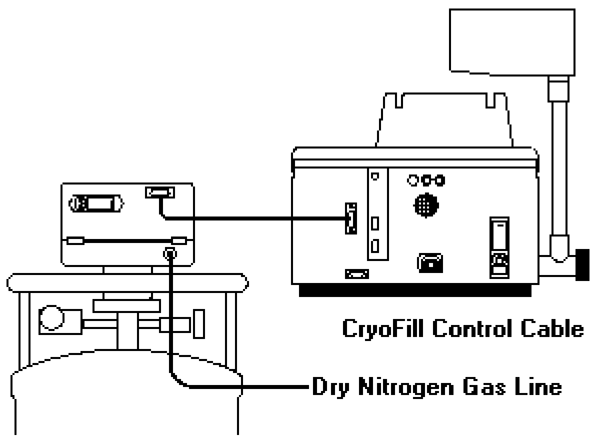
WARNING: **Check that the pressure gauge on the liquid nitrogen tank reads zero before opening the vent.**

1. Open the vent on the supply tank.

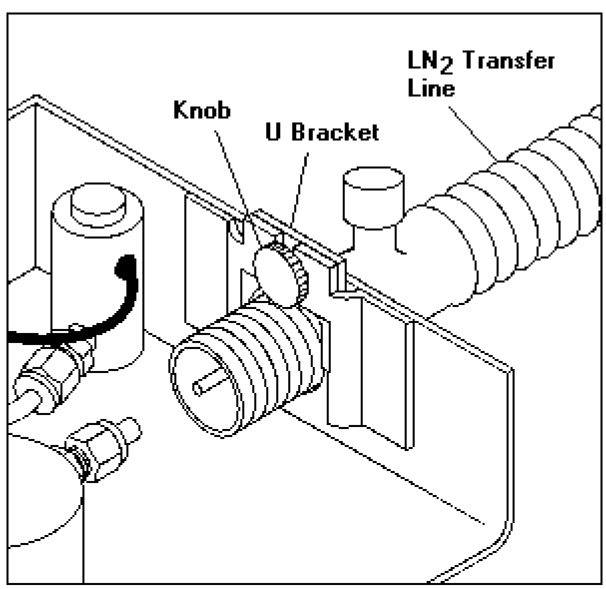


(Figure shows the control box up off its resting place in order to see the vent.)

2. At the adjustable control panel, press the **Menu** button to display DSC OPERATION on the display panel.
3. Press the **Menu** button to display CLEAN FURNACE?.
4. Press either the **Scroll Down** or the **Scroll Up** button three times. You should see CryoFill LN2 ACCY on the display.
5. Press the **Menu** button to see TURN CryoFill OFF?
6. Press the **Enter** button to turn the CryoFill off and return to the parent menu.
7. Turn the power to the CryoFill control box off.
8. Remove the following from the control box:
    a. power cord
    b. communications cable to Pyris 1 DSC
    c. dry nitrogen gas connection



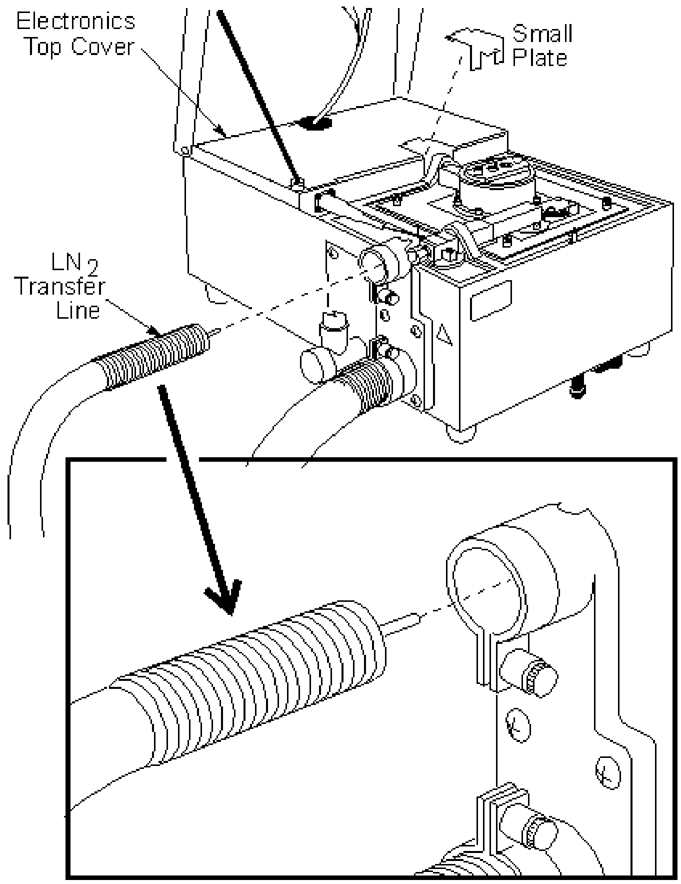
9. Lift the control box lid and loosen the knob holding the U-bracket that in turn is holding the LN2 transfer line in place. Remove the U-bracket.



10. Disconnect the LN2 transfer line from the control box by pulling it out.

WARNING: DO not disconnect the LN2 transfer line while it is cold. It should be at room temperature.

11. Loosen the top black knob on the mounting assembly on the side of the Pyris 1 DSC that is holding the LN2 transfer line in place.
12. Disconnect the LN2 transfer line from the top hole of the mounting assembly and set aside.

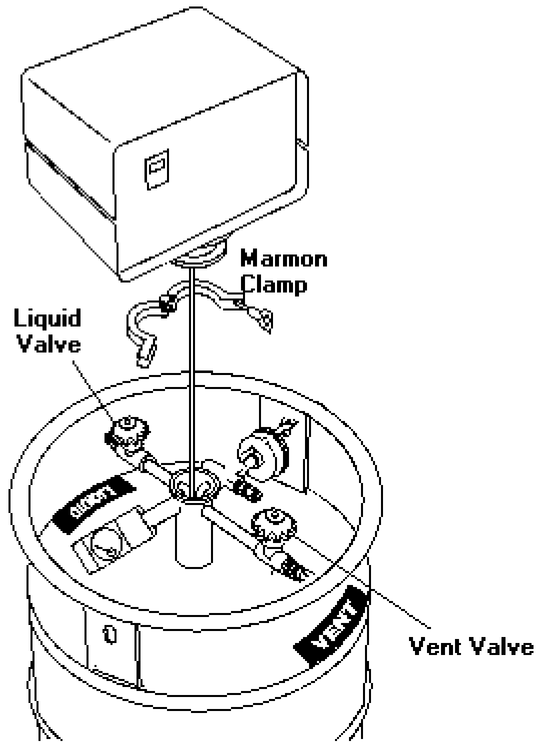


13. Move the tank to where the liquid nitrogen supply is located.

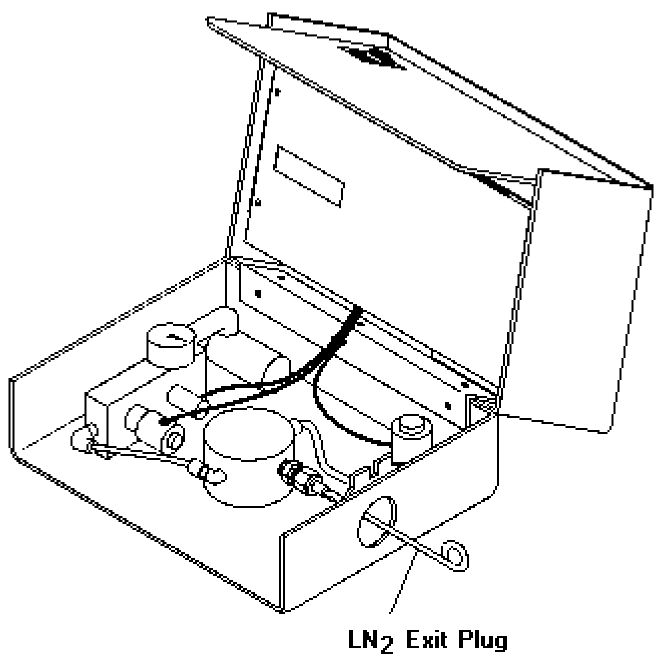
Fill the LN2 Tank

WARNING: Make sure that the VENT valve on the LN2 tank is open.

1. Open the valve labeled LIQUID on the LN2 tank in order to let liquid nitrogen in.



2. Connect a standard LN2 transfer line between the tank's LIQUID valve and a low-pressure LN2 source.
3. Insert the liquid nitrogen exit plug into the exit valve of the LN2 control box, i.e., the connector socket for the liquid nitrogen transfer line that goes to the Pyris 1 DSC, to prevent leakage of the LN2 while filling the tank.



4. Open the valve on the LN2 source and fill the tank. The tank is full when the level indicator reads full. Keep the tank's vent open during filling.

WARNING: When the liquid nitrogen tank becomes full, liquid nitrogen will spill out from the vent. Take care that you do not get splattered with liquid nitrogen.

5. Shut off the valve of the LN2 source.
6. Close the input LIQUID valve of the LN2 tank.
7. Wait for the tank to stabilize and for the transfer line connector to the tank to warm up.
8. Wearing a glove, remove the transfer line from the tank and from the source.
9. Close the VENT valve on the LN2 tank.
10. Remove the exit plug from the connector.

WARNING: Do not remove the exit plug until the septum is warm. Otherwise damage to the septum will occur.

11. Roll the tank back to the lab.

After Refilling the LN2 Tank

After you have refilled the LN2 tank and have returned it to your lab:

1. Take the end of the transfer line without the black cap and, holding it horizontally, insert the stainless steel needle all the way into the Teflon septum protruding out of the top hole in the mounting assembly on the analyzer. Be sure that the transfer line is pushed all the way into the septum and the black hose covers the septum.
2. Adjust the black knob on the mount assembly as needed to allow adjustment of the transfer line. Tighten the black knob.
3. With the lid of the control box open, reconnect the liquid nitrogen transfer line to the transfer line connector.
4. Replace the U-bracket in its slot and tighten the knob. Close the lid.
5. Reconnect the communications cable from the Pyris 1 DSC to the control box.
6. Reconnect the power cord to the control box to the power source.
7. Reconnect the pneumatic dry nitrogen gas connection to the back of the control box.
8. Make sure that the vent is closed, then turn on the power to the control box.
9. Make sure the power to the Pyris 1 DSC is on and that the lid is closed. At the adjustable control panel turn the CryoFill on.

WARNING: Do not fill the dewar in the Pyris 1 DSC with the lid open.

The dewar in the Pyris 1 DSC will now fill with liquid nitrogen.

Intracoolers 1P and 2P

The Perkin Elmer Intracoolers 1P and 2P provide a convenient way of cooling the sample holder enclosure cover of the Pyris 1 DSC. Intracooler 1P is a single-stage closed-loop circulating heat exchanger and Intracooler 2P is a dual-stage closed-loop circulating heat exchanger. Each contains a compact mechanical refrigeration unit coupled to the unique cylindrical expansion chamber by means of a rugged, insulated stainless-steel flexible line. The expansion chamber mounts directly on the Pyris 1 DSC sample holder enclosure block with a special bolt. The refrigerant is a widely available, nontoxic, non-CFC refrigerant that can continuously produce block temperatures as low as –55°C in the case of Intracooler 1P and –85°C in the case of Intracooler 2P. This permits operation of the Pyris 1 DSC to –30°C and –65°C, respectively. The Intracoolers should be placed beneath or next to the bench with the Pyris 1 DSC.

NOTE: The lowest temperature obtainable will depend on the ambient temperature and voltage in your laboratory.

Precautions for Intracoolers 1P and 2P

- Do not lift or move the Intracooler by pulling the flexible line.
- Do not make any sharp bends in the flexible line.
- Do not block or restrict air flow to the vents on the Intracooler.
- Do not operate the Intracooler above 32°C.
- Do not operate at line voltage outside 110 V ± 10% or 220 V ± 10% in 50-Hz operation.

Parts Required for Intracoolers 1P and 2P

The following parts are required for operation of the Pyris 1 DSC with an Intracooler 1P or 2P:

Intracooler 1P	N537-2180 (115 V, 50 or 60 Hz) N537-2181 (230 V, 50 or 60 Hz)
Intracooler 2P	N537-2182 (115 V, 50 or 60 Hz) N537-2183 (230 V, 50 or 60 Hz)

The following replacement parts are available:

0319-1720	Insulator Disk
0319-1527	Expansion Chamber Bolt
0319-1719	Split Bushing (2)

Installing an Intracooler 1P or 2P

To install an Intracooler 1P or 2P into a Pyris 1 DSC, follow the procedure below:

Before Installing an Intracooler

1. If your Pyris 1 DSC has the cold finger attached, it must be removed. Follow the instructions in **Remove the Cold Finger** and return here.

NOTE: If your Pyris 1 DSC has the turbulent chamber installed, it must be removed. Follow the instructions in **Remove the Turbulent Chamber** (below) and return here.

2. Place the Intracooler on the floor to the left of the bench where the Pyris 1 DSC is located. Make sure that there is a 4-in. clearance near the vents of the Intracooler to provide air for cooling the unit.

   For best results, the unit should be operated at a normal ambient temperature of 22°C (74°F). Higher temperature ambients will not permit the unit to reach its maximum low temperature. Never operate the Intracooler in ambients above 32°C (90°F).

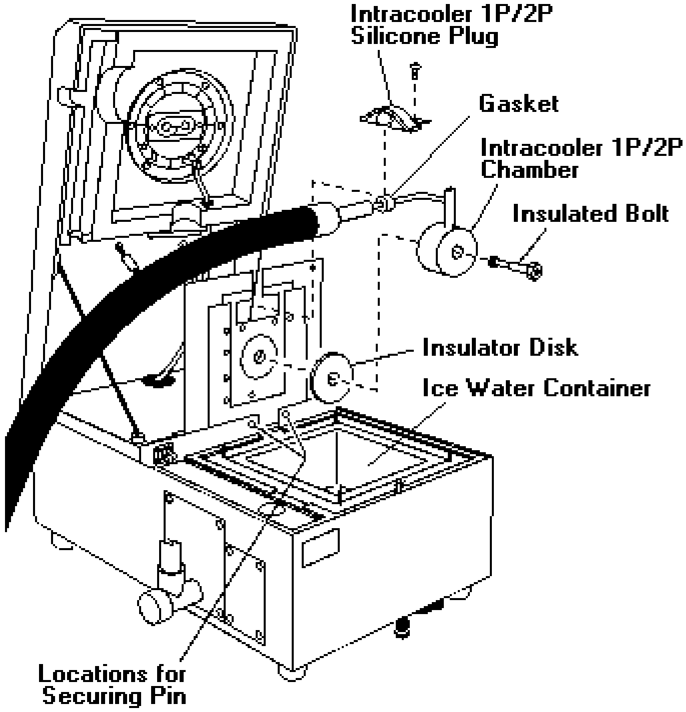
3. Shut down the Pyris 1 DSC system and remove the line power.

Intracooler Installation

At this point:

- your Pyris 1 DSC should be open with the lid propped up
- there should be no silicone plug in place
- the clamp on the left side of the epoxy plate for ambient operation should be removed
- the epoxy plate for ambient operation should be in the groove with no device (turbulent chamber or cold finger) attached

1. The pin for retaining the epoxy plate should be in the hole on the left side of the electronics compartment wall. If you just removed a cooling device, move the pin from the right hole to the left.
2. Lightly coat the top surface (that surface closest to the tube inlet) of the Intracooler expansion chamber with silicone grease. Do the same to the bottom surface of the Pyris 1 DSC sample holder block.
3. Place the insulator disk on the top surface of the Intracooler expansion chamber.
4. Angle the bar holding the Intracooler expansion chamber through the slit in the front of epoxy plate. Position the expansion chamber/insulator disk near the bottom of the sample holder and turn counterclockwise about 90° so that the bar is perpendicular with the left side of the epoxy plate.
5. Take the insulated bolt kit and place the split bushing around the insulated bolt beneath the head. Insert the bolt through the expansion chamber and insulator disk.



6. Fasten the expansion chamber and the insulator disk to the Pyris 1 DSC sample holder block by turning the hex bolt clockwise using a wrench. **DO NOT** overtighten the bolt.
7. Remove the retaining pin.
8. Holding the sides of the sample holder, lift the epoxy plate up and out of the groove (be aware of the tubing) and lower it onto the four standoffs.
9. Fasten the plate with the knurled nuts.
10. Place the foamy gasket (P/N N537-3108) over the inlet hose of the Intracooler in the position where the silicone plug will be placed.

    Look for the rectangular cut near two screw holes in the epoxy plate. This is where the silicone plug is to go.
11. Place the silicone plug for the Intracooler over the foamy gasket and secure the plug to the epoxy plate with the two Posidrive screws.
12. Close the small lid that covers the sample holder's tubing and electrical connections.
13. While holding the analyzer's lid, remove the prop rod and place it back on the top of the electronics compartment.
14. Close the lid and push the latch to the right and then back to lock the lid.
15. Slide the sample holder enclosure cover forward and push the handle down to lock the cover.

Reconnecting the Pyris 1 DSC after Intracooler Installation

1. Plug in the line power for the Pyris 1 DSC.
2. Make sure that the REFRIGERATION switch on the Intracooler is in the OFF (down) position. Plug the Intracooler into line power.

3. Set the purge gas flow rate to 20 psi (at the regulator).

Operating the Pyris 1 DSC Using an Intracooler 1P or 2P

Before operating the Pyris 1 DSC with an Intracooler 1P or 2P installed for subambient operation, see the precautions to be taken.

1. Make sure that the sliding sample holder enclosure cover is in the locked position, i.e., it should be all the way forward and the handle should be down.
2. Make sure that the AirShield is ON.
3. Make sure the nitrogen purge gas to the sample holder flows at 20 cc/min (regulator pressure of 20 psi).
4. Start up the Pyris software.
5. Program the Pyris 1 DSC temperature to 25°C.
6. Allow the system to equilibrate for a minimum of 30 minutes. Do not attempt to analyze materials or calibrate the Pyris 1 DSC before the system equilibrates since baseline repeatability may not be achieved.
7. Check the baseline over the temperature range of interest and optimize the baseline, if necessary.
8. Check the temperature and calorimetric calibration after the Pyris 1 DSC has equilibrated if this is the first time using the system with an Intracooler.

   Metal calibration standards or any of the subambient calibration reference materials can be used for calibration, depending on the temperature range of interest. Volatile sample pans (P/N 0219-0062) should be used for all liquid and organic solid calibration materials to prevent vaporization and condensation in the sample holder cavity.

   NOTE: Try to load sample and calibration materials into the Pyris 1 DSC when the furnace temperatures are at room temperature or higher. If you must open the sample holder enclosure cover, make sure that the AirShield is on. Do not keep the cover open for more than a minute since frost will form in front of the cleats and around the AirShield. If this should occur, use the acid brush provided to sweep away any frost, making sure to keep it away from the sample holders.

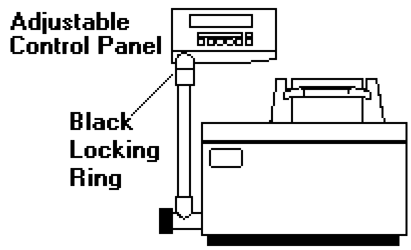
9. Load your encapsulated sample and reference materials into the sample holders and perform your experiment. Observe the following precautions:
   - When operating the Intracooler 1P or 2P, it is recommended that the Pyris 1 DSC be left on at all times and the temperature of the sample holders be left at 50°C overnight. Leave the sample holder purge gas on.
   - The samples used for calibration must be a minimum 99.999% pure. Even small levels of impurity can influence the temperature and/or energy of the transition.
   - The Intracoolers are not thermostatically controlled but operate at “constant cooling.” Thus, the block temperature will change with a change in ambient temperature. Also, the cooling efficiency of the Intracoolers will depend on the ambient temperature. Optimum operation is realized at an ambient of 22°C. Under no circumstances should the Intracoolers be operated in ambient temperatures above 32°C.
   - When properly installed, the Intracoolers may be operated for long periods of time. Occasionally, however, it may be necessary to power off the Intracooler so that minor frost that may have built up can be eliminated.

Removing an Intracooler 1P or 2P

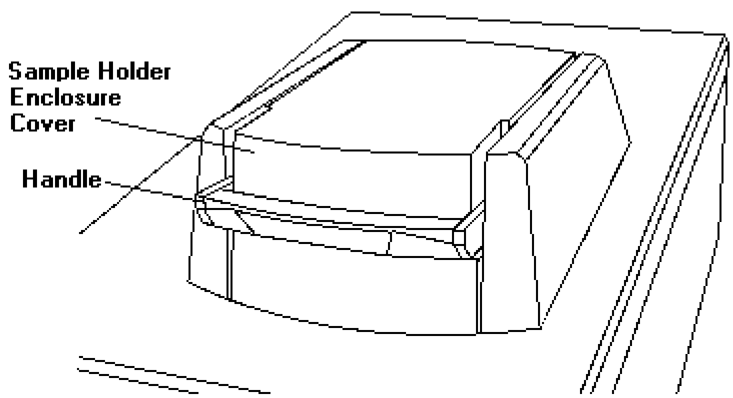
In order to use a turbulent chamber or the cold finger, or to convert your system from ambient to subambient using the CryoFill LN2 Cooling System, you may first have to remove the Intracooler 1P or 2P. To do so, perform the following steps.

> **NOTE:** If you wish to use the Pyris 1 DSC in subambient operation using the CryoFill Liquid Nitrogen Cooling System, the system has to be installed by a Perkin Elmer service engineer. Once installed, you can switch from CryoFill to ambient operation and from ambient to subambient operation on your own.

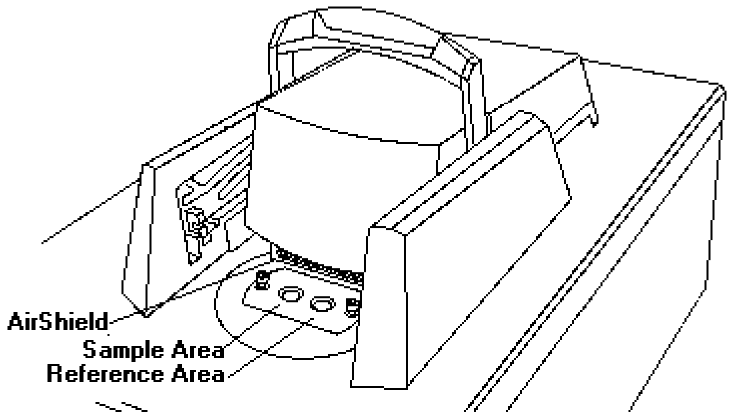
1. Turn off the power to the Pyris 1 DSC.
2. Loosen the black ring on the adjustable control panel's pole by turning it counterclockwise. Swing the panel 90° or enough so that it is out of the way of the analyzer's lid when lifted.



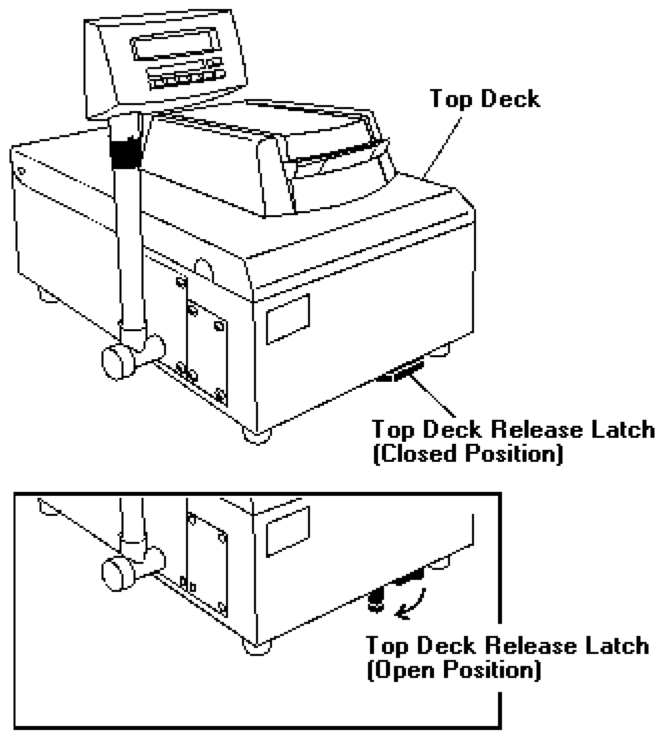
3. Lift the handle on the sliding sample holder cover to disengage the cover.



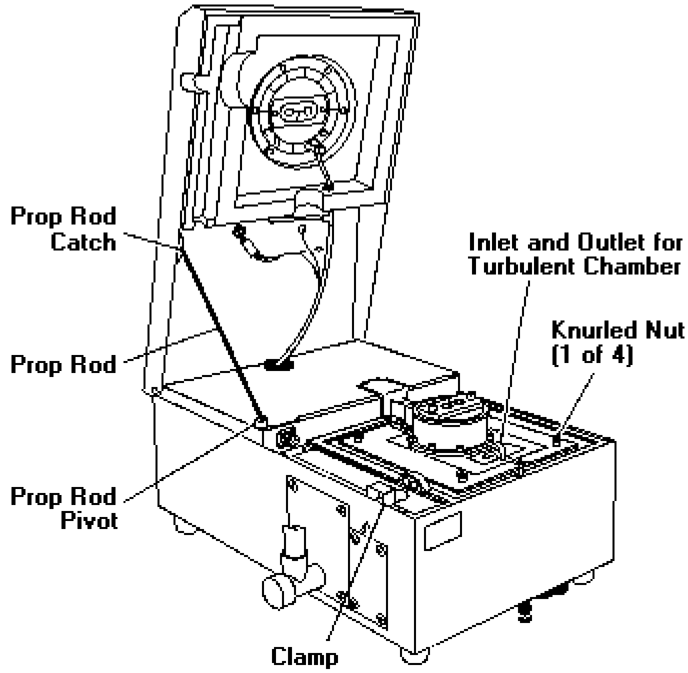
4. Slide the cover back as far as possible to expose the sample holders.



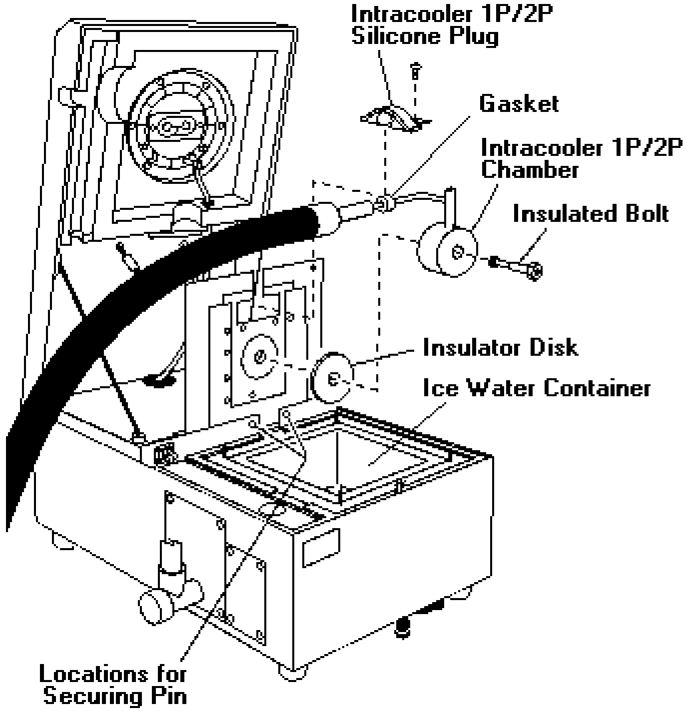
5. Pull the latch at the bottom of the Pyris 1 DSC out and push it all the way to the left to unlock the analyzer's lid.



6. Lift the lid up and prop it up with the prop rod by inserting the rod in the prop rod holder on the underside of the analyzer lid.



7. Remove the two Posidrive screws that secure the silicone plug keeping the Intracooler hoses in place.
8. Remove the foamy gasket from the inlet hose and store in a safe place.
9. Lift up and slide back the small cover at the front of the electronics compartment that covers the tubes leading to the sample holder.
10. Remove the four brass knurled nuts from the front of the epoxy plate for ambient operation.
11. Slowly lift the epoxy plate with the Intracooler attached up and off of the four screws by holding onto the sample holder with both hands. After the plate clears the screws, begin to angle the back of the plate down while continuing to lift it up. **BE CAREFUL** of the tubing attached to the sample holder and the Intracooler.
12. Align the two holes in the epoxy plate with the holes in front of the groove in the front of the electronics compartment. Gently lower the back edge of the epoxy plate into the groove.
13. Insert the retainer pin through the holes. This will keep the epoxy plate in place when the Intracooler is removed.
14. Remove the Intracooler expansion chamber from the sample holder block by removing the bolt. The split bushing and the insulator disk between the chamber and the sample holder can now be removed.



The Pyris 1 DSC is now ready for installation of a turbulent chamber or a cold finger. If you are preparing your system for using the CryoFill, return to removing the epoxy plate for ambient operation.

From Ice Water Operation to Turbulent Chamber

The Pyris 1 DSC is shipped with the cold finger attached and its associated silicone plug installed. The white plastic ice water bucket is also in place. To change the Pyris 1 DSC from ice water operation using the cold finger to operation using the turbulent chamber, perform the two steps below:

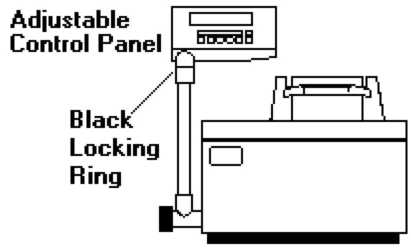
- **Remove the Cold Finger**
- **Install the Turbulent Chamber**

Remove the Cold Finger

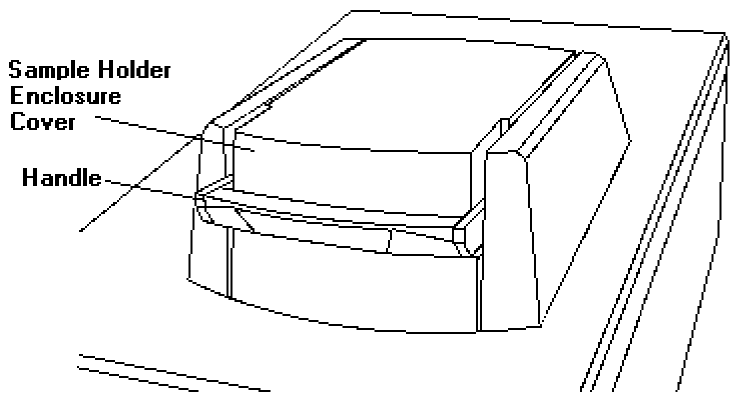
Since the Pyris 1 DSC is shipped with the cold finger attached and the ice bucket in place, in order to change the cooling device, you will have to remove the cold finger from the sample holder unless you are going to use the CryoFill. To remove the cold finger from the sample holder, follow the procedure below.

NOTE: If you wish to use the Pyris 1 DSC in subambient operation using the CryoFill Liquid Nitrogen Cooling System, it will have to be initially installed by a Perkin Elmer service engineer. Once installed, you can switch from subambient to ambient operation and from ambient to subambient operation of the Pyris 1 DSC on your own.

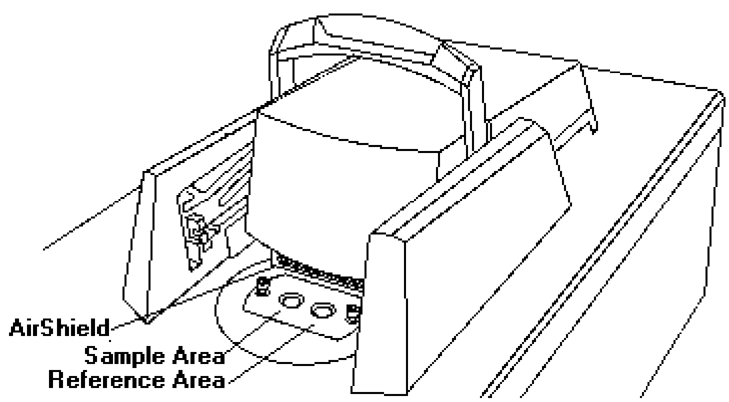
1. Loosen the black ring on the adjustable control panel's pole by turning it counterclockwise. Swing the panel 90° or enough so that it is out of the way of the analyzer's lid when lifted.



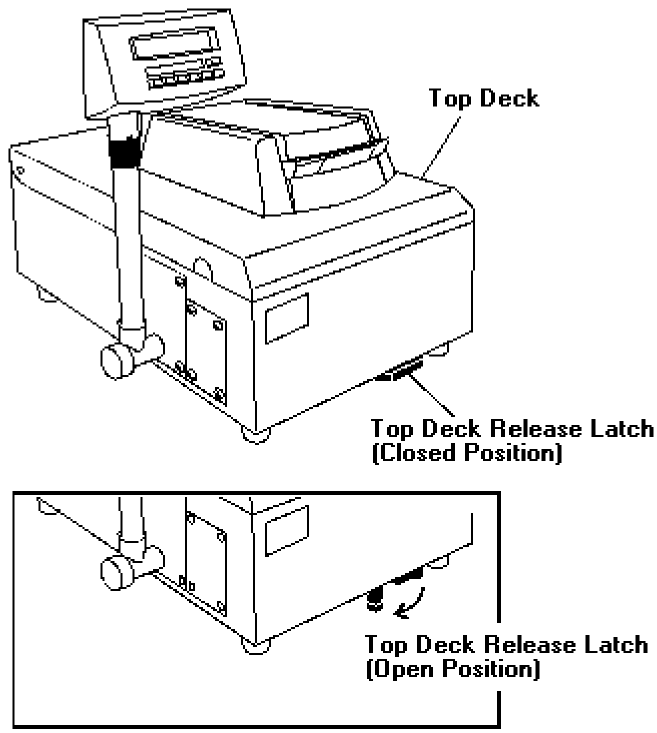
2. Lift the handle on the sliding sample holder enclosure cover to disengage the cover.



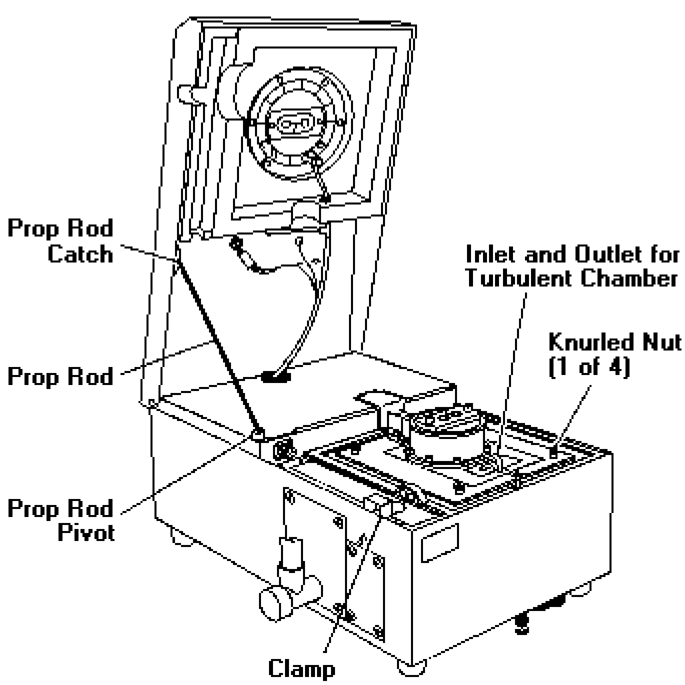
3. Slide the enclosure cover back as far as possible to expose the sample holders.



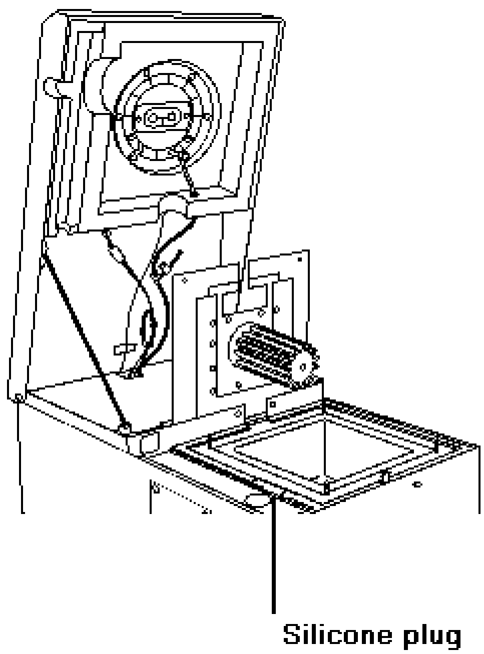
4. Pull the latch at the bottom of the Pyris 1 DSC out and push it all the way to the left to unlock the analyzer's lid.



5. Lift the lid up and prop it up with the prop rod by inserting the rod in the prop rod holder on the underside of the analyzer lid.



6. Lift up and slide back the small cover at the front of the electronics compartment that covers the tubes and electrical leads leading to the sample holder.
7. Remove the four brass knurled nuts from the front of the epoxy plate for ambient operation.
8. Slowly lift the epoxy plate up and off of the four screws by holding onto the sample holder with both hands. After the plate clears the screws, begin to angle the back of the plate down while continuing to lift it up. **BE CAREFUL** of the tubing attached to the sample holder.
9. Align the two holes in the epoxy plate with the holes in the front of the groove in the electronics compartment. Gently lower the back edge of the epoxy plate into the groove.



10. Insert the retainer pin through the holes on the right side of the epoxy plate. This will keep the epoxy plate in place when the cold finger is removed.
11. Grasp the cold finger and turn counterclockwise to unscrew it from the sample holder.
12. Remove the two Posidrive screws holding the blank silicone plug in place on the left side of the analyzer deck. Remove the plug.
13. Store the cold finger and its silicone plug for future use.

You can now install a turbulent chamber or an Intracooler 1P or 2P.

Install a Turbulent Chamber

After removing a cold finger or an Intracooler 1P or 2P from the sample holder, you can install a turbulent chamber. The epoxy plate for ambient operation should still be in the holding groove.

1. Remove the retaining pin from the hole on the right side of the electronics compartment.
2. Lift the epoxy plate up and align the screw hole on the left side of the plate with the left hole on the electronics compartment. Lower the plate back into the groove.
3. Insert the retaining pin through the holes.

4. Take the turbulent chamber and coat the top (indicated by "UP" with an arrow on the side of the chamber) with silicone grease.
5. Attach the Tygon tubing to the inlet (lower) and exit (upper) copper tubes on the turbulent chamber.
6. Bring the tubing through the slit in the epoxy plate so that it is above the plate as you place the turbulent chamber against the bottom of the sample holder. Turn the turbulent chamber so that the connectors and tubing are facing toward the left of the instrument.
7. While holding the chamber in place with one hand, attach the chamber to the sample holder with the bolt provided.
8. Remove the retaining pin from the epoxy plate.
9. While grasping the two sides of the epoxy plate or the sides of the sample holder on top of the plate, whichever is easier for you, **CAREFULLY** lift the plate from the groove. Once it clears the groove, begin to angle the front side of the plate down as you move the plate toward you. **BE CAREFUL** not to pull on the tubes attached to the sample holder. Make sure the Tygon tubes on the turbulent chamber are out of the way of the plate. They should be above the plate and facing the left side of the instrument.
10. Lower the epoxy plate for ambient operation onto the four standoffs. Replace the knurled nuts on the screws, tightening them to finger-tightness.
11. Attach the silicone plug (P/N N537-0141) to the analyzer deck with the two Posidrive screws provided.
12. Thread the Tygon tubes through the holes in the silicone plug.
13. Place the square clamp over the Tygon tubing to the left of the silicone plug and attach it to the epoxy plate with the two Posidrive screws. This clamp keeps the tubing securely in place as it exits the analyzer.
14. Attach the hoses of the turbulent chamber as follows:
    - If using tap water, connect the inlet hose (attached to the lower copper tube extending from the turbulent chamber) to the tap and the exit hose (attached to the upper copper tube) to a drain.
    - If using a water circulator, connect the inlet hose to the outlet of the circulator and the exit hose to the circulator's inlet.
15. While holding up the lid, remove the prop rod and place it back on the top of the electronics compartment.
16. Lower the lid, making sure the Tygon tubing from the turbulent chamber is exiting the analyzer in the proper place on the left side of the instrument.
17. Push the latch at the bottom of the Pyris 1 DSC to the right and back to lock the lid.
18. Slide the sliding sample holder enclosure cover forward until it stops. Lower the handle to lock the cover in place.
19. Return the adjustable control panel to its original position and turn the black ring clockwise to lock the panel into position.

Pyris 1 DSC is now ready for ambient operation with a turbulent chamber.

From Subambient to Ambient Operation

To convert the Pyris 1 DSC from subambient operation using the CryoFill LN2 Cooling System back to ambient operation, you must perform the following steps:

NOTE: The CryoFill LN2 Cooling System must be initially installed by a Perkin Elmer service engineer. Once installed, you can switch from the CryoFill to ambient operation and vice versa on your own.

- **Disconnect the CryoFill Control Box from Pyris 1 DSC**
- **Remove the Epoxy Plate for Subambient Operation**
- **Install the Epoxy Plate for Ambient Operation**

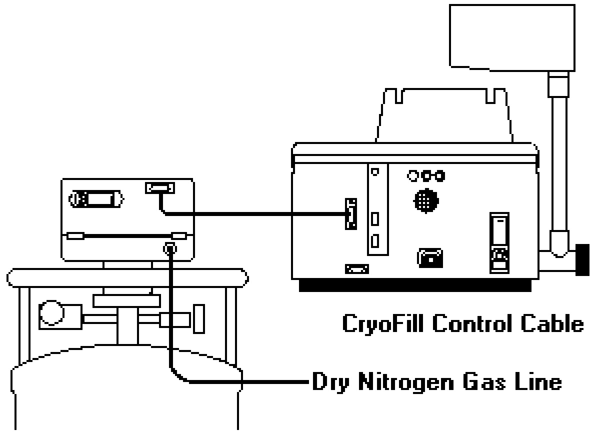
After performing these steps, your analyzer is ready to use a cold finger, an Intracooler, or turbulent chamber as a cooling device.

Disconnect the CryoFill Control Box from Pyris 1 DSC

The following procedure is for disconnecting the CryoFill control box and liquid nitrogen transfer line from Pyris 1 DSC in order to change operation from subambient to ambient.

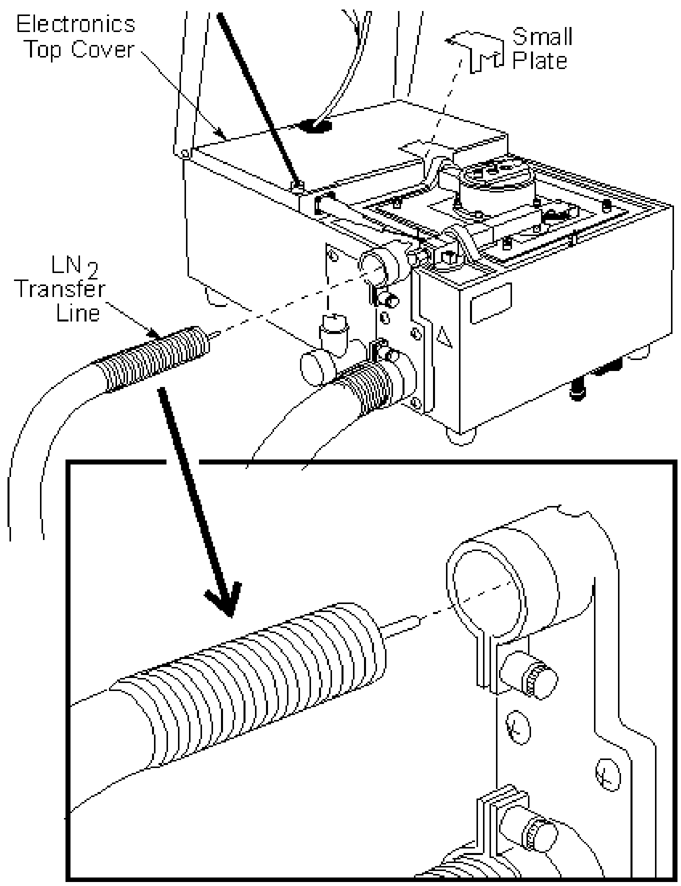
WARNING: **Before disconnecting the CryoFill control box from the Pyris 1 DSC, try to use up all of the liquid nitrogen in the analyzer's dewar. Otherwise, vent off any remaining LN2 before starting the conversion from subambient to ambient operation.**

1. At the adjustable control panel, press the **Menu** button to display DSC OPERATION on the display panel.
2. Press the **Menu** button to display CLEAN FURNACE?.
3. Press either the **Scroll Down** or the **Scroll Up** button three times. You should see CryoFill LN2 ACCY on the display.
4. Press the **Menu** button to see TURN CryoFill OFF?
5. Press the **Enter** button to turn the CryoFill off and return to the parent menu.
6. Turn off the power to the CryoFill control box.
7. Turn off the power to the Pyris 1 DSC.
8. Remove the communications cable connecting the CryoFill control box to Pyris 1 DSC from the RS232 connector at the back of the analyzer.

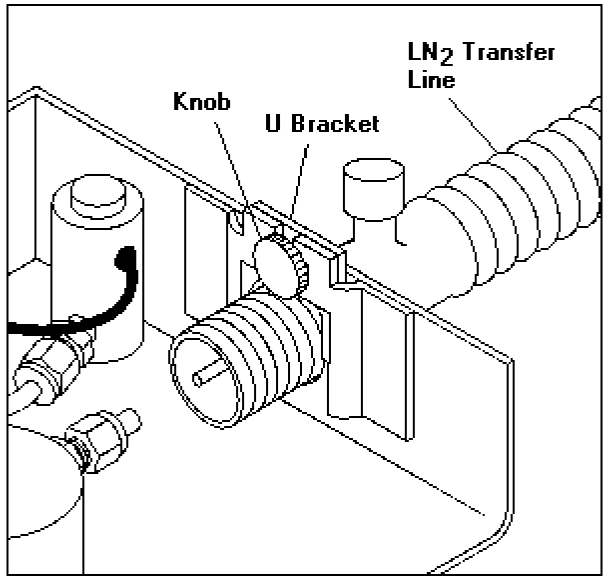


9. Loosen the upper black knob on the transfer line mounting assembly on the left side of the Pyris 1 DSC that is securing the transfer line.
10. Remove the liquid nitrogen transfer line from the Teflon septum in the top hole of the transfer line mounting assembly.

WARNING: Do not disconnect the LN2 transfer line while it is cold. It should be at room temperature.

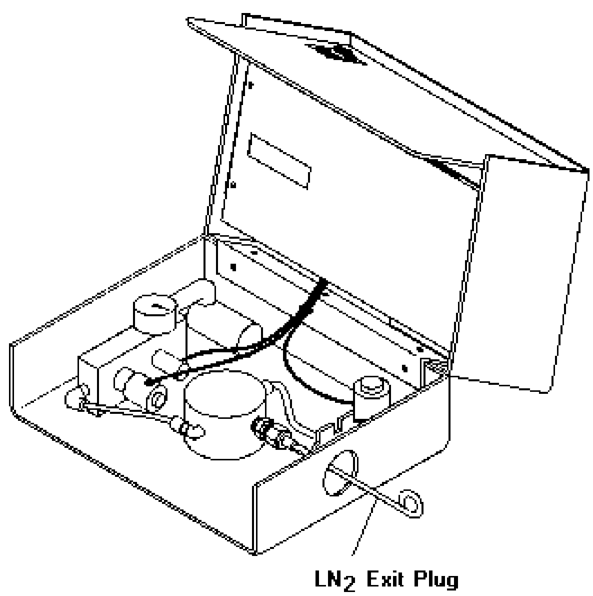


11. Open the control box lid and loosen the black knob holding the U bracket that is keeping the transfer line in place. Remove the U bracket.
12. Remove the liquid nitrogen transfer line from the transfer line connector on the control box.



Set the transfer line aside in a safe place.

13. Open the VENT valve on the LN2 tank.
14. Insert the exit plug into where the LN2 transfer line was just removed to prevent leakage.



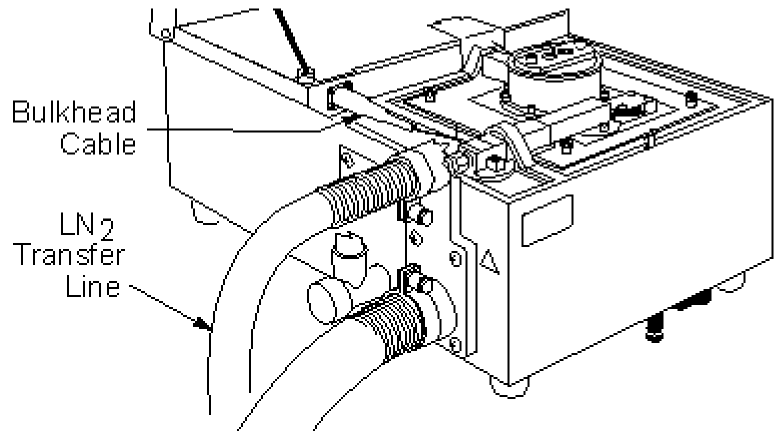
15. Disconnect the dry nitrogen pneumatic gas connection from the control box.
16. Disconnect the power cord from the power source.

17. Wrap up the cord and cables to get them out of the way and move the tank to a storage area.

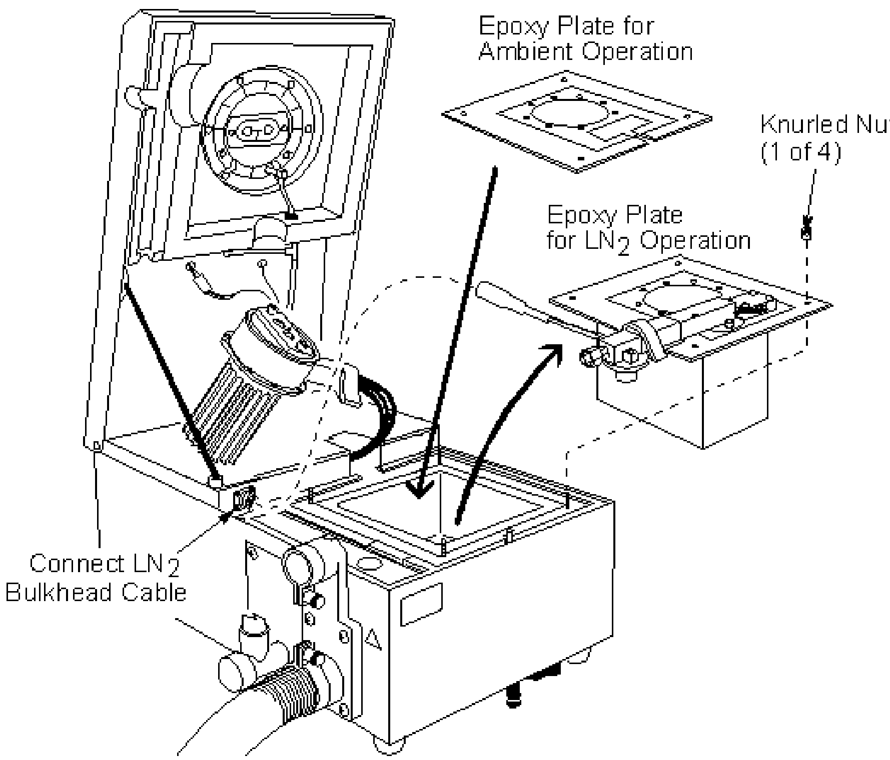
You do **not** need to remove the exhaust tube from the Pyris 1 DSC.

Remove the Epoxy Plate for Subambient Operation

1. Make sure that the power to the instrument is off.
2. Open the analyzer lid and prop up the lid with the prop rod.
3. Disconnect the bulkhead cable from the bulkhead receptacle.



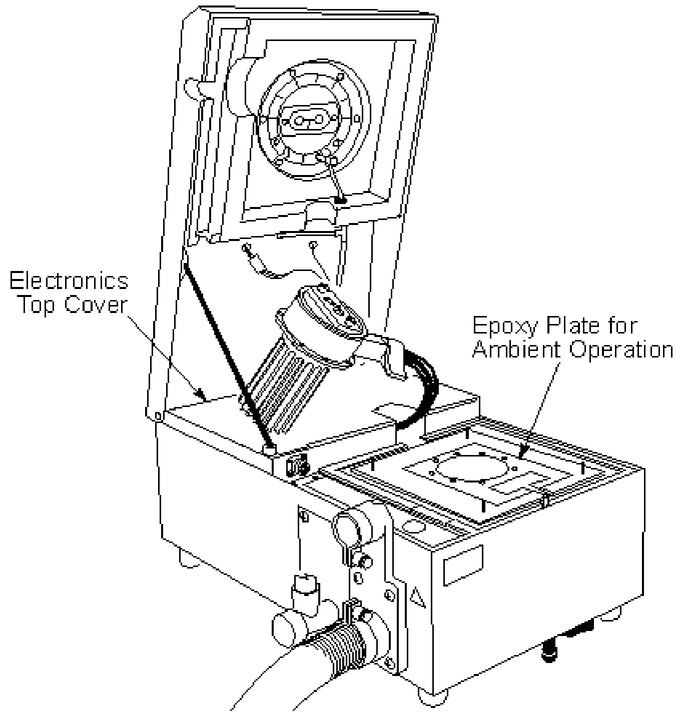
4. Lift up and remove the small plate that covers the electrical connections to the sample holder.
5. Remove the eight (8) Phillips head screws securing the sample holder to the epoxy plate for subambient operation.
6. Remove the two Posidrive screws securing the rear square clamp that holds the sample holder onto the epoxy plate (near the electronics compartment). Remove the clamp.
7. Lift the sample holder with cold finger attached and carefully place it on the cover to the electronics chassis. Take care not to pull the connections from the board.



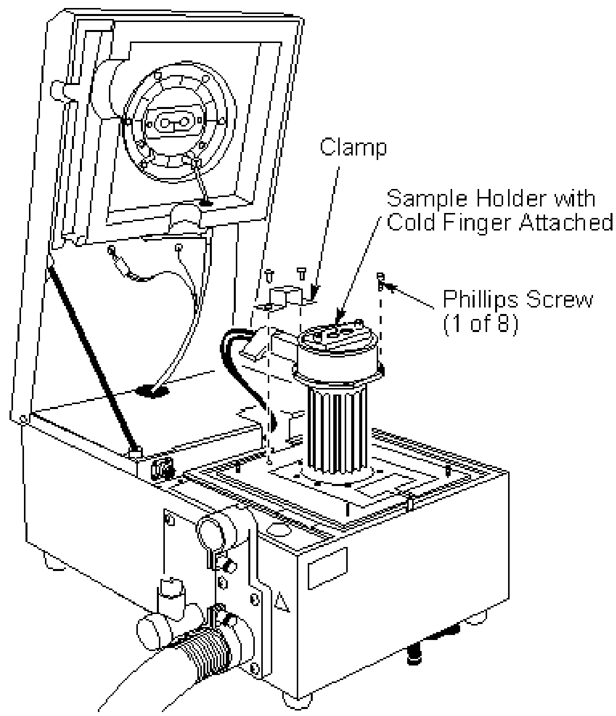
8. Remove the four knurled nuts that secure the epoxy plate.
9. Lift the epoxy plate from the four screws and remove. While lifting the epoxy plate, make sure to clear the liquid nitrogen exhaust hole from the small hole in the vent epoxy plate on the left-hand side of the analyzer.
10. Replace the ice water bucket into the analyzer.
11. Remove the drip tray from underneath the left side of the analyzer.

Install the Epoxy Plate for Ambient Operation

1. Take the epoxy plate for ambient operation and position it on the four standoff screws.



2. Take the sample holder with the cold finger attached and lower it onto the epoxy plate. Secure it with the eight (8) Phillips head screws.
3. Replace the rear clamp used to secure the sample holder to the epoxy plate and secure with the two Posidrive screws.



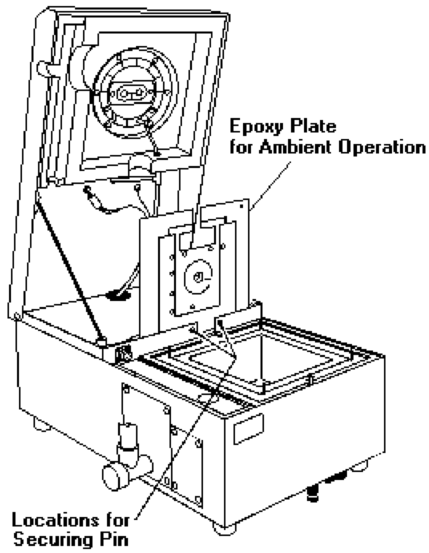
4. If you are going to use the cold finger for ambient operation,
   a. secure the epoxy plate with the four knurled nuts,
   b. secure the blank silicone plug with the two Posidrive screws,
   c. replace the small plate that covers the electrical connections to the sample holder,
   d. fill the bucket with ice water,
   e. close the analyzer lid by lowering the prop rod, press down on the lid, and push the hood latch all the way to the right.

Your instrument is now ready for you to install a turbulent chamber or an Intracooler 1P or 2P.

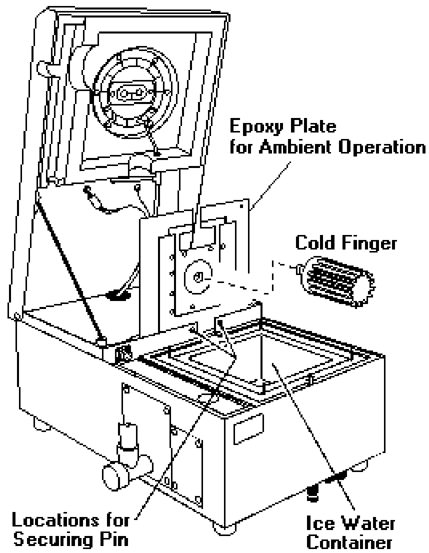
Install the Cold Finger

After removing the turbulent chamber or an Intracooler 1P or 2P, you can now install the cold finger for ice water operation of the Pyris 1 DSC or for subambient operation using the CryoFill LN2 Cooling System. The epoxy plate for ambient operation should still be in the groove.

1. Remove the retaining pin from the right side of the epoxy plate.



2. Align the holes on the left side of the epoxy plate and the electronics compartment.
3. Insert the retaining pin through the holes.
4. Coat the top of the cold finger with silicone grease.
5. With both hands, hold the cold finger while aligning the screw-like knob on its top side with the hole on the underside of the sample holder. Attach the cold finger by turning it clockwise. **DO NOT** overtighten the cold finger.



6. Remove the retainer pin from the electronics compartment side.
7. While grasping the two sides of the epoxy plate or the sides of the sample holder on top of the plate, whichever is easier for you, **CAREFULLY** lift the plate from the groove. Once it clears the groove, begin to angle the front side of the plate down as you move the plate towards you. **BE CAREFUL** not to pull the tubes attached to the sample holder.
8. Lower the epoxy plate for ambient operation onto the four standoff screws. Replace the knurled nuts on the screws, tightening them to finger tightness.
9. Attach the blank or flat silicone plug to the analyzer deck with the two screws that secured the turbulent chamber's plug.
10. Fill the ice bucket through the hole in the epoxy plate.
11. While holding up the analyzer deck, remove the prop rod and place it back on the top of the electronics compartment.
12. Lower the lid and push the latch to the right and back to lock the lid.
13. Slide the sliding sample holder enclosure cover forward until it stops. Lower the handle to lock the cover in place.
14. Return the adjustable control panel to its original position and turn the black ring clockwise to lock the panel into position.

The Pyris 1 DSC is now ready for ambient operation with ice water as the coolant. If you are preparing your system for using the CryoFill, return to **Removing the Epoxy Plate for Ambient Operation.**

From Ambient to CryoFill Operation

If you had the CryoFill Liquid Nitrogen Cooling System installed by a Perkin Elmer service engineer and then switched to ambient operation (following the instructions given in the topic **From Subambient to Ambient Operation**), you can reconfigure your Pyris 1 DSC for subambient operation using the CryoFill LN2 Cooling System once again. Perform the following steps:

- **Remove the Epoxy Plate for Ambient Operation**
- **Install the Epoxy Plate for Subambient Operation**
- **Connect the CryoFill Control Box to Pyris 1 DSC**
- **Connect Dry Nitrogen Gas to CryoFill Control Box**

Remove the Epoxy Plate for Ambient Operation

To use the Pyris 1 DSC in subambient mode with the CryoFill Liquid Nitrogen Cooling System, you will have to first remove the epoxy plate for ambient operation.

1. If your Pyris 1 DSC currently has a turbulent chamber installed, **remove the turbulent chamber** (see below). Once the turbulent chamber is removed from the epoxy plate for ambient operation, return here.

   If your Pyris 1 DSC currently has an Intracooler 1P or 2P installed, remove the Intracooler. Once the Intracooler is removed from the epoxy plate for ambient operation, return here.
2. If you just removed a turbulent chamber or an Intracooler, you now must install the cold finger. Once the cold finger is installed, return here.

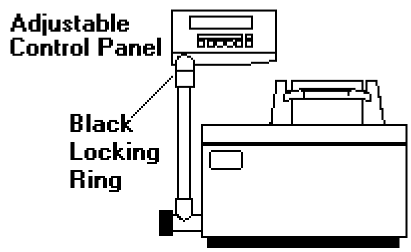
   Now the Pyris 1 DSC should have the cold finger attached to the sample holder and the epoxy plate for ambient operation should be in the holding groove.
3. Remove the retaining pin holding the epoxy plate in place.
4. Lift the epoxy plate with sample holder out of the groove and place it on the four standoff screws.
5. Lift up and remove the small plate that covers the electrical connections to the sample holder.
6. Remove the eight (8) Phillips head screws securing the sample holder to the epoxy plate for ambient operation.
7. Remove the two (2) Posidrive screws securing the rear square clamp that holds the sample holder onto the epoxy plate (near the electronics compartment). Remove the clamp.
8. Lift the sample holder with cold finger attached and carefully place it on the cover to the electronics chassis. Take care not to pull the connections from the board.
9. Lift the epoxy plate from the standoffs and remove. Store it for later use.
10. Remove the plastic bucket and store for later use.

Remove the Turbulent Chamber

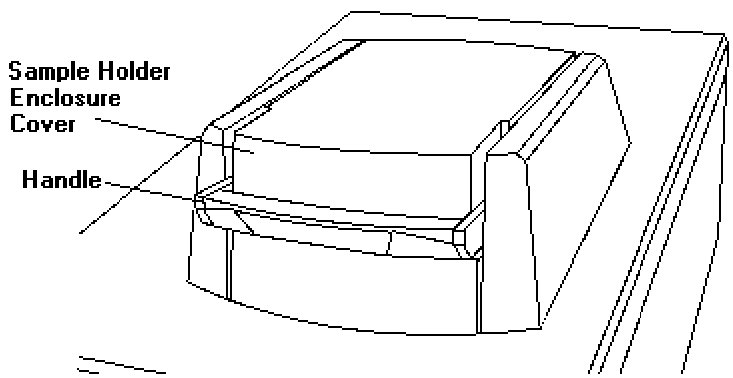
In order to change to another cooling device, you may have to remove the turbulent chamber. Follow the procedure below.

NOTE: If you wish to use the Pyris 1 DSC in subambient mode using the CryoFill Liquid Nitrogen Cooling System, the CryoFill will have to be installed initially by a Perkin Elmer service engineer. Once installed, you can switch from subambient to ambient operation and from ambient to subambient operation of the Pyris 1 DSC on your own.

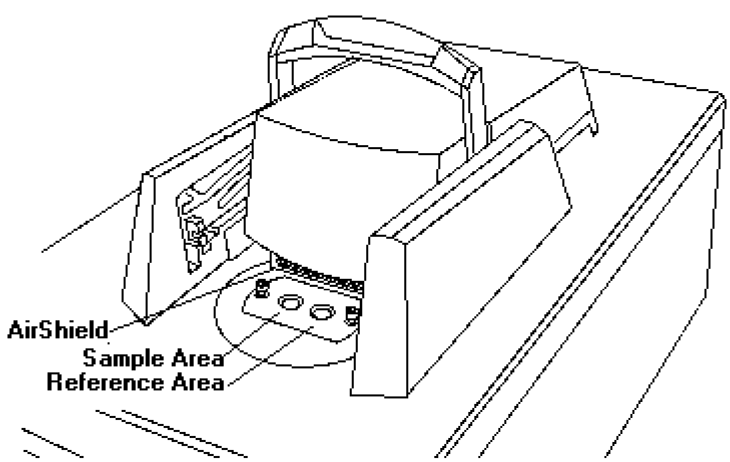
1. Loosen the black ring on the adjustable control panel's pole by turning it counterclockwise. Swing the panel 90° or enough so that it is out of the way of the lid when lifted.



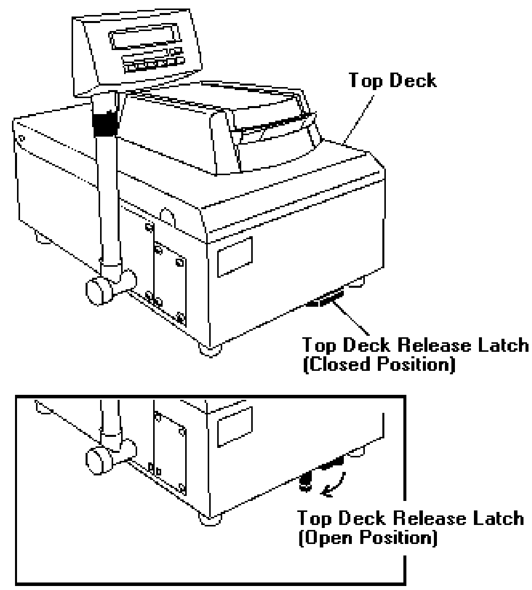
2. Lift the handle on the sliding sample holder enclosure cover up to disengage the cover.



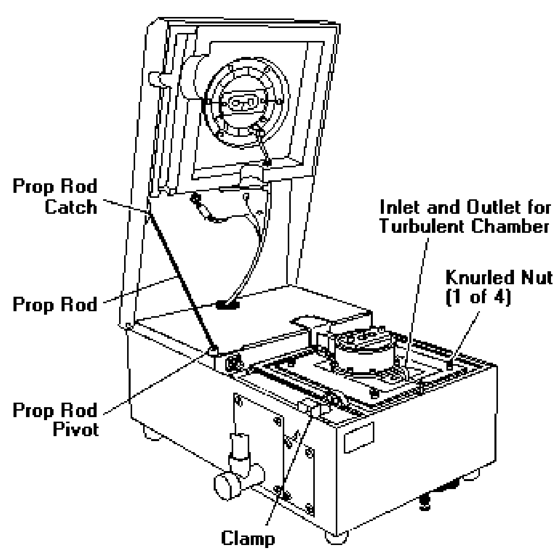
3. Slide the cover back as far as possible.



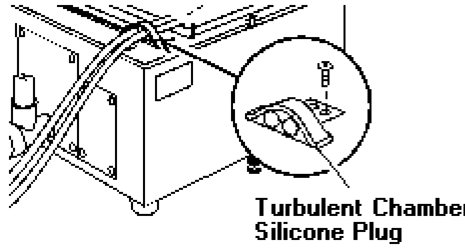
4. Pull the release latch at the bottom of the Pyris 1 DSC out and push it all the way to the left to unlock the analyzer's lid.



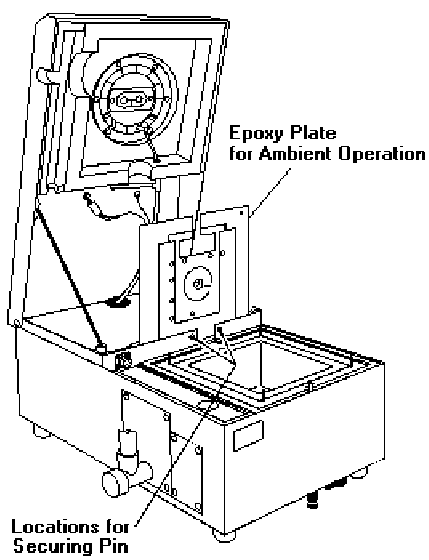
5. Lift the lid up and prop it up with the prop rod by inserting the rod in the prop rod holder on the underside of the analyzer lid.



6. Remove the two Posidrive screws from the turbulent chamber's silicone plug on the left side of the analyzer deck. (This plug is bell-curve shaped.) Lift the plug off of the deck. The Tygon tubing should remain in the plug.



7. Remove the two Posidrive screws holding down the clamp on the left side of the epoxy plate for ambient operation. Remove the clamp.
8. Lift up and slide back the small cover at the front of the electronics compartment that covers the tubes and electrical connections leading to the sample holder.
9. Remove the four brass knurled nuts from the top of the epoxy plate for ambient operation.
10. Remove any sample pans, covers, and sample holder lids from the sample holders before lifting the epoxy plate.
11. Slowly lift the epoxy plate up and off of the screws by holding onto the sample holder with both hands. After the plate clears the screws, begin to angle the back of the plate down while continuing to lift it up. **BE CAREFUL** of the tubing attached to the sample holder.
12. Align the two holes in the epoxy plate with the holes in the front of the groove in the electronics compartment. Gently lower the back edge of the epoxy plate into the groove.



13. Insert the retainer pin through the holes on the right-hand side. This will keep the epoxy plate in place when the turbulent chamber is removed.
14. Remove the insulated bolt securing the turbulent chamber to the sample holder block and remove the chamber.
15. Clear the turbulent chamber from the Pyris 1 DSC.
16. Store the turbulent chamber and silicone plug, with tubing attached to the chamber and going through the plug, for future use.

The Pyris 1 DSC is now ready for installation of a cold finger or an Intracooler. If you are preparing your system for using the CryoFill, return to **Removing the Epoxy Plate for Ambient Operation.**

Install the Epoxy Plate for Subambient Operation

To use the CryoFill Liquid Nitrogen Cooling System, you must install the epoxy plate for subambient operation when converting your system from ambient back to the CryoFill.

1. Locate the epoxy plate for use with the CryoFill LN2 Cooling System and place it over the standoffs that hold the plate.
2. Adjust the plate so that the liquid nitrogen exhaust hole is aligned with the hole in the small epoxy plate and the exhaust tube.
3. Secure the epoxy plate with the four knurled nuts.
4. Connect the bulkhead cable to the bulkhead receptacle.
5. Replace the sample holder with cold finger attached into the epoxy plate for subambient operation and secure it with the 8 Phillips head screws.
6. Replace the rear clamp used to secure the sample holder using the two Posidrive screws.
7. Replace the small plate that covers the electrical connections to the sample holder.
8. Close the analyzer lid by lowering the prop rod and placing it on the top of the electronics chassis.
9. Press down on the lid and push the hood latch all the way to the right to lock the lid.
10. Slide the drip tray underneath the left side of the analyzer where the exhaust vent and transfer are located. The drip tray traps any water from frost that might build up on the transfer line.

Connect the CryoFill Control Box to Pyris 1 DSC

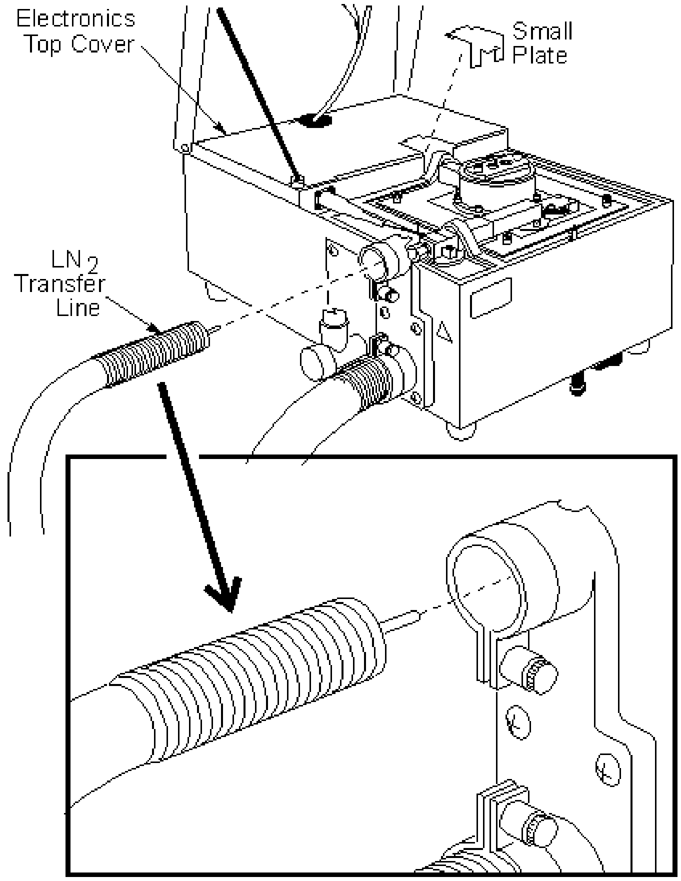
Since the CryoFill LN2 System was initially installed by a Perkin Elmer service engineer, when you switched your system to ambient mode, you should have followed the instructions in **Disconnect the CryoFill Control Box from Pyris 1 DSC**. If that is the case, the cord and cables should still be connected to the CryoFill LN2 control box and the LN2 storage tank should be in the area. You can now connect the CryoFill LN2 control box to your Pyris 1 DSC.

1. Take the CryoFill control cable (N537-0755) attached to the control box and attach the loose male end to the back of the Pyris 1 DSC at the CryoFill port.
2. Take the power cable attached to the receptacle on the control box and plug it into an electrical outlet.
3. Take the liquid nitrogen transfer line and connect the end without the black cap to the Pyris 1 DSC by performing the following steps:
   a. Loosen the black knob next to the top hole in the mounting assembly.
   b. While holding the transfer line horizontally, insert the stainless steel needle all the way into the Teflon septum in the top hole of the transfer line mounting assembly.

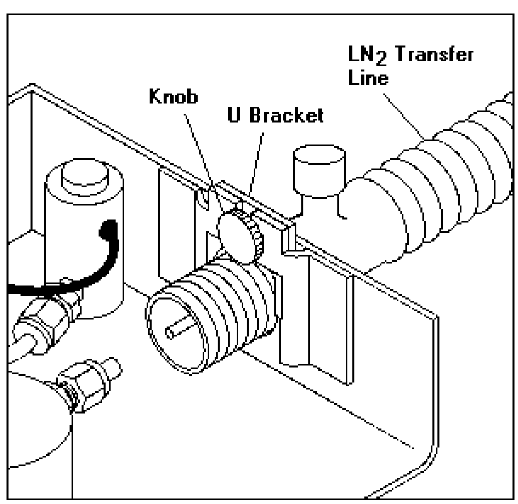
      CAUTION: Be sure that the transfer line is pushed all the way into the septum and the black hose covers the septum.

   c. Adjust the black knob on the mounting assembly as needed to allow adjustment of the transfer line.

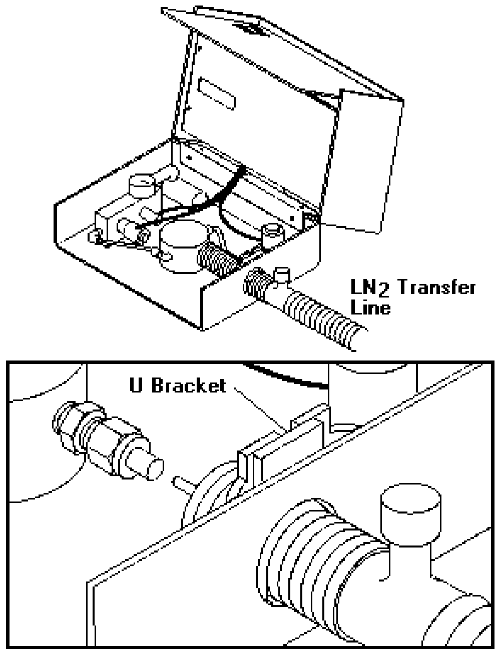
d. Tighten the black knob.



4. Make sure the liquid nitrogen exit plug is not in the control box.
5. Loosen the knob securing the U-bracket on the LN2 control box and then remove the U-bracket.



6. Insert the LN2 transfer line into the septum on the control box.

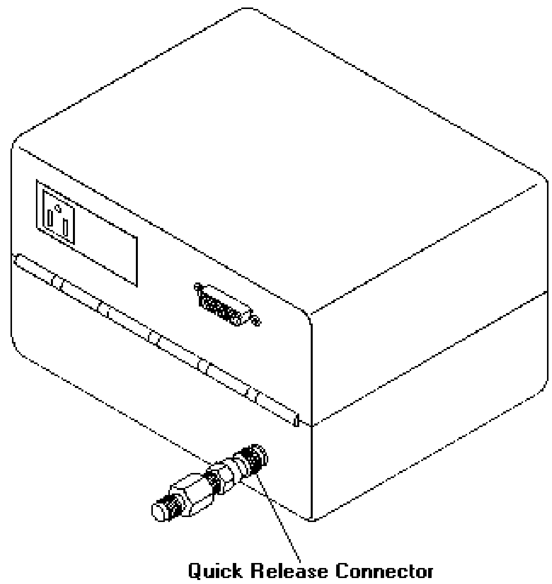


CAUTION: Be sure that the transfer line is pushed all the way into the septum and the black hose covers the septum.

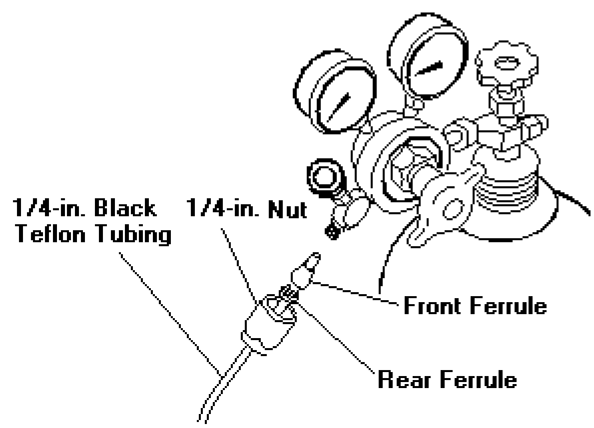
Connect Dry Nitrogen Gas to CryoFill Control Box

In setting up the Pyris 1 DSC for subambient operation using the CryoFill LN2 Cooling System, the final step is connecting the dry nitrogen gas to the CryoFill control box. The dry nitrogen gas is necessary to pressurize the liquid nitrogen tank at 69 kPa (10 psi). The gas supply should have been prepared by the Perkin Elmer service engineer when the CryoFill System was initially installed.

1. Connect the dry nitrogen pneumatic gas connection to the Quick Connector at the back of the control box.



2. Connect the other end of the tube, with the 1/4-in. Teflon tubing, to the gas cylinder.



3. Turn on the power to the Pyris 1 DSC.
4. Turn on the power to the CryoFill control box.
5. At the adjustable control panel, press the **Menu** button to display DSC OPERATION on the display panel.
6. Press the **Menu** button to display CLEAN FURNACE?
7. Press either the **Scroll Down** or the **Scroll Up** button three times. You should see CryoFill LN2 ACCY on the display.
8. Press the **Menu** button to see TURN CryoFill ON?
9. Press the **Enter** button to turn the CryoFill on and return to the parent menu.
10. Press the **Scroll Up** button to go to the Cover Heater display.
11. Press the **Menu** button to display TURN HEATER ON?
12. Press the **Enter** button to turn the cover heater on and return to the parent menu.

13. Press the **Scroll Up** button to go to the AirShield display.
14. Press the **Menu** button to display TURN AirShield ON?
15. Press the **Enter** button to turn the AirShield on and return to the parent menu.
16. Press the **Exit** button to return to the default display.

Maintenance

The Pyris 1 DSC needs little routine maintenance other than proper treatment as a sensitive electromechanical device. Occasionally, the sample holder cups may become coated with sample residue and a cleaning procedure may be necessary. The guard ring inserts may also become coated with residue. They can be wiped with a cotton swab moistened with isopropyl alcohol or an appropriate solvent.

See the topic on how to **Insert and Remove the Guard Rings** in Pyris Multimedia Presentations Help.

The following topics contain information on maintaining your Pyris 1 DSC:

- **Cleaning the Sample Holder**
- **Sample Holder Treatment**
- **Removing/Replacing the Sample Holder**
- **Removing/Replacing the Sample Holder Cover O-Ring**
- **Cleaning the Sliding Sample Holder Enclosure Cover**
- **Cleaning the Furnace**

Cleaning the Sample Holder

1. Since the sample holder assemblies are the hottest components in the cavity area, they will usually remain clean and free of condensates. However, samples may ooze out of sample pans and accidentally spill into the sample holder cup. Such materials should be removed at low temperatures whenever possible by using tweezers or moistened cotton swabs.
2. Solvents should not be applied directly in the holder cup. Moisten a cotton swab with solvent (e.g., ethanol or isopropyl alcohol or other appropriate solvent) and then clean the sample cup or upper portion of the cavity wall by using the cotton swab. Periodically check the underside of the sliding sample holder enclosure cover for the Pyris 1 DSC, and, if necessary, clean it in a similar fashion.
3. Any residual sample that has carbonized may remain in the sample holder cup after it has been cleaned with solvent. To remove the residue, leave the sliding sample holder enclosure cover open to expose the holders to the air. While observing the residue, program the DSC to 600°C at 100°C/min. Most carbonaceous or organic materials will burn off in the neighborhood of 600°C. If necessary, leave the instrument temperature at 600°C for not more than 5 – 10 minutes to burn off the residue.
4. Materials not free of metal or metal-containing compounds may irreversibly alloy with the sample holder. If some alloying has occurred, the holder will remain usable, although minor peaks may appear on the baseline at high temperatures.

Sample Holder Treatment

Handling the sample holder correctly is important to the proper operation of the sample holder and subsequently the DSC. The DSC sample holder assemblies are rugged and should last indefinitely if treated as any sensitive electronic device should be treated and if used as directed. The sample holder’s performance will decline or fail for any of the following reasons:

- **Application of Excessive Mechanical Force**
- **Exceeding Melting Temperature of Containers**
- **Mechanical Breaking or Shorting of Sample Holder Leads**
- **Chemical or Physical Attack of Sample Holder Materials by Samples**

Application of Excessive Mechanical Force

The holders are mounted on a hollow platinum-iridium post that may bend or break off if excessive vertical or horizontal force is applied to the assembly. Do not attempt to force sample holder lids into the cups. Always reshape any sample holder lids that do not fit into the sample cups without the use of excessive mechanical force.

Exceeding Melting Temperature of Containers

Aluminum sample pans will melt and alloy with the platinum-iridium holders at the melting point of aluminum. **DO NOT USE ALUMINUM SAMPLE PANS AT TEMPERATURES ABOVE 600°C.**

Mechanical Breaking or Shorting of Sample Holder Leads

The heater and sensor leads to each sample holder assembly are thin platinum ribbons that extend at right angles from the cup a short distance toward the cavity wall. Then they bend downward vertically to where they are soldered to feed-through posts at the bottom of the cavity wall. When looking straight down into the cavity, these leads should be visible at the point where they emerge from the cup. The leads may be partly covered by small sections of insulating aluminum oxide cloth protuding from the interior of the sample holder. The leads must never be disturbed or they may break and cause an open circuit or may wipe against the sample holder and cause a short circuit.

WARNING: **The possibly catastrophic consequences of disturbing the sample holder leads emphasize the importance in exercising great care when cleaning or “poking around” the cavity area. Never use a cotton swab or similar object to clean the cavities below the level of the sample cup. An effective cleaning tool is a No. 9 cork borer (0.5 in. i.d.) that is wrapped first with double-sided adhesive tape and then with a layer of slightly moistened paper towel. Insert this cleaning tool vertically all the way into the cavity and rotate it to clean the wall of condensates or other contamination. A 0.5-in.-i.d. tube will not touch the sample holder leads but will make good contact with the cavity walls.**

- **Open-Circuited Sensor:** The sensor will have infinite resistance and the instrument will react as if a malfunctioning sample holder is at “infinite temperature,” which means that no program power will be provided to the holders so that they will not heat.

- **Short-Circuited Sensor:** The sensor will have zero resistance and the instrument will react as if the sample holders require program power to elevate them to the program temperature. In trying to meet this demand, the instrument will apply maximum heater power to both holders and will drive them ballistically to their maximum temperatures. Both holders will glow red hot, and, if aluminum pans are present, they will melt and destroy the holders.
- **Open-Circuited Heater:** The sample holder will not heat and the sensor will detect its abnormally low temperature. The average power circuit will apply power to drive the average temperature of the sample and reference holders to the desired program temperature. Therefore, the reference holder will overheat, and, if the program temperature is high enough, it may heat to maximum temperature. If aluminum is present in the reference holder, it may melt and destroy the holder.

Chemical or Physical Attack of Sample Holder Materials by Samples

All the materials used to construct the sample holders (platinum–iridium, pure platinum, and aluminum oxide) are subject to chemical attack by very few chemical materials.

CAUTION: Platinum–iridium and platinum will alloy with most metals at sufficiently high temperatures.

Chemical attack may produce discoloration or spotting of the sample holder and, in severe cases, may destroy it. Metals, samples containing metals, or samples that may decompose to metallic products should not be heated to temperatures much above their melting points. The proper sample containers must be used. Since gold also alloys readily with most metals, never use gold or platinum pans for metal samples. Use aluminum pans for such samples when programming the temperature up to 600°C. Above this temperature use graphite pans. Aluminum oxide sample containers, tantalum pans, and others that can be homemade, may also be used.

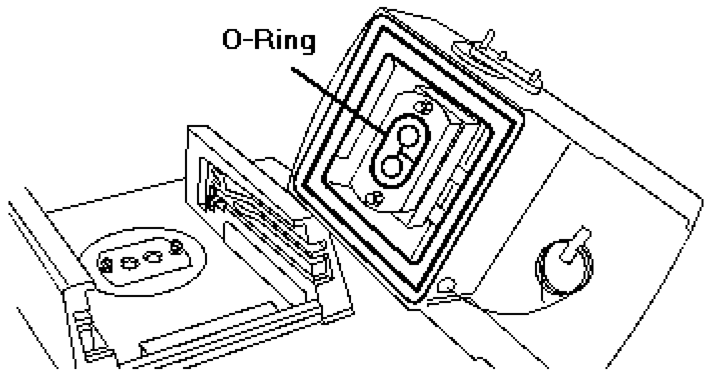
Cases of sample holder failure caused by deposition of carbonaceous vapors or other conducting vapors on electrical components of the sample holders should occur rarely, if at all. Condensation on the relatively hot sample holders is unlikely. The purge gas flow tends to sweep vapors up and away from the leads, and sample sizes of a few milligrams will usually not generate large enough quantities of condensable vapors per unit time to saturate the surrounding atmosphere. However, it is possible to short out the sample holder in this way by using excessive sample sizes, programming at excessive speed, using a low purge flow rate, and programming to excessively high temperatures. Always avoid mistreating the sample holders for maximum safety and maximum lifetime of the sample holder assembly.

Removing/Replacing the Sample Holder

SAMPLE HOLDER REPLACEMENT IS BEST PERFORMED BY A PERKIN ELMER SERVICE ENGINEER.

Removing/Replacing the Sample Holder Cover O-Ring

The sliding sample holder enclosure cover has a silicone O-ring in the side that faces the sample holder:



This O-ring should not need any maintenance. It is made of strong material so cleaning solvents will not bother it. However, if it does degrade over time and needs replacing, follow these steps:

1. Lift the handle of the sliding sample holder enclosure cover up until it stops.
2. Push the cover back along the rails until it stops.
3. Unlatch the analyzer lid.
4. Lift the lid up and, while doing so, pull the sample holder enclosure cover forward to close it. Push the handle down to lock the cover.
5. Lift the prop rod off the top of the electronics compartment and prop up the analyzer lid by putting the end of the rod in the hole on the left side of the lid.
6. Remove the O-ring from the recessed channel at the bottom of the enclosure cover using a sharp object such as a thin screwdriver.
7. Install the new O-ring in the channel.
8. While holding up the lid, place the prop rod back on top of the electronics compartment.
9. While slowly bringing down the lid, push the sliding sample holder enclosure cover handle up and slide the cover back until it stops.

CAUTION: When opening and closing the Pyris 1 DSC lid, the sliding cover MUST be back, exposing the sample holder.

10. Bring the lid all the way down and engage the latch to lock it.
11. Bring the sliding cover forward and push down the handle to lock it into place.

Cleaning the Sliding Sample Holder Enclosure Cover

Over time the underside of the sliding sample holder enclosure cover, like the sample holders, may need to be cleaned. Periodically check this area, along with the sample holders, and, if necessary, clean it. Cleaning should be done at low temperatures whenever possible by using tweezers or cotton swabs moistened with an appropriate solvent such as isopropyl alcohol. To clean the sliding sample holder enclosure cover, perform the following steps:

1. Turn the power to the Pyris 1 DSC off.

2. Loosen the black ring on the adjustable control panel's pole by turning it counterclockwise. Swing the panel 90° or enough so that it is out of the way of the lid when lifted.
3. Lift the handle on the sliding sample holder cover up to disengage the cover.
4. Slide the cover back as far as possible.
5. Pull the latch at the bottom of the Pyris 1 DSC out and push it all the way to the left to unlock the analyzer's lid.
6. Pull the sliding cover forward and push the handle down to relock the cover.
7. Lift up the analyzer's lid and prop it up with the prop rod by inserting the rod in the prop rod holder on the underside of the lid.
8. Moisten a cotton swab with a solvent such as isopropyl alcohol and clean the area. Make sure no particles fall down into the sample holder area. You may also want to remove the O-ring to clean that area as well.
9. When finished cleaning, remove the prop rod from the holder and place it on top of the electronics compartment. Make sure that it is not lying across the recess on the front left side of the compartment top. That accommodates the prop rod holder in the lid.
10. Lower the lid but **DO NOT TRY TO LOCK IT**.
11. Lift the handle on the sliding cover and push the cover back as far as it will go.
12. Push the latch at the bottom of the analyzer to the right and then push it in to lock the lid.
13. Pull the sliding cover forward and push the handle down to lock the cover in place.

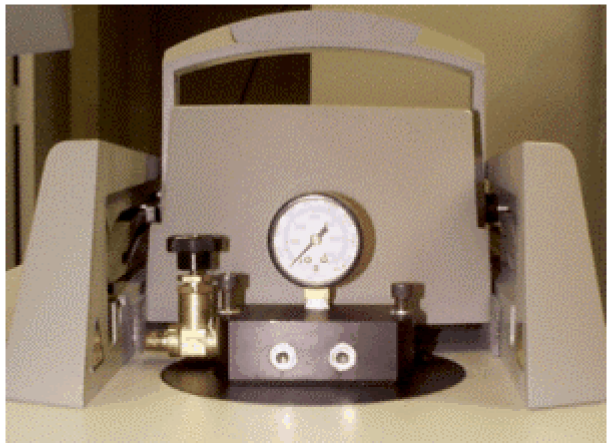
Cleaning the Furnace

The Pyris 1 DSC furnace can be cleaned by using the Clean Furnace procedure accessed either from the **Clean Furnace** button on the control panel or the Clean Furnace option on the adjustable control panel's menu (see the topic in **Adjustable Control Panel**). The Clean Furnace procedure applies only if the residue is organic and is known to be free of metal or metal-containing compounds.

Accessories

Pyris 1 DSC High Pressure Cell

The Pyris 1 DSC High Pressure Cell is an accessory for performing heating, cooling, and isothermal measurements at elevated pressures. The measuring system operates with two independently controlled sample holders which are made of a platinum–iridium alloy and is incorporated in the Pyris 1 DSC.



The sample enclosure block is pressure-sealed by use of a high-pressure cell cover. The pressure of the accessory is 0 – 4200 kPa (0 – 608.69 psi; 0 – 42 bar), while the temperature range of the cell is 40 – 500°C (104 – 932°F). Allowable purge gases for the system include oxygen, argon, helium, carbon dioxide, air and any other inert or reactive gas. An ambient liquid cooling system (e.g., turbulent chamber) can be attached to the sample holder.

WARNING: **Pressure above 4200 kPa (6.8.69 psi; 42 bar) will damage the sample holder assembly and the pressure gauge and may cause personal injury.**

- **Safety Precautions**
- **Installation**
- **Calibration**
- **Operating with the Pyris 1 DSC High Pressure Cell**
- **Maintenance**
- **Parts**
- **Troubleshooting**

Safety Precautions for the High Pressure Cell

WARNING: **Do not use explosive gases or gases that form explosive mixtures with air to pressurize the Pyris 1 DSC high pressure cell. Do not operate the device in an explosive atmosphere.**

The following precautions must be observed when using the high pressure cell on the Pyris 1 DSC:

- Pressure should not exceed 4200 kPa (608.69 psi; 42 bar).
- Do not modify the high pressure cell in any way.
- Clearly label gas cylinders and their contents as well as lines leading from them. Confusion can result in dangerous reactions: gas poisoning, fire, or explosion.
- Ensure that only authorized and properly trained personnel use the device.
- Store cylinders in accordance with the regulations and standards applicable to your locality, state, and country.
- When cylinders are stored in storage rooms, the storage room should be well ventilated and dry. Ensure that there is adequate ventilation to prevent the formation and accumulation of dangerous gases. This is particularly important in small or confined areas.
- Do not store cylinders near elevators, aisles, or in locations where heavy, moving objects may strike or fall against them.
- Use and store cylinders away from exits and exit routes.
- Locate cylinders away from heat sources, including heat lamps. Compressed gas cylinders should not be subjected to temperatures above 52°C (130°F).
- When storing cylinders outdoors, they should be stored above ground on a suitable floor and protected against temperature extremes (including the direct sunlight).
- Store cylinders in the upright position, fastened securely to an immovable bulkhead or permanent wall.

Handling Cylinders

- Use a suitable hand truck to move cylinders after making sure that the container cap is secured and the cylinder is properly fastened to the hand truck.
- Use only approved regulators, tubing, and gas line connectors. When connecting fittings, make sure the proper pressure regulator provided for each type of gas is used.
- Place gas lines where they will not be damaged or stepped on and where objects will not be dropped on them.
- Do not refill gas cylinders.
- Check the condition of the gas lines and connectors regularly. Broken and leaking gas lines can whip around violently and cause injuries. The gas streaming from them can cause poisoning, fires, or explosion.

Safety Checks

1. Before pressurizing the system, place the high pressure cover on the cell.
2. Secure the cover with the two bolts.
3. Close the pressure release valve.
4. Pressure the system slowly, using the pressure regulator on the gas cylinder.

5. Release pressure by shutting off the gas at the cylinder and then slowly open the pressure release valve.

NOTE: A wrench supplied with the accessory may be required to open the pressure release valve.

High Pressure Cell Installation

Unpacking

Carefully unpack the Pyris 1 DSC High Pressure Cell kit. Keep the packing materials for possible future storage or reshipment.

Requirements

WARNING: **The Pyris 1 DSC High Pressure Cell must not be operated in an explosive atmosphere.**

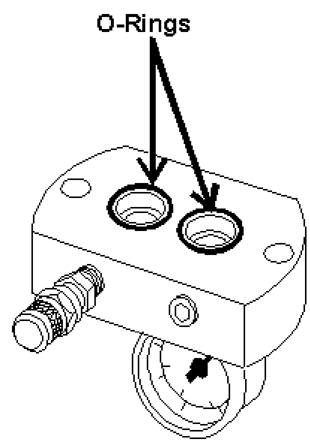
The following items are required for installing and operating the high pressure cell:

- 1 gas cylinder (connection to a central gas supply is also possible)
- 1 pressure regulator
- gas supply tubing

Connecting the Gas Supply

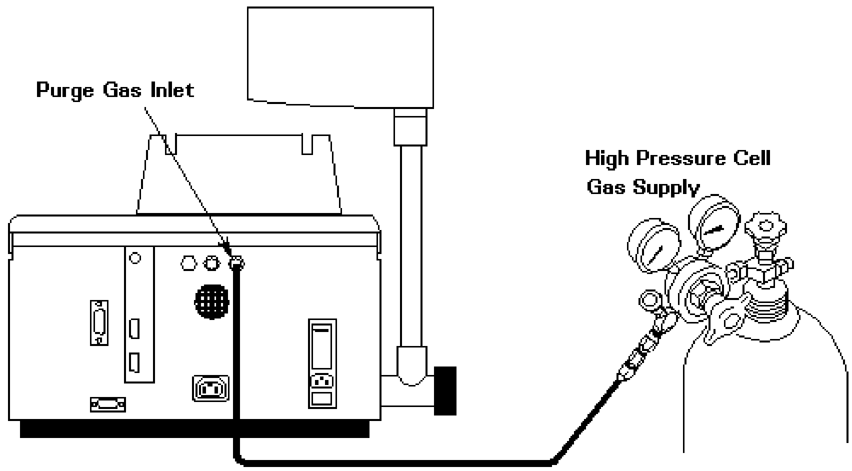
Observe the local safety rules when installing and operating gas cylinders. In order to connect the gas supply to the cell, perform the following steps:

1. Place the O-rings (P/N 0992-1024) into the grooves in the bottom of the high pressure cover.



2. Connect the “A” restrictor (P/N 0154-1496) to the regulator on the gas cylinder.
3. Connect the pressure snubber (P/N 0992-3395) to the “A” restrictor.

4. Connect one end of the high pressure gas supply tubing (P/N 0992-3173) to the pressure snubber.
5. Connect the other end of the high pressure gas supply tubing to the purge gas connector on the back of the Pyris 1 DSC.



Connecting an Ambient Cooling System

During an extended heating period, the temperature of the entire sample enclosure block rises. This may alter the baseline and the results obtained. Connecting a turbulent chamber or a cold finger to be used with ice water to the bottom of the sample holder solves this problem.

The Pyris 1 DSC is shipped with the cold finger in place. If the cold finger has been removed, see one of the topics listed below for instructions on how to switch back to either the cold finger or the turbulent chamber.

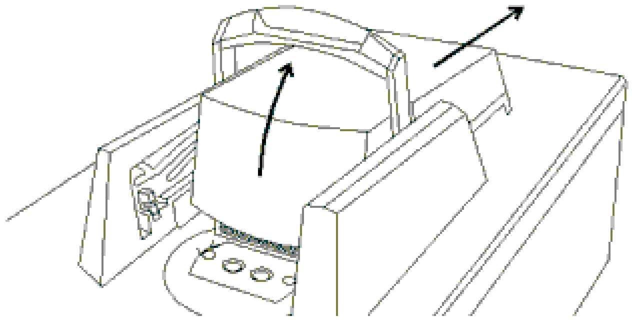
- **From Subambient to Ambient Operation**
- **Install the Turbulent Chamber**
- **Install the Cold Finger**

Once the turbulent chamber is set up, i.e., the supply and return lines are attached at both ends, adjust the flow rate on the coolant supply to obtain the necessary flow rate to cool the cell. Once the cold finger is in place, fill the reservoir with cool tap water.

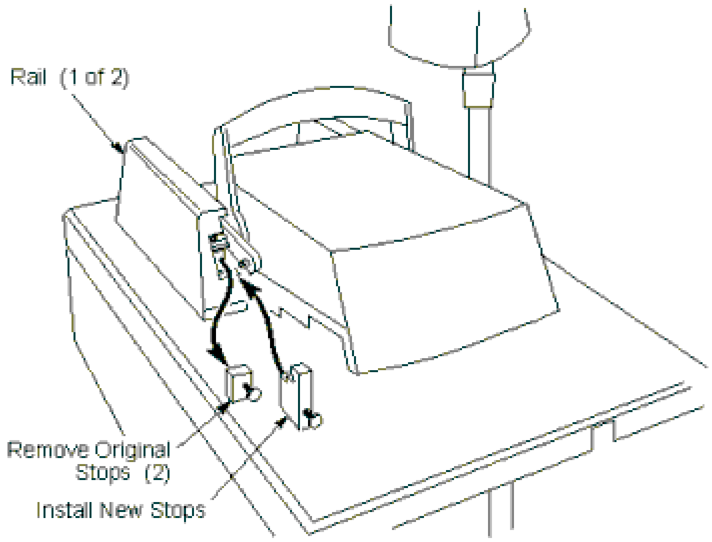
NOTE: Any noncorrosive, noncombustible fluid may serve as the coolant.

Connecting the HPC Cover to the Sample Holder

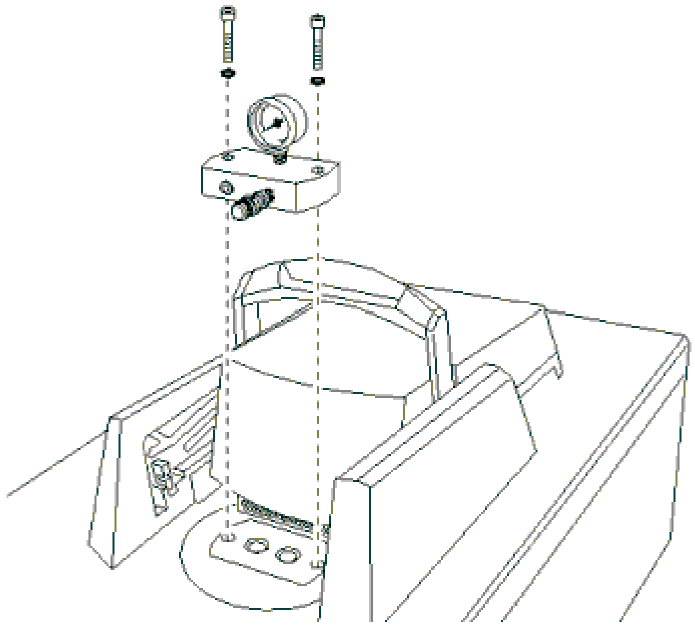
1. Make sure that the analyzer's deck is latched closed. Lift the sliding sample holder enclosure cover handle and slide the cover back to expose the sample holder.



2. Unscrew the sample holder cover retainers in the back of the cover.
3. Install the black exterior retainers in place of the retainers removed in step 2.



4. Push the latch at the bottom of the Pyris 1 DSC to the left and lift up the deck. Place the prop rod in position to keep the lid up.
5. Remove the four retaining screws from the foam insulation ring around the sample holder enclosure block.
6. Remove the two set screws from either side of the sample holder block.
7. Remove the clasps and spring washers.
8. Remove guard ring inserts from the sample holders.
9. Reinstall the foam insulation ring.
10. Close the analyzer deck and push the latch to the right.
11. Prepare and install the aluminum sample holder covers.
12. Place the high pressure cover on top of the sample holder, aligning the screw holes in the cell with those in the sample holder.
13. Place one shaft spacer (P/N 0990-7842) on each bolt (P/N 0991/3466).
14. Insert the two bolts into the holes on the high pressure cover. Tighten the bolts to a specified torque (120 in. lb.) using the torque wrench provided (P/N 0992-3394).



15. Close the pressure release valve on the high pressure cover.
16. If a flow controller is attached to the cell, close the valve going to the controller.
17. Open the valve on the gas cylinder. Slowly increase the pressure until the desired pressure is reached. Do not adjust the pressure above 4200 kPa (608.69 psi; 42 bar).
18. Watch the pressure increase on the pressure gauge. Build up pressure to 4200 kPa maximum.
19. Close the pressure release valve on the gas cylinder. Observe the pressure reading on the pressure gauge. The pressure displayed on the gauge must not drop. If gas leaks out of the pressure release valve, tighten the pressure release valve using the wrench provided.
20. If the cell is still leaking, make sure that the high pressure cover is placed correctly on the sample enclosure block and that the O-ring in the cover are seated correctly and are not damaged.
21. Slowly open the pressure release valve. Depressurize the sample holder enclosure fully.

WARNING: **Never try to pressurize the cell without the high pressure cover secured.**

WARNING: **Never open the high pressure cover when it is pressurized. Open the pressure release valve and depressurize the enclosure fully before opening the cover. Carefully read the pressure gauge on the high pressure cover to make sure there is no pressure.**

22. Once the cell has been depressurized, open the high pressure cover.
23. Solve any problems that occurred. Recheck the pressure sealing as necessary.

High Pressure Cell Calibration

We recommend that you calibrate the Pyris 1 DSC after installing the high pressure cell before performing any runs.

NOTE: When performing a calibration, use the same parameters that you plan to use for the analysis, i.e., pressure, atmosphere, and heating rate.

1. Prepare the sample holder covers and the enclosure.
2. Build up pressure in the sample holder enclosure.
3. Set up a method using the parameters you plan to use during a subsequent analysis.
4. Start the calibration of the Pyris 1 DSC.

Operating with the Pyris 1 DSC High Pressure Cell

Operating the Pyris 1 DSC with the high pressure cell attached consists of the following steps:

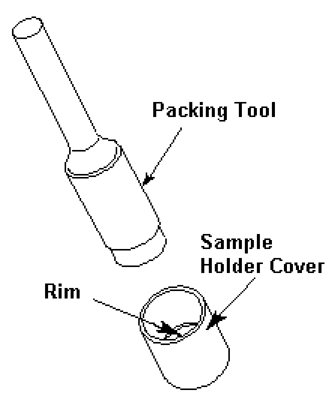
Prepare Sample Holder Covers and Enclosure

CAUTION: Do not hit, bend, or use force when working on the sample holders. The measuring and heating connections could be damaged. Do not scratch the sample holders since this could also damage them.

Before using the cell, be sure that the sample holder covers are clean and that they have been properly packed with glass wool. In order to properly prepare the sample holder covers, perform the following steps:

1. Check the sample holder covers and, if necessary, clean them using a cotton swab moistened with an appropriate solvent, e.g., acetone.
2. If the sample holders are already packed with glass wool, make sure that the glass wool is clean. If the glass wool is not clean, remove it with tweezers and replace it.
3. Using tweezers, place three fresh glass wool pads into both sample holder covers.
4. Pack the glass wool into the sample holder covers, making sure that it is completely under the rim of the covers. Use both ends of the black packing tool as needed to pack the glass wool.

CAUTION: Fibers from the glass wool must not protrude above the rim of the sample holder cover.



Preparing the Sample for the High Pressure Cell

1. Place the sample loading tray on top of the sample enclosure block.

   CAUTION: Do not drop the sample pans into the enclosure cavity when you insert or remove samples. This could damage the sample holders. Always use the sample loading tray when placing sample pans into the sample holders.

2. Use tweezers to place an empty pan and cover (if needed for your experiment) into the reference (right-hand side) holder.

   NOTE: When using sample pan covers, be sure that they are made of the same material as the pan.

   CAUTION: Do not use aluminum sample pans above 500°C (932°F) since the pans will alloy with the surfaces of the sample holders and damage them.

   CAUTION: Never touch the sample, pan, or cover with your fingers. Oils from the skin can affect the data. Always use tweezers to place the sample into the pan, the pans into the sample holders, and the covers onto the sample pans.

   CAUTION: Do not confuse the sample holder and the reference sample holder of the sample enclosure block. This will substantially alter the baseline. The sample holder is on the left and the reference holder is on the right, as you face the analyzer.

3. Place solid samples into a new pan using tweezers. Use a syringe or small glass rod for liquid samples. If necessary, place a cover on the sample pan.

   CAUTION: When analyzing unknown samples, use very small amounts (1 mg or less).

   NOTE: The sample should be weighed before placing the sample pan into the sample holder.

   NOTE: To obtain reproducible results, distribute the sample evenly over the bottom of the pan and place samples in the pan the same way for all runs.

4. Place the pan containing the sample into the sample holder.
5. Place the platinum sample holder covers in position.
6. Remove the sample loading tray.
7. Carefully place the prepared aluminum sample holder covers with glass wool over the sample holders and gently push them down as far as possible. Do not force them; they could become jammed in the sample holder block.

   NOTE: It is important that you use the same sample holder cover that was previously used over the sample holder and the reference holder each time. Do not mix them up.

Pressurizing the Sample Holder Enclosure

1. Place the high pressure cover on the sample holder enclosure.
2. Position the bolts in the two holes and tighten using the torque wrench provided.
3. Close the pressure release valve. If necessary, use the wrench.
4. If a flow controller is attached to the cell, close the valve leading to the controller.
5. Slowly open the pressure regulator on the gas cylinder to the desired pressure. Pressure should not exceed 4200 kPa.
6. Watch the pressure increase on the pressure gauge on the high pressure cell cover. Build up pressure to the desired value.
7. If a flow controller is used, slowly open and adjust its valve to obtain the desired pressure.
8. Begin the measurement when the pressure has stabilized.

Reducing the Pressure without a Flow Controller Attached

1. If you want to continue operating at a lower pressure, slowly open the pressure release valve.
2. Adjust the pressure on the pressure regulator of the gas cylinder to obtain the desired operating pressure.
3. Close the pressure release valve.

Reducing the Pressure with a Flow Controller Attached

1. Close the valve to the flow controller.
2. Slowly open the pressure release valve, adjust the pressure on the pressure regulator of the gas cylinder, then close the pressure release valve.
3. Open the valve to the flow controller and let the pressure stabilize at the desired new valve. Read the pressure on the pressure gauge attached to the high pressure cell cover.

Obtaining Data with Pyris 1 DSC and High Pressure Cell

1. Create your method in Pyris Software for Windows.
2. Prepare the sample holder covers.
3. Load the sample pans into the sample and reference holders. Use the sample loading tray when loading and removing pans.
4. Place platinum sample holder covers in position.
5. Place the aluminum sample holder covers with glass wool over the sample and reference holders. Be sure to use the sample cover for each holder that you used previously.
6. Place the high pressure cell cover on the cell and build up the pressure in the cell.
7. Start the method from the Pyris 1 DSC control panel.
8. At the end of the run, close the regulator valve on the gas cylinder.
9. Slowly open the pressure release valve on the high pressure cell cover.
10. Completely depressurize the sample enclosure block. Read the pressure from the pressure gauge attached to the high pressure cell cover.

WARNING: Never open the high pressure cell cover while it is pressurized.

11. Remove the aluminum sample holder covers.
12. Place the sample loading tray on the sample enclosure block and carefully remove the sample pans using tweezers.

CAUTION: Do not drop the pans into the enclosure cavity when removing or inserting. This could damage the sample holders. Always use the sample loading tray when the sample holders are exposed.

Maintenance of the Pyris 1 DSC High Pressure Cell

To clean the Pyris 1 DSC high pressure cell, perform the following steps:

1. Check the sample holders for contamination from samples.
2. Remove sample residue using tweezers or cotton swabs. Pay special attention to metal residues.
3. Be sure to remove the sample pans from the sample holders. Metal residues can destroy the sample holders.
4. Clean the sample holders with an appropriate solvent, e.g., acetone or ethanol. Do not pout the solvent directly into the holders; moisten cotton swabs with the solvent. Remove condensates with a suitable solvent.

CAUTION: Do not hit, bend, or use force when working on the sample holders. The measuring and heating connections could be damaged. Take care not to scratch the sample holders; scratches can damage them.

5. Organic residues can decompose. Heat up the sample holders to 500°C (932°F). This is done at ambient pressure without the high pressure cell cover in place.

High Pressure Cell Parts List

The Pyris 1 DSC High Pressure Cell Kit (P/N N537-0162) contains the following items:

NOTE: A regulator is needed for the gas cylinder but is not supplied.

Part Number	**Description**	**Quantity**
0992-4041	Dry box stopper screw	2
0992-4042	Dry box stopper	2
N537-0160	High pressure cell cover	1
N519-1123	Dome	1
B050-0724	Sample holder cover	2
B050-0722	Glass wool	1 pkg.
0992-1024	O-ring (Viton)	12
0992-3173	Tubing – flex high pressure	1
0990-3087	Connector – male	1
N537-3094	Sample loading tray	1

0154-1496	“A” restrictor	1
0992-3395	Pressure snubber	1
0991-3466	Bolt – high tensile	2
0990-7842	Shaft spacer	2
0990-3394	Wrench – torque	1
0990-7274	Wrench – open end	1
0993-6286	Operating Instructions	1

Troubleshooting for the High Pressure Cell

Here are some remedies if you experience some trouble with the high pressure cell:

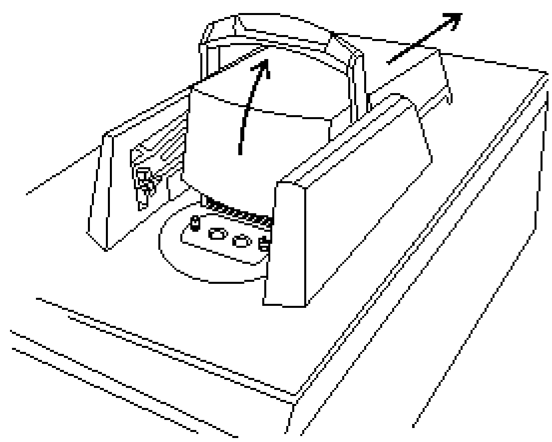
Error	Possible Cause	Remedy
Baseline changes substantially.	Glass wool, sample holder cover, or sample holders dirty.	Replace glass wool; clean sample holder covers and sample holders with solvent. Restore baseline and swap the sample holder covers.
Spikes in the curve.	Glass wool in sample holder covers dirty.	Replace glass wool or restore baseline.
	Sample enclosure block not closed correctly.	Check O-rings and pressure release valve.
	Pressure fluctuations. Connections or high pressure tubing not airtight. O-rings deformed.	Replace and connect tubing correctly. Check and replace O-rings.
	Pressure regulator of the gas cylinder defective.	Check and replace if necessary.
	Glass wool not fully under the rims inside the sample holder covers.	Check, replace glass wool correctly.
Noise on the baseline.	Glass wool, sample holder cover, or sample holders dirty.	Replace glass wool; clean sample holder covers and sample holders with solvent.
	Contaminated sample holder.	Clean holder with solvent or by heating to 600°C (1112°F).
	Vibrations	Place high pressure cell on a table free from vibration. Test the cell with pressure by measuring the baseline when the sample enclosure block is depressurized.

Pyris 1 DSC Flow Through Cover (P/N N537-4039)

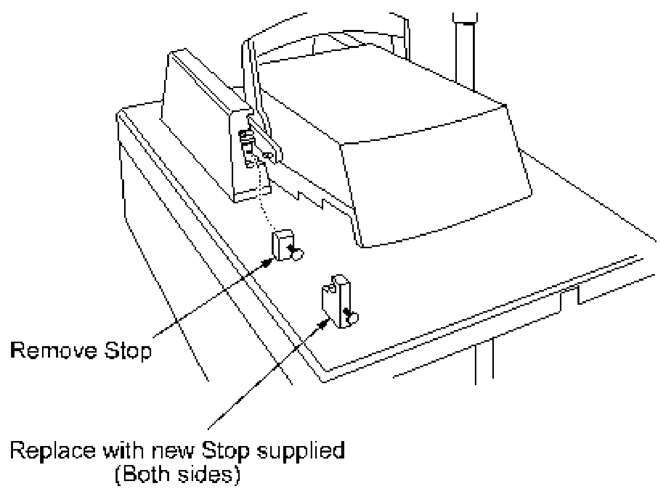
The Pyris 1 DSC Flow Through Cover is an add-on cover, not a replacement to the sliding cover. It provides a short path for volatiles or decomposition products and purge gas to exit from the sample holder directly through the top of the cover. This allows the use of more rapid purge rates or the collection of decomposition products with minimal condensation loss. It should be used with samples that undergo decomposition, dehydration, or oxidation in order to minimize condensation of reaction products in the Pyris 1 DSC block and to minimize the calorimetric effect from the evolution of products whose thermal conductivity is different from that of the purge gas.

The Flow Through Cover attaches easily to the Pyris 1 DSC as follows:

1. Slide the sample enclosure cover back until both guide arms are exposed:

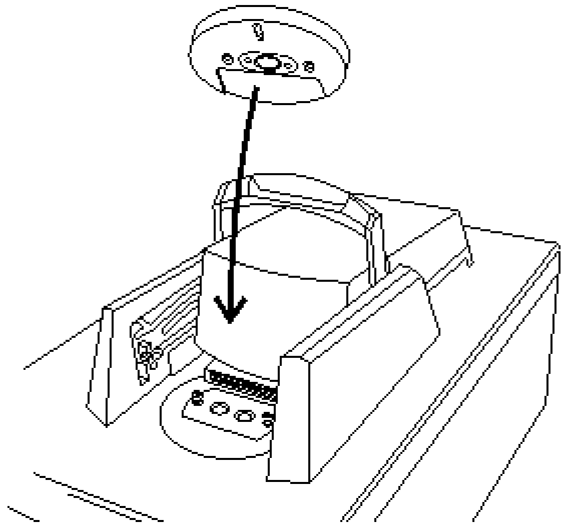


2. Remove the two retaining stoppers or blocks for the sliding enclosure cover at the back of the cover by unscrewing the screws and pulling the stops out.
3. Insert a screw into each new stop (P/N N537-4042) and insert the stop at the back of the enclosure cover.

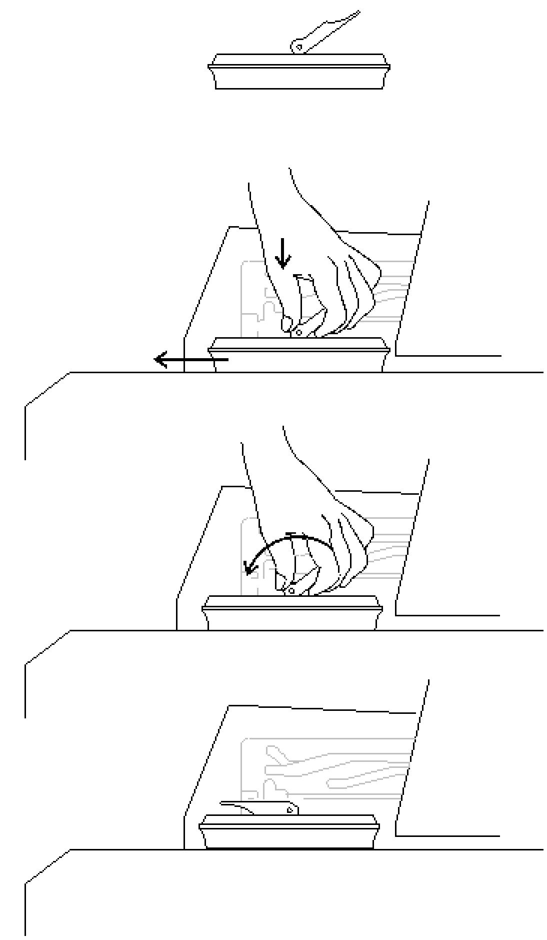


The first guide arm and bearing are behind the new blocks. The sliding enclosure cover should not move.

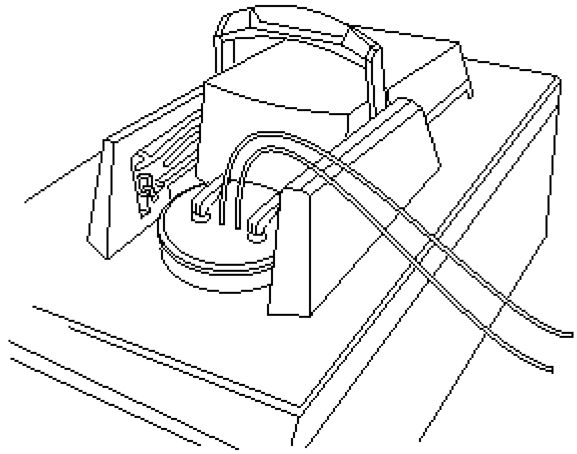
4. Position the flow through cover over the sample holder.



5. Using your thumbs, push down on both levers simultaneously and slide the cover toward the front of the instrument. Lock the cover in place by pushing the levers down.



6. Place the silicone exhaust lines on each of the vents on top of the cover and let the tubes hang off to the side.



7. Place the protective dome over the cover with the tubes exiting through the slot on the side of the dome. Lead both tubes to a laboratory vent or hood so that laboratory personnel will not have to breath potentially dangerous products of decomposition.

WARNING: **An efficient laboratory ventilation system to remove vapors that may be produced when performing analyses must be provided.**

8. Close off the normal purge gas outlet using a termination fitting.

It is recommended that the normal purge rate be increased from 20 to 50 cc/min to facilitate sweeping of reactive gases and other evolved products. Rates above 100 cc/min may be used but some degradation of performance (increased noise and temperature uncertainty) may be observed. This accessory is primarily for use in above-ambient investigations. Subambient operation is limited by the effects of condensation.

After installing the flow through cover and setting the desired purge rate, you should perform Temperature Calibration.

Operating with the Flow Through Cover

Here are some helpful operating points for decomposition analysis using the Pyris 1 DSC flow through cover:

- Use small sample sizes: 1 mg for polymer decomposition; 0.1 – 0.5 mg for explosives. Use slow heating rates if a large exotherm is expected. A large sample and a rapid heating rate may allow the reaction to run away. Under these conditions it may be possible to exceed the melting temperature of aluminum sample pans which may result in requiring an expensive procedure to replace the calorimeter assembly. For high temperature work, use platinum or ceramic sample pans.
- For quantitative analysis of reactions which evolve products, consider the use of a purge gas that minimizes the thermoconductivity effect. For example, if CO_2 is evolved, consider using CO_2 as the purge gas.
- For OIT testing, contact the Perkin Elmer Thermal Analysis Product Department for detailed information.
- The Pyris 1 DSC is not the instrument of choice for the study of decomposition reactions. In general, thermogravimetric analysis (TGA) is a better analytical technique for investigating

decomposition processes. The high-sensitivity performance of the Pyris 1 DSC may be adversely affected by the evolution of condensable or reactive products.

- It is advisable to terminate any decomposition reaction as soon as possible after the onset temperature is reached.
- For collecting the decomposition products, close off the reference outlet line or collect products from both sides. Note that there is no restriction between the sample and the reference outlets. Thus, the addition of a small restriction on the sample side will cause all purge gas to be vented through the reference side. In general, it is better to treat the two purge outlets in identical fashion so as not to introduce asymmetry into the purge system.
- For repeated use of the Pyris 1 DSC for decomposition analysis, it is recommended that you clean the underside of the Pyris 1 DSC cover. It may also be necessary to remove, clean, and replace the guard ring inserts.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

If you are going to use a TAGS or a GSA 7, you must install a filter dryer for each of the purge gases you will attach to the TAGS or the GSA 7. The filter dryer is installed between the gas tank and the TAGS. The newer filter dryer offered by Perkin Elmer is the Drierite Compressed Air Filter Dryer Accessory.

WARNING: FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F

- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl bowl
- Anodized aluminum head
- 1/4" pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

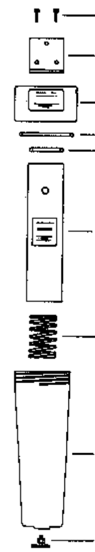
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this filter drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4" pipe fitting using Teflon (Registered DuPont Trademark) tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4" pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2-1/2" o.d. O-ring on top of cartridge.

8. Place the 4" o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD S0AP AND WATER. D0 NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis. It can be used with the Pyris 1 DSC.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the rear of the Pyris 1 DSC at the connector labeled Gas Selector Accessory.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Connecting a TAGS to a Pyris 1 DSC

The Thermal Analysis Gas Station allows you to use up to four gases for the purge gas. You can switch gases in the gas program of the method. This topic discusses how to connect the gas lines to the TAGS and Pyris 1 DSC.

Preparing the Laboratory for TAGS

The TAGS will accept the same laboratory conditions suited for the Pyris 1 DSC. Some precautions are

- Do not place the TAGS in direct sunlight or close to heating and cooling units.
- The temperature of the area should be between 10°C and 35°C.
- Relative humidity should be between 20% and 80% and noncondensing.
- The TAGS requires little bench space. The electrical power consumption is only 8 VA max. Therefore, it can be connected directly to the instrument's power line. The supply must be smooth, clean, and free of transient voltages over 40 V. The operating frequency is 50 – 60 Hz.

Unpacking the TAGS

The TAGS is shipped in one box and is surrounded by foam on all sides. To unpack the TAGS, follow the steps below:

1. Remove the power cable, the serial cables, and the manual from the box.
2. Remove the foam insert from the box.
3. Remove the upper foam piece.
4. Remove the TAGS from the box.
5. Remove all remaining packing material.

Installing the TAGS

Setting the Correct Voltage

Upon delivery, the TAGS is set at a voltage of 220/240 V. Changing the input voltage should be done at the AC inlet plug.

1. Remove the fuse holder with a small screwdriver by gently prying it out of the compartment. Once the holder is out far enough to grasp, slide the fuse holder out of the slot.
2. Rotate the fuse holder 180°.
3. Remove the fuse from the 220-V position (100 mA Slow Blow) and insert a 200 mA Slow Blow fuse for 110 V (it goes into the right side when "110 – 120 V" is in the upright position.

4. Reinsert the fuse holder.
5. Make sure that the arrow next to the voltage label is pointing at the white line below the fuse holder.

Connecting the Gases

The procedure below assumes that there are four gas supplies for the purge gas and that the gas supplies (and filter dryers, if used) are already connected.

1. Connect a length of 1/8-in. Teflon tubing from the Gas A supply to the input #1 connector on the rear of the TAGS.
2. Connect a length of 1/8-in. Teflon tubing from the Gas B supply to the input #2 connector.
3. Connect a length of 1/8-in. Teflon tubing from the Gas C supply to the input #3 connector.
4. Connect a length of 1/8-in. Teflon tubing from the Gas D supply to the input #4 connector.
5. Connect a length of 1/8-in. Teflon tubing to the OUT connector on the TAGS using a Swagelok fitting.
6. Connect the other end of the tubing from the OUT connector to the **Purge Gas Inlet** connector on the Pyris 1 DSC.

Connecting the Pyris 1 DSC, TAGS, and Computer

1. Attach one of the two 9 male/9 female RS232 cables provided with the TAGS to COM1 or COM2 port on the computer.
2. Attach the other end of that cable to the RS232 IN connector on the TAGS.
3. Connect the other 9 male/9 female cable to the RS232 OUT connector on the TAGS.
4. Connect the 9 male/25 female adapter to the other end of that second cable.
5. Attach the 25-pin end of the 9 male/25 male cable to the adapter.
6. Connect the 9-pin end of the cable to the **RS232 Computer** connector on the rear of the Pyris 1 DSC.

Turbulent Chamber

A cold water turbulent chamber can be used with the Pyris 1 DSC. Circulating baths and refrigerated liquid chillers can be attached to the turbulent chamber as well. The Pyris 1 DSC includes a turbulent chamber in its shipment. A turbulent chamber keeps the sample holder at a temperature in the range of 25°C. For optimum results, the flow rate of the coolant must be constant and there should be no circulating air bubbles in the system.

Pyris 1 DSC Part Numbers

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Spares Kit (N537-0100)

Part No.	Description	Quantity
0219-0041	Standard aluminum pans and covers	1 pkg. of 400
0250-6483	Teflon purge line tubing (1/8 in.)	11 ft
0250-6515	Tygon tubing for AirShield (1/4 in.)	10 ft
0250-6519	Tygon tubing	20 ft
0319-0033	Indium reference material	1
0319-0036	Zinc reference material	1
0401-0109	Acid brush	1
0419-0299	Platinum sample holder covers with holes	2
N537-0543	Turbulent chamber	1
0990-3094	Tube insert	2
0990-3212	Reducing bushing	2
0990-3428	NPT male connector (1/4 in. Swagelok)	2
0990-3434	NPT male connector (1/8 in. Swagelok)	2
0990-8134	Teflon thread sealant tape	1 pkg.
0990-8138	Tweezers (hooked)	1
0992-0008	Clamps for turbulent chamber tubing	2
0998-1613	Fuse, Slow Blow 3AG, 1.6 A, 250 V	2
0998-1626	Fuse, Slow Blow 3AG, 3 A, 250 V	2
0998-8986	Power cord (for North America)	1
0999-1641	Fuse, Metric Slow Blow, 1.6 A, 250 V	4
N519-0762	Calibration Sample Kit	1
N537-0134	Seal Assembly	1
N537-0135	Plate Assembly - Ice	1
N537-2169	Pack – Spares kit	1
N537-2178	Securing Pin	1
N537-2184	Aluminum funnel for inserting ice	1
N537-2186	Label	1
N537-3115	Siphon for removal of water	1
N822-1178	RS 232 cable	1

Pyris 1 DSC Autosampler

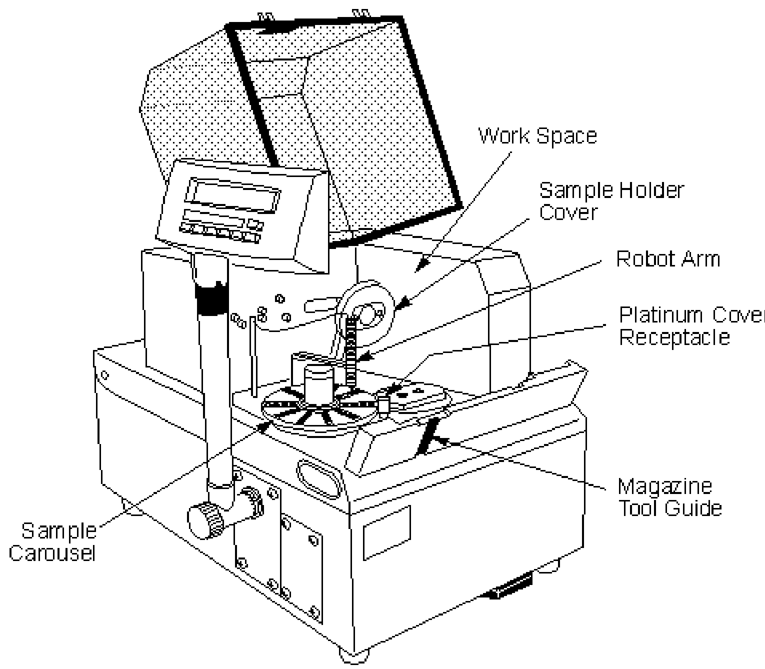
The Pyris 1 DSC Autosampler is an accessory that attaches to the top of the Pyris 1 DSC after removing the lid. The sample holder fits into the baseplate of the autosampler and is closed by a special sample holder cover built into the autosampler. It connects directly to auxiliary port on the Pyris 1 DSC via an RS 232 cable while the Pyris 1 DSC is connected to the computer by another RS 232 cable. The autosampler can also connect directly to the computer if there are at least two COM ports. With the Pyris Player software you can create play lists of steps that automate analyses. The autosampler's tray has 48 sample positions but with Pyris Player you can create play lists that contain steps to analyze up to 1000 samples.

Autosampler programs are set up and executed by the Pyris Player software. Play lists can be created that can run all 48 samples, perform tolerance tests on the results, and print out the data automatically. The Sample Group function in a play list is designed with autosamplers in mind. You can have all the sample pans tared and all the the samples weighed through the Sample Group page of the play list.

You control the autosampler through the Pyris software. Some functions can be performed from the Pyris 1 DSC adjustable control panel, however, you need to exit the Pyris 1 DSC application software before using the panel to control the autosampler.

The autosampler consists of the following major components:

- the working space, which includes the sample tray, robot arm, robot arm lifting mechanism, lever gear, platinum cover receptacle, and sample holder cover, is located under a protective cover
- the pneumatic system, which includes the switching valves for the mechanical movement of the robot arm, robot arm lifting mechanism, and sample holder cover
- the electrical system, which includes the power supply, control electronics, and RS-232 interface
- **rear panel** controls and connections



If you want to use a purge gas to purge the autosampler working space, you can connect a purge gas line to the rear of the autosampler. Connect the tubing from the purge gas source to the connector at the back of the autosampler.

The needle valve is used to regulate the amount of purge gas permitted into the diffuser. Initially, the needle should be closed all the way. Slowly open up the valve to permit purge gas in.

The entire Pyris 1 DSC autosampler can be tilted back as follows:

1. Loosen the upper black ring just below the adjustable control panel and rotate the control panel so that it faces to the left.
2. Make sure that all sample pans are removed from the tray and platinum lids are removed from the lid receptacle.
3. Make sure that the autosampler cover is locked in position with the two cover locks that are hidden by the horizontal front panel.
4. Pull the release latch at the bottom of the Pyris 1 DSC forward and push it all the way to the left. This unlocks the analyzer deck.
5. Carefully lift the deck up and secure it with the prop rod.

Observe the following precautions when you perform this procedure:

WARNING: **The autosampler and analyzer deck are very heavy. When tilting them up, hold the Pyris 1 DSC down with your other hand. Be absolutely certain the autosampler cannot fall backward by accident or drop down.**

WARNING: **Never try to lift the autosampler by the recess at the horizontal front panel. This can damage the tray mechanism.**

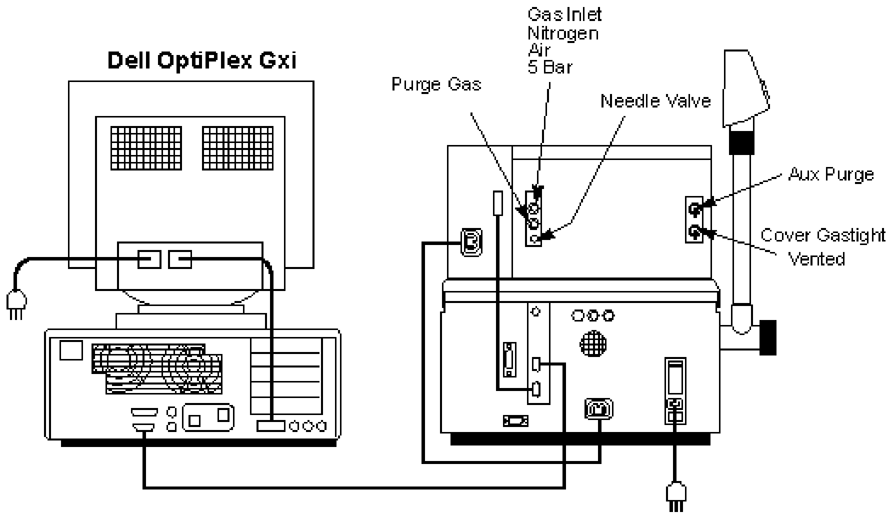
Opening the analyzer deck provides easy access to the sample holder and the interior of the Pyris 1 DSC for the installation of the turbulent chamber or accessories for subambient analyses, i.e., CryoFill or Intracooler.

The following topics discuss the Pyris 1 DSC Autosampler in more detail:

- **Warnings and Safety Practices**
- **How the Autosampler Works**
- **Sample Handling**
- **Running a Play List**
- **Troubleshooting**
- **Maintenance**

Rear Panel

The Pyris 1 DSC Autosampler rear panel contains the following controls and connections:



- power cord connection
- RS-232 connector used to connect to the AUX port on the Pyris or the COM2 port on the computer
- gas inlet connection for pneumatic gas (nitrogen or air, 5 bar) to operate the robot arm
- a purge gas inlet connector to connect the purge gas for the working space
- auxiliary purge switch to open or close the flow of the purge gas to the working space
- vent switch to open or close the vent to the working space
- needle valve regulates the flow of purge gas into the working space

Warnings and Safety Practices

WARNING: Be sure that all instrument operators read and understand the following precautions. It is advisable to post a copy of these precautions on or near the Pyris 1 DSC Autosampler itself.

The following precautions must be observed when using the Pyris 1 DSC Autosampler. These are in addition to the precautions that apply to the Pyris 1 DSC.

WARNING: Your Pyris 1 DSC Autosampler is designed to be installed by a Perkin Elmer Service Engineer. Do not try to perform any installation procedure beyond what is mentioned in this manual.

WARNING: Observe all local safety regulations when connecting the components of your Pyris 1 DSC Autosampler system to each other and to the local electrical supply.

⚠ **WARNING:** **Observe all local safety regulations concerning the handling of gas cylinders when connecting the gas supply to the autosampler.**

⚠ **WARNING:** **Never operate the Pyris 1 DSC Autosampler in an explosive atmosphere.**

CAUTION: When tilting the Pyris 1 DSC Autosampler back, be absolutely certain it cannot fall backward accidentally.

CAUTION: Never try to lift the Pyris 1 DSC Autosampler by the recess in the horizontal front panel. The tray could be damaged.

CAUTION: Make sure that all cables and tubes are clear when lowering the autosampler down to be closed.

CAUTION: Never use the Autosampler Control dialog box to control the autosampler while the autosampler is loading or returning a sample or reference pan.

How the Autosampler Works

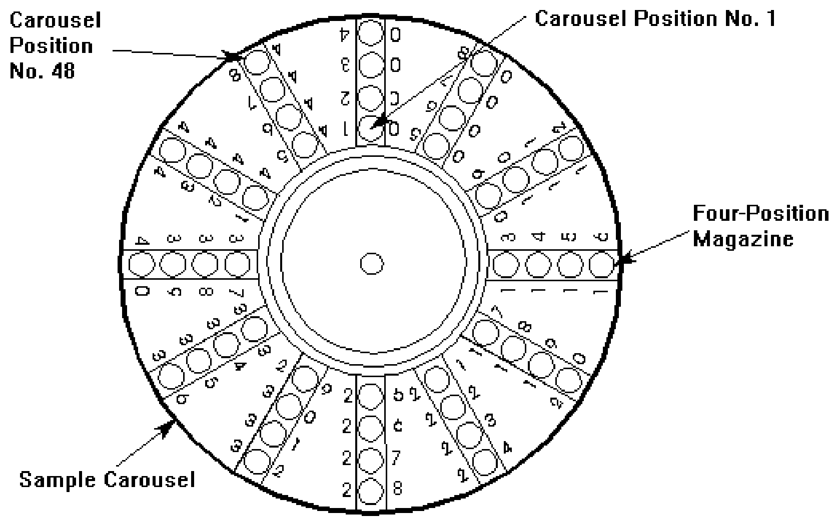
Working Space

The working space is covered by a transparent, air-tight clear plastic cover. The cover is closed tight by two locks accessible when the horizontal front panel is flipped forward. The cover permits observation of the movements of the mechanical components, protects the mechanical components and sample pans from dust, and allows the working space to be filled with dry, compressed air or nitrogen.

Filling the working space with dry, compressed air or dry nitrogen helps avoid condensation or ambient humidity on the sample pans, sample holder, and other mechanical parts during subambient analyses. The analyses can take place in an inert atmosphere (nitrogen), thus preventing air from penetrating the working space.

Sample Tray

The sample tray is driven by a stepper motor. Samples are loaded into sealed sample pans, which are then loaded into magazines. Each magazine can hold up to four sealed capsules; the sample tray holds 12 magazines. The tray can thus hold up to 48 samples at one time. The magazines are arranged radially on the tray; a concentric magnet ring in the center of the tray prevents the magazines from slipping out. Magazine positions are labeled 1 through 48 for easy identification of the samples.



Magazines can be pulled out and inserted through a small port in the working space cover so you can change samples without affecting the dry atmosphere in the working space.

Movement Control

The moving components of the autosampler, i.e., robot arm, robot arm lifting mechanism, and lever gear, are activated by electromagnetic and pneumatic drives. An electric motor controls the horizontal movements of the robot arm by means of the lever gear. The vertical movements of the robot arm and lifting mechanism are activated by the pneumatic system, which uses compressed air or dry nitrogen as the working gas. You can use the same gas supply for the purge gas and the pneumatic gas.

Robot Arm

The tip of the robot arm is made of a special silicon rubber that contains carbon. Both the robot arm and the lifting head are conductive and grounded to avoid electrostatic discharges. This prevents the sample capsule from sticking to the robot arm when it is set down into the magazine.

In addition to picking up the sample capsules and platinum covers, the robot arm functions as a sensor. During a typical work cycle, the autosampler uses the robot arm to check whether the magazine position is empty and whether the platinum cover is positioned properly. An error is reported back to Pyris if there is a problem.

Sample Holder Cover

The sample holder cover is activated by the pneumatic system in an elbow lever mechanism and contains an O-ring that seals the Pyris 1 DSC sample holder cover gas-tight.

Autosampler Work Cycle

When you begin a sample run by starting a play list created in Pyris Player, the autosampler performs a typical work cycle, which includes the following steps:

NOTE: The autosampler DOES NOT check for the presence of a platinum lid on the receptacle and whether the sample cell in the sample holder is empty before loading a sample pan. You must be sure that a lid is in the receptacle and that the cell is empty. It also DOES NOT check whether the sample position to which the sample pan is to be returned is occupied.

1. Before the start of a run, the robot arm is in its starting position, i.e., above tray position 3 (the third sample pan position from the center of the tray in the magazine that faces the sample holder).
2. The play list entry **Load Sample** starts.
3. The sample holder cover opens.
4. The sample tray transports the magazine with the specified sample to the sampling position (magazine directed toward the sample holder).
5. The robot arm picks up the specified sample and brings it to the sample cell in the sample holder.
6. The robot arm releases the sample into the sample cell.
7. The robot arm picks up the platinum lid from the receptacle.
8. The platinum lid is transported to the sample cell.
9. The platinum lid is placed on the sample cell.
10. The robot arm positions itself over position 3.
11. The sample holder cover closes.
12. The robot arm moves to the platinum lid receptacle and is lowered. The working gas is switched off.
13. The sample is analyzed according to the method specified in the play list.
14. The play list command **Return Sample** starts the following sequence of events:
15. The robot arm moves to its starting position as the sample holder cover opens.
16. The working gas switches on.
17. The robot arm moves to the sample cell and picks up the platinum lid.
18. The robot arm brings the platinum lid to its receptacle and deposits it in the receptacle.
19. The robot arm moves to the sample cell and picks up the sample pan.
20. The robot arm carries the sample pan back to the sample tray, which has rotated the appropriate magazine into position, and releases it into the specified position (it does not have to be the same position from which it was removed).
21. The robot arm returns to the starting position; the sample holder cover remains open.
22. If the next sample in the play list is in a different magazine, the sample tray moves this magazine to the sampling position.
23. Steps 5 – 22 are repeated for each sample in the play list until all of the samples specified are analyzed.
24. After the last sample in the play list is analyzed, the robot arm returns to the starting position and the sample holder cover closes.

Sample Handling

Handling samples encompasses the following topics:

- **Preparing Samples**
- **Loading Samples for the First Time**
- **Changing Samples**

Preparing Samples for Pyris 1 DSC Autosampler

When preparing your samples for analysis in the Pyris 1 DSC autosampler, take the following variables into consideration:

- **Sample size**
- **Sample atmosphere**
- **Temperature range**
- **Scanning rate**

See these topics in the "Operating Variables and Sample Handling" section.

In addition to these considerations, the autosampler has specific requirements for the choice of sample pan and cover and for the method of sample encapsulation.

Sample Pans

Sample pans and covers of various capacities and wall thicknesses are available from Perkin Elmer. These pans and covers will resist the varying internal pressures of sample capsules without deformation. Click here to see a list of sample pans and covers that can be used in the Pyris 1 DSC autosampler.

CAUTION: Never touch the sample or the sample pan and cover with your bare fingers. Contamination from human body oils and greases can affect your results. Always use tweezers or the Perkin Elmer Standard Suction Manipulator (P/N B014-3263) or the Special Suction Manipulator (P/N B014-2512).

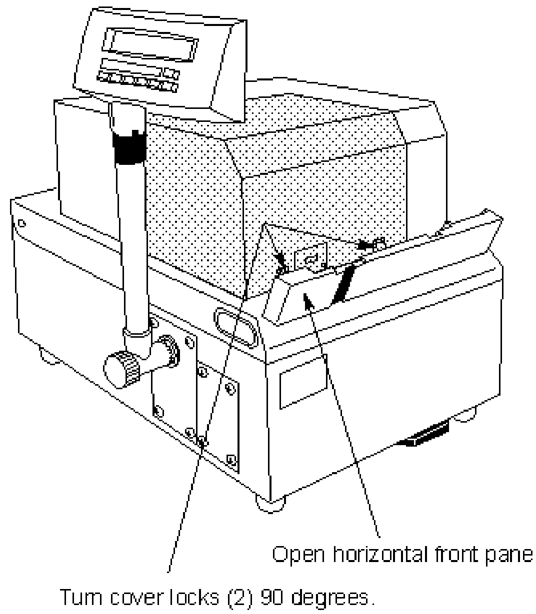
Sample Encapsulation

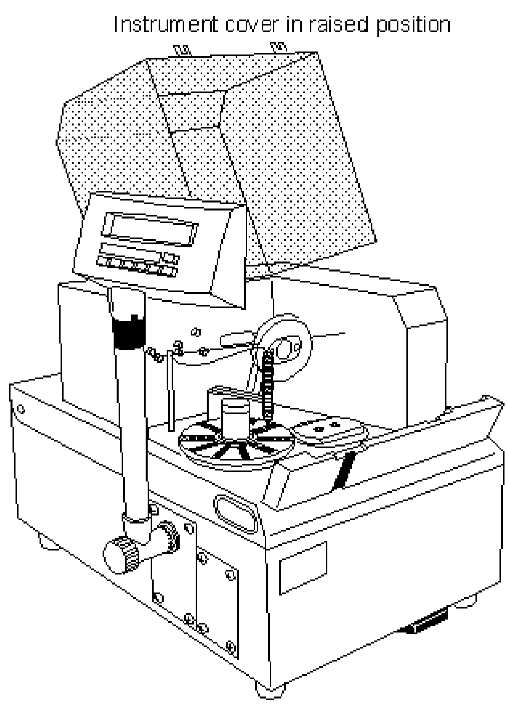
The Universal Crimper Press (P/N B013-9005) is required for preparing samples for the autosampler. The instructions provided with the press or in the topic **How to Use a Universal Crimper Press** describe how to use the accessory to encapsulate your samples.

Loading Samples in Pyris 1 DSC Autosampler for the First Time

Use the procedure below to load samples into the sample tray if you have not used the Pyris 1 DSC autosampler yet.

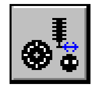
1. Make sure that the autosampler is turned on and the pneumatic or working gas is turned on and adjusted to the correct pressure (~70 – 80 psi).
2. If you are using a purge gas, check that it is connected to the elbow connector properly.
3. Open the protective cover by lifting it up and back. If the protective cover is locked, open the front horizontal panel by flipping it up and turn both locks 90° to release the cover.



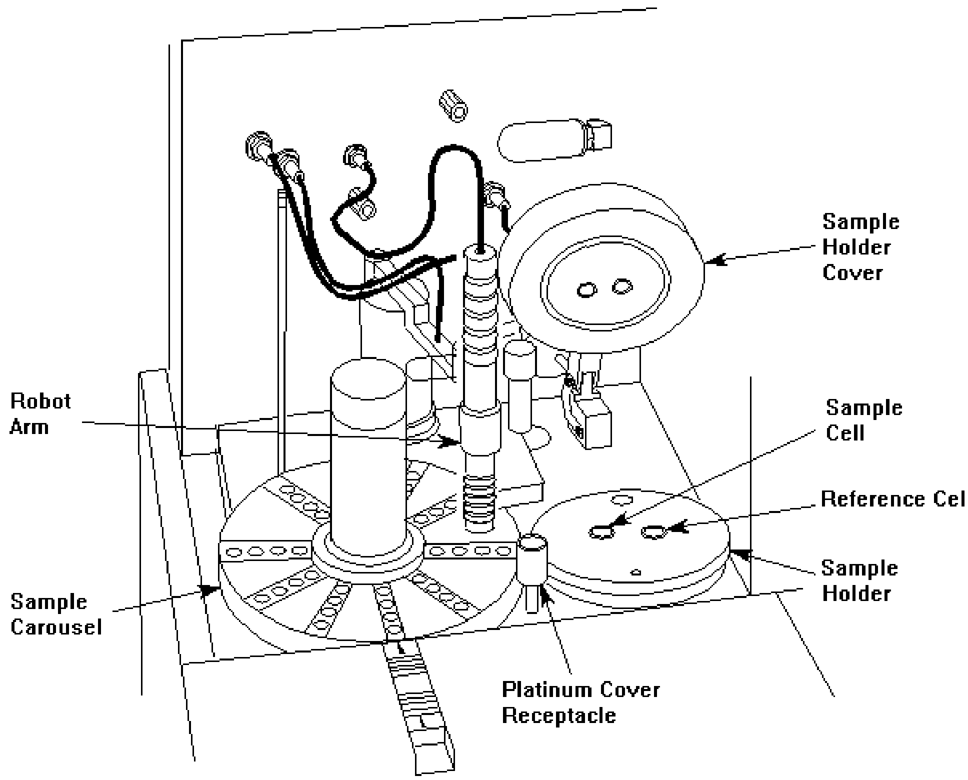


NOTE: If you are using a subambient cooling device such as the CryoFill or Intracooler, open the working space *before* you cool down the system. If the cooling device is already on and the Pyris 1 DSC is already at subambient temperatures, use the sample changing procedure described in Changing Samples.

4. Click on the **Autosampler Control** button in the control panel:



5. In the Autosampler Control dialog box, click on **Open Cover** to open the sample holder cover.
6. Using a suction manipulator (P/N B014-3263 or B014-2512) or tweezers, insert an empty, sealed sample capsule into the reference cell of the sample holder.



7. Cover the reference cell with a platinum cover (supplied with the autosampler). Make sure the sample cell of the sample holder is empty.

NOTE: If you are going to perform high-precision specific heat, you must be very particular about the reference sample pan and the platinum lid used. You can use the Load Reference command in a play list to load a precisely measured reference sample pan from a specified position in the tray and then cover it with a precisely weighed platinum lid from the receptacle.

8. Place a platinum cover on the platinum lid receptacle.
9. Prepare your samples as described in Preparing Samples and Sample Preparation.
10. Insert the magazines into their numbered positions in the sample tray.

CAUTION: The numbers on the magazines must correspond with the numbers on the sample tray.

11. Using a suction manipulator, insert the sealed sample pans into the desired tray locations.

12. Close the autosampler cover and lock it by turning the two locking bolts. Put the horizontal front panel back down to cover the bolts.
13. Open the purge gas line and adjust the pressure.

NOTE: If you plan to operate the Pyris 1 DSC autosampler at subambient temperature, click here for important information.

Preparing the Pyris 1 DSC Autosampler for Subambient Operation

If you plan to use the Pyris 1 DSC Autosampler for subambient applications, observe the following guidelines:

- Connect the cooling system you are using as described in the topic Subambient Operation and subtopics.
- The sample holder can be cooled down as far as –170°C with the CryoFill, –30°C with the Intracooler 1, –65°C with the Intracooler 2, and –70°C with the CCA 7.
- Before turning on the cooling system, purge the autosampler working space. Purging the autosampler working space prevents air humidity condensation on the sample holder. To purge the working space, first make sure that the operating gas is connected and adjusted to the correct pressure and the protective cover is closed and locked. Move the AUX Purge switch on the back of the autosampler to the On position. Purge the working space with dry operating gas (compressed air or nitrogen) for at least 15 minutes.
- Keep the autosampler protective cover closed and locked while operating your cooling system. Change samples in the magazines using the Autosampler Control dialog box and the magazine changing port and tool.
- Note that the Pyris software will not permit a **Load Sample, Return Sample, Load Reference**, or **Return Reference** to be executed in a play list until the sample or reference temperature is about 50 – 55°C. This is the safe temperature range for sucker tip to pick up the sample pan. If the sample or reference temperature is not in that range at the end of a run (you can make the End Condition in the method Go To Temp of 30°C in the Set End Condition section), the software will program the Program Temperature up to the default of 50°C. When the sample or reference temperature is within plus or minus 5°C of the Program Temperature, the load or unload step in the play list will begin.

Changing or Adding Samples

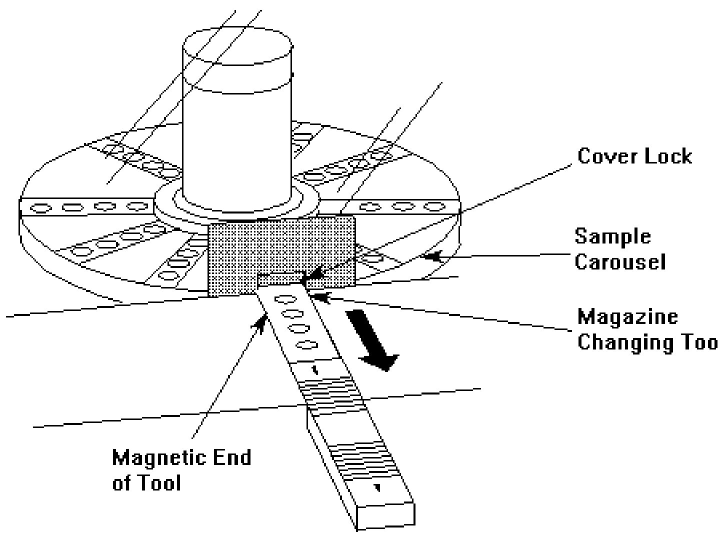
You may need to change samples in or add samples to the sample tray while a run is in progress. For example, you may want to run a play list that analyzes more than the maximum 48 samples that the tray can hold. However, to prevent humidity and dust from entering the autosampler's working area, or if the Pyris 1 DSC is in subambient mode, you should keep the protective cover closed and locked at all times. To access the sample tray to remove and add pans, the protective cover is equipped with a port through which magazines can be removed and inserted easily.

To change or add samples while a method step or a postrun data analysis step in the play list is being performed, you do not have to pause the play list. Use the Autosampler Control dialog box to select the tray position number that you want to access. The tray will rotate into position so that you can remove the magazine that contains the desired sample position. Magazines are removed and inserted with a magazine-changing tool – a flat, rectangular metal piece with a magnetic end.

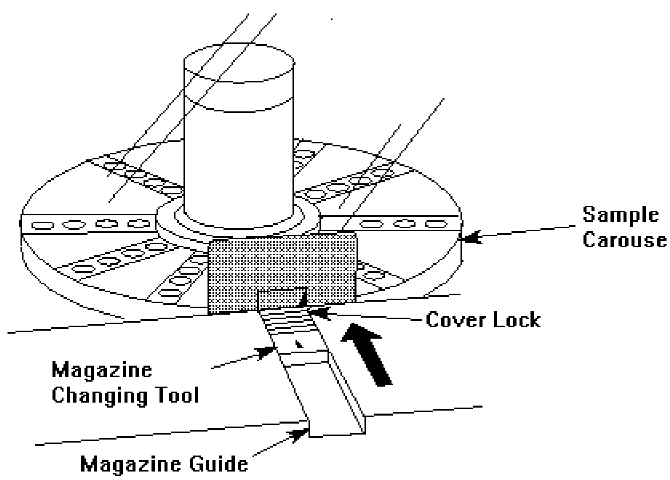
CAUTION: Do not try to change samples or add samples while the play list is executing a Load Sample, Return Sample, Load Reference, or Return Reference command.

Use the following procedure to change samples in or add samples to the tray during a run:

1. Click on the **Autosampler Control** button in the control panel.
2. In the Autosampler Control dialog box, enter the tray location in which you want to insert a sample pan or pans and click on the **Move Carousel to Location** button. The tray rotates so that the magazine containing that location is facing the insertion slot at the front of the analyzer.

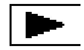


Removing a Magazine

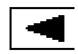


Inserting a Magazine

3. Position the magazine-changing tool in the magazine guide so that the magnetic end faces the port. The magnetic end of the magazine-changing tool looks like this:



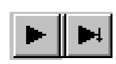
4. Push the magazine-changing tool through the port until it contacts the magazine in the tray.
5. Pull the magazine-changing tool with the magazine attached out of the port.
6. Add sample pans to or remove samples pans from the magazine using a suction manipulator or tweezers.
7. Place the magazine in the magazine guide.
8. Position the magazine-changing tool in the magazine guide so that the nonmagnetic end faces the port. The nonmagnetic end looks like this:



9. Push the magazine back through the port.
10. When finished with one magazine, return to the Autosampler Control dialog box and either enter another location that is in another magazine to add or change samples or click on the **Close** button.

Running a Play List

Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items – Load Sample, Run Method, Return Sample – there is a Sample Group. This simplifies grouping like samples together (as you would have in a sample tray of an autosampler) that use the same test method and data analysis within a play list. A Sample Group consists of a Sample List and a Data Analysis List. A specific method is selected for the samples in the Sample List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization and calculations for automatic data analysis. Display curve allows the last run data set to be recalled. Playback of the play list begins by clicking on the **Start** or **Start at Current Step** button on the Player toolbar. They are the two leftmost buttons on the Player toolbar:



NOTE: If you select the **Start at Current Step** button, the focused item must be a main-level item, i.e., Prepare Sample, Data Analysis, or Sample Group. However, if in a Sample Group, you can start a play list if the current item is a Sample line.

Before starting a play list, perform the steps below:

1. Review the safety and warning notes in **Warnings and Safety Practices**.
2. Verify that the purge and pneumatic gas tubing for the autosampler and the Pyris 1 DSC and the tubing for a cooling device, if applicable, are properly connected.
3. Verify that the electrical and cable connections between the autosampler and Pyris and all other cables are properly connected.
4. Turn on the power to the system components in the following order:
   - computer
   - GSA 7 or TAGS if present
   - autosampler

- Pyris 1 DSC
- CryoFill control box if present
- printer

5. Turn on the pneumatic gas for the autosampler and the purge gas for the Pyris 1 DSC; adjust pressures.
6. Prepare your samples as described in Sample Preparation.
7. Load the samples into the sample tray and the reference sample and platinum lid into the reference cell as described in Loading Samples for the First Time. Place a platinum lid onto the receptacle.
8. If using purge gas for the autosampler, turn on the purge gas and adjust the pressure.
9. If using a subambient cooling device, follow the instructions in Operating the Pyris 1 DSC Using an Intracooler 1P or 2P or Operating the Pyris 1 DSC with Liquid Nitrogen.
10. Start up Pyris Software for Windows and either create a new play list or load an existing play list you wish to use to run your samples.
11. Start the play list. While the play list is running, you can
    a. edit the entries in the play list that have not been executed yet
    b. view the progress of the run in the Instrument Viewer window
    c. print a collapsed or expanded version of the play list or history
    d. while in a Sample Group, you can continue to add, insert, or delete samples while the list if running, below the currently running line.

NOTE: If you select Go To Load, Go To Temp, or Hold at Temp from the Control Panel while a play list is running, the current sample run will end and the playback of the play list ends.

The View Play List page gives an expanded view of the Edit Play List page and provides a macro view of all the sequences.

The View Sample List page is a spreadsheet-style view of the samples of this play list. It includes all the sample information such as ID, method used, location, etc.

The View History page is a historical view of the completed steps of the play list and the Sample History page is a historical view of the samples run.

Troubleshooting

When there is a problem with the Pyris 1 DSC Autosampler, a status message or code is displayed at the bottom of the Pyris window in the Status Bar and also in the Status Panel, if you have Autosampler Status selected for display. The error message also will appear in the History file of a play list run.

Two errors that occur more often than others are

1. When the autosampler is first powered on, the tray moves so that the sample 1 position faces the magazine-changing port and the robot arm is moved over the tray. Occasionally, if the robot arm started at the platinum cover receptacle, there may be too much friction in the linkage system to let the robot arm move. When this occurs, the autosampler reports an error and cannot continue. Either repeatedly power the autosampler off and on until the arm becomes unstuck or open the protective cover and carefully push the robot arm into movement.

2. After many cycles, the robot arm may fumble the platinum cover (and, less frequently, sample pans). When this occurs, the autosampler reports an error and cannot continue. Open the protective cover and replace the platinum cover.

If an error, such as those listed below, occurs while you are running a play list, the play list will stop; you will have to restart the play list after resolving the problem. If data is being collected at the time, click on the **Stop Method** button on the Control Panel. In that case, the data from that step in the play list will be lost. The History page can be used to determine what samples were run prior to the error.

To help resolve a problem causing the error message, the following topics give the possible causes and solutions for some of the more common errors that could occur while using the autosampler:

No Working Gas Pressure

Possible Causes	**Solutions**
The working gas is not connected.	Connect the working gas to the Gas Inlet port on back of the autosampler. Set the inlet pressure to 500 kPa (5 bar).
The inlet pressure is < 400 kPa (4 bar).	Set the inlet pressure to 500 kPa (5 bar).
The internal gas tube has a leak or has slipped from its nozzle.	1. Turn the Pyris 1 DSC/autosampler off, disconnect the power cord, and remove the rear cover. 2. Check the internal gas tube for leaks; repair if necessary. 3. Check the position of the internal gas tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the Pyris 1 DSC/autosampler.
Switch for inlet pressure is defective.	Contact a Perkin Elmer Service Engineer.

Robot Arm Not in Raised Position

Possible Causes	**Solutions**
The upper pressure tube at the robot arm has slipped off or has a leak.	Tighten the upper pressure tube.
The upper knurled screw in the center of the lifting head is loose.	Tighten the upper knurled screw.
The switchover valve is hung up.	Turn the Pyris 1 DSC/autosampler off and on (several times if necessary).
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.
The robot arm throttle is not adjusted properly.	Contact a Perkin Elmer Service Engineer.

Robot Arm Not in Lowered Position

Possible Causes	Solutions
The lower pressure tube at the robot arm has slipped off or has a leak.	Tighten the lower pressure tube.
The lower knurled screw in the center of the robot arm is loose.	Tighten the lower knurled screw.
The switchover valve is hung up.	Turn the Pyris 1 DSC/autosampler off and on (several times if necessary).
The robot arm throttle is not adjusted properly.	Contact a Perkin Elmer Service Engineer.

Sample Cover Could Not Be Closed

Possible Causes	Solutions
The switchover valve is hung up.	Turn the Pyris 1 DSC/autosampler off and on (several times if necessary).
The lifting cylinder gas tube has a leak or has slipped off.	1. Turn off the Pyris 1 DSC/autosampler, disconnect the power cord, and remove the rear cover. 2. Check the lifting cylinder pressure tube for leaks; repair if necessary. 3. Check the position of the lifting cylinder pressure tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the power to the Pyris 1 DSC/autosampler.
The lifting cylinder is defective.	Contact a Perkin Elmer Service Engineer.
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.

Sample Cover Closed But Not Sealed

Possible Causes	Solutions
An object is caught between the sample holder and the sample holder cover.	Inspect the sample holder and remove the object as follows: **NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and the method. Enter 50°C in Go To Temp field and click on **Go To Temp** button. Once the sample temperature has reached 50°C, you can perform the steps below: 1. Click on the **Autosampler Control** button on the control panel. 2. Click on the **Open Cover** button in the dialog box.

	3. Open the protective cover of the autosampler and remove the object. 4. Close the protective cover. 5. Click on the **Close Cover** button in the dialog box. 6. Restart the play list.
The sample holder cover O ring is defective.	Inspect the sample holder cover O ring and replace it if necessary.
The purge gas inlet for the sample holder is open and no gas pressure can build up.	Connect the gas tube for the sample holder purge to the gas supply or interconnect the gas lines.
The lifting cylinder gas tube has a leak or has slipped off.	1. Turn off the Pyris 1 DSC/autosampler and remove the rear cover. 2. Check the lifting cylinder pressure tube for leaks; repair if necessary. 3. Check the position of the lifting cylinder pressure tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the power to the Pyris 1 DSC/autosampler.
The sample holder is out of alignment.	Contact a Perkin Elmer Service Engineer.
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.

Robot Arm in Wrong Position

Possible Causes	**Solution**
The coding disk/light barrier is defective.	Contact a Perkin Elmer Service Engineer.

Tray Not in Correct Position

Possible Causes	**Solutions**
The lever gear/sample tray is blocked; it will not reach the end position of any particular movement within 5 min.	Check the lever gear/sample tray movement for obstructions and, if necessary, remove the obstruction. **NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and a method. Enter 50°C in Go To Temp field and click on **Go To Temp** button. Once the sample temperature has reached 50°C you can perform the procedure above.
The coding disk/light barrier for the level gear/sample tray is defective.	Contact a Perkin Elmer Service Engineer.

Sample/Platinum Cover Could Not Be Found

Possible Causes	Solutions
The sample pan or platinum cover in the sample holder cannot be picked up (i.e., it is stuck) by the robot arm.	**NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and a method. Enter 50°C in Go To Temp field and click on **Go To Temp** button. Once the sample temperature has reached 50°C you can perform the steps below: 1. Open the protective cover and loosen the pan or cover, leaving it in the sample holder. Close the protective cover. 2. Restart the play list.
The sample pan or platinum cover is deformed or the sample pan cover was crimped asymmetrically, causing the robot arm to lose the sample pan.	1. Open the protective cover and verify that the sample pan is deformed; replace it with a newly encapsulated sample. Close the protective cover. 2. Restart the play list.
The location in the sample tray is empty.	1. Open the protective cover and verify that the sample position is empty; place an encapsulated sample in the magazine. Close the protective cover. 2. Alternatively, click on the Autosampler Control button and in the dialog box enter the empty position in the Select Carousel Position field and click on Move Carousel to Location. The tray will rotate to place the magazine with the empty position so you can remove it through the magazine port and insert a sample pan. Return the magazine back into the tray. Click on **Close** to close the dialog box. 3. Restart the play list.
The platinum lid is dropped when unloading a sample.	1. Open the protective cover and replace the lid on the sample holder. Close the protective cover. 2. Restart the play list.
The suction tip on the robot arm is defective.	Replace the suction tip.

Final Position of Autosampler Not Achieved

Possible Causes	**Solutions**
The lever gear/sample tray is blocked; it will not reach the end position of any particular movement within 5 min.	Check the lever gear/sample tray movement for obstructions and, if necessary, remove the obstruction. **NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and a method. Enter 50°C in Go To Temp field and click on **Go To Temp** button. Once the sample temperature has reached 50°C you can perform the procedure above.
The coding disk/light barrier for the level gear/sample tray is defective.	Contact a Perkin Elmer Service Engineer.

Sample Number/Tray Position Out of Range

Possible Causes	**Solutions**
You entered a number >48 in the play list for Load Sample or Load Reference or for Select Carousel Location field in the Autosampler Control dialog box.	Reenter a number less than or equal to 48 in the appropriate field.

Sample Position of Magazine Occupied

Possible Causes	**Solutions**
The autosampler found something (e.g., a sample pan) located in a magazine position that should be empty.	1. Click on the **Autosampler Control** button in the Control Panel. 2. In the Select Carousel Position, enter the number of the sample tray position that contains the object you want to remove. 3. Click on the Move Carousel to Location field. The tray will rotate so that the magazine with the specified position faces the magazine port. 4. Using the magazine-changing tool, remove the magazine and remove the unwanted object. Reinsert the magazine. 5. Click on the **Close** button in the Autosampler Control dialog box.

Sample Cell/Reference Cell Already Occupied

Possible Causes	Solutions
The autosampler found a sample pan in the sample holder, when it should be empty.	1. Click on the **Autosampler Control** button in the Control Panel. The Autosampler Control dialog box appears. 2. Enter an empty sample tray position in the Select Carousel Position field. 3. Click on Unload Sample to Location to have the pan deposited in the empty position. 4. Click on the **Close** button in the Autosampler Control dialog box.

No Platinum Cover Available

Possible Causes	Solutions
A sample that has already been run is still in the sample holder.	1. Click on the **Autosampler Control** button in the Control Panel. The Autosampler Control dialog box appears. 2. Enter an empty sample tray position in the Select Carousel Location field. 3. Click on the Unload Sample to Location button to have the pan deposited in the empty position. 4. Click on the **Close** button in the Autosampler Control dialog box. 5. Restart the play list.
During a run, the platinum cover was not placed in its receptacle.	1. Open the protective cover and use the suction tip tool to place the platinum cover in its receptacle. 2. Restart the play list.

Syntax Error

Possible Causes	Solutions
Transmission error.	Retry the play list.

Command Not Recognized

Possible Causes	Solutions
Transmission error.	Retry the play list.

Carrier Detect Signal Dropped

Possible Causes	Solutions
Transmission error.	Check the RS-232 cable between the autosampler and the Pyris 1 DSC.

Maintenance

- The suction tip (P/N B013-8618) on the Pyris 1 DSC autosampler arm picks up debris over time. It should be checked about every 200 cycles. If it is dirty, it should be replaced.
- Clean the platinum lids with ether or alcohol on a cotton swab. Be sure that the lid is dry before using it again.

Observe the following guidelines when cleaning the autosampler:

- The exterior surfaces of the autosampler are resistant to diluted acids but not to concentrated acids.
- The painted surfaces and protective cover are not resistant to organic solvents.
- To clean surfaces under the protective cover, i.e., the working space, use only a cloth dampened with clear water to which a small amount of commercial household cleaning agent was added. Wipe these areas dry with a soft, dust-free cloth or tissue.
- With a medium soft brush, remove any dust that has accumulated in the folds of the robot arm bellow or in the magazine groove of the sample tray.

CAUTION: Over time the robot arm might become misaligned with respect to the sample tray and sample holder cells. If the sample pans and platinum lid are not being deposited properly, e.g., the lid may be at an angle on top of the sample pan, your autosampler should be realigned by a Perkin Elmer service engineer. The autosampler is realigned by running an alignment program from the Autosampler Diagnostic Panel dialog box and using special alignment tools.

Chapter 2
DSC 7

Overview

The Perkin Elmer DSC 7 Differential Scanning Calorimeter is connected to a computer via a TAC 7/DX Thermal Analysis Instrument Controller. The Pyris Software for Windows installed on the computer controls the DSC 7 via temperature control programs. The DSC 7 is used for the calorimetric measurement, characterization, and analysis of thermal properties of materials. Using Pyris Software for Windows, you can program the DSC 7 to run from an initial to a final temperature through transitions in the sample material such as melting, glass transitions, solid-state transitions, or crystallization. The temperature range is scanned by changing the temperature at a linear rate in order to study these endothermic and exothermic reactions of samples. The DSC 7 also can be used to gather and analyze data from isothermal reactions such as crystallization or curing.

The standard configuration DSC 7 is equipped with an insulated reservoir that allows the use of an ice water bath for standard operation from 25°C to 725°C. You can access the reservoir through the stainless steel reservoir access plate that is just in front of the sample holder on the top deck of the analyzer.

To load the ice water into the DSC 7, remove the reservoir access plate and add ice and water until the reservoir is approximately 75% full. Replace the reservoir access plate. The DSC 7 will cool down and equilibrate for approximately 30 minutes after adding the ice water coolant. The coolant will allow continuous operation of the DSC 7 for 12 to 18 hours.

In the ambient configuration, the DSC 7 is provided with a tap water circulating chamber. This is used for operation from approximately 50°C to 725°C. Tap water must be conveniently located near the DSC 7 and must be continuously circulated during instrument operation.

Additional cooling accessories are available for extending the temperature range to the subambient:

- Intracoolers 1 and 2 allow operation to –30°C and –65°C, respectively. The Intracoolers are mechanical refrigeration units that automatically maintain a constant DSC 7 sample holder temperature.
- The CCA 7 Liquid Nitrogen Controlled Cooling Accessory allows operation to –140°C. The CCA 7 is a liquid nitrogen-based system that automatically controls the DSC 7 sample holder environment over a broad temperature range.
- The DSC 7 Dry Box Assembly and Liquid Nitrogen Subambient Accessory allow operation of the DSC 7 to –170°C.

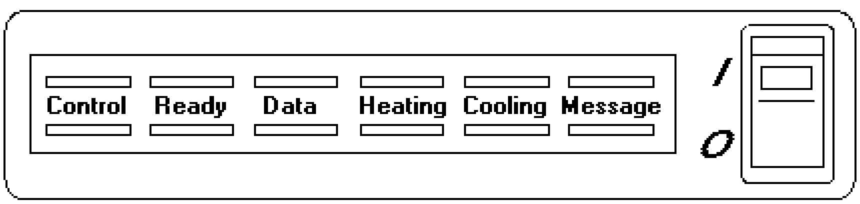
An additional option for the DSC 7 is the autosampler, called the DSC 7 Robotics System, that attaches to the DSC 7 and is controlled by the Pyris software. The Robotics System allows you to perform fully automated DSC analyses. The robotics system's carousel has 48 sample positions but you can program it to run up to 1000 samples. It is useful in laboratories that analyze multiple samples of the same or different types. With the Pyris Player software, you can create "play lists" that contain multiple methods for analyzing one sample; multiple samples to be analyzed by one method, and many ways to perform postrun data analysis.

The features of the DSC 7 and the Robotic System are discussed in the following topics:

- **Safety Precautions**
- **Sample Holder**
- **Baseline Adjustment**
- **Operating Variables and Sample Handling**
- **Calibration**
- **Subambient Operation**
- **Maintenance**
- **Accessories**
- **Part Numbers**
- **Robotics System**

Operating Controls and Status Indicators

The DSC 7 has two controls and six status indicators. The status indicators are on the front panel, seen below, along with the power button.



The controls of the DSC 7 are found behind the front drop-down panel. The Balance control dial is used to optimize the curvature of the baseline. The Slope control dial is used to adjust the slope of the baseline.

Control

The DSC 7 temperature sensors are in control of the temperature; power is being supplied to the furnace to maintain the program temperature selected in the software.

The Control light should always be illuminated during the main section of a temperature program run in order to obtain accurate data. If the Control light goes out during a cooling run, the data after that will not be accurate.

The temperature at which the Control indicator will illuminate and, therefore, the acceptable range of the DSC 7, is dependent on the type of coolant or cooling accessory that is being used. For example, with an ice water bath in the DSC 7 reservoir, temperatures as low as 25°C can be attained. With liquid nitrogen as the coolant, temperatures as low as –170°C can be attained.

Ready

While blinking, Ready means that the program temperature of the DSC 7 has reached the Load Temperature defined in Pyris Software for Windows. When the Control light is also lit, the temperature program is ready to begin. When the Ready light is lit continuously, it means that power is supplied to the DSC 7.

Upon startup of the system, the Ready indicator should be the only indicator that is illuminated. In some cases, the Control indicator may also be illuminated upon startup depending on the heatsink and coolant temperatures.

Data

When the Data light is on, data are being collected from the analyzer. The Data light remains unlit at all other times.

Heating

When the Heating light is illuminated, the DSC 7 is heating under temperature program control at the selected rate.

Cooling

When the Cooling indicator is illuminated, the DSC 7 is cooling under temperature program control at the selected rate.

Message

When the Message indicator is blinking, an error or informative message must be acknowledged at the computer.

Power

The power switch is on the right side of the front panel. When illuminated, line power is being supplied to the DSC 7.

Balance

The Balance control is found behind the front drop-down panel. It is used to optimize the curvature of the baseline.

Slope

The Slope control is found behind the front drop-down panel. It is used to adjust the slope of the baseline.

Theory

The theory of operation of the DSC 7 is based on the Perkin Elmer power-compensated “null-balance” DSC principle in which energy absorbed or evolved by the sample is compensated for by adding or subtracting an equivalent amount of electrical energy to a heater located in the sample holder.

Platinum resistance heaters and thermometers are used in the DSC 7 to measure the energy and temperature. The continuous and automatic adjustment of heater power (energy per unit time) necessary to keep the sample holder temperature identical to that of the reference holder provides a varying electrical signal equivalent to the varying thermal behavior of the sample. This measurement is made directly in differential power units [milliwatts (mW)], providing true electrical energy measurement of peak areas. In conventional “heat flux” DSC designs, the magnitude of peak areas depends directly on the magnitude of the thermal constants in the system, which vary as a function of temperature. The Perkin Elmer DSC design measures the energy of a transition directly, providing more accurate calorimetric information.

Safety Precautions

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

The following precautions must be observed when using the DSC 7 Differential Scanning Calorimeter:

- Never turn the computer off until the following message appears:

  It’s now safe to shut off your computer.

- Never press the **Reset** button on the computer if the Pyris software appears to malfunction. Press the **Ctrl–Alt–Del** keys simultaneously and select the Task Manager to close Pyris Software for Windows.
- Never remove the outer instrument cover or side panels on the DSC 7 without shutting the instrument down and disconnecting its power cord from the power source.
- Always use helium as the purge gas when liquid nitrogen is used in the subambient reservoir of your DSC 7. At other times, use nitrogen, argon, oxygen, or air as the purge gas.
- Do not immerse the purge gas exit line in a liquid since the liquid may be drawn back into the sample holder.

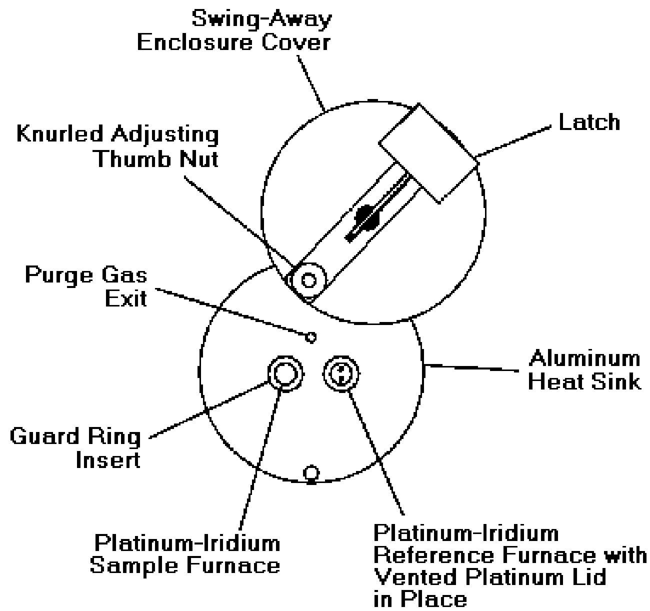
Always observe the precautions indicated for the use of the various cooling devices.

- Only high-quality purge gases should be used with the DSC 7. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for the removal of any moisture from the purge gases.
- Always observe the startup or shutdown procedures with the DSC 7 and all related instruments.
- Do not use aluminum sample pans above 600°C. Since aluminum melts at 660°C, the pans will alloy with and destroy the sample holders. Entering a temperature above 600°C will cause the computer to display a dialog box with a cautionary message. Always make sure that you are not using aluminum pans in either the sample of reference cell before extending the DSC 7 temperature range above 600°C.
- Always encapsulate indium, tin, lead, and zinc standards in aluminum or graphite pans. These metals will alloy with gold, copper, or platinum pans.
- Always use the sample handling tray or guard ring inserts when removing or inserting sample pans or sample holder covers into the DSC 7 sample holder. This will protect sample holder leads.

- It is very important that nothing fall down into the cavity surrounding the sample holders. If this does occur, turn off the power to the DSC 7 **IMMEDIATELY** and call your local P-E Service Representative for instructions.
- Do not force the platinum sample holder covers to fit the sample holders and do not deform the covers in any way. The sample holder covers should fit easily and loosely into the holders. When required, reform the covers with the reforming tool.

Sample Holder

The DSC 7 sample holder is on the top of the DSC 7.



Swing-Away Enclosure Cover

The swing-away enclosure cover, with its rubber O-ring seal, seals the furnaces from the external atmosphere during an analysis. If the cover seems too loose, use the knurled nut to adjust the tightness.

Knurled Adjusting Thumb Nut

Use the knurled adjusting thumb nut to adjust the tightness of the enclosure cover. Do not overtighten the nut as that can damage the sample enclosure cover.

Purge Gas Exit

The purge gas exits from the tiny hole on the top of the aluminum heatsink.

Guard Ring Insert

The guard ring insert (P/N 0319-0236) for the DSC 7 is positioned around the outside of the furnace. It prevents samples, pans, and other foreign objects from falling into the cavity around the furnace. In general, the guard rings should remain in place under all circumstances. However, since baseline stability is slightly better without the guard rings when using liquid nitrogen, there may be a few applications (e.g., specific heat capacity) for which you may want to remove them.

Note the slit. The slit must be aligned with the purge gas exit hole. Once you are sure that there is proper alignment, with the end of a tweezers gently press down on the guard ring until it is flush with the top of the heat sink.

Platinum - Iridium Sample Furnace

There are two low-mass platinum - iridium cells (furnaces) embedded in a large aluminum heatsink. The left furnace is used for encapsulated sample materials; the right furnace is used for reference materials. For example, the right furnace typically contains an empty sample pan and lid or an empty sample container of the type used in the sample cell.

To gain access to the sample and reference furnaces:

1. Lift the latch on the sample holder enclosure cover.
2. Swing the enclosure cover to the side to access the sample and reference furnaces.

Platinum - Iridium Reference Furnace with Vented Platinum Lid in Place

Vented platinum lids are used to cover both the sample and the reference furnaces. These lids should be similarly oriented when they are placed in their respective furnace. The lids should fit snugly into the furnace but should not be forced. If a lid is bent or deformed, use the Sample Holder Cover Reforming Tool (P/N 0319-0030) to reform the lids. Whenever sample lids or sample materials are removed or placed into the instrument, make certain that either the guard ring inserts are permanently in place or the sample handling tray (P/N 0419-0303) is used.

Sample Holder Cover Reforming Tool (P/N 0319-0030)

DSC performance is enhanced with sample holder covers that are formed properly and are not degraded by extended usage. This tool allows the analyst to reform the platinum covers to their original dimensions.

Aluminum Heat Sink

The aluminum heat sink provides a constant temperature environment for the sample and reference calorimeters.

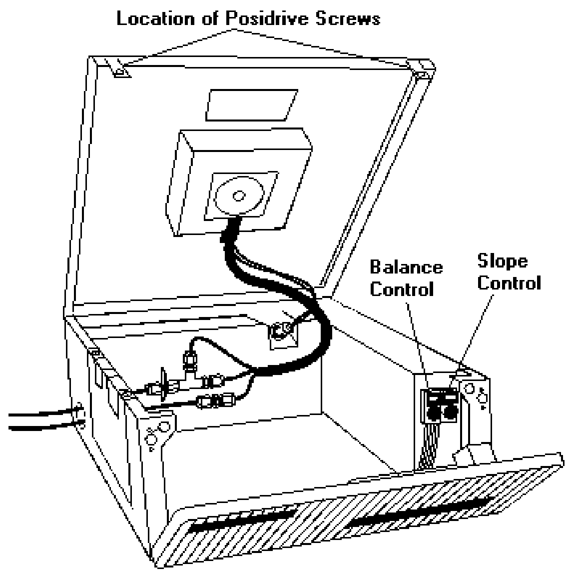
Latch

To gain access to the sample furnaces, lift the latch on the sample holder enclosure cover.

See **Maintenance** for more information on cleaning and replacing the DSC 7 sample holder.

Baseline Adjustment

Optimization of the DSC 7 baseline involves adjusting two controls that are located behind the DSC 7 front panel: Balance and Slope. To access these controls, grasp the upper corners of the DSC 7 front panel and pull forward. You now have access to the baseline controls and purge gas connectors. The two baseline controls are in the upper-right-hand corner.



NOTE: The readings on both of these baseline controls may be adjusted between approximately 5 and 80. Do not attempt to exceed this range on either control.

CAUTION: Do NOT force the Balance or the Slope control beyond the point where the upper or lower stops are reached. Forcing the control beyond its limits may destroy the mechanism. Do not attempt to adjust the control before disengaging the lock.

To optimize the DSC 7 baseline using the Balance and Slope controls, perform the following three procedures:

- **Manual Baseline Optimization**
- **Baseline Curvature Correction**
- **Baseline Slope Adjustment**

Manual Baseline Optimization

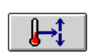
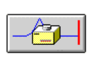
1. Determine the temperature range over which the baseline is to be optimized. It is not necessary to optimize the baseline over the entire temperature range if most of your experiments will be performed over only a smaller portion of the range. For example, if your work involves the analysis of the melting profiles of thermoplastic materials, you will generally be working in the range of 50°C – 250°C. Thus, you need to optimize the instrument only between those temperatures.

2. Select the coolant or cooling accessory that will be used for your experiment. An ice water bath, liquid nitrogen, Intracoolers, or a CCA 7 can be used. Allow enough time for the system

to equilibrate after selecting the desired coolant or cooling accessory. This example assumes you are optimizing your baseline over the temperature range of 50°C – 250°C.

3. Set the purge gas to a flow rate of approximately 20 – 30 cc/min.
4. Set the Balance and Slope controls to a reading of 040 before proceeding.

NOTE: The Balance and Slope controls were set during initial installation of the DSC 7. If the settings for the DSC 7 have been changed or accidentally moved, set both controls to 040 before proceeding.

5. Remove all sample and reference containers from the sample and reference cells. Replace with the vented platinum covers.
6. In the Pyris software, click on the DSC 7 button in the Pyris Manager to open the DSC 7 Instrument Application.
7. From the Method Editor, select the Program tab.
8. Click on the **End Condition** button to select the action that the system will take at the end of the experiment. Select **Go To Temp**; 30°C is the default.
9. Select the Initial State tab.
10. In the Initial State page, make sure that the Baseline File box is deselected, i.e., the is no "x" in it. Any filename displayed will be grayed out. The remaining options on this page should be left in the default state.
11. Select **Save Method As** from the **File** menu and enter a method name to store the selected options (if desired).
12. The Initial State page should still be displayed. In the Set Initial Values box, enter **50** for Temperature.
13. In the Data Collection box, make sure that the Sample Rate is Standard.
14. Select the Program tab.
15. Click on the **Add a step** button and select **Temperature Scan**.
16. In the Rate box the entry should be **40**.
17. In the **To** field, enter **250**.
18. Select **Save** from the File menu (or click on the disk icon on the standard toolbar).
19. Click on the **Go To Temperature** button  in the DSC 7 Control Panel. The instrument will now program to 50°C and hold there. After a short time the Ready and Control status indicators on the DSC 7 will change appropriately. The Y Value signal will stabilize.
20. Click on the **Start** button  in the Control Panel to start the run. The light will change from red to green. On the DSC 7 the Control, Ready, Data, and Heating status indicators should light. This means that the DSC 7 is in temperature control, data are being collected, and a heating experiment is being performed.

Baseline Curvature Correction

After running the baseline, you now determine the amount of curvature correction that must be made to the Balance control by performing the following steps:

1. With the Data Analysis screen still displayed, from the **Calc** menu select **Peak**.

2. Enter a Left Limit value and a Right Limit value in the dialog box, selecting points near the beginning and the end of the curve. Select the Peak Height option in the dialog box.
3. Click on the **Calculate** button.
4. In the Adjust Baseline dialog box, click on the **Calculate** button.
5. A peak calculation is performed and displayed in the Data Analysis page. Note the peak height value.
6. Invert the peak height value (i.e., if the height is 11.2 mW, make it –11.2 mW) and add this value to the current Balance value.
7. Adjust the Balance control setting to the newly calculated value by opening the front cover and turning the Balance knob appropriately.

NOTE: Changes made to the DSC's Balance control setting affect the temperature calibration. If you change the setting, check the temperature calibration.

8. Select and then perform another baseline run as described above. Most of the baseline curvature should be gone by now.

Baseline Slope Adjustment

After removing the baseline curvature, baseline slope can be removed. To correct the slope, perform the following steps:

1. Set up and start a run using the method you saved while performing the manual baseline optimization.
2. When the run is approximately half finished (i.e., the temperature is approximately 150°C), slowly turn the Slope control until the baseline displayed on the screen is straight and parallel with the X axis.

The baseline is now optimized.

Operating Variables and Sample Handling

In differential scanning calorimetry, the object of an experiment is to record the heat flow to or from a sample over a linearly changing temperature range or at a single isothermal temperature. The goal is to obtain accurate data in a minimum period of time. The following topics illustrate some of the techniques that can be used to obtain the most accurate DSC data in a minimum amount of time.

- **Sample Preparation**
- **Sample Pans**
- **Sample Encapsulation**
- **Sample Size**
- **Sample Atmosphere**
- **Temperature Range**
- **Scanning Rate**
- **Sample Loading**

- **Specific Heat Analysis**

Sample Preparation

The differential scanning calorimeter can analyze solid or liquid samples. Solid samples can be in film, powder, crystal, or granular form. Although quantitative accuracy will remain the same regardless of sample shape, the qualitative appearance of a run may be affected by the sample configuration. Proper sample preparation that maximizes the contact surface between the pan and the sample will reduce the resistance of the sample to heat flow through the DSC temperature sensors and will result in maximum peak sharpness and resolution.

The best sample shapes for optimum performance are thin disks or films or fine granules spread in a thin layer on the bottom of the pan. Materials such as polymer films can be conveniently prepared by cutting out sections of the film with a standard paper punch or cork borer. Solid materials can be sliced with a razor or knife.

See "how to" topics **Prepare Solid Sample, Prepare Liquid Sample,** and **Prepare Powdered Sample** in the Pyris Multimedia Presentations Help for more information.

Sample Pans

After preparing the sample, it must be placed into a sample pan which will then be placed in the sample holder for analysis. The following types of sample pans are available:

- **Standard Sample Pans**
- **Specialty Sample Pans**
- **Volatile Sample Pans**
- **Large-Volume O-Ring-Sealed Stainless-Steel Pans**
- **High-Pressure Capsules**
- **Robotic System Sample Pans**

DSC Sample Pan Table

This table lists sample pans available for the DSC 7 for most thermal analysis applications. Associated with each pan, where applicable, is the crimper to be used with that particular pan.

Sample Pan	Operating Range	Vol. (µL)	Pressure	Crimper/ Sealer	Qty.	Part No.
Std. aluminum pans *	–170°C to 600°C		1 atm, 100 kPa	0219-0048	400	0219-0041
Std. gold pans *	–170°C to 725°C		1 atm, 100 kPa	0219-0048	10	0219-0042
Std. copper pans*	–170°C to 725°C		1 atm, 100 kPa	0219-0048	200	0319-0026
Alumina pans	–170°C to 725°C		1 atm, 100 kPa	Not required	6	N519-0180
Platinum pans	–170°C to 725°C		1 atm, 100 kPa	Not required	4	0319-0024
Graphite pans	–170°C to 725°C		1 atm, 100 kPa	Not required	4	0319-0025
Volatile aluminum pans *	–170°C to 600°C	20	2 atm, 200 kPa	0219-0061	400	0219-0062

Volatile aluminum pans (pierced)*	–170°C to 600°C	20	1 atm, 100 kPa	0219-0061	100	N519-0788
Volatile gold pans*	–170°C to 725°C	20	2 atm, 200 kPa	0219-0061	10	0219-0080
Stainless-steel/O-ring*	–40°C to 300°C	60	24 atm, 2400 kPa	0990-8467	20 1000	0319-1047 0319-0029
Original high-pressure stainless steel	–170°C to 400°C	30	150 atm, 15,000 kPa	Not available		0419-1761
Original high-pressure gold-plated	–170°C to 400°C	30	150 atm, 15,000 kPa	Not available		0419-1760
High-pressure stainless steel	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2901
High-pressure gold-plated	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2902
High-pressure titanium	–170°C to 400°C	30	150 atm, 15,000 kPa	B018-2864	5	B018-2903
DPA 7	–170°C to 600°C	12.5	1 atm, 100 kPa	Not Required	100	B018-6859
Robotic aluminum	–170°C to 600°C	10	1 atm, 100 kPa	B013-9005	400	B014-3015
Robotic aluminum	–170°C to 600°C	30	1 atm, 100 kPa	B013-9005	400	B014-3016
Robotic aluminum	–170°C to 600°C	50	1 atm, 100 kPa	B013-9005	400	B014-3017
Robotic aluminum	–170°C to 600°C	40	1 atm, 100 kPa	B013-9005	400	B014-3021
Robotic aluminum (vented)	–170°C to 600°C	30	1 atm, 100 kPa	B013-9005	400	B014-3018
Robotic aluminum (vented)	–170°C to 600°C	50	1 atm, 100 kPa	B013-9005	400	B014-3019
Robotic gold	–170°C to 725°C	50	1 atm, 100 kPa	B013-9005	10	B014-3024
Robotic aluminum	–170°C to 600°C	10	2 atm, 200 kPa	B013-9005	400	B016-9319
Robotic aluminum	–170°C to 600°C	25	1 atm, 100 kPa	B013-9005	400	B017-4937
Robotic aluminum	–170°C to 600°C	30	2 atm, 200 kPa	B013-9005	400	B014-3020
Robotic aluminum	–170°C to 600°C	50	2 atm, 200 kPa	B013-9005	400	B016-9320
Aluminum*	–170°C to 600°C	12.5	2 atm, 200 kPa	B013-9005	400	B016-9321

* These pans may also be crimped using the Universal Crimper Press with the appropriate sealing insert.

Standard Sample Pans

Standard sample pans come in three different types:

- **Aluminum Sample Pans**
- **Copper Sample Pans**
- **Gold Sample Pans**

In the standard sample pan, the sample is completely enclosed in a highly conductive capsule and distributed in a thin layer such that the internal resistance in the sample itself is very small. The sample pan material and the sample holder design have very small thermal resistance. This contributes to superior thermal conductivity and temperature control between the sample and the sample pan. Because of the high thermal conductivity of aluminum, copper, and gold, heat transfer is maximized. This means that the sample size can be minimized. Because of their high temperature capabilities, platinum and gold can be used to 725°C.

CAUTION: Never exceed 600°C when using aluminum sample pans. Since aluminum melts at 660°C, an aluminum sample pan will alloy with and destroy the sample holder.

The metal standards (indium, tin, lead, and zinc) must be encapsulated in aluminum pans because they will alloy with gold, copper, or platinum pans.

Aluminum Sample Pans (P/N 0219-0041)

Aluminum sample pans are used with nonvolatile solid samples such as plastics and polymers. They are used routinely for applications such as polymer melting, crystallization, glass transitions of polymers, thermoplastics, and thermosets. The pans are crimped but not hermetically sealed. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Gold Sample Pans (P/N 0219-0042)

Gold sample pans are used for any nonmetal, i.e., ceramics, coal, soils, or minerals. They can also be used for any material that reacts with aluminum or which must be heated over 600°C. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Copper Sample Pans (P/N 0319-0026)

Copper sample pans can be used for samples that have transitions that occur above the temperature range of the aluminum pans. Typical applications for the use of copper sample pans include antioxidant testing, thermoset and thermoplastic glass transition, and melting point determinations. These pans are crimped with the Standard Sample Pan Crimper Press (P/N 0219-0048) and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

See the topics on how to **Crimp the Sample Pan by Hand** or by **Using the Standard Sample Pan Crimper** in the Pyris Multimedia Presentations Help.

Specialty Sample Pans

There are three specialty sample pans for use with a DSC:

Alumina Sample Pans (P/N N519-0180)

Alumina sample pans are used for oxidation studies of metals, minerals, ceramics, clays, and soils. They can also be used for melting and eutectic diagrams in the metals industry. The associated covers are pierced. A crimper is not required.

Graphite Sample Pans (P/N 0319-0025)

Graphite sample pans should be used in an inert atmosphere. They are used primarily with metals, but not for oxidation studies. They have extremely good heat transfer properties that yield sharp transition curves. A crimper is not required.

Platinum Sample Pans (P/N 0319-0024)

Use platinum sample pans and covers when gold and graphite pans and covers are not suitable. These pans are used to analyze mineral, soil, and ceramic samples. They are also used to allow a chemical reaction where platinum acts as a catalyst. Platinum sample pans are easy to clean and are reusable. A crimper is not required.

Volatile Sample Pans

Volatile sample pans are used with volatile solid or liquid samples which exert significant vapor pressure at the temperature of interest. They are available in both aluminum (P/N 0219-0062) and gold (P/N 0219-0080). The benefits of the volatile sample pan are as follows:

- They can withstand an internal pressure of 2 atm (30 psi).
- Aqueous solutions can be scanned up to and through 100°C to observe solute behavior.
- The heats of fusion of materials that sublime can be determined can be determined accurately.
- The effect of an enclosed atmosphere on thermal behavior of a sample can be observed.
- Capsules have an effective volume of 20 μL.
- Gold pans are available for samples requiring temperatures above 600°C.
- Covers with 50-μL-pinhole lids are available [Vapor Pressure Sample Pan Kit (P/N N519-0788)] for measurement of boiling points, heats of vaporization, and sublimation temperatures.

The major applications for this type of pan are purity analyses (i.e., pharmaceuticals, melting of lipids or liquid crystals) and phase transitions, heat of vaporization, and boiling points.

See the topic on how to **Seal a Volatile Sample Pan Using a Volatile Sample Sealer** in the Pyris Multimedia Presentations Help.

Large-Volume O-Ring-Sealed Stainless-Steel Pans

Large-volume O-ring-sealed stainless-steel sample pans (P/N 0319-0218) contain the sample in a sealed environment throughout an experiment. The Viton O-ring allows formation of a seal which suppresses the vaporization of a solvent or contains a volatile reaction product, thereby eliminating the interfering effects of the heat of vaporization. The benefits of this type of sample pan are

- sealed capsules prevent any mass loss
- capsules can withstand an internal pressure of 24 atm
- high internal pressure capability allows water samples to be heated higher than 100°C
- capacity of 60 µL
- large sample capacity yields higher sensitivity
- operating range of –40°C to 300°C unless otherwise limited by sample vapor pressure

The large-volume O-ring-sealed stainless-steel sample pan is used in the study of aqueous biological solutions (such as protein in water) where the dilution requires large sample sizes and water vaporization must be suppressed. They are also used in epoxy and phenolic curing and vulcanization of rubber where the loss of volatiles can otherwise make interpretation difficult.

See the topic on how to **Seal a Large-Volume O-Ring Stainless-Steel Sample Pan** in the Pyris Multimedia Presentations Help.

NOTE: A large-volume O-ring-sealed stainless-steel sample pan can be sealed with a **universal crimper press** with optional sealing insert (P/N B050-5340) or with a **quick press** with the quick press spacer (P/N 0319-1047).

Large-Volume Stainless-Steel Capsule Kit (P/N 0319-0021)

This kit contains the quick press (P/N 0990-8467), the quick press spacer die (P/N 0319-1047), guard ring inserts (1 pair) (P/N 0319-0236), and 20 pans, covers, and O-rings (P/N 0319-0218).

High-Pressure Capsules

For DSC runs where high pressure is expected in the capsule, we recommend that you use reusable high-pressure capsules. The reusable high-pressure capsules and sealer are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used. The capsules are safe to handle because of a bursting disk on the cover of the capsule that allows the sample to escape if the pressure should exceed 150 atm.

There are three high-pressure capsules available:

Stainless-Steel High-Pressure Capsules (P/N B018-2901)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number contains 5 capsules and 20 copper seals.

Gold-Plated High-Pressure Capsules (P/N B018-2902)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number consists of 5 capsules and 20 gold-plated copper seals.

Titanium High-Pressure Capsules (P/N B018-2903)

For use in DSC runs where high-pressure is expected in the capsule, e.g., explosive materials. Part number consists of 5 capsules and 20 titanium seals.

Some of the benefits of using the high-pressure capsule are

- permits suppression of the endothermic signal resulting from the volatilization of sample material or from the volatilization or decomposition of reaction by-products
- permits the study of explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used
- availability in stainless steel, gold-plated steel, and titanium
- can withstand an internal pressure of 150 atm maximum
- broad temperature range, subambient to 400°C
- reusable after sealing
- used in hazard testing

The high-pressure capsules are sealed with the high-pressure capsule sealing tool (P/N B018-2864).

See the topic on how to **Use the High-Pressure Capsule Sealing Tool** in the Pyris Multimedia Presentations Help.

Robotic System Sample Pans

The robotic system sample pans are used to prepare sample materials for the robotic system for the DSC 7. Pans are available in sealed or vented versions. These pans can also be used for nonrobotic systems. The Universal Crimper Press (P/N B013-9005) provides high-quality sample crimping for the robotic system sample pans.

Below is a list of robotic system pans available:

1-bar (100 kPa) maximum internal pressure

Part No.	Capacity	Thickness	Description
B014-3015	10 µL	0.1 mm	sealed aluminum pans (400)
B014-3016	30 µL	0.1 mm	sealed aluminum pans (400)
B014-3017	50 µL	0.1 mm	sealed aluminum pans (400)
B014-3018	30 µL	0.1 mm	vented aluminum pans (400)
B014-3019	50 µL	0.1 mm	vented aluminum pans (400)
B014-3024	50 µL	0.1 mm	sealed gold pans (10)

2 bar (200 kPa) maximum internal pressure

B014-3020	25 µL	0.15 mm	sealed aluminum pans (400)
B014-3021	50 µL	0.15 mm	sealed aluminum pans (400)

See the topic on how to **Crimp the Robotic System Sample Pan Using the Universal Crimper Press** in the Pyris Multimedia Presentations Help.

Robotic System Sample Pan Covers

B014-3003	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (400)
B014-3004	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (400)
B014-3040	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (2000 covers)
B014-3050	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (800)

Starter Kits

B014-6340	Aluminum Sample Pan Starter Kit. Contains 50 of each type of robotic system aluminum sample pan and 350 sample pan covers.
B014-3030	Aluminum Sample Pan Starter Kit. Contains 400 of each type of robotic system aluminum sample pan and 2000 sample pan covers.

Pan and Cover Kit (2 atm, 200 kPa)

B016-9320	Aluminum sample pans and covers, 30-µL internal volume (400 pans and covers)
B016-9321	Aluminum sample pans and covers, 50-µL internal volume (400 pans and covers)
B700-1015	Aluminum sample pan cover with centered 2.5-mm hole. Designed for OIT measurements and for experiments that require an open sample pan for maximum exposure of the sample to the gaseous environment. Can be used with 30- or 50-µL Robotic System aluminum sample pan bottoms (400 lids)
B700-1014	Aluminum sample pan cover with centered 0.05-mm hole. Designed for vapor pressure and boiling point measurements. Can be used with 10-, 30-, or 50-µL Robotic System aluminum sample pans (400 lids)

Sample Encapsulation

The standard aluminum sample pans and covers provided with the DSC are suitable for most thermal analysis applications. The most common method of sample encapsulation is crimping the sample pan cover in place. The normal procedure yields a tightly, but not hermetically, sealed pan. When maximum contact of the sample with the atmosphere is necessary, perforate the sample pan cover with tweezers before crimping or use a cover punched from a 60/100 mesh screen.

For materials that emit volatile byproducts or need to be hermetically sealed, additional sample pans are available.

See the topic on **How to Encapsulate Samples** in the Pyris Multimedia Presentations Help.

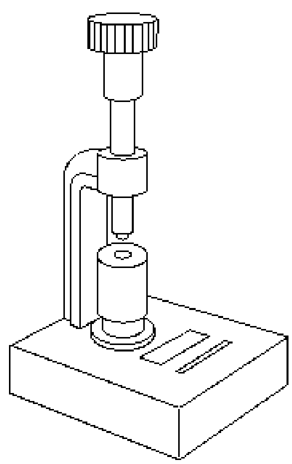
Crimpers

The following table lists the crimpers available for the sample pans used with a DSC for most thermal analysis applications.

Crimper	Use	Part No.
Standard sample pan crimper press	Crimps covers on Standard Sample Pans of aluminum, gold, and copper. Has a replaceable crimper head	0219-0048
Volatile sample sealer	Used with Volatile Sample Pans	0219-0061
High-pressure capsule sealing tool	Used with High-Pressure Capsules	B018-2864
Universal crimper press	Used with the Large-Volume O-Ring-Sealed Stainless-Steel Pans and Volatile Sample Pans. This is accomplished by using appropriate sealing insert.	B013-9005
Quick Press	Used with Large-Volume O-Ring-Sealed Stainless-Steel Pans	0990-8467

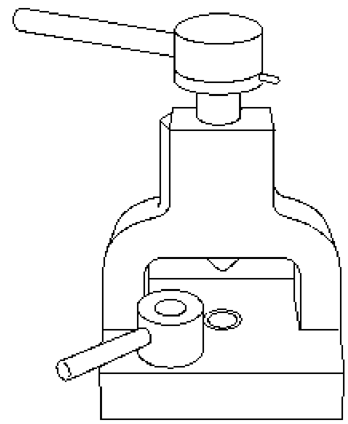
Standard Sample Pan Crimper Press

The Standard Sample Pan Crimper Press (P/N 0219-0048) is used to crimp covers on standard sample pans of aluminum, gold, and copper for use with the DSC 7. Samples are crimped in pans but are not hermetically sealed. The design incorporates a replaceable crimper head.



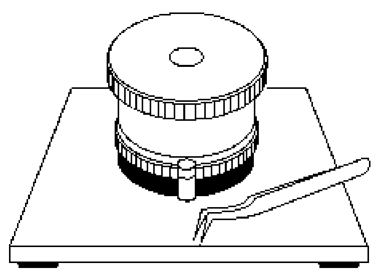
Volatile Sample Sealer

The Volatile Sample Sealer (P/N 0219-0061) is used to seal volatile sample pans. The pans and sealer are used with volatile solid or liquid samples that exert significant vapor pressure in the temperature range of interest. For example, aqueous solutions can be scanned up to and through 100°C to observe solute behavior. The heats of fusion of materials which sublime (e.g., camphor) can be determined accurately using these sample pans. Additionally, the effect of an enclosed atmosphere (e.g., water vapor evolved in dehydration below 100°C) on thermal behavior of a sample can be observed.



High-Pressure Capsule Sealing Tool

The high-pressure capsule sealing tool (P/N B018-2864) is used to seal the reusable high-pressure capsules. Those capsules are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used.



The capsules that can be sealed by this tool are

- **stainless-steel high-pressure capsules**
- **gold-plated stainless-steel high-pressure capsules**
- **titanium high-pressure capsules**

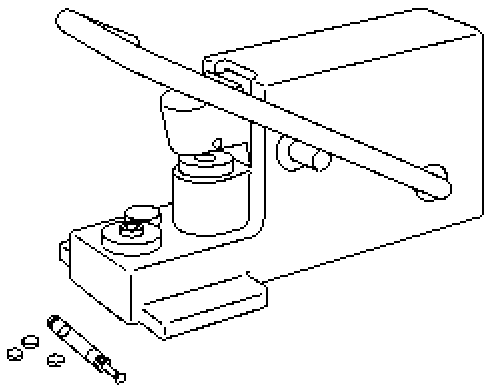
Vapor Pressure Sample Pan Kit (P/N N519-0788)

This kit includes 100 volatile aluminum pans and 100 pierced covers. A 50-µm-diameter hole is centered in the cover. Use this kit for more reproducible measurements of boiling points, heats of vaporization, and sublimation temperatures. Vapor pressure studies can be conducted when using these pans and covers with the high-pressure DSC.

Universal Crimper Press

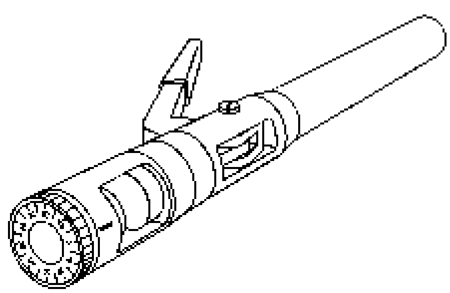
The Universal Crimper Press (P/N B013-9005) is used for sealing all of the Robotic System sample pan types as well as most of the other DSC sample pans when used with the appropriate optional sealing insert. Other pans that can be crimped by a universal crimper press are

Part No.	DSC Sample Pan Type
B050-8921	Standard Sample Pans
B050-5340	Large-Volume O-Ring Sealed Stainless-Steel Sample Pans
B014-4637	Volatile Sample Pans
B013-9033	Robotic System Sample Pans (included with the Universal Crimper Press)
B017-4929	12.5-L, 2-atm aluminum sample pans



Quick Press

The Quick Press (P/N 0990-8467) is used to crimp large-volume O-ring sealed stainless-steel sample pans (P/N 0319-0218) which are used with samples that vaporize or contain a volatile reaction product in the temperature range of interest. Application areas include the study of dilute aqueous solutions or curing reactions at temperatures below 300°C (e.g., phenolic or epoxy resins).



Sample Size

The quantity of sample that can be analyzed by the DSC is limited only by the volume of the sample pan that you use. However, the sample size in conjunction with the scanning rate and the sensitivity (Y range) will affect the quality of the results.

With the DSC, running a large sample at a fast scanning rate will improve the usable sensitivity. Running a small sample at a slow scanning rate will improve the resulting peak resolution. Most samples run on the DSC analyzer will be in the 0.5–30-mg range.

NOTE: Use small samples of 1 mg or less for decomposition studies and other analyses in which the sample may tend to contaminate the sample holders or in which the behavior of the sample has not previously been investigated. Also, use a flow through cover in such analyses.

Sample Atmosphere

CAUTION: Always use helium as the purge gas when liquid nitrogen is used for subambient operation. Never leave the purge gas exit line immersed in a liquid.

You can control the atmosphere in which the encapsulated sample is run by using a purge gas to displace or introduce reactive gases into the sample furnaces. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another, always check the temperature and energy calibration. Flow rates of 20 – 30 cc/min of purge gas are recommended. These flow rates will be realized by inlet purge gas rates of 20 – 30 psi.

Argon or nitrogen of 99.9% minimum purity is recommended for purging the sample holders when operating at ambient temperatures and above. **The gas must be dry.** A size 1A cylinder equipped with a suitable regulator is recommended. The regulator should be equipped with a shutoff valve at the outlet. The shutoff valve should have a 1/4-in. NPT male thread on the outlet side for connection to the DSC's purge gas line. A purge gas flow restrictor should not be placed in the line as this is contained in the purge gas system within the instrument.

For degradation studies or for experiments on samples that offgas during transition or reaction, a Flow Through Cover Kit (P/N 0319-0062) is required on the DSC 7. The Flow Through Cover permits the purge gas and volatiles to exit directly through the top of the DSC 7 cover. The recommended flow rates of the purge gas are 40 – 50 cc/min. These flow rates will be realized by inlet purge gas pressures of 40 – 50 psi. Air or oxygen may be used as the purge gas.

Argon and nitrogen are the recommended purge gases for Intracoolers 1 and 2 and for the CCA 7 Liquid Nitrogen Controlled Cooling Accessory used with the DSC 7. Helium is the recommended purge gas for liquid nitrogen subambient operation. Recommended flow rates of the purge gas are 20 – 30 cc/min. These flow rates will be realized by inlet purge gas pressures of 20 – 30 psi.

The GSA 7 Gas Selector Accessory is a computer-controlled gas-switching device. The GSA 7 permits the automatic switching of sample purge gases at an operator-selected time or temperature during an analysis. You are allowed two gas changes per temperature program step with the GSA 7.
The TAGS (Thermal Analysis Gas Station) may also be used for automatic switching of four sample purge gases using a predefined operation program that you set up in the method. You are allowed up to ten gas changes per temperature program step with the TAGS.

Temperature Range

The temperature range for your analysis depends on the sample and the applications of your results. The DSC 7 can be used to analyze samples from a temperature of –170°C (when used with a Liquid Nitrogen Accessory and dry box) to 725°C (in ambient operation). Isothermal analyses also can be performed at any selected temperature in the range of the instrument.

CAUTION: Do not use aluminum sample pans when programming the DSC to run above 600°C. Since aluminum melts at 660°C, the pans will alloy with and destroy the sample holders. Always make sure that you are not using aluminum pans in either the sample or the reference cell when you want the temperature of the DSC to exceed 600°C.

Scanning Rate

The DSC will allow scanning rates of 0.1°C/min to 500°C/min in steps of 0.1°C/min. Proper selection of scanning rate will increase efficiency of your analysis at the desired sensitivity. Generally, slower scanning rates improve the peak resolution while faster scanning rates improve the usable sensitivity.

NOTE: Scanning rates greater than 100°C/min are normally used only for rapidly heating or cooling the DSC to starting temperatures or to selected isothermal temperatures. Typical experimental scanning rates range from 5°C/min to 40°C/min.

Sample Loading

Prior to loading the sample into the DSC 7 sample holder, the sample must undergo sample preparation and encapsulation. In addition to encapsulating the sample, it is recommended that a reference capsule be used in the reference furnace during the DSC experiment. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated.

Load the sample and reference pans into the DSC 7 as follows:

1. Remove the draft shield and open the sample holder enclosure by lifting the latch and swinging the enclosure cover to the side.
2. Note the orientation of the platinum sample holder covers. Remove them from the sample holders with a pair of tweezers.
3. Using tweezers, place the encapsulated sample in the left sample holder and place an empty pan and cover (reference) in the right sample holder.
4. Replace the sample holder covers back on the sample holders, oriented as before. Maintaining the sample holder covers’ orientation improves the baseline repeatability, especially at high temperatures.
5. Close the sample holder enclosure, lock it into place, and replace the draft shield.

CAUTION: When loading or removing sample materials from the sample holder, make certain that the guard ring inserts are in place. If you are not using guard ring inserts, you must use the sample handling tray (P/N 0419-0303) which is provided with the DSC 7. Failure to use the guard ring inserts or the sample handling tray may cause the sample holder covers or sample pans to drop inside the sample holder cavity, damaging the sample holder.

See the topic on how to **Insert the Guard Rings** in the Pyris Multimedia Presentations Help.

Specific Heat Analysis

The determination of specific heat capacity by a power-compensated DSC analyzer is an important tool in thermal analysis. Heat capacity is an intrinsic property of a material. It is the amount of energy needed to increase a unit quantity (e.g., 1 g, 1 mole) of material 1°C. All materials have specific heat. If there is no phase transition or reaction, the specific heat is a positive number that gradually changes with temperature.

Specific heat is fully normalized output of a DSC, i.e., the displacement during a scan is equal to the product of the heat capacity times the sample weight times the scanning rate plus the no-sample baseline. DSC data is sometimes misinterpreted because just one heat flow run was performed. The data from that run may contain information about the instrument itself such as the specific heat of the reference side of the sample holder. This problem can be fixed by performing a baseline run with no sample using the same conditions to be used for the sample. This constitutes the classical two-curve Cp method. This is illustrated here by finding the specific heat of the reference standard sapphire using the DSC 7.

The following steps were performed in order to obtain the specific heat of sapphire:

1. Prepare the DSC 7.
2. Place an empty standard aluminum sample pan and lid in each sample holder in order to run a no-sample baseline.
3. Enter the parameters into the Preferences pages and the Method Editor. Use a Sample Weight of 0 for the baseline run:

Purge Gas	Nitrogen at 20 cc/min
Cooling Device	Ice bath
Sample Weight	28.12 mg
Initial Temperature	50°C
Y Initial	0 mW
Equilibrate Temp.	0.01°C
Heat Flow	0.01 mW
Wait no longer than	15 min
Method Program	Isothermal – Scan – Isothermal
Heating/Cooling Rate	10°C/min
Temperature Range	50 – 100°C, 100 – 150°C, . . . , 250 – 300°C

4. Click on the **Start Method** button to start the baseline “no sample” run.
5. Find the sapphire standard in the Specific Heat kit (P/N 0219-0136) and encapsulate it in the standard aluminum sample pan used in the baseline run.
6. Enter the sample weight of 28.12 mg into the Method Editor.
7. Click on the **Start Method** button to start the sample run.
8. After data collection is complete, click on the **Data Analysis** button on the toolbar.
9. Using Add Data in the File menu, select the two data files you just collected. The baseline curve should be the lower curve and the sample curve should be the top curve.

10. The Step Select feature should be activated. Make sure that a checkmark appears next to Step Select in the Curves menu. If it does not, click on Step Select.
11. If you have data from multiple scanning steps, the Start Time at Zero feature should not be checked in the Curves menu.
12. The data is now in the form such that you can calculate the specific heat for each iso–scan–iso step.
13. Make sure that the sample curve is the active curve (heavy line display). Click on Heat Flow in the Curves menu. The Step Select dialog box is displayed.
14. From the list of steps, select adjacent iso–scan–iso steps to create a segment. Click on **OK**. If your method had 11 steps, you should be able to define 3 segments.
15. Repeat steps 11 and 12 for the baseline curve but use Baseline Heat Flow from the Curves menu.
16. Click on a segment of the sample curve and choose Multiple Curve from the Specific Heat submenu in the Calc menu.
17. The default selection for the baseline curve should be the segment of the curve that coincides with the segment of the sample curve selected. Click on **OK** or press **Enter** to accept the selection.
18. The specific heat curve and its ordinate scale is added to the display.
19. In the case of multiple-step data, repeat steps 15 and 16 for each scanning step (iso–scan–iso segment).
20. Change the X scale to Temperature using Rescale X in the Display menu or in the toolbar.

You are finished finding the specific heat of sapphire.

Operational Suggestions for Accurate Cp Analysis

To obtain a more accurate specific heat measurement, try to follow the suggestions below:

- **DSC Block Stability:** The DSC analyzer block temperature should be stable. For above-ambient specific heat, use ice water as the coolant. Water alone is less satisfactory because the water slowly warms up over time. An Intracooler is good for subambient work. Allow sufficient equilibration time after changing the heat sink temperature before running a baseline. One hour should be sufficient.
- **Purge Gas:** Use dry nitrogen or argon for the purge gas. Defective or inferior regulators can result in irregular purge flow with resultant baseline wanderation. Avoid using flow restrictors and flow meters on the inlet or outlet purge during data acquisition.
- **Calibration:** When performing a two-curve Cp analysis, it is important that the instrument be carefully calibrated. Single-point calibration is not sufficient. Use high-purity melting standards to bracket the temperature range of interest. Use the melting energy of an accurately weighed specimen of indium for energy calibration.
- **Preconditioning of the DSC:** Before performing the first of several high-sensitivity or high-accuracy DSC analyses, it it good practice to heat the analyzer to the highest temperature that will be achieved during your analysis, hold the temperature briefly, and then return to the Load temperature. Use the **Go To Temp** button on the control panel.
- **Temperature Program:**
    - Isothermal steps must be long enough to allow heat flow equilibration which depends on sample size and pan type. Two to three minutes is usually sufficient for step length.
    - The initial isotherm must be at least 30°C above block temperature.

- No physical or chemical process (e.g., decomposition, moisture loss) should occur during either isotherm.
- Do not try to cover a wide temperature range between isothermal steps. The isothermal steps behave like a reference for heat flow. By aligning the isothermal data, which is done automatically by the software, the effects of a slight baseline shift are minimized. If a wide temperature range must be used, it is preferable to divide the analysis into several steps, selecting the steps so that they bracket regions with transitions.
- During the scanning step, for a small sample, use a rapid heating rate (e.g., 20°C/min); for very large samples, use a low rate (e.g., 5°C/min). This ensures uniform heating of the sample.

- **Sample Pans:** You must use the same sample pan and lid for the sample run that you use for the no-sample baseline run. This assures that there will be no Cp error caused by weight difference. Use crimpable standard aluminum pans. For volatile samples, use a sealed pan that will not burst from the pressure of escaping volatiles. If possible, use a vacuum oven to remove the volatiles prior to the run so that you can use a crimpable sample pan.
- **Sample Size:** The sample size depends on the desired heating rate. A sample of 5 – 25 mg is normal.
- **Reference Side:** Place an empty pan in lid in the reference holder for both the sample run and the baseline run.
- **Platinum Sample Holder Covers:** Always use the platinum sample holder covers (in addition to the sample pan lids). Before beginning specific heat analysis, identify one with a distinguishing mark to help prevent mixing up the two lids. Take care to use the same lid on the same side consistently.

Calibration

Calibration of the DSC 7 for temperature and energy is accomplished by running high-purity standard and reference materials with known temperature and energy transitions. (Perkin Elmer ships reference materials in the Spares kit with each analyzer.) The data obtained after running these materials are used in the calibration programs to automatically calibrate the DSC. Once the calibration is performed, the analyzer will be calibrated continuously, even when the system is turned off. Unless major analyzer condition changes are made, the DSC should remain calibrated.

Calibration Precautions

The following precautions must be observed when the calibrating a DSC 7:

- Do not use aluminum pans above 600°C since they will alloy with and contaminate the sample holders.
- All metal reference materials (indium, tin, lead, zinc) must be encapsulated in aluminum pans because they will alloy with gold or platinum pans.
- All inorganic reference materials (potassium sulfate, potassium chromate) should be encapsulated in gold pans.
- For the best temperature repeatability, the reference material should be flattened and placed in the center of the sample pan and then encapsulated. The sample pan should be placed in the center of the sample holder.

- When using subambient reference materials, it is recommended that they be encapsulated in volatile sample pans for a tight, hermetic seal (P/N 0219-0062 and 0219-0080 and volatile sample sealer 0219-0061).
- When using liquid nitrogen or an Intracooler, always observe the precautions for liquid nitrogen use.

When to Calibrate the DSC

Once you have performed the automatic calibration programs, the temperature and heat flow (energy) calibration of the DSC should remain unchanged for an extended period, provided there are no changes in the instrument operating conditions. (Periodic checks of the calibration typically show variations of <1% after many days.) There are a few conditions that will change either the temperature or the energy calibration and cause the need to recalibrate:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Run a reference material in the new range to determine if the current calibration is still valid.
- If the Balance is adjusted, recalibration may be necessary. Always optimize the baseline before the instrument is calibrated.
- If the purge gas type or purge gas flow rate is changed, the calibration should be checked for highest accuracy.
- If the coolant or cooling accessory used with the DSC is changed, recalibration may be necessary.
- Since temperature calibration depends slightly on scan rate, final calibration should be performed at the scan rate that you will use for your experiments. As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating rates, or heating and cooling rates, it is better to calibrate at the slowest rate to be used.
- If the instrument has been turned off for a long time (i.e., weeks or months), the instrument may appear to need recalibration. Condition the DSC by performing several heating and cooling runs with the sample holder empty and then check the calibration by running reference materials.

DSC Calibration Routines

The following calibration routines are available for the DSC 7:

- **Calibrate Temperature**
- **Calibrate Heat Flow**
- **Calibrate Furnace**

NOTE: If you need to **optimize the baseline** using the Balance and Slope controls, do so before calibrating the analyzer.

As part of maintaining proper operating conditions for your instrument, you should calibrate it when it is first installed and when you change the sample holder. The recommended routine to use is the Temperature calibration routine. It is recommended also that the DSC be calibrated every three to six months after installation.

Calibrate Temperature

1. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. Restore the default Temperature calibration by selecting Temperature from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the All command.
3. Select the **Save and Apply** button in the Calibration window to send the default calibration values to the analyzer and save the current calibration file.
4. Select **Close** to close the Calibration window.
5. Complete a scan for each reference material under the same conditions that you run your samples.
6. Perform a Peak Area calculation and include the Onset temperature. Record the ΔH (J/g) and Onset results; you will need the Onset result for Temperature calibration and the ΔH result for Heat Flow calibration.
7. Repeat steps 5 and 6 for each additional reference material to be used.
8. Select the Calibrate command in the **View** menu. The Calibration window appears.
9. Select the Temperature tab. Enter the name of the reference material used, the expected Onset value, and the Onset result just measured for each reference material.
10. Select the check box in the Use column for each reference material to be used in the calibration.
11. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the current calibration file.
12. Go on to the next calibration procedure by clicking on its tab or select **Close** to close the Calibration window and begin using the new calibration values.

Calibrate Heat Flow

1. While using the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. Restore the default Heat Flow calibration by selecting Heat Flow from the Restore menu. If you performed a Temperature calibration just prior to starting a Heat Flow calibration and selected All from the Restore menu, then you do not need to restore the default Heat Flow calibration here.
3. Select **Save and Apply**.
4. Select **Close**.
5. Complete a scan using a reference material or use one that was run for the Temperature calibration.
6. Perform a Peak Area calculation and note the ΔH (J/g) result. You can also use the ΔH result recorded for one of the reference materials used in the Temperature calibration.
7. In the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
8. Select the Heat Flow tab. In the Calibration table, enter the name of the reference material used, the expected ΔH value, the ΔH result just measured, the weight of the reference material used, and the name of the method file used for the run.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration value to the analyzer and save the current calibration file.

10. Go on to the next calibration procedure by clicking its tab or select **Close** to close the Calibration window and begin using the new calibration values.

Calibrate Furnace

This calibration is available only if the instrument has DDSC capability.

1. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. If applicable, complete the Temperature calibration.
3. Remove the pans from sample and reference holders.
4. Select the Furnace tab in the Calibration window.
5. In the Minimum field, enter a minimum temperature that is below your normal operating region.
6. In the Maximum field, enter a maximum temperature that is above your normal operating region.
7. Select the **Begin Calibration** button.
8. Wait the designated time for completion of the Furnace calibration.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.
10. Select **Close** to close the Calibration window and begin using the new calibration values.

DSC Calibration Reference Materials

Your company may have to comply with ISO 9000. Perkin Elmer Thermal Analysis calibration materials are ISO 9000 compatible. They are called Reference Materials. ISO has two classifications for calibration materials: Reference Materials and Certified Reference Materials.

Reference Materials (RMs) comply with ISO for instrumentation calibration. They are available from Perkin Elmer and Perkin Elmer provides documentation with each RM that cross references the material’s lot code. This declaration should be filed with all other material certificates and documents in your ISO file.

ISO Guide 30 defines a RM as a

> “Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.”

Certified Reference Materials (CRMs) are traceable to national or international standards through an unbroken chain of comparisons. Not every calibration material is a CRM. CRMs can be very expensive and are not available for all thermal analysis techniques.

If you are not sure what material to use, then check with your Quality Assurance Department and discuss the differences between RMs and CRMs. If you find that you need CRMs, you may order them from the National Institute of Standards and Technology (301 975-6776).

Reference Material E is a standard epoxy–glass composite known as FR4. It is bisphenol A-type epoxy supported by four layers of woven E-type glass fibers. This type of material can be assayed by DSC thermal analysis. The supporting glass permits analysis well above the glass transition temperature. The epoxy resin exhibits mechanical relaxations down to liquid nitrogen temperatures. It is highly crosslinked and changes little under normal storage and handling conditions. Reference material E has been analyzed by various techniques and has been found to agree with the manufacturer's specifications. Assay numbers are not indicated on the samples. Recent lots indicate a storage modulus of 1.1e10 to 1.8e10 Pa.

The different reference materials for operation and calibration of your DSC in the ambient to 725°C temperature range and for subambient operation are outlined in the following two topics.

Calibration Reference Materials from Ambient to 725°C

Very-high-purity (>99.9%) metals or inorganic materials are typically used to calibrate the temperature and energy axes of the DSC when operating in the temperature range from ambient to 725°C. Two high-purity metal reference materials [indium (P/N 0319-0033) and zinc (P/N 0319-0036)] are supplied with your instrument for calibration purposes. Other reference materials are also available from Perkin Elmer. The nominal transition temperatures and energies for each of these reference materials recommended for calibration are as follows:

NOTE: Temperature and energy transition values may vary slightly from one lot number to another. Refer to the documentation provided with the reference material to determine the values for that lot number.

Reference Material	**Part No.**	**Transition Point (°C)**	**Transition Energy (J/g)**
Indium	0319-0033	156.60	28.45
Tin	0319-0034	231.88	60.46
Lead	0319-0035	327.47	23.01
Zinc	0319-0036	419.47	108.37
Potassium Sulfate	na	585.0 ± 0.5	33.26
Potassium Chromate	na	670.5 ± 0.5	35.56

Also available from Perkin Elmer are kits of encapsulated reference materials:

0219-0045 includes a large and a small indium, tin, and lead

N519-0762 includes indium and zinc

The selection of the proper reference materials is crucial for the optimal performance of the DSC over the temperature range used for your experiments. Always select a reference material that encompasses that temperature range. For example, if you operate the DSC from 50°C to 200°C, use indium, which melts at 156.60°C.

If you wish to calibrate the energy as well as the temperature, you must accurately weigh the reference material before the run. Typical weights for standards and reference materials range from 1 to 10 mg. The accuracy and precision to which you weigh the sample are directly related to those of the energy measurements made on the DSC.

NOTE: The zinc reference material should not be heated more than twice.

Calibration Reference Materials for Subambient Operation

Very-high-purity (>99.999%) organic materials can be used to calibrate the temperature when operating the DSC in the temperature range from –170°C. See the table below for a list of recommended subambient calibration standards that can be used with your DSC. Normally, indium is the reference material that is recommended for energy calibration for all operating conditions.

Substance	Transition	Transition Temp. (°C)	Transition Energy (J/g)
Cyclopentane	Crystal	–151.16	69.45(a)
Cyclopentane	Crystal	–135.06	4.94(a)
Cyclohexane	Crystal	–87.06	79.58(b)
Cyclohexane	Melt	6.54	31.25(b)
n-Heptane	Melt	–90.56	140.16(c)
n-Octane	Melt	–56.76	182.0(c)
n-Decane	Melt	–29.66	202.09(c)
N-Dodecane	Melt	–9.65	216.73(c)
n-Octadecane	Melt	28.24	241.42(d)
Hexatriacontane	Crystal(*)	72.14	18.74(d)
Hexatriacontane	Crystal	73.84	60.25(d)
Hexatriacontane	Melt	75.94	175.31(d)
P-Nitrotoluene	Melt	51.64	

(*) Shows some thermal history dependence
(a) Aston, J.G. et al., *JAC*, **65**, 341 (1943)
(b) Aston, J.G. et al., *JAC*, **65**, 1035 (1943)
(c) Finke, H.L. et al., *JAC*, **76**, 33 (1954)
(d) Schaerer, A.A. et al., *JAC*, **77**, 2017 (1955)

1. The materials listed here, if used for calibration, must be of 99.999% minimum purity as even small levels of impurity can affect the temperature and/or energy of the transition.
2. These substances must be encapsulated in volatile sample pans. To improve peak resolution, place a small piece of aluminum foil over your sample before sealing the pan.
3. If the peaks are not sharp (as in indium), the sample may be impure and the temperatures you measure may not be correct. Use a higher purity sample or check the purity of the sample by an alternate technique such as gas chromatography.

Subambient Operation

The DSC 7 can be operated in the subambient mode. Perkin Elmer offers subambient accessories to satisfy most temperature requirements. There are four options for the DSC 7 that extend the temperature range downward to –30°C, –65°C, –140°C, and –170°C. A dry box is required for all subambient operations, and the four temperature ranges can be reached with the Intracooler 1, Intracooler 2, CCA 7, and the Liquid Nitrogen Accessory, respectively. Subambient performance of DSC 7 is comparable to ambient performance with respect to noise, resolution, baseline linearity, and repeatability.

In order to identify which subambient accessory your DSC requires, you must determine what temperature you would regularly cool to.

Subambient operation of the DSC 7 is obtained by using either liquid nitrogen or an Intracooler 1 or 2.

Liquid nitrogen allows operation to temperatures as low as –170°C.

Intracoolers 1 and 2 provide a convenient means of cooling the sample holder enclosure block of the DSC 7. Intracooler 1 is a single-stage closed-loop circulating heat exchanger and Intracooler 2 is a dual-stage closed-loop circulating heat exchanger. Each contains a compact mechanical refrigeration unit coupled to the unique cylindrical expansion chamber by means of a rugged, insulated stainless steel flexible line. The expansion chamber mounts directly on the DSC 7 sample holder enclosure block with special bolts. The refrigerant is a widely available, nontoxic, non-CFC refrigerant that can continuously produce block temperatures as low as –55°C in the case of the Intracooler 1 and –85°C in the case of Intracooler 2. This permits operation of the DSC 7 to –30°C or –65°C, respectively. The Intracooler should be placed on the floor either beneath or next to the bench with the DSC 7. To optimize the sample handling environment, a dry box must be installed on your DSC 7.

NOTE: The lowest temperature obtainable will depend on the ambient temperature and voltage in your laboratory.

- The DSC 7 Dry Box Assembly with Irises (N519-0226) or the DSC 7 Dry Box with Gloves (N519-0738) is required for operation of the DSC 7 with liquid nitrogen.
- The Liquid Nitrogen Level Indicator Kit (P/N N519-0185) is required for operation of the DSC 7 with liquid nitrogen.
- It is also recommended that the filter dryer accessory be used with all purge gases to eliminate moisture from the purge gases.
- In addition to the Dry Box Assembly and the Liquid Nitrogen Level Indicator Kit, the ambient DSC 7 (P/N N519-0245, N519-0246, N519-0247) must have a reservoir assembly and cold finger added for operation with liquid nitrogen.

Precautions for Liquid Nitrogen Use

Performance of the DSC 7 in the subambient mode should be similar to performance of the instrument in ambient mode with respect to noise, baseline linearity, baseline repeatability, resolution, etc. To achieve this level of performance, however, when using **liquid nitrogen** as a coolant, the following requirements must be met:

- Helium **must** be used as the purge gas for the sample holder. A gas such as nitrogen or argon should **not** be used because it has a condensation temperature close to that of the coolant, and thus, the increased density of that gas may lead to anomalous and nonrepeatable baselines.
- The helium purge gas **must** be exceptionally dry (use of the filter dryer accessory is recommended). Condensation may occur on the walls of the sample holder enclosure and affect baseline performance if the purge gas is not exceptionally dry.
- The dry box **must** be free of leaks and purged with dry nitrogen.
- On the DSC 7, the red rubber O-ring located in the swing-away sample holder enclosure lid **must** be removed for operation with liquid nitrogen. The sample holder enclosure purge gas exit line must be blocked with a Swagelok plug to prevent backdiffusion of moist gas into the enclosure.
- The use of liquid nitrogen as a coolant will require recalibration of both temperature and calorimetric sensitivity, which will be affected by a few degrees and a few percentage points, respectively.

- Always use extreme caution when working with liquid nitrogen. It can cause severe burns. Use the loading funnel provided with the accessory.
- Never overfill the liquid nitrogen coolant reservoir.
- Use of liquid nitrogen as a coolant requires that the instrument be placed in a well-ventilated area.
- Use protective gloves when handling samples and when opening the sample holder as these areas get extremely cold. Since hands may be a source of moisture, use plastic gloves when placing hands inside the dry box on the DSC 7.
- Always leave the DSC 7 and TAC 7/DX on overnight when operating in the subambient mode. This will help prevent moisture buildup in the sample holder. The sample holder temperature should be left on between 50°C and 100°C to prevent condensation inside the sample holder overnight. Make sure that the dry box is purged with dry gas before and during liquid nitrogen operation.

Installing Liquid Nitrogen Accessories

To install liquid nitrogen accessories into a **DSC 7**, see the following topics:

- **Dry Box Assembly**
- **Liquid Nitrogen Indicator Kit**

In addition to athe Dry Box Assembly and Liquid Nitrogen Level Indicator, ambient DSC 7 analyzers must have a reservoir assembly and a cold finger added for operation with liquid nitrogen. To install the accessory for using liquid nitrogen in an **ambient DSC 7**, see

- **Ambient DSC 7 Reservoir Kit**

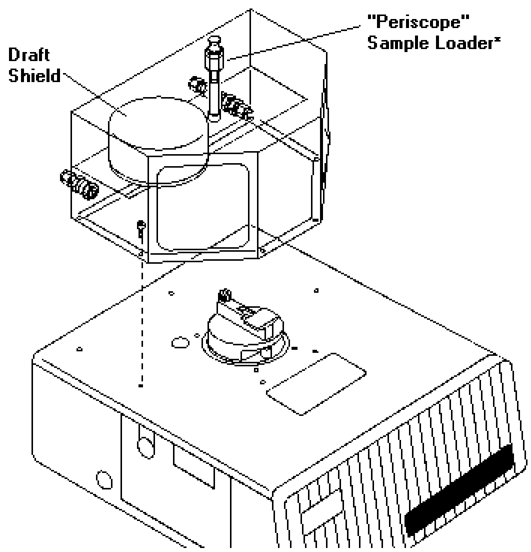
DSC 7 Dry Box Assembly

To optimize the sampling environment of the DSC 7 when operating in subambient mode, you **must install the dry box assembly**. The DSC 7 Dry Box Assembly with Irises (P/N N519-0226) and Dry Box Assembly with Gloves (P/N N519-0738) are to be installed **into a full-range or an ambient DSC 7** for operation at four temperature ranges: –30°C and above, –65°C and above, –140°C and above, and –170°C and above.

The dry box must be attached to the subambient analyzer deck (P/N N519-0139) before it is installed.

Assembling the Dry Box/Analyzer Deck Assembly

1. Shut down the DSC 7 system and remove the line power. Disconnect the ANALOG and DIGITAL cable connections (P/N N519-0103 or N519-0310) from the back of the DSC 7.
2. Place the subambient analyzer deck (P/N N519-0139) on a flat surface. (This is the deck with predrilled holes supplied with the DSC 7 Dry Box Assembly.)
3. Place the draft shield on the shelf inside the dry box.

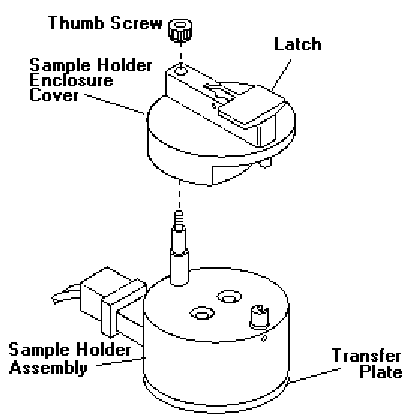


4. Place the dry box gasket on the analyzer deck so that the seven holes in the gasket match the seven holes drilled in the deck.
5. Place the dry box on the deck so that the holes in the dry box, gasket, and analyzer deck line up.
6. Use the seven dry box hold-down screw clamps to mount the dry box on the analyzer deck. Securely fasten all screw clamps. Tightening the screw clamps with your fingers will be sufficient. Do not overtighten.

You can now install the dry box and analyzer deck assembly.

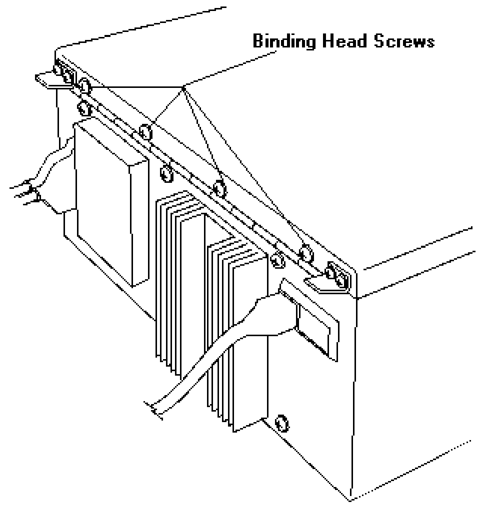
Installing the Dry Box/Analyzer Deck Assembly

1. Open the sample holder enclosure cover.
2. Loosen and remove the thumbscrew and remove the sample holder enclosure cover.



3. Remove all sample pans and sample holder lids from the sample holder.
4. Remove the plastic ring around the sample holder assembly.

5. Using a 1/8-in. Allen wrench, unscrew and remove the four Allen screws around the DSC 7 sample holder.
6. Turn the DSC 7 on the bench so that the left side of the instrument (i.e., the side with the two openings) is facing you. On the back of the analyzer, remove the four binding head screws and their washers securing the deck hinge to the cover. Lower the hinge.



7. Open the front panel of the DSC 7 and remove the two binding screws and their washers securing the front of the deck.
8. Lift off the analyzer deck.
9. Place the subambient analyzer deck with the dry box assembly attached to it on the DSC 7. On the back of the analyzer, raise the hinge and replace the four binding head screws and their washers to secure the hinge to the deck. Make sure that the hinge screws are secured tightly.

Liquid Nitrogen Level Indicator Kit

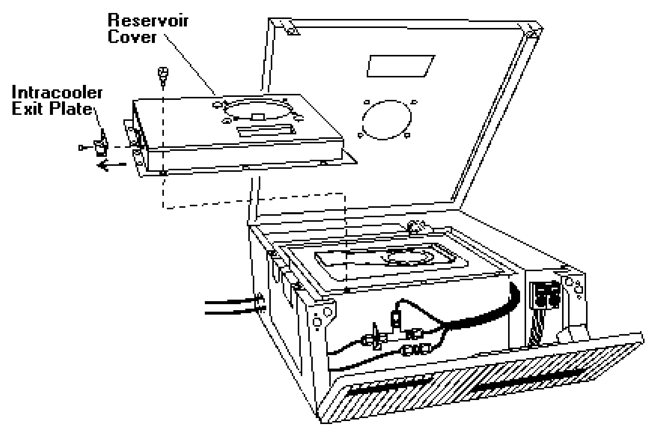
To operate a full-range or an ambient DSC 7 in subambient mode at –170°C and above with liquid nitrogen as the coolant, you must install the Liquid Nitrogen Level Indicator Kit (P/N N519-0185). The kit contains the following items:

Part No.	Description	Quantity
0419-0276	LN2 float and shaft	1
N519-1459	Cap for shaft	1
0250-5815	4-in. o.d. x 3/16-in. i.d. copper tubing	6 ft
0250-6240	insulation for copper tubing	6 ft
0990-3276	3/8-in. to 1/4-in. reducing union for connecting copper tubing to the reservoir	1

Installing the Liquid Nitrogen Indicator Kit

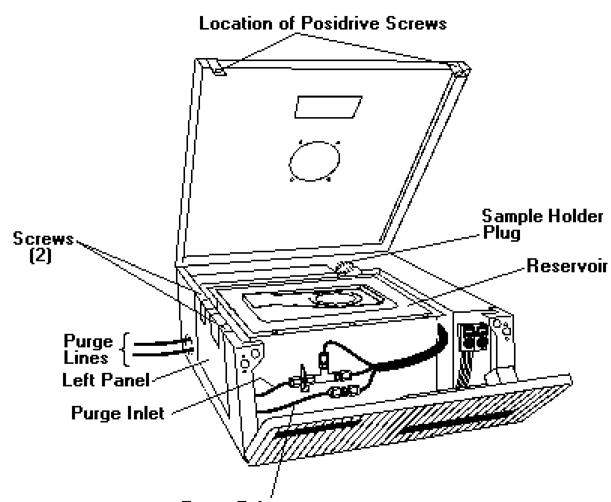
1. If you have not done so already, block off the purge gas exit (EXHAUST) line with a Swagelok plug to prevent back-diffusion of moist gas into the enclosure.

2. Raise the analyzer deck. The deck has two small tabs that allow it to remain stationary while in the vertical position without being propped up. Be careful not to jar the analyzer, however, since the deck may fall.
3. Unscrew and remove the 15 screws securing the top of the reservoir and remove the reservoir cover.

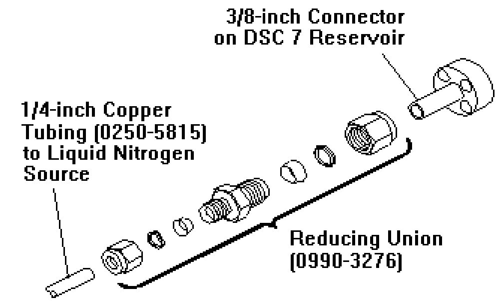


NOTE: Two longer shoulder screws are inserted into the Intracooler exit plate on the left side of the reservoir cover. Keep these screws separate from the others.

4. Pass the shaft of the liquid nitrogen float (P/N 0419-0276) through the hole in the reservoir cover, making sure that the markings on the shaft face the front of the DSC 7. Replace the reservoir cover and float assembly, securing it with the 15 screws.
5. Loosen the two small screws which hold the left side door of the analyzer closed and open the door by swinging it down.

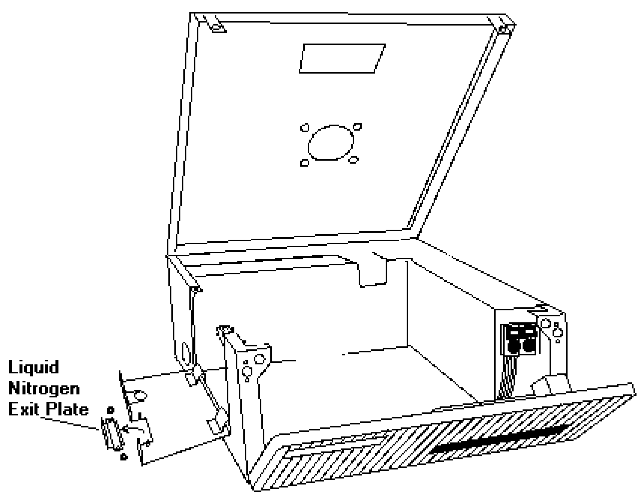


6. If attached, remove the Tygon drain tube from the left side of the reservoir.
7. Using the reducing union (P/N 0990-3276), connect the copper tubing to the filling tube on the left-hand side of the reservoir. Cover the copper tubing with the tubing insulator provided.



The copper tubing serves as the liquid nitrogen inlet to the reservoir. The outer end of the insulated copper line should be connected to a low [172.5 kPa (25 psi)] pressurized tank of liquid nitrogen.

8. Remove the two nuts and washers from the liquid nitrogen exit opening on the left access door of the DSC 7.



Once these nuts and washers are removed, the plate on the door can be removed. (The liquid nitrogen exit opening plate is the smaller of the two plates on the door.) Removing this plate provides the chassis exit opening for the insulated copper liquid nitrogen line.

9. Close the left access door of the DSC 7 and secure it in place by tightening the two securing screws.

10. Lower the analyzer deck, making sure that the float shaft does not bind on the deck.

    a. Make sure the sample holder is centered in the analyzer deck's opening. If it is not, position the reservoir until the sample holder is centered.

    b. Make sure the screw holes for replacing the four Allen head screws around the sample holder are aligned with the holes in the reservoir cover.

    c. When the sample holder and screw holes are centered, lift the analyzer deck and tighten the four reservoir mounting screws at the sides of the reservoir to lock the reservoir into position. Lower the analyzer deck.

11. Reinstall the front two screws and washers that secure the deck. Install the four Allen screws around the sample holder.

12. Close the front panel. Screw the cap (P/N N519-1459) onto the float shaft.

NOTE: Do **NOT** replace the plastic guard ring around the sample holder. Keep the guard ring inserts in place when using liquid nitrogen as a coolant. However, since baseline stability is slightly better when the guard rings are not in place, there are a few applications, e.g., specific heat capacity, for which you may want to remove them. Use the sample handling tray (P/N 0419-0303) provided for loading or removing samples from the sample holder.

13. Remove the red rubber O-ring seal from the groove on the sample holder enclosure cover. This O-ring is not used when operating with liquid nitrogen.
14. Install the sample holder enclosure cover.
15. Place the insulated reservoir access panel (P/N 0419-0261) over the opening at the front of the analyzer deck. Secure the access panel assembly in place with the two screws provided.

After installing the Liquid Nitrogen Indicator Kit, you must set up the DSC 7 for operation.

Setting Up the DSC 7 for Liquid Nitrogen Operation

1. Connect the ANALOG and DIGITAL cable connections (P/N N519-0103 or N519-0310) at the back of the DSC 7.
2. Plug in the DSC 7 to line power.
3. Set the helium purge gas flow rate to 20 psi at the tank regulator. Connect a source of dry nitrogen to the purge gas fitting on the back of the dry box.
4. Connect the end of the insulated copper tubing leaving the left side of the DSC 7 to the liquid nitrogen delivery source.

Ambient DSC 7 Reservoir Kit

To operate an **ambient** DSC 7 at subambient temperatures using liquid nitrogen, you **must** install the Ambient DSC 7 Reservoir Kit. The Ambient DSC 7 Reservoir Kit (P/N N519-0312) includes the following parts:

Part No.	**Description**	**Quantity**
0250-6519	Tygon tubing	6 ft
0419-0274	Reservoir with cover and cold finger	1
0992-0008	Hose clamp	10
N519-0506	Weld assembly	1
N519-1880	Gasket	1
0993-9973	Instructions	1

Installing the Reservoir Kit in an Ambient DSC 7

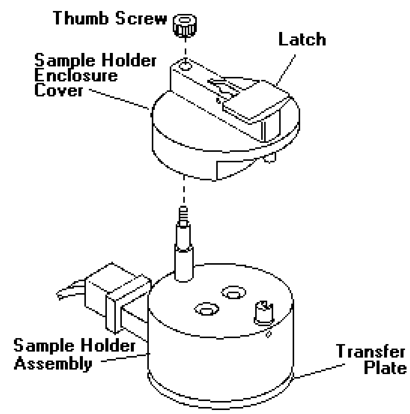
To install the DSC 7 Reservoir Kit (P/N N519-0312) in an ambient DSC 7 in order to operate at subambient temperatures down to –170°C, perform the following two steps:

- **Remove the Sample Holder Assembly**
- **Install the Reservoir Kit**

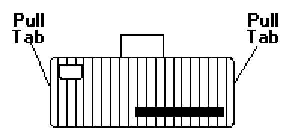
Remove the Sample Holder Assembly

Before installing the reservoir kit in an ambient DSC 7, you must remove the sample holder assembly and reservoir cover as follows:

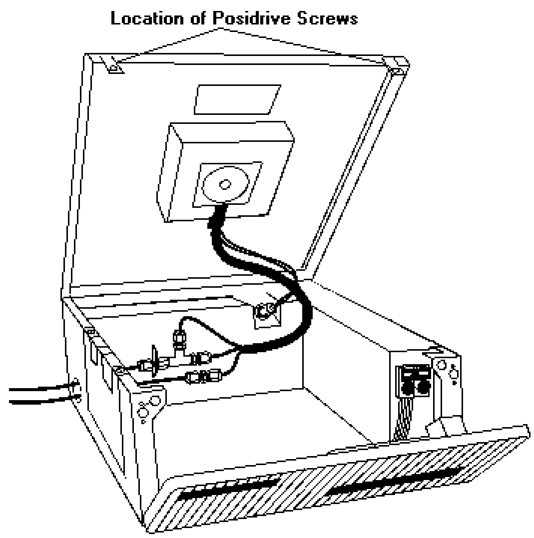
1. Shut down the DSC 7 system and remove line power. Remove the ANALOG and DIGITAL cables connected at the back of the DSC 7 (P/N N519-0103 or N519-0310).
2. Turn off the purge gas flow to the DSC 7.
3. Remove the draft shield. Open the sample holder enclosure cover. Loosen and remove the thumbscrew which secures the enclosure cover and remove the sample holder enclosure cover.



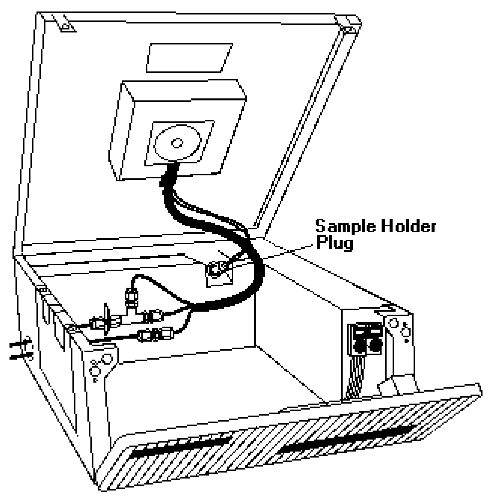
4. Remove the plastic ring located around the sample holder assembly.
5. Open the front panel of the DSC 7.



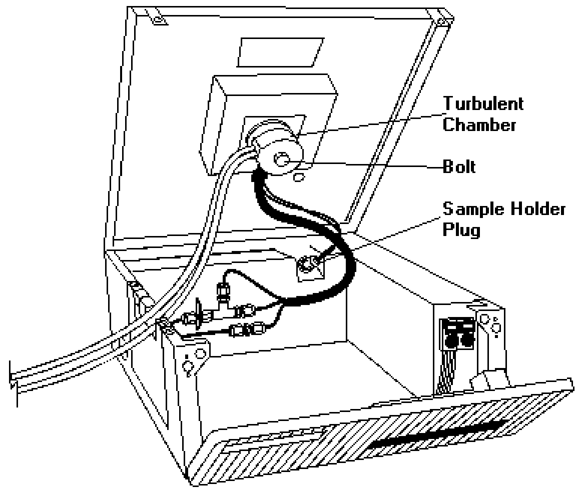
6. Remove the two screws securing the analyzer deck. Lift up the analyzer deck.



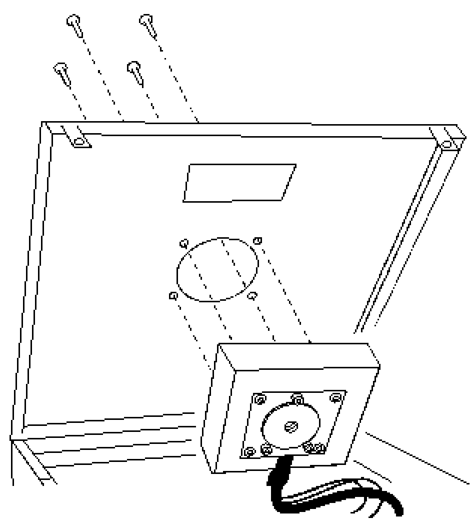
7. Disconnect the sample holder electrical connections from the chassis plug marked BLOCK. Swivel the plug clamp to release its locking tabs.



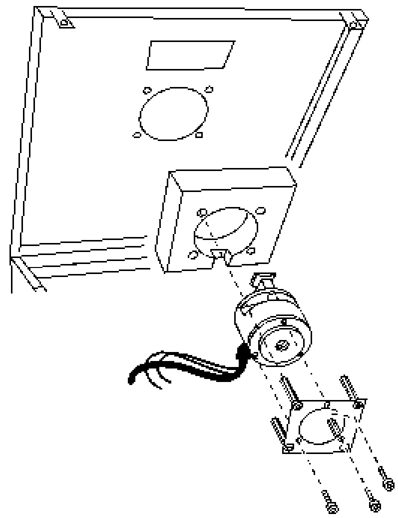
8. Remove the two screws securing the purge gas fitting to the back chassis wall and remove the entire fitting.
9. Working from the underside of the DSC 7 analyzer deck, remove the hex bolt securing the turbulent chamber to the sample holder block and remove the chamber.



10. Lower the DSC 7 analyzer deck part way. While supporting the base of the sample holder with one hand, remove the four Allen head screws around the sample holder. You should now be able to lower and remove the entire sample holder assembly and mounting brackets from the analyzer deck.



11. Unscrew and remove the three Allen screws that attach the insulator mounting plate to the sample holder assembly. Separate the insulator block foam, sample holder assembly, and transfer plate.

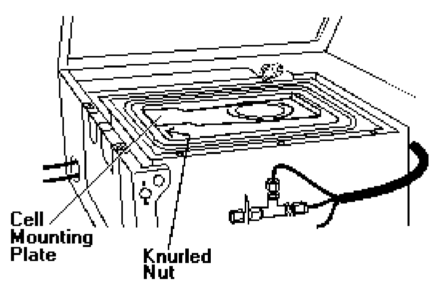


Install the Reservoir Kit

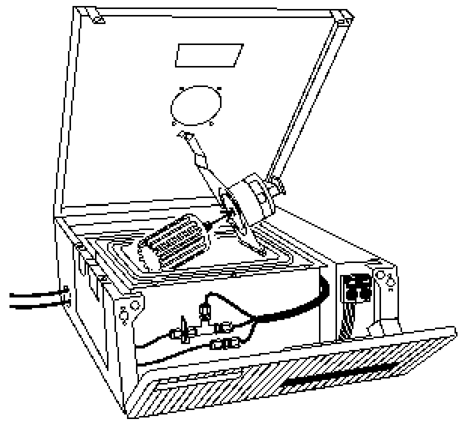
1. Remove any screws securing the reservoir cover to the reservoir and remove the reservoir cover.
2. Place the reservoir assembly into the DSC 7. The reservoir should be oriented so that the mounting brackets are located toward the sides of the DSC 7 and the two screw studs for mounting the purge gas fitting are facing toward the front of the instrument.
3. Install the four reservoir mounting screws, two on the left side and two on the right side of the reservoir, into the screw holes on the base of the DSC 7. Do NOT tighten these screws down all of the way.

NOTE: It may be necessary to adjust the height of the reservoir assembly before securing it to the DSC 7 in order to provide an adequate seal between the O-ring on the reservoir cover and the analyzer deck. On a newer DSC 7, a gasket takes the place of the O-ring, but an adequate seal is still required. This adjustment is made by loosening the screws that attach the reservoir mounting brackets to the reservoir and raising or lowering the position of these brackets.

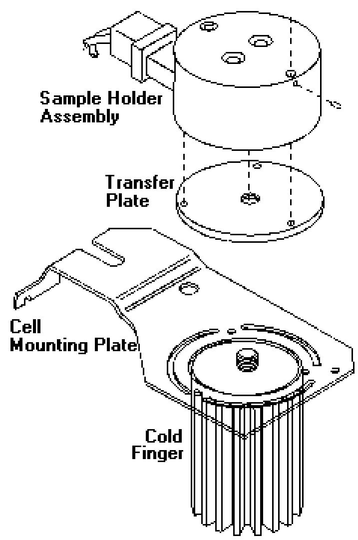
4. Loosen the knurled nut located on the inside left wall of the reservoir.



5. Swing up the cell mounting plate.



6. Using the three Allen screws and their washers (the insulator washers are not needed), fasten the transfer plate and the sample holder assembly to the cell mounting plate. Make sure that the sample holder harness leads out to the rear portion of the reservoir.



7. Coat the top of the cold finger with silicone grease (not provided) and screw the cold finger into the transfer plate. Swing the cell mounting plate back into position and tighten the knurled locking nut.
8. Run the purge gas lines and the entire purge gas fitting around the right side of the reservoir. Mount the purge gas fitting to the two screw studs on the front of the reservoir using the nuts and washers provided.
9. Connect the sample holder electrical connector to the chassis plug marked BLOCK. Swivel the plug clamp to lock it into place.
10. Install the reservoir cover and secure it using the 15 screws provided. Make sure that the two longer screws are fastened at the Intracooler exit plate on the left side of the reservoir cover. Install the O-ring in the groove on the top of the reservoir cover. The O-ring must seal against the deck lid. On a newer DSC 7, a gasket takes the place of the O-ring, but an adequate seal is still required.

11. If you intend to operate the DSC 7 with an ice water bath in the reservoir, lower the analyzer deck. (If you intend to operate the DSC 7 with liquid nitrogen as the coolant, you must first remove the ambient analyzer deck and replace it with the Subambient Analyzer Deck/Dry Box Assembly).
    a. If the sample holder is not centered in the opening of the analyzer deck, position the reservoir until the sample holder is centered.
    b. Make sure that the screw holes for replacing the four Allen head screws around the sample holder are aligned with the holes in the reservoir cover.
    c. When the sample holder and screw holes are properly centered, raise the analyzer deck and tighten the four reservoir mounting screws at the sides of the reservoir to lock the reservoir into position.
    d. Lower the analyzer deck.
12. Reinstall the front two screws and washers that secure the analyzer deck. Install the four Allen head screws around the sample holder. Close the front panel.
13. Replace the sample holder enclosure cover and thumbscrew.
14. Turn on and adjust the flow of the desired purge gas.
15. Reconnect the ANALOG and DIGITAL cable connections (P/N N519-0310) at the rear of the DSC 7. Plug in the DSC 7 to line power.

Operating the DSC 7 with Liquid Nitrogen

Prior to operating the DSC 7 with liquid nitrogen as the coolant, see the precautions listed above.

To operate the DSC 7 in subambient mode with liquid nitrogen as the coolant, perform the following steps:

1. Make sure that the dry box assembly and the liquid nitrogen level indicator have been installed.
2. Purge the dry box with dry nitrogen for several minutes. Adjust the flow of nitrogen into the dry box until the dry box entrance flaps show a positive pressure deflection. It is preferable to use dry box gloves instead of irises. If you have an older dry box with the iris design, you may wish to upgrade the dry box with the dry box glove kit (P/N N519-0504) that is offered by Perkin Elmer.
3. Make sure your helium sample purge gas is flowing at 20 cc/min (regulator pressure of 20 psi).
4. Slowly begin to introduce liquid nitrogen into the reservoir. Do not try to fill the reservoir too quickly. The recommended procedure is to fill the reservoir through the insulated copper tubing connected to the side of the reservoir. This tubing can be connected directly to a pressurized tank of liquid nitrogen, minimizing the dangers of exposing skin and eyes to liquid nitrogen.

WARNING: **Use protective gloves when turning the valves on the pressurized tank of liquid nitrogen.**

If a pressurized tank of liquid nitrogen is not available, you may pour liquid nitrogen from a dewar directly into the reservoir after removing the insulated reservoir access panel. This procedure, however, is much less convenient and increases your risk of injury due to spillage of the liquid nitrogen.

WARNING: If you choose to pour liquid nitrogen into the reservoir, it is recommended that you use the funnel and wear protective gloves, a face shield, and a rubber apron.

5. First fill the reservoir to approximately 75% capacity. Because all of the components of the reservoir and the sample holder take time to cool down to liquid nitrogen temperatures, you may have to fill the reservoir frequently at first. Keep the reservoir approximately 75% full during the initial cool down cycle. Do not attempt to analyze materials or calibrate the DSC before the system equilibrates since baseline repeatability may not be achieved.
6. As you operate throughout the day, keep the reservoir 50% or more full. Never let the liquid nitrogen level drop below 50% of the reservoir's capacity. Also, check the tightness of the seven dry box hold-down screw clamps during the first few hours of operation. As the analyzer deck and gasket cool down, the screws may become loose causing the dry box to leak at the seal.
7. Lower the dry box purge gas rate to approximately 10 cc/min.
8. Check the baseline over the temperature range of interest and optimize the baseline (if necessary).
9. Perform a temperature calibration after the DSC 7 has equilibrated. Suitable subambient calibration standards are in Calibration Reference Materials for Subambient Operation. Volatile sample pans (P/N 0219-0062) should be used for all liquids and organic solids to prevent vaporization and condensation in the sample holder cavity.

   CAUTION: Always load sample and calibration materials into the DSC 7 sample holder at room temperature or higher. Never open the sample holder enclosure cover when the sample holders are below room temperature since this can cause condensation in the sample holder environment.

10. Load your sample and reference materials into the DSC 7.
11. Observe the following precautions while performing your experiment:
    - Do not place your hands inside the dry box assembly during an experiment; the equipment may be disturbed.
    - When operating with liquid nitrogen as coolant, it is recommended that the instrument be left on at all times and the temperature of the DSC 7 sample holders be left at 50°C overnight. Leave the sample holder purge gas and dry box purge flowing.
    - The samples used for temperature calibration must be minimum 99.9% pure as even small levels of impurity can influence the melting point.
    - A small amount of frost buildup may be noted around openings and seams of the dry box and on the sample holder enclosure cover and the heat sink. This is normal. You can carefully scrape away this frost with a razor blade, making certain that you do not accidentally force any of the frost into the sample furnaces.

Precautions for Intracoolers 1 and 2

- Do not lift or move the units by pulling the flexible line.
- Do not make any sharp bends in the flexible line.
- Do not block or restrict air flow to the vents on the units.
- Do not operate the units in ambients above 32°C.
- Do not operate at line voltage outside 110 V +10% or 220 V +10% in 50-Hz operation.
- A Dry Box Assembly (P/N N519-0226 or N519-0738) is required when using Intracooler 1 or 2.

Parts Required for Intracoolers 1 and 2

The following parts are required for operation of the DSC 7 with an Intracooler 1 or 2:

Intracooler 1	0319-0205 (115 V, 50 or 60 Hz) 0319-0206 (230 V, 50 or 60 Hz)
Intracooler 2	0319-0207 (115 V, 50 or 60 Hz) 0319-0208 (230 V, 50 or 60 Hz)

A DSC 7 Dry Box Assembly **must** be used when operating the DSC 7 with Intracoolers 1 and 2. The Dry Box Assembly includes the following parts:

N519-0107	Dry Box Assembly
N519-1056	Dry Box Gasket
0990-7509	Dry Box Hold Down Screw Clamps (7)
N519-0139	Analyzer Deck

The following replacement parts are available:

0319-1720	Insulator Disk (included in Insulated Bolt Kit P/N 0319-0247)
0319-1527	Expansion Chamber Steel Bolt (included in Insulated Bolt Kit P/N 0319-0247)
0319-1719	Split Bushing (included in Insulated Bolt Kit P/N 0319-0247)

Installing an Intracooler for a Full-Range DSC 7

To install an Intracooler 1 or 2 for use with a full-range DSC 7, perform the following steps:

- **Assemble the Dry Box/Analyzer Deck Assembly for the Intracooler**
- **Install the Dry Box/Analyzer Deck Assembly for the Intracooler**
- **Install the Intracooler into the Full-Range DSC 7**
- **Reconnect the DSC 7 after Intracooler Installation**

Before installing the Intracooler,

1. Place the Intracooler on the floor to the left of the DSC 7. Make sure that there is a 4-in. clearance near the vents of the Intracooler to provide air for cooling the unit.

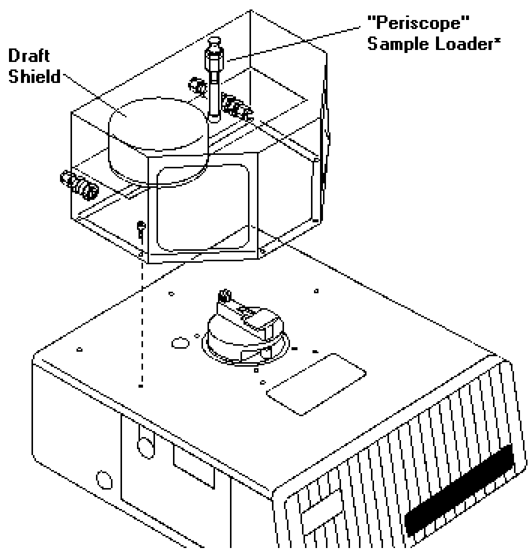
For best results, the unit should be operated at a normal ambient temperature of 22°C (74°F). Higher temperature ambients will not permit the unit to reach its maximum low temperature. Never operate the unit in ambients above 32°C (90°F).

2. Shut down the DSC 7 system and remove line power. Disconnect the ANALOG and DIGITAL cable connections (P/N N519-0103 or N519-0310) from the back of the DSC 7.

Assemble the Dry Box/Analyzer Deck Assembly for the Intracooler

The dry box needs to be assembled onto the subambient analyzer deck before it is attached to the analyzer. Place any large objects such as the draft shield into the dry box before attaching the dry box to the analyzer deck. This is particularly important if you are going to use the dry box with gloves. Once the dry box with gloves is attached to the analyzer deck, you will have to remove a glove to place items like tweezers into the dry box area.

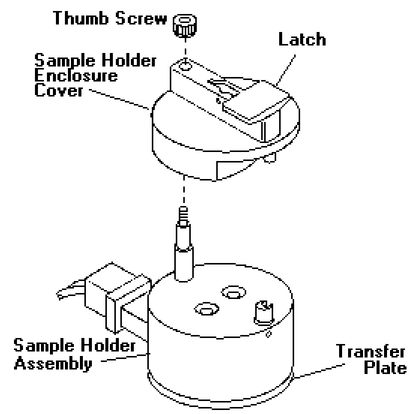
1. Place the analyzer deck on a flat surface. (This is the deck with the predrilled holes supplied with the DSC 7 Dry Box Assembly.)
2. Place the draft shield on the shelf inside the dry box.



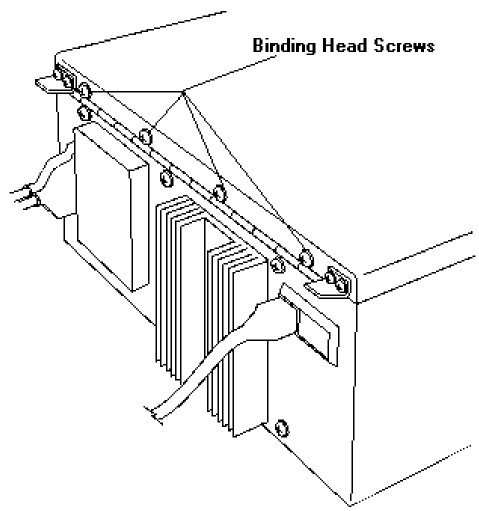
3. Place the dry box gasket on the subambient analyzer deck so that the seven holes in the gasket match the seven holes drilled in the deck.
4. Place the dry box on the deck so that the holes in the dry box, gasket, and analyzer deck line up.
5. Use the seven dry box hold-down screw clamps to mount the dry box on the analyzer deck. Securely fasten all screw clamps with your fingers. Do not overtighten.

Installing the Dry Box/Analyzer Deck Assembly for the Intracooler

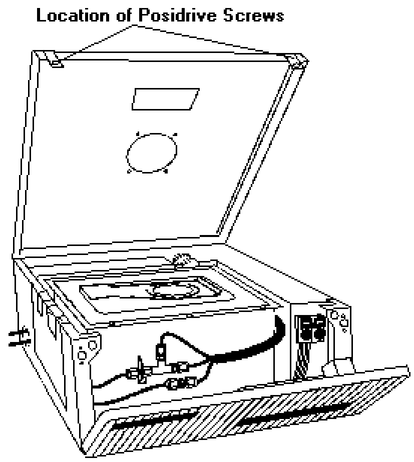
1. Open the sample holder enclosure cover.
2. Loosen and remove the thumbscrew and remove the sample holder enclosure cover.



3. Remove all sample pans and sample holder lids from the sample holder.
4. Remove the plastic ring around the sample holder assembly.
5. Using a 1/8-in. Allen wrench, unscrew and remove the four Allen screws around the DSC 7 sample holder.
6. Turn the DSC 7 on the bench so that the left side of the instrument (i.e., the side with the two openings in it) is facing you. On the back of the analyzer, remove the four binding head screws and their washers securing the deck hinge. Lower the hinge.



7. Open the front panel of the DSC 7 and remove the two Posidrive screws and their washers securing the front of the deck.



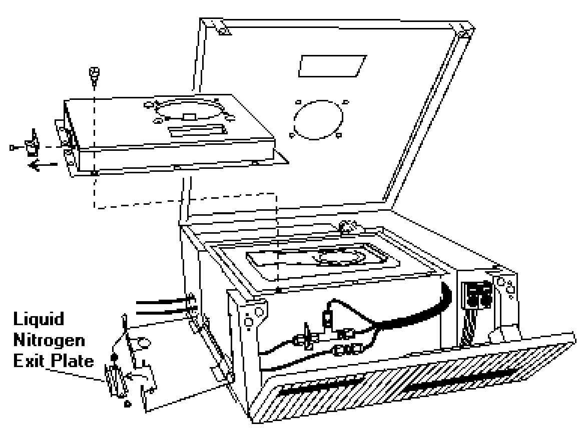
8. Lift off the analyzer deck. Remove the reservoir access panel from this deck (it is clipped in place) and install it on the deck that has the dry box assembly.
9. Place the subambient analyzer deck with the dry box assembly attached to it on the DSC 7. On the back of the analyzer, raise the hinge and replace the four binding head screws and their washers to secure the hinge to the deck.

NOTE: Make sure that the hinge screws are secured tightly.

Install the Intracooler in a Full-Range DSC 7

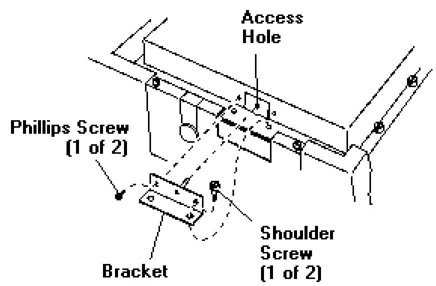
1. Lift the analyzer deck.

   The deck has two small tabs that allow it to remain stationary while in the vertical position without being propped up. Be careful not to jar the analyzer, however, since the deck may fall.
2. Loosen the two small screws that hold the left-side door of the analyzer closed and open the door by swinging it down. Remove the two nuts and washers from the Intracooler exit opening on this door (the Intracooler exit plate is the larger of the two plates on this door). Once these nuts and washers are removed, the plate on the door can be removed. (Removing this plate provides the chassis exit opening for the Intracooler line.)

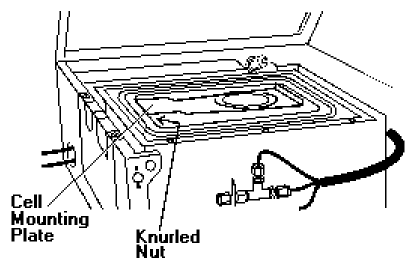


3. Unscrew the 15 screws securing the top of the reservoir and remove the cover.
4. Place the cover on a flat surface and remove the two small screws securing the metal Intracooler exit plate on the left-hand side of the cover. Remove the plate.
5. With a screwdriver, gently pry out the rectangular insulation plug located under the metal plate that was just removed. If the insulation plug is difficult to remove, score the outer edges with a razor blade.

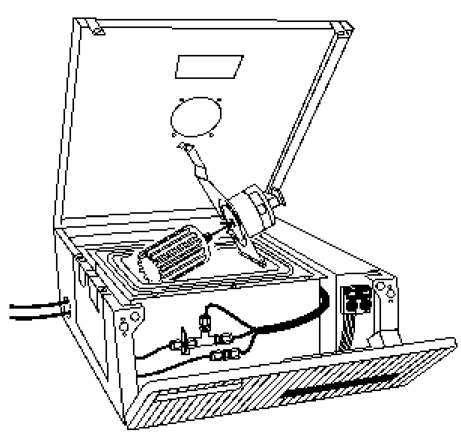
   Removing this plate and insulation plug provides the reservoir exit opening for the Intracooler line.



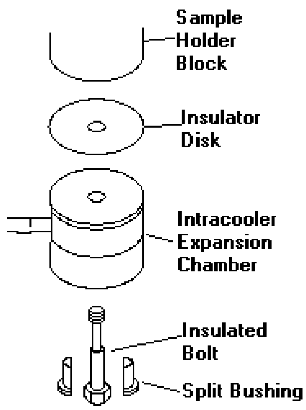
6. Loosen the knurled nut located on the inside wall of the reservoir and swing up the cell mounting plate.



7. Remove the cold finger by holding the sample holder firmly with one hand and turning the cold finger in a counterclockwise direction with the other hand.

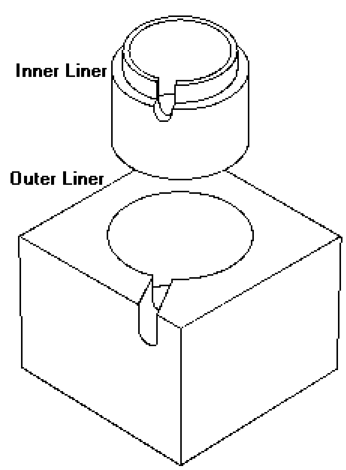


8. Lightly coat the top surface (that surface closest to the tube inlet) of the Intracooler expansion chamber with silicone grease. Do the same to the bottom surface of the DSC 7 sample holder block.
9. Place the insulator disk on the top surface of the Intracooler expansion chamber.
10. Find the split bushing and bolt in the insulated bolt kit. Place the two bushing pieces around the bolt beneath the bolt's head.
11. Fasten the expansion chamber and insulator disk to the DSC 7 sample holder block using the bolt with split bushing.



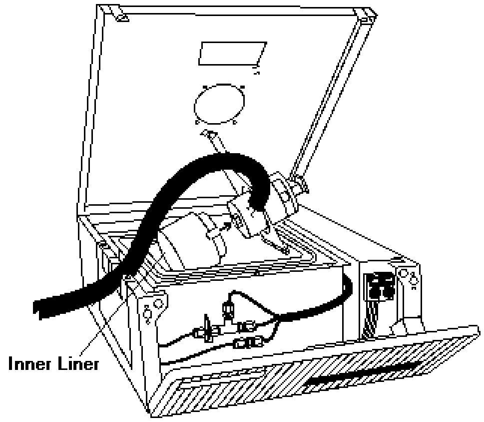
The flexible line near the top of the expansion chamber should extend directly out to the left. Be careful not to severely bend or twist the flexible line.

The Intracooler expansion chamber comes with an insulator. The insulator is made up of two pieces: the inner liner and the outer liner.



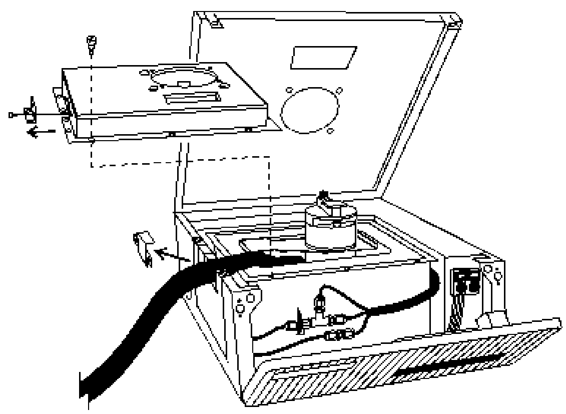
The inner liner is round and fits over the expansion chamber. The outer liner is cubical and fits around the inner liner. However, you will not use the outer liner in the full-range DSC 7.

12. Remove the inner liner from the outer liner and store the outer liner away.
13. Place the inner liner of the expansion chamber insulator over the chamber. Make sure that the flexible line is in the cutout groove in the liner.



14. Swing the sample head back into the reservoir and tighten the knurled locking nut on the inside wall of the reservoir. The inner liner should rest on the base of the reservoir.

15. Run the flexible line from the Intracooler out through the top left-hand side of the reservoir.



16. Replace the reservoir cover, making sure that the flexible line from the Intracooler and the sample holder connections exiting the back of the reservoir are not constricted. Replace the 15 screws that secure the cover of the bath.

17. Close the left access door of the DSC 7 and secure it in place by tightening the two securing screws on the chassis.

18. Lower the analyzer deck. Reinstall the front two screws and washers that secure the deck. Install the four Allen screws around the sample holder. Close the front panel.

Reconnecting the DSC 7 after Intracooler Installation

1. Connect the ANALOG and DIGITAL cable connections (P/N N519-0310) at the back of the DSC 7. Plug in the DSC 7 to line power.

2. Make certain the REFRIGERATION switch on the Intracooler is in the off (down) position. Plug the Intracooler into line power.

3. Set the purge gas flow rate to 20 psi (at the regulator). Connect a source of dry nitrogen to the purge gas fitting on the back of the dry box.

Installing an Intracooler in an Ambient DSC 7

The following topics describe installation of an Intracooler 1 or 2 into an ambient DSC 7:

- **Removing the Analyzer Deck and Sample Holder Assembly**
- **Assembling the Dry Box/Analyzer Deck Assembly for the Intracooler**
- **Installing the Dry Box/Analyzer Deck Assembly onto the Ambient DSC 7**
- **Intracooler Installation into the Ambient DSC 7**
- **Reconnecting the DSC 7 after Intracooler Installation**

Before installing the Intracooler,

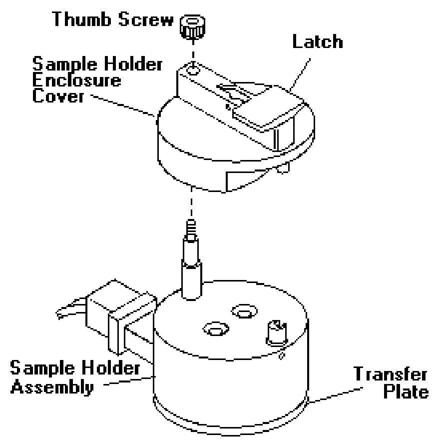
1. Place the Intracooler on the floor to the left of the DSC 7. Make sure that there is a 4-in. clearance near the vents of the Intracooler to provide air for cooling the unit.

   For best results, the unit should be operated at a normal ambient temperature of 22°C (74°F). Higher temperature ambients will not permit the unit to reach its maximum low temperature. Never operate the unit in ambients above 32°C (90°F).

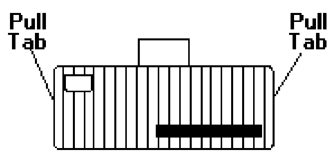
2. Shut down the DSC 7 system and remove line power. Disconnect the ANALOG and DIGITAL cable connections (P/N N519-0103 or N519-0310) from the back of the DSC 7.

Remove the Analyzer Deck and Sample Holder Assembly

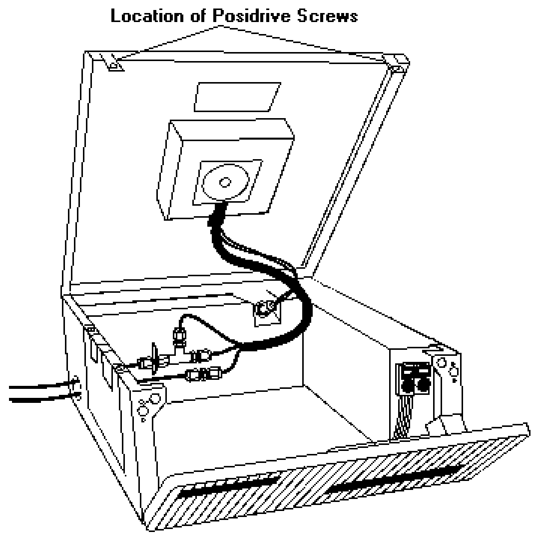
1. Remove the draft shield. Open the sample holder enclosure cover. Loosen and remove the thumbscrew and remove the sample holder enclosure cover.



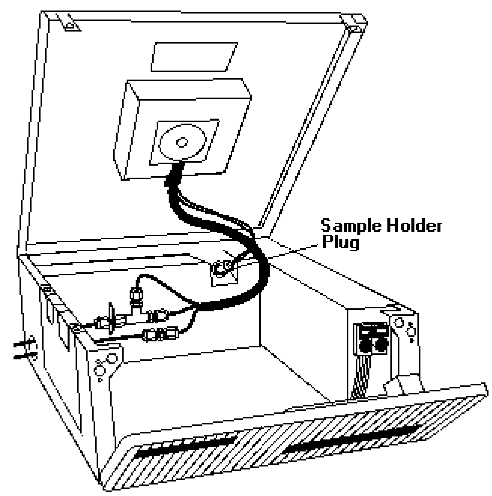
2. Remove all sample pans and sample holder lids from the sample holders.
3. Remove the plastic ring around the sample holder assembly and store away.
4. Open the front panel of the analyzer.



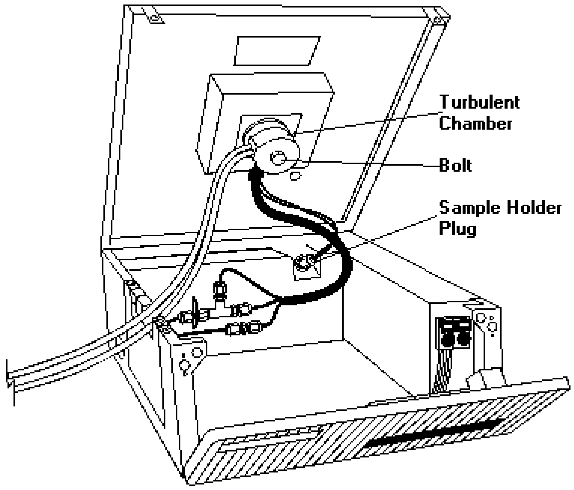
5. Remove the two Posidrive screws securing the analyzer deck. Lift the analyzer deck.



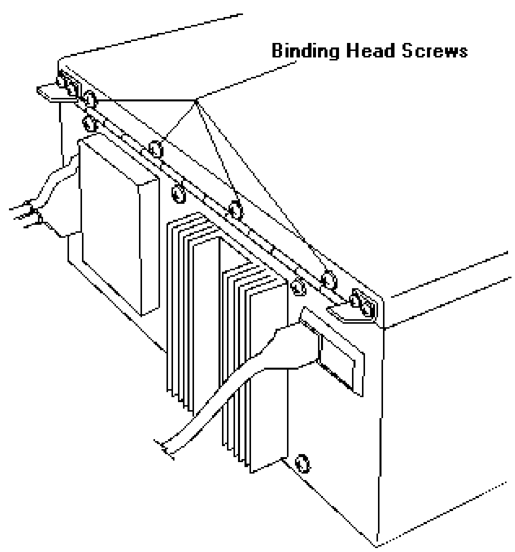
6. Disconnect the sample holder electrical connections from the chassis plug marked BLOCK. Swivel the plug clamp to release its locking tabs.



7. Remove the two purge gas lines connected to the purge gas fitting on the back chassis wall.
8. Working from the underside of the DSC 7 cover, remove the hex bolt securing the turbulent chamber to the sample block and remove the chamber.



9. Lower the analyzer deck. At the back of the analyzer, remove the four binding head screws and their washers securing the deck hinge to the deck. Lower the hinge.



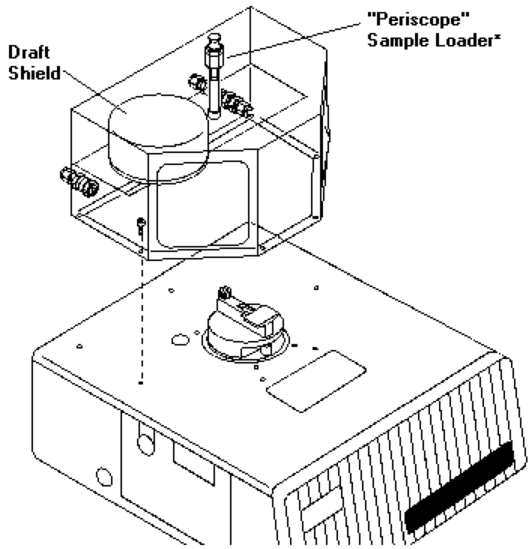
10. Lift off the analyzer deck and sample holder assembly.

11. Unscrew and remove the four Allen head screws around the DSC 7 sample holder. You should now be able to remove the entire sample holder assembly and mounting bracket from the cover.

Assemble the Dry Box/Analyzer Deck Assembly for the Intracooler

The dry box needs to be assembled onto the subambient analyzer deck before it is attached to the analyzer. Place any large objects such as the draft shield into the dry box before attaching the dry box to the analyzer deck. This is particularly important if you are going to use the dry box with gloves. Once the dry box with gloves is attached to the analyzer deck, you will have to remove a glove to place items like tweezers into the dry box area.

1. Place the analyzer deck on a flat surface. (This is the deck with the predrilled holes supplied with the DSC 7 Dry Box Assembly.)
2. Place the draft shield on the shelf inside the dry box.



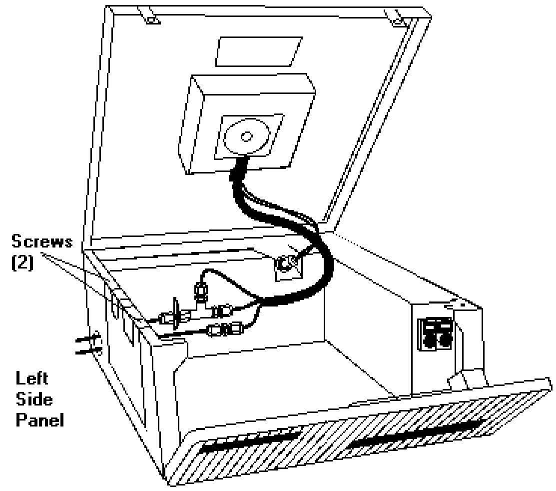
3. Place the dry box gasket on the subambient analyzer deck so that the seven holes in the gasket match the seven holes drilled in the deck.
4. Place the dry box on the deck so that the holes in the dry box, gasket, and analyzer deck line up.
5. Use the seven dry box hold-down screw clamps to mount the dry box on the analyzer deck. Securely fasten all screw clamps with your fingers. Do not overtighten.

Install the Dry Box/Analyzer Deck Assembly onto the Ambient DSC 7

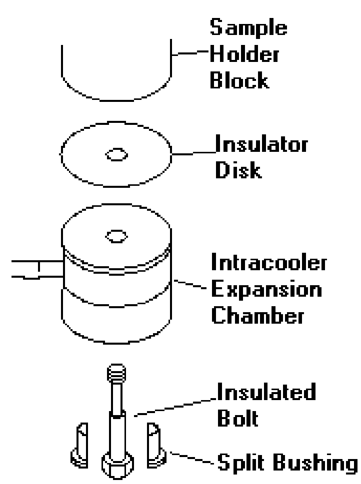
1. Attach the sample holder assembly to the dry box/analyzer deck assembly using the four Allen head screws.
2. Place the dry box/analyzer deck assembly with the sample holder attached onto the DSC 7. Lift the hinge on the back of the analyzer and replace the four binding head screws and their washers to secure the hinge to the deck. Make sure that the hinge screws are secured tightly.
3. Lift the deck and connect the purge gas lines to the purge gas fitting on the back chassis wall. Connect the sample holder electrical connector to the chassis plug marked BLOCK.

Intracooler Installation into the Ambient DSC 7

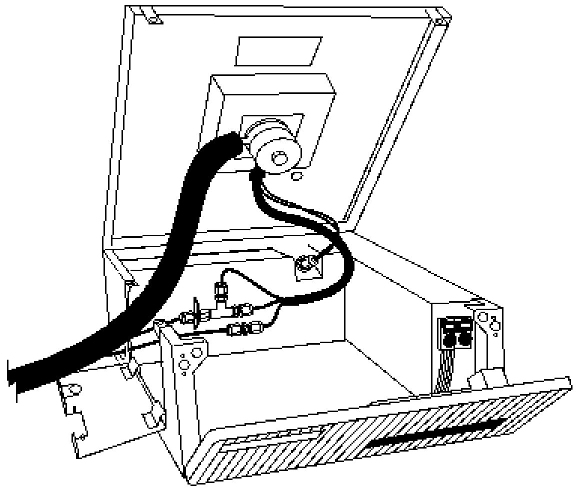
1. Loosen the two small screws that hold the left side door of the analyzer closed and open the door by swinging it down.



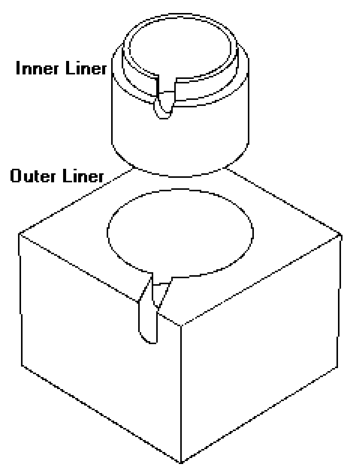
2. Remove the two nuts and washers from the Intracooler exit opening on this door (the Intracooler exit plate is the larger of the two plates on this door). Once these nuts and washers are removed, the plate on the door can be removed. (Removing this plate provides the chassis exit opening for the Intracooler line.)
3. Lightly coat the top surface (that surface closest to the tube inlet) of the Intracooler expansion chamber with silicone grease. Do the same to the bottom surface of the DSC 7 sample holder block.
4. Place the insulator disk on the top surface of the Intracooler expansion chamber.
5. Find the split bushing and bolt in the insulated bolt kit. Place the two bushing pieces around the bolt beneath the bolt's head.
6. Fasten the expansion chamber and insulator disk to the DSC 7 sample holder block using the bolt and split bushing.



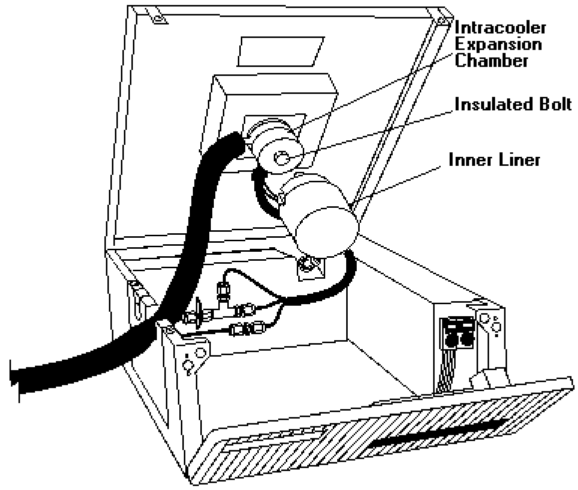
The flexible line near the top of the expansion chamber should extend directly out to the left. Be careful not to severely bend or twist the flexible line.



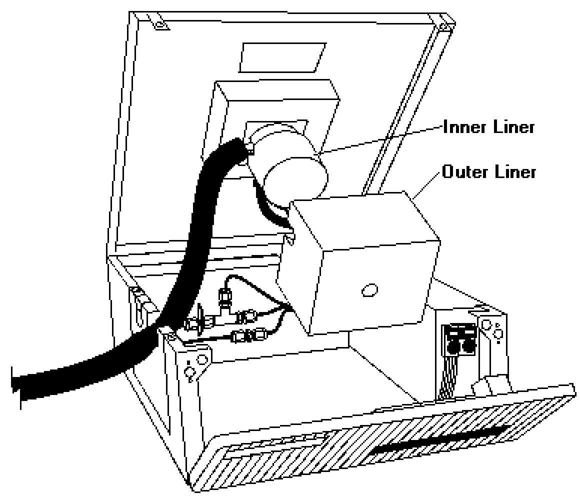
The Intracooler expansion chamber comes with an insulator. The insulator is made up of two pieces: the inner liner and the outer liner. The inner liner is round and fits over the expansion chamber. The outer liner is cubical and fits around the inner liner.



7. Remove the inner liner from the outer liner.
8. Place the inner liner over the expansion chamber. Make sure that the flexible line is in the cutout groove in the liner.



9. Position the outer liner slightly over the inner liner, making sure that the flexible hose line sits in the cutout of the outer liner.



It may be necessary to push back some of the insulation on the flexible hose line so that it fits inside of the cutout. It is **NOT** necessary to push the outer liner all of the way onto the inner liner at this time.

10. Close the left access door of the DSC 7 and secure it in place by tightening the two securing screws on the chassis.
11. Run the flexible line from the Intracooler out through the top left-hand side of the analyzer through the Intracooler exit opening.
12. Lower the analyzer deck. The inner liner with the Intracooler expansion chamber will seat itself within the outer liner and the outer liner will rest on the base of the DSC 7.
13. Reinstall the front two screws and washers that secure the deck.
14. Reattach the sample holder enclosure cover and thumbscrew to the sample holder assembly.

Reconnecting the DSC 7 after Intracooler Installation

1. Connect the ANALOG and DIGITAL cable connections (P/N N519-0310) at the back of the DSC 7. Plug in the DSC 7 to line power.
2. Make certain the REFRIGERATION switch on the Intracooler is in the off (down) position. Plug the Intracooler into line power.
3. Set the purge gas flow rate to 20 psi (at the regulator). Connect a source of dry nitrogen to the purge gas fitting on the back of the dry box.

Operating the DSC 7 Using an Intracooler 1 or 2

Prior to operating the DSC 7 with an Intracooler 1 or Intracooler 2 installed for subambient operation, see the precautions listed under the **Subambient Operation** section.

1. Make sure that the dry box assembly has been installed.
2. Purge the dry box with dry nitrogen for several minutes. Adjust the flow of nitrogen into the dry box until the dry box entrance flaps show a positive pressure deflection.
3. Make sure your nitrogen purge gas to the sample holder flows at 20 cc/min (regulator pressure of 20 psi).
4. Start up the system.
5. Program the DSC 7 temperature to 25°C.
6. Allow the system to equilibrate for a minimum of 30 min. Do not attempt to analyze materials or calibrate the DSC 7 before the system equilibrates, since baseline repeatability may not be achieved.
7. Check the baseline over the temperature range of interest and optimize the baseline (if necessary).
8. Check the temperature and calorimetric calibration after the DSC 7 has equilibrated if this is the first time using the system with an Intracooler.

   Metal calibration standards or any of the subambient calibration reference materials can be used for calibration, depending on the temperature range of interest. Volatile sample pans (P/N 0219-0062) should be used for all liquid and organic solid calibration materials to prevent vaporization and condensation in the sample holder cavity.

WARNING: **When using an Intracooler 1 or 2, always load sample and calibration materials into the DSC 7 sample holder at room temperature or higher. Never open the sample holder enclosure cover when the sample holders are below room temperature. This could cause condensation in the sample holder environment.**

9. Load your sample and reference materials into the DSC 7 and perform your experiment. Observe the following precautions:

   - Do not place your hands inside the dry box assembly during an experiment; the experiment may be disturbed.
   - When operating the Intracooler 1 or 2, it is recommended that the DSC 7 be left on at all times and the temperature of the DSC 7 sample holders be left at 50°C overnight. Leave the sample holder purge gas on.

- The samples used for calibration must be minimum 99.9% pure as even small levels of impurity can influence the temperature and/or energy of the transition.
- A small amount of frost buildup may be noted on the sample holder enclosure cover and the heat sink. This is normal. On the sample holder, you can carefully scrape away this frost with a razor blade, making certain that you do not accidentally force any of the frost into the sample furnaces.
- The Intracoolers are not thermostatically controlled but operate at “constant cooling.” Thus, the block temperature will change with a variation in ambient temperature. In addition, the cooling efficiency of the Intracoolers will depend on the ambient temperature. Optimum operation is realized at an ambient of 22°C. Under no circumstances should the Intracoolers be operated in ambients above 32°C.

When properly installed, the Intracoolers may be operated continuously. However, some condensation will undoubtedly form on the DSC 7 sample holder enclosure block. This should not affect the operation of the DSC 7 as long as the condensation does not enter the sample holder cavities. Consequently, samples should be introduced into the sample holder when the holder temperature is at room temperature. It is recommended that the Intracooler be turned off and the holder temperature be left at 100°C overnight whenever condensation buildup is extreme.

Calibration Reference Materials for Subambient Operation

Very-high-purity (>99.999%) organic materials can be used to calibrate the temperature when operating the DSC in the temperature range from –170°C. See the table below for a list of recommended subambient calibration standards that can be used with your DSC. Normally, indium is the reference material that is recommended for energy calibration for all operating conditions.

Substance	**Transition**	**Transition Temp. (°C)**	**Transition Energy (J/g)**
Cyclopentane	Crystal	–151.16	69.45(a)
Cyclopentane	Crystal	–135.06	4.94(a)
Cyclohexane	Crystal	–87.06	79.58(b)
Cyclohexane	Melt	6.54	31.25(b)
Water	Melt	0.00	333.88
n-Heptane	Melt	–90.56	140.16(c)
n-Octane	Melt	–56.76	182.0(c)
n-Decane	Melt	–29.66	202.09(c)
N-Dodecane	Melt	–9.65	216.73(c)
n-Octadecane	Melt	28.24	241.42(d)
Hexatriacontane	Crystal(*)	72.14	18.74(d)
Hexatriacontane	Crystal	73.84	60.25(d)
Hexatriacontane	Melt	75.94	175.31(d)
P-Nitrotoluene	Melt	51.64	

(*) Shows some thermal history dependence
(a) Aston, J.G. et al., *JAC*, **65**, 341 (1943)
(b) Aston, J.G. et al., *JAC*, **65**, 1035 (1943)
(c) Finke, H.L. et al., *JAC*, **76**, 33 (1954)
(d) Schaerer, A.A. et al., *JAC*, **77**, 2017 (1955)

1. The materials listed here, if used for calibration, must be of 99.999% minimum purity as even small levels of impurity can affect the temperature and/or energy of the transition.
2. These substances must be encapsulated in volatile sample pans. To improve peak resolution, place a small piece of aluminum foil over your sample before sealing the pan.
3. If the peaks are not sharp (as in indium), the sample may be impure and the temperatures you measure may not be correct. Use a higher purity sample or check the purity of the sample by an alternate technique such as gas chromatography.

Maintenance

The DSC 7 needs little routine maintenance other than proper treatment as a sensitive electromechanical device. Occasionally, the sample holder cups may become coated with sample residue and a cleaning procedure may be necessary. If the guard ring inserts become dirty, you can clean them with a cotton swab dipped in isopropyl alcohol or appropriate solvent to remove any sample residue. See the topic on how to **Remove and Insert Guard Rings** in the Pyris Multimedia Presentations Help.

The following topics contain information on maintaining your DSC 7:

- **Cleaning the Sample Holder**
- **Sample Holder Treatment**
- **Sample Holder Enclosure Cover O-Ring Installation**
- **Replacing the Sample Holder**
- **Clean the Furnace**
- **Intracooler Maintenance**

Cleaning the Sample Holder

1. Since the sample holder assemblies are the hottest components in the cavity area, they will usually remain clean and free of condensates. However, samples may ooze out of sample pans and accidentally spill into the sample holder cup. Such materials should be removed at low temperatures whenever possible by using tweezers or moistened cotton swabs.
2. Solvents should not be applied directly in the holder cup. Moisten a cotton swab with solvent (e.g., ethanol or isopropyl alcohol or other appropriate solvent) and then clean the sample cup or upper portion of the cavity wall by using the cotton swab. Periodically check the underside of the sample holder enclosure lid and, if necessary, clean it in a similar fashion.
3. Any residual sample that has carbonized may remain in the sample holder cup after it has been cleaned with solvent. To remove the residue, leave the sample holder enclosure lid open to expose the holders to the air. While observing the residue, program the DSC to 600°C at 100°C/min. Most carbonaceous or organic materials will burn off in the neighborhood of 600°C. If necessary, leave the instrument temperature at 600°C for not more than 5 – 10 minutes to burn off the residue.

CAUTION: This procedure applies only if the residue is organic and is known to be free of metal or metal-containing compounds.

Materials not free of metal or metal-containing compounds may irreversibly alloy with the sample holder. If some alloying has occurred, the holder will remain usable, although minor peaks may appear on the baseline at high temperatures.

Sample Holder Treatment

Handling the sample holder correctly is important to the proper operation of the sample holder and subsequently the DSC. The DSC sample holder assemblies are rugged and should last indefinitely if treated as any sensitive electronic device should be treated and if used as directed. The sample holder's performance will decline or fail for any of the following reasons:

- **Application of Excessive Mechanical Force**
- **Exceeding Melting Temperature of Containers**
- **Mechanical Breaking or Shorting of Sample Holder Leads**
- **Chemical or Physical Attack of Sample Holder Materials by Samples**

Application of Excessive Mechanical Force

The holders are mounted on a hollow platinum-iridium post that may bend or break off if excessive vertical or horizontal force is applied to the assembly. Do not attempt to force sample holder lids into the cups. Always reshape any sample holder lids that do not fit into the sample cups without the use of excessive mechanical force.

Exceeding Melting Temperature of Containers

Aluminum sample pans will melt and alloy with the platinum-iridium holders at the melting point of aluminum. **DO NOT USE ALUMINUM SAMPLE PANS AT TEMPERATURES ABOVE 600°C.**

Mechanical Breaking or Shorting of Sample Holder Leads

The heater and sensor leads to each sample holder assembly are thin platinum ribbons that extend at right angles from the cup a short distance toward the cavity wall. Then they bend downward vertically to where they are soldered to feed-through posts at the bottom of the cavity wall. When looking straight down into the cavity, these leads should be visible at the point where they emerge from the cup. The leads may be partly covered by small sections of insulating aluminum oxide cloth protuding from the interior of the sample holder. The leads must never be disturbed or they may break and cause an open circuit or may wipe against the sample holder and cause a short circuit.

WARNING: **The possibly catastrophic consequences of disturbing the sample holder leads emphasize the importance in exercising great care when cleaning or "poking around" the cavity area. Never use a cotton swab or similar object to clean the cavities below the level of the sample cup. An effective cleaning tool is a No. 9 cork borer (0.5 in. i.d.) that is wrapped first with double-sided adhesive tape and then with a layer of slightly moistened paper towel. Insert this cleaning tool vertically all the way into the cavity and rotate it to clean the wall of condensates or other contamination. A 0.5-in.-i.d. tube will not touch the sample holder leads but will make good contact with the cavity walls.**

- **Open-Circuited Sensor:** The sensor will have infinite resistance and the instrument will react as if a malfunctioning sample holder is at "infinite temperature," which means that no program power will be provided to the holders so that they will not heat.

- **Short-Circuited Sensor:** The sensor will have zero resistance and the instrument will react as if the sample holders require program power to elevate them to the program temperature. In trying to meet this demand, the instrument will apply maximum heater power to both holders and will drive them ballistically to their maximum temperatures. Both holders will glow red hot, and, if aluminum pans are present, they will melt and destroy the holders.
- **Open-Circuited Heater:** The sample holder will not heat and the sensor will detect its abnormally low temperature. The average power circuit will apply power to drive the average temperature of the sample and reference holders to the desired program temperature. Therefore, the reference holder will overheat, and, if the program temperature is high enough, it may heat to maximum temperature. If aluminum is present in the reference holder, it may melt and destroy the holder.

Chemical or Physical Attack of Sample Holder Materials by Samples

All the materials used to construct the sample holders (platinum–iridium, pure platinum, and aluminum oxide) are subject to chemical attack by very few chemical materials.

CAUTION: Platinum–iridium and platinum will alloy with most metals at sufficiently high temperatures.

Chemical attack may produce discoloration or spotting of the sample holder and, in severe cases, may destroy it. Metals, samples containing metals, or samples that may decompose to metallic products should not be heated to temperatures much above their melting points. The proper sample containers must be used. Since gold also alloys readily with most metals, never use gold or platinum pans for metal samples. Use aluminum pans for such samples when programming the temperature up to 600°C. Above this temperature use graphite pans. Aluminum oxide sample containers, tantalum pans, and others that can be homemade, may also be used.

Cases of sample holder failure caused by deposition of carbonaceous vapors or other conducting vapors on electrical components of the sample holders should occur rarely, if at all. Condensation on the relatively hot sample holders is unlikely. The purge gas flow tends to sweep vapors up and away from the leads, and sample sizes of a few milligrams will usually not generate large enough quantities of condensable vapors per unit time to saturate the surrounding atmosphere. However, it is possible to short out the sample holder in this way by using excessive sample sizes, programming at excessive speed, using a low purge flow rate, and programming to excessively high temperatures. Always avoid mistreating the sample holders for maximum safety and maximum lifetime of the sample holder assembly.

Sample Holder Enclosure Cover O-Ring Installation

The following procedure is for installing the O-ring in the sample holder enclosure cover:

1. Lift off the draft shield to expose the sample holder enclosure.
2. Lift the latch of the swing-away lid to release the cover, then rotate the cover to the right.
3. Remove the knurled thumb nut holding down the cover and lift off the cover.
4. Install the O-ring in the channel on the bottom of the enclosure cover.
5. Mount the enclosure cover in position.
6. Replace and tighten the knurled thumb nut.

Sample Holder Replacement

The DSC 7 sample holder assemblies consist of a matched pair of sample holders mounted in an aluminum enclosure block. They include electrical leads and purge lines. There are two categories of sample holder assemblies that can be replaced in the field. Individual sample holders or matched pairs cannot be replaced in the field.

Sample holder assemblies available from Perkin Elmer are

0319-0007	replacement sample holder assembly with holders matched and tested to pass all specifications from –170°C to 725°C
0319-0006	replacement sample holder assembly with holders matched and tested to meet all specifications from –70°C to 725°C
0319-0151	replacement sample holder tested to meet all specifications for a temperature range of ambient to 725°C

NOTE: All sample holders have the same precision and accuracy. They are selected for slightly different baseline performance over the temperature range. Each sample holder operates over the full temperature range of the DSC 7.

The replacement procedure differs depending on whether the DSC 7 is equipped with a reservoir for full-range (including subambient) use or with a turbulent water chamber for ambient use only.

WARNING: **This replacement procedure should be performed only by Perkin Elmer factory-trained personnel. If you perform the procedure without proper training, any damage or injury that occurs from error or misunderstanding of these instructions is not the responsibility of the Perkin Elmer LLC.**

Replacing the Sample Holder Assembly on a Full-Range DSC 7

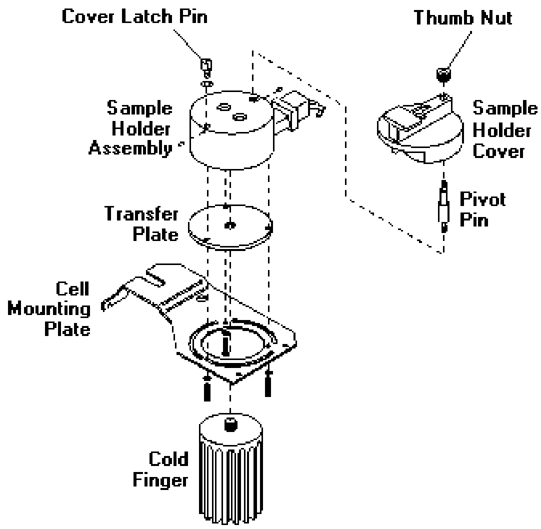
Replace the sample holder assembly on the full-range DSC 7 (P/N N519-0243, N519-0242, N519-0244) by performing the following two steps:

- **Removing the Sample Holder Assembly on a Full-Range DSC 7**
- **Installing a New Sample Holder Assembly in a Full-Range DSC 7**

NOTE: Before beginning, make sure that the power switch on the DSC 7 is in the OFF position and remove the line power. Also, make sure there is no purge gas flow to the DSC 7.

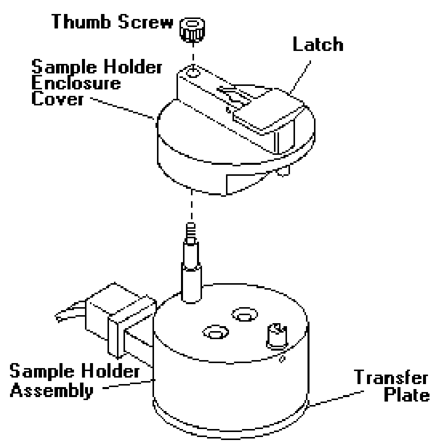
Removing the Sample Holder Assembly on a Full-Range DSC 7

Sample Holder Assembly as used with a full-range DSC 7



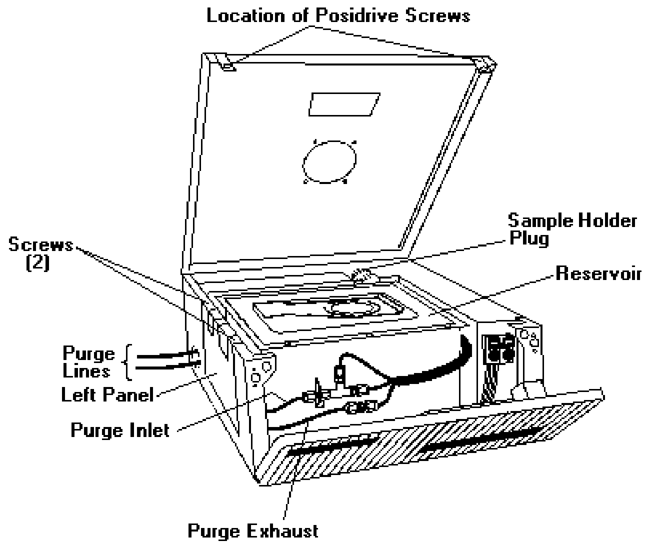
WARNING: Before beginning, make sure that the power switch on the DSC 7 is in the OFF position and remove the line power. Also, make sure there is no purge gas flow to the DSC 7.

1. Remove the draft shield. Open the sample holder enclosure cover. Loosen and remove the thumbscrew and remove the sample holder enclosure cover.



2. Remove the guard rings, if present, from the sample cells. Using the sample handling tray, remove all sample pans and sample holder lids from the sample holders.
3. Remove the plastic ring around the sample holder assembly.
4. Using a 5/32-in. Allen wrench, unscrew and remove the four Allen screws around the DSC 7 sample holder. Open the front panel and remove the additional two binding head screws and their washers securing the cover. Lift the cover.

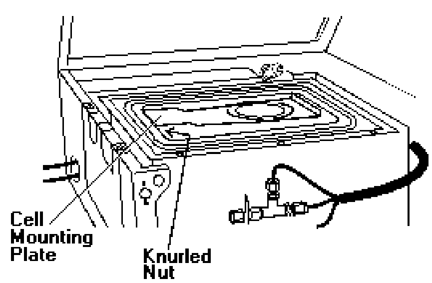
5. Disconnect the sample holder electrical connector from the chassis plug marked BLOCK. Swivel the plug clamp to release its locking tabs.
6. Using two 7/16-in. wrenches, disconnect the purge gas lines at the front of the reservoir.



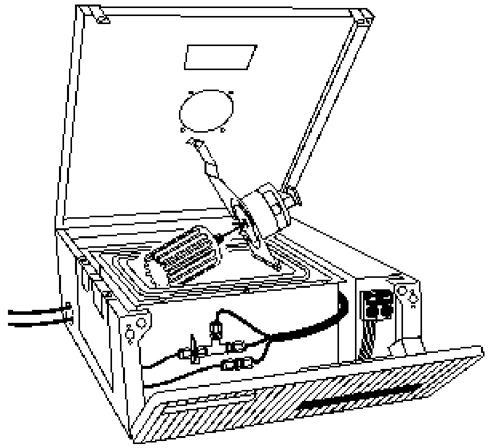
7. Remove the O-ring (on older DSC 7 units) or rubber gasket (on newer DSC 7 units) from the reservoir cover. Unscrew and remove the 15 screws securing the top of the reservoir. Remove the reservoir cover.

NOTE: Two longer shoulder screws are inserted into the Intracooler exit plate on the left side of the reservoir cover. Keep these screws separate from the others.

8. Using a 5/64-in. Allen wrench, remove the set screw that secures the pivot pin in the sample holder assembly and then unscrew and remove the pin. Install the pivot pin in the new sample holder; use the set screw to hold it in place.
9. Using a 1/16-in. Allen wrench, remove the set screw that secures the enclosure cover catch pin and then remove the pin and its washer. The pin is press fit and not threaded into the sample holder assembly. Install the enclosure cover catch pin and screw in the new sample holder.
10. Loosen the knurled nut located on the inside left wall of the reservoir.



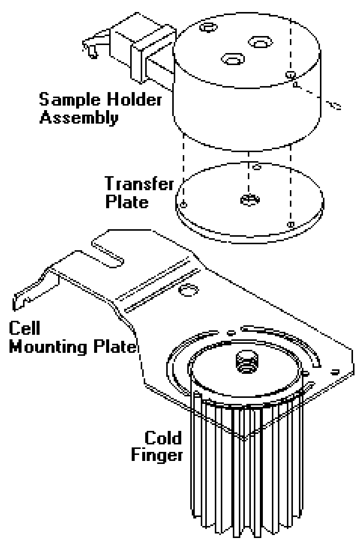
11. Swing up the cell mounting plate.



12. Remove the cold finger by holding the sample holder assembly firmly with one hand and turning the cold finger in a counterclockwise direction with the other hand.

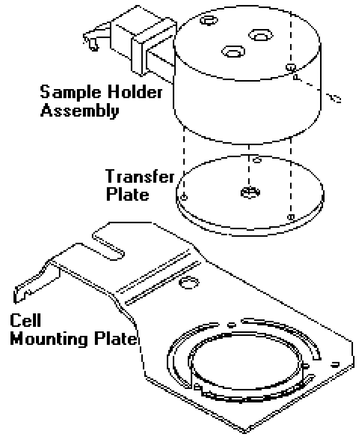
    NOTE: If an Intracooler is connected, it must be removed.

13. While supporting the sample holder block, use a 5/32-in. Allen wrench to unscrew and remove the three screws and washers that attach the sample holder assembly to the cell mounting plate. Remove the transfer plate from the sample holder.

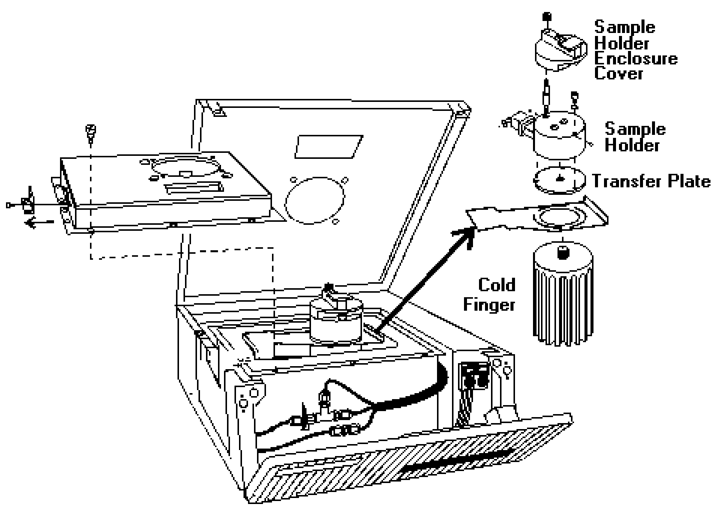


Installing a New Sample Holder Assembly in a Full-Range DSC 7

1. Make sure that the transfer plate is coated with silicone grease. Press the coated transfer plate to the new sample holder assembly.
2. Using the three Allen screws and their washers, fasten the transfer plate and the new sample holder assembly to the cell mounting plate. Make sure that the sample holder harness leads out to the rear portion of the reservoir.



3. Screw the cold finger into the transfer plate.
4. Swing the cell mounting plate back into position and tighten the knurled locking nut.
5. Reinstall the reservoir cover using the 15 shoulder screws. Make sure that the longer screws are fastened at the Intracooler exit plate. Reinstall the O-ring on the reservoir cover.
6. Reconnect the block harness and the purge gas lines. Note that the sample holder purge lines are connected at the restrictor and that the single exit line (with marked tag) is connected to the Teflon exit line. After finger-tightening the fittings, tighten them an additional quarter turn using two 7/17-in. wrenches.



7. Close the DSC 7 cover and reinstall the front two screws and washers that secure the cover. Install the four Allen screws around the sample holder and close the front panel. Reinstall the plastic ring around the sample holder.
8. Reattach the sample holder enclosure cover and thumbscrew to the sample holder assembly and check the latching mechanism for smoothness of operation. The catch mechanism will require adjustment if the cover is difficult to close or moves when the latch is closed. Sufficient adjustment is usually achieved by loosening the set screw securing the catch pin and rotating the catch pin until the latch closes firmly so the cover cannot move. If positive

latching is not achieved by rotating the catch screw, adjust the height of the pivot pin after loosening its securing set screw.

9. Reinstall the guard ring inserts and platinum sample holder covers.
10. Turn on the purge gas and set the flow rate to 20 psi at the regulator.
11. Plug in the DSC 7 to line power.

Replacing the Sample Holder Assembly on an Ambient DSC 7

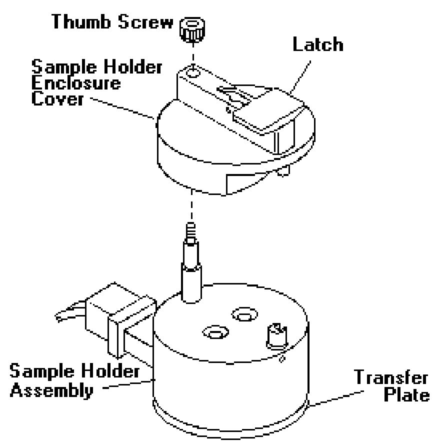
Replace the sample holder assembly on an ambient DSC 7 (P/N N519-0246, N519-0245, N519-0247) by performing the following two steps:

- **Removing the Sample Holder Assembly on an Ambient DSC 7**
- **Installing a New Sample Holder Assembly in an Ambient DSC 7**

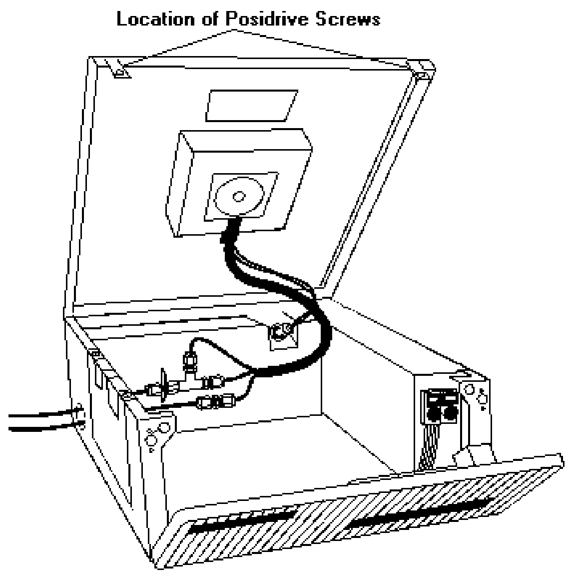
NOTE: Before beginning, make sure that the power switch on the DSC 7 is in the OFF position and remove the line power. Also, make sure there is no purge gas flow to the DSC 7.

Removing the Sample Holder Assembly on an Ambient DSC 7

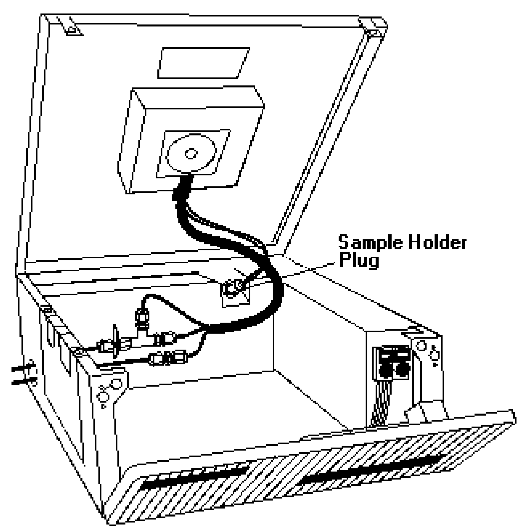
1. Remove the draft shield. Open the sample holder enclosure cover. Loosen and remove the thumbscrew and remove the sample holder enclosure cover.



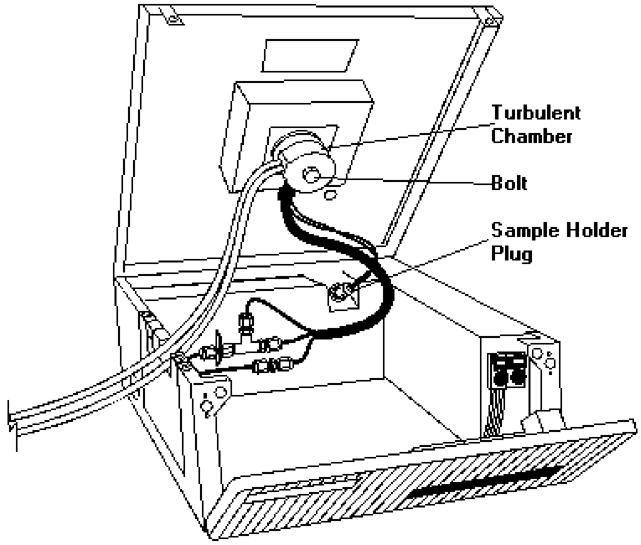
2. Remove the guard rings, if present, from the sample cells. Using sample handling tray, remove all sample pans and sample holder lids from the sample holders.
3. Remove the plastic ring around the sample holder assembly.
4. Open the front panel and remove the two Posidrive screws securing the cover. Lift the cover.



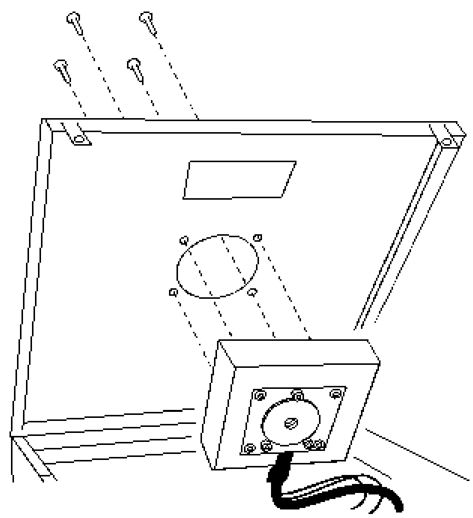
5. Disconnect the sample holder electrical connections from the chassis plug marked BLOCK. Swivel the plug clamp to release its locking tabs.



6. Disconnect the purge gas lines at the chassis.
7. Working from the underside of the DSC 7 cover, remove the hex bolt from the turbulent chamber and remove the chamber.



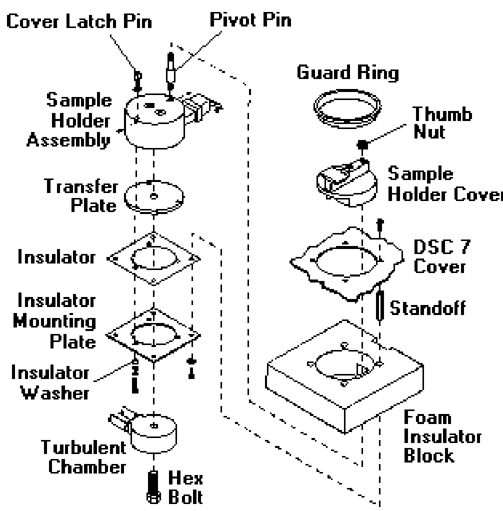
8. While supporting the sample holder assembly, loosen and remove the four binding head screws at the insulator mounting plate and remove the sample holder assembly.



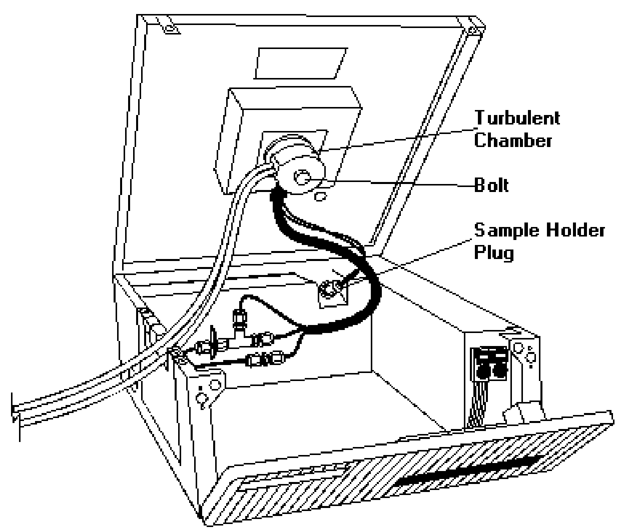
Installing a New Sample Holder Assembly in an Ambient DSC 7

1. While supporting the sample holder assembly, unscrew and remove the three Allen screws that attach the insulator mounting plate to the sample holder assembly. The transfer plate also detaches from the sample holder assembly. Make note of the order in which the steel washers and insulator washer are oriented on these screws. Maintain this order when installing the screws into the new sample holder.

Sample Holder Assembly as Used with an Ambient DSC 7



2. Remove the set screw that secures the pivot pin in the sample holder assembly and then remove the pin. Install the pivot pin in the new sample holder using the set screw to hold it in place.
3. Remove the set screw that secures the enclosure cover catch pin and then remove the pin. The pin is press fit and not threaded into the sample holder assembly. Install the enclosure cover catch pin and screw in the new sample holder.
4. Replace the turbulent chamber and secure it with the hex bolt.
5. Reconnect the block harness and the purge gas lines. Note that the sample holder purge lines are connected at the restrictor, and the single exit line (with marked tag) is not connected. After finger-tightening the fittings, tighten them an additional quarter turn using two 7/16-in. wrenches.



6. Replace other components for normal operation.
7. Close the DSC 7 cover. Replace the front two screws that secure the cover. Close the front panel.
8. Reattach the sample holder enclosure cover and thumbscrew to the sample holder assembly and check the latching mechanism for smoothness of operation. The catch mechanism will require adjustment if the cover is difficult to close or if the cover can move when the latch is closed. Sufficient adjustment is usually achieved by loosening the set screw that secures the catch pin and rotating the catch pin until the latch closes firmly so the cover cannot move. If a positive latching is not achieved by rotating the catch screw, adjust the height of the pivot pin after loosening its securing set screw.
9. Reinstall the guard ring inserts and platinum sample holder covers.
10. Turn on the purge gas and set the flow rate to 20 psi at the regulator.
11. Plug in the DSC 7 to line power.

Clean the Furnace

You can access the Clean Furnace procedure for the DSC 7 by clicking on the **Clean Furnace** button on the control panel.

Intracooler Maintenance

The Intracooler refrigeration system is a hermetically sealed freon system which should require no periodic maintenance except for possible cleaning of the air-cooled condenser if it collects an excessive amount of dust.

In the event of a refrigerant leak, the repair could easily be performed by a local refrigeration repair service. The leak should first be located and sealed. (The flexible line will withstand soft soldering. Take care not to overheat the line, however, and do not attempt silver brazing.) The unit should then be evacuated to 5 mTorr for 24 hours before recharging. The type of refrigerant and static charge are listed on the inside cover. If such service is not available, contact a Perkin Elmer representative.

If the unit does not have a leak and does not reach maximum low temperature, it may be a result of low voltage or high ambient temperature. Measure the voltage at the wall receptacle with the Intracooler ON. If the voltage is below 104 V for 60-cycle operation or below 100 V for 50-cycle operation, try another receptacle. Low voltage can result from heavy electrical equipment on the same line or the use of a long extension cord.

WARNING: **Only very experienced electrical engineers or technicians should attempt to measure line voltage. Failure to take proper precautions could result in serious injury.**

Accessories

DSC 7 Flow Through Cover Kit

The Flow Through Cover Kit (P/N 0319-0062) for the DSC 7 provides a short path for volatiles and/or decomposition products to exit from the DSC sample holder. The cover replaces the standard swing-away cover. It should be used with samples that undergo decomposition, dehydration, oxidation, etc., especially if the thermal conductivity of the products of the reaction is different than that of the purge gas.

To operate the DSC 7 with the flow through cover in place, it is recommended that the normal purge rate be increased from 20 to 50 cc/min to facilitate sweeping of reactive volatile gases.

The DSC calibration constant should be checked by melting indium at the higher flow rate.

Installation of the DSC 7 Flow Through Cover Kit

The Flow Through Cover Kit (P/N 0319-0062) contains the following parts:

Part No.	Description	Quantity
0319-0061	Replacement Cover Assembly	1
0319-0288	Outlet Tube Assembly	2
0319-1666	Sample Holder Exit Fitting	3
0990-2102	16-in. i.d. x 5/32-in. o.d. O ring	6
0990-2205	3/4-in. i.d. x 7/8-in. o.d. O ring	4
0993-9234	Instructions	1

To install the flow through cover, proceed as follows:

1. Remove the standard DSC swing-away cover assembly after loosening the one large knurled securing screw.
2. Assemble the flow through cover kit as follows:
   a. Hold the replacement cover assembly upside down and install the 7/8-in. o.d. O rings in the cutouts in the bottom of the assembly.
   b. Place a 5/32-in. o.d. O ring over the short end of an outlet tube assembly and, while holding the cover assembly upside down, place the end of the outlet tube assembly and O ring in one of the holes in the cover assembly.
   c. Turn the cover assembly over (right side up) and place a sample holder exit fitting over the outlet tube assembly and screw it into the hole in the cover assembly.
   d. Repeat steps b and c for the second hole in the cover assembly.
3. Secure the replacement cover assembly with the one large knurled screw removed in step 1.
4. Insert guard ring inserts, if available, to provide a small annular space to increase effective purge gas flow rates.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis. It can be used with the DSC 7.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the plug attached to the N519-0310 analog cable at the back of the TAC 7/DX Thermal Analysis Instrument Controller. The TAC 7 then connects to the analyzer.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step. Instructions for installing the TAGS are in Installation Help.

CCA 7 Liquid Nitrogen Controlled Cooling Accessory

The Controlled Cooling Accessory (CCA 7) is a subambient accessory for the DSC 7. It is closed-loop LN2 cooling system that allows you to dial in the desired sample holder block temperature on the CCA 7 control unit. Using the CCA 7, the DSC 7 can be run from –140°C. To optimize the sample environment, a dry box must be installed on the DSC 7 and the sample holder enclosure cover O-ring must be removed.

The CCA 7 automatically controls the flow of liquid nitrogen from a storage tank (dewar) to a circulating chamber attached to the instrument. Built-in control circuitry allows the precise control of heat sink temperature for enhanced subambient operation.

Key features of the CCA 7 include:

- fully automated control of liquid nitrogen during subambient operation
- convenient and simple operation to liquid nitrogen subambient temperatures
- reduced consumption of liquid nitrogen

Refer to the CCA 7 manual for full instructions on installation.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

WARNING: FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 1/4" pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

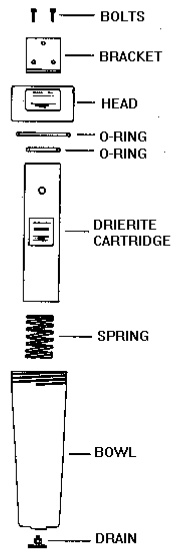
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this filter drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4" pipe fitting using Teflon (Registered DuPont Trademark) tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4" pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.

7. Place the 2-1/2" o.d. O-ring on top of cartridge.
8. Place the 4" o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD S0AP AND WATER. D0 NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

TAC 7/DX Thermal Analysis Instrument Controller

The TAC 7 controls the analyzer and digitizes the analog output from the detector before sending it on to the computer.

Turbulent Chamber

A cold water turbulent chamber can be used with the DSC 7. Circulating baths and refrigerated liquid chillers can be attached to the turbulent chamber as well. The ambient DSC 7 comes with the turbulent chamber attached to the sample holder. A turbulent chamber keeps the sample holder at a temperature in the range of 25°C. For optimum results, the flow rate of the coolant must be constant and there should be no circulating air bubbles in the system.

DSC 7 Part Numbers

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Parts Provided with the DSC 7

Below is a list of the parts that come with your DSC 7. Your instrument should be one of the following:

	115 V	230 V	100 V
DSC 7 Differential Scanning Calorimeter	N519-0243	N519-0242	N519-0244
DSC 7 Ambient Differential Scanning Calorimeter	N519-0246	N519-0245	N519-0247

Spares and Accessories Kit (N519-0147)

Part No.	Description	Quantity
0219-0041	Aluminum Pans and Covers (400 of each)	1
0319-0033	Indium Calibration Reference Material	1
0319-0036	Zinc Calibration Reference Material	1
N519-0274	Draft Shield Assembly	1
N519-1333	Gasket for Draft Shield Assembly	1
0419-0299	Vented Platinum Sample Holder Cover	2
0419-0303	Sample Handling Tray	1
0990-3212	1/8-in. to 1/4-in. NPT Reducing Bushing (female)	1
0990-3434	1/8-in. NPT Male Connector	1
0990-8138	Tweezers	1
0998-1614	Fuse, 2-A, 250-V Slow Blow (DSC 7 for 100 and 115 V)	2
0998-1611	Fuse, 2-A, 250-V Slow Blow (DSC 7 for 230 V)	4
0998-1610	Fuse, 0.5-A, 250-V Slow Blow (DSC 7 Power Supply)	2
0998-4330	Grommet	1
0990-3004	Hose Clamp	1
0250-6519	Tygon Tubing, 3/8-in. i.d. by 1/2-in. o.d.	6 ft

DSC 7 Sample Holder Accessories

Part No.	Description	Quantity
0319-0031	Vented Platinum Sample Holder Covers	6
0419-0299	Vented Platinum Sample Holder Covers	2
0319-0236	Guard Ring Inserts	2
0319-0062	Flow Through Cover	1
0319-0030	Sample Holder Cover Reforming Tool	1
0990-2206	O-Ring Seal for DSC 7 Sample Holder Enclosure Cover	1
N519-0348	DSC 7 Sample Holder Enclosure Cover	1

DSC 7 Sample Pans and Crimping Accessories

Standard Crimper Press

Part No.	Description	Quantity
0219-0048	Standard Sample Pan Crimper Press	1
0219-1171	Replacement Crimper Head for Standard Sample Pan Crimper Press	1

Sample Pans for Standard Crimper Press

Part No.	Description	Quantity
0219-0041	Standard Aluminum Sample Pans and Covers	400 each
0219-0042	Standard Gold Sample Pans and Covers	10 each
0319-0024	Standard Platinum Sample Pans Covers (where gold or graphite pans are not suitable)	4 each
0319-0026	Standard Copper Sample Pans and Covers	200 each
0319-0025	Standard Graphite Sample Pans and Covers	4 each

Volatile Sample Sealer

Part No.	Description	Quantity
0219-0061	Volatile Sample Sealer Assembly for sealing volatile solids or liquids in aluminum or gold volatile sample pans	1
0219-1295	Replacement Lower Die for Volatile Sample Sealer	1
0219-1296	Replacement Upper Die for Volatile Sample Sealer	1

Sample Pans for Volatile Sample Sealer

Part No.	Description	Quantity
0219-0062	Volatile Sample Pans and Covers (aluminum) for volatile solid and liquid samples. Capsules have an effective volume of 20 µL and can withstand internal pressures up to 3 atm.	400 each
0219-0080	Volatile Sample Pans and Covers (gold) for volatile solid and liquid samples which are reactive with aluminum. Capsules have an effective volume of 20 µL and can withstand internal pressures up to 3 atm.	10 each

Stainless Steel Capsules

Part No.	Description	Quantity
0319-0021 includes:	Large Volume Stainless Steel O-Ring Sealed Capsules and Sealing Kit, which includes:	

0319-0218	Large Volume Stainless Steel O-Ring Sealed Pans, Covers, and O-Rings	20 each
0319-0236	Guard Ring Inserts	1
0990-8467	Quick Press	1
0319-1047	Quick Press Spacer Die	1
0319-0218	Large Volume Stainless Steel O-Ring Sealed Pans, Covers, and O-Rings (internal volume of 75 µL; operating range 40°C to 300°C unless otherwise limited by sample vapor pressure, not to exceed 350 psi. If properly sealed, will contain water to 225°C)	20 each
0319-0029	Large Volume Stainless Steel O-Ring Sealed Pans, Covers, and O-Rings	1000 each

High-Pressure Capsules

Part No.	Description
B018-2864	High Pressure Capsule Sealing Tool
B018-2901	Stainless Steel High Pressure Capsules
B018-2902	Gold Plated Stainless Steel High Pressure Capsules
B018-2903	Titanium High Pressure Capsules
B018-2904	Copper Seals
B018-2905	Gold Plated Copper Seals

Accessories for Use with Older Style High Pressure Capsules

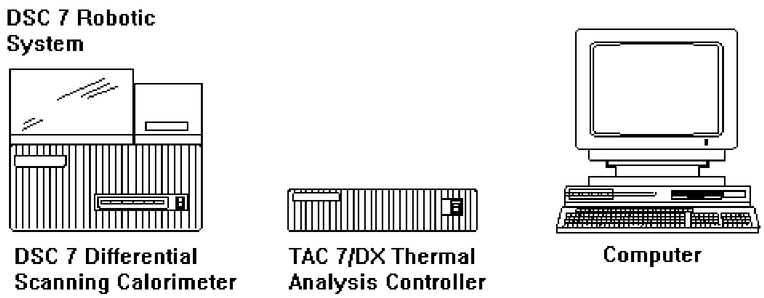
Part No.	Description
0419-1757	High Pressure Capsule Sealing Tool
0419-1761	Stainless Steel High Pressure Capsules
0419-1760	Gold Plated Stainless Steel High Pressure Capsules
0419-1759	Copper Seals
0419-1758	Gold Plated Copper Seals

Additional Spares and Supplies

Part No.	Description	Quantity
0319-0018	Calibration Sample Kit (includes indium, tin, lead, zinc, potassium sulfate, and potassium chromate)	1
0219-0136	Specific Heat Kit	1
0319-0039	Filter Dryer Accessory	1
N537-0103	Drierite Compressed Air Filter Dryer	1
0240-0084	Pressure Regulator for Oxygen	1

0240-0085	Pressure Regulator for Helium and Nitrogen	1
0290-1624	Float Displacement Flow Meter	1
0990-5147	Silicone Grease	1

DSC 7 Robotic System



The DSC 7 Robotic System is a robot autosampler attached to the top of a DSC 7 Differential Scanning Calorimeter, which is connected to a TAC 7/DX Thermal Analysis Instrument Controller which itself is connected to a computer that runs Pyris Software for Windows. The DSC 7 sample holder fits into the baseplate of the robotic system and is closed by a special sample holder cover built into the robotic system. The robotic system unit connects to the auxiliary port of the TAC 7/DX controller by an RS 232 cable. One TAC is used for both the DSC 7 analyzer and the robotic system. It can also connect directly to the computer at the COM2 port, if it exists.

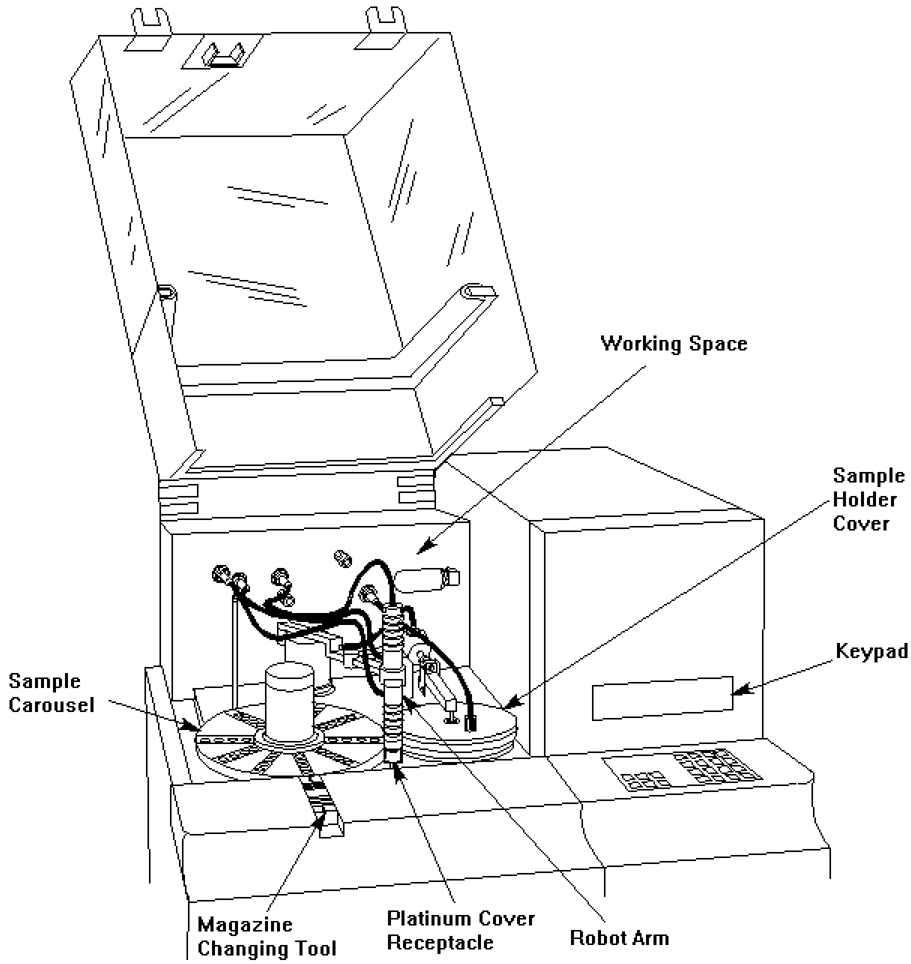
With the Pyris Player software, the robotic system becomes fully automated for DSC analyses. The robotic system's tray has 48 sample positions but with Pyris Player you can program it to run up to 1000 samples.

All robotic system programs are set up and executed by the Pyris Player software. If you need to perform an operation manually, you can switch to manual mode via the **Reset + Manual** keys on the robotic system's keypad. Manual operations that require switching to manual mode include changing samples in the sample tray, opening and closing the sample holder cover, and running single samples.

Special robot commands can be used on the robotic system's keypad if you want to use the DSC 7 as a stand-alone, i.e., without the robotic system, or if you need to clear an error without opening the protective cover.

The robotic system consists of the following major components:

- the working space, which includes the sample tray, robot arm, robot arm lifting mechanism, lever gear, platinum cover, and sample holder cover, is located under a protective cover
- the pneumatic system, which includes the switching valves for the mechanical movement of the robot arm, robot arm lifting mechanism, and sample holder cover
- the electrical system, which includes the power supply, control electronics, and RS 232 interface
- keypad and display
- rear panel controls and connections



The entire robotic system can be tilted back by opening the DSC 7 front panel, removing the two Phillips-head locking bolts from the DSC 7 (these bolts secure the front of the robotic system to the DSC 7), then gripping the robotic system and tilting it back. This provides easy access to the lower side of the sample holder and to the interior of the DSC 7 for the installation of accessories for subambient analyses. Observe the following precautions when you perform this procedure:

CAUTION: Switch off the analyzer before preparing the DSC 7 for tilting the robotic system.

CAUTION: The robotic system is heavy; when tilting it back, hold the DSC 7 down, Be absolutely certain the robotic system cannot fall backward by accident or drop down.

CAUTION: Never try to lift the robotic system by the recess at the horizontal front panel. This can damage the tray mechanism.

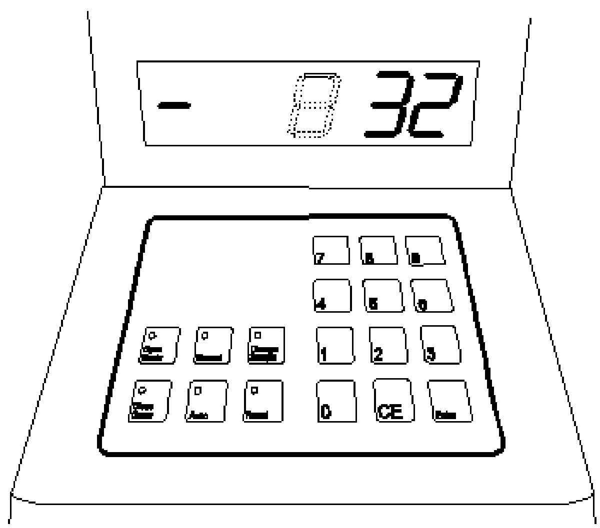
CAUTION: When folding the robotic system down, be sure that no electrical cables or tubes are squeezed.

The following topics discuss the DSC 7 Robotic System in more detail:

- Warnings and Safety Practices
- How the Robotic System Works
- Operating Modes
- Robotic System Controls
- Sample Handling
- Running a Play List
- Robot Commands
- Troubleshooting
- Maintenance
- Parts Provided

Robotic System Keyboard and Display

The front panel includes a keyboard and a display window.



The keypad has numeric and function keys. The function keys are used to switch the robotic system between auto and manual operation, for opening and closing the sample holder, and for changing samples. The function keys each contain a small LED that lights when the key is active. The numeric keys are used to enter tray numbers when changing samples and to manually move the robot arm.

The display shows the current operating status (auto or manual), numerical values used when manually changing samples, and the display of error codes.

The display window has two parts: the status display on the left and the numerical display on the right.

Status Display

The status display is a single-characters display of the current operational status of the robotic system. The codes are

–	robotic system is in manual mode
H (Host)	robotic system is in auto mode
O (Open)	Sample holder cover is being opened
L (Lock)	Sample holder cover is being closed
S (Sample)	**Change Sample** key is active
E (Error)	An error has occurred

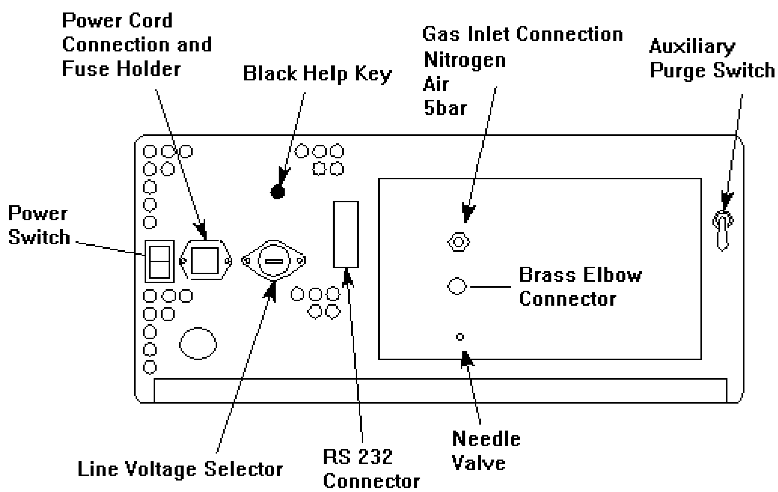
Numerical Display

The numerical display is a three-digit display showing:

- currently selected tray position numbers used in the **Change Sample** key operation
- numerical part of the robotic system error codes

Robotic System Rear Panel Controls

The robotic system rear panel contains the following controls and connections:



- power switch
- power cord connection and fuse holder; connect power cord and change the main fuse
- line voltage selector for selecting appropriate AC line voltage for your location
- RS 232 connector used to connect the RS 232 cable from the TAC AUX port
- gas inlet connection for the pneumatic gas
- elbow connector to connect purge gas for the working space. You can connect the exhaust tubing from the CCA 7 cooling accessory.
- auxiliary purge switch to open or close the flow of the purge gas to the working space

- Help button which is used in troubleshooting error codes
- Needle valve regulates the amount of purge gas permitted into the diffuser in the working space. Initially, the needle should be closed all the way. Slowly open up the valve to permit purge gas into the working space.

Warnings and Safety Practices

WARNING: **Be sure that all instrument operators read and understand the following precautions. It is advisable to post a copy of these precautions on or near the DSC 7 Robotic System itself.**

The following precautions must be observed when using the DSC 7 Robotic System. These are in addition to the precautions that apply to the DSC 7.

WARNING: **Your DSC 7 Robotic System is designed to be installed by a Perkin Elmer Service Engineer. Do not try to perform any installation procedure beyond what is mentioned in this manual.**

WARNING: **Observe all local safety regulations when connecting the components of your DSC 7 Robotic System to each other and to the local electrical supply.**

WARNING: **Observe all local safety regulations concerning the handling of gas cylinders when connecting the gas supply to the robotic system.**

WARNING: **Never operate the DSC 7 Robotic System in an explosive atmosphere.**

CAUTION: When tilting the DSC 7 Robotic System back, be absolutely certain it cannot fall backward accidentally.

CAUTION: Never try to lift the DSC 7 Robotic System by the recess in the horizontal front panel. The tray could be damaged.

CAUTION: Make sure that all cables and tubes are clear when lowering the autosampler down to be closed.

CAUTION: Never use the Autosampler Control dialog box to control the Robotic System while it is loading or returning a sample pan.

How the Robotic System Works

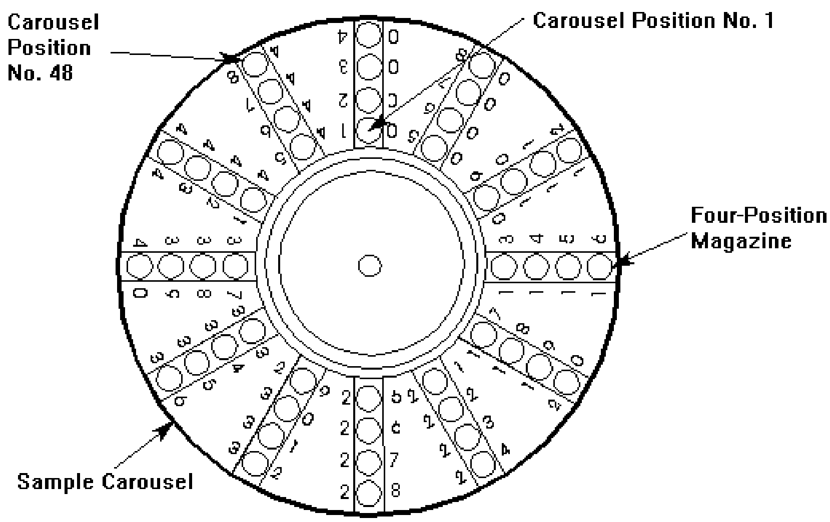
Working Space

The working space is covered by a transparent, air-tight clear plastic cover. The cover is closed tight by two locks accessible when the horizontal front panel is pulled forward. The cover permits observation of the movements of the mechanical components, protects the mechanical components and sample capsules from dust, and allows the working space to be filled with dry, compressed air or dry nitrogen.

Filling the working space with dry, compressed air or dry nitrogen helps avoid condensation or ambient humidity on the sample capsules, sample holder, and other mechanical parts during subambient analyses. The analyses can take place in an inert atmosphere (nitrogen), thus preventing ambient air from penetrating the working space.

Sample Tray

The sample tray is driven by a stepper motor. Samples are loaded into sealed sample pans, which are then loaded into magazines. Each magazine holds up to four sealed capsules; the tray holds 12 magazines. The tray can hold up to 48 samples at any one time. The magazines are arranged radially on the tray; a concentric magnet ring in the center of the tray prevents the magazines from slipping out. Magazine positions are labeled 1 through 48 for easy identification of the samples.



Magazines can be pulled out and inserted through a small slot in the working space cover so you can change samples without affecting the dry atmosphere in the working space.

Movement Control

The moving components of the robotic system excluding the sample tray, i.e., robot arm, robot arm lifting mechanism, and lever gear, are activated by electromagnetic and pneumatic drives. An electric motor controls the horizontal movements of the robot arm by means of a lever gear. The vertical movements of the robot arm and lifting mechanism are activated by the pneumatic system, which uses compressed air or dry nitrogen as the working gas. This is the same gas used for the purge gas.

The robot arm picks up a sample pan and lifts up the platinum cover by aspirating them at the lower part of the lifting arm, where they are held. The sample pan and platinum cover are released by the pneumatic system by the application of low pressure.

Robot Arm

The tip of the robot arm is constructed from a special silicon rubber that contains carbon. Both the robot arm and the lifting head are conductive and grounded to avoid electrostatic discharges. This prevents the sample capsule from sticking to the robot arm when it is set down into the magazine.

In addition to picking up the sample capsules and platinum covers, the robot arm functions as a sensor. During a typical work cycle, the robotic system uses the robot arm to check whether the magazine position is empty and whether the platinum cover is properly positioned. An error code is reported back to Pyris if there is a problem.

Sample Holder Cover

The sample holder cover is activated by the pneumatic system in an elbow lever mechanism and contains an O-ring that seals the DSC 7 sample holder cover gas-tight. An error code is reported back to Pyris when the cover does not close properly and the seal is not tight.

Robotic System Work Cycle

When you begin a sample run by starting a play list created with the Pyris Player Editor, the robotic system performs a typical work cycle, which includes the following steps:

1. Before the start of a run, the robot arm is in its starting position, i.e., above tray position 3 (the third sample pan position from the center of the tray in the magazine that faces the sample holder).
2. The play list entry **Load Sample** begins.
3. The sample holder cover opens.
4. The robot arm verifies that the platinum cover is located in its receptacle.
5. The robot arm verifies that the sample cell in the sample holder is empty.
6. The sample tray transports the magazine containing the specified sample to the sampling position (facing the sample holder).
7. The robot arm picks up the specified sample and carries it to the sample cell in the sample holder.
8. The robot arm releases the sample into the sample cell.
9. The robot arm picks up the platinum lid from the receptacle.
10. The platinum lid is transported to the sample cell.
11. The robot arm places the platinum cover onto the sample cell.
12. The robot arm positions itself over position 3.
13. The sample holder cover closes.
14. The robot arm moves to the platinum cover receptacle where it is lowered. The working gas is switched off.
15. The sample is analyzed according to the method specified in the play list.

The play list command **Return Sample** starts the following sequence of events:

16. The robot arm moves over to position 3 as the sample holder cover opens.
17. The working gas is switched on.
18. The robot arm moves to the sample cell and picks up the platinum lid.
19. The robot arm carries the platinum lid to its receptacle and places it into the receptacle.
20. The robot arm moves to the sample cell and picks up the sample pan.
21. The robot arm carries the sample pan back to the sample tray, which has rotated the appropriate magazine into position, and releases it into the specified position (it does not have to be the same position from which it was removed).
22. The robot arm returns to starting position 3; the sample holder cover remains open.
23. If the next sample in the play list is in a different magazine, the sample tray moves this magazine to the sampling position.
24. Steps 5 – 22 are repeated for each sample until all of the samples specified in the play list are analyzed.
25. After the last sample in the play list is analyzed, the robot arm returns to the starting position and the sample holder cover closes.

DSC 7 Robotic System Operating Modes

The DSC 7 Robotic System has two operating modes: auto and manual. Both operating modes are selected at the robotic system keypad. The current operating mode is displayed as a single character on the display window.

Auto Mode

In auto mode, the robotic system is controlled by Pyris software. While the DSC 7 Robotic System is in auto mode, you can run a play list that automatically runs multiple samples and performs postrun analyses on the collected data. Through the Autosampler Control dialog box, you can control the loading and unloading of specific samples in the sample tray for individual sample analysis. However, you must go into Manual mode to open and close the sample holder cover. The **Manual** and **Reset** keys are the only keys active on the keypad while in auto mode.

Manual Mode

In manual mode, the movements of the sample tray and sample holder cover are controlled via the robotic system keypad. You can open and close the sample holder cover, load and unload reference samples to and from the reference cell, rotate the tray to change samples, and use the Robotic System Help routines. **The Robotic System must be switched to manual mode before you can perform these operations.** All of the keys on the keypad are active when in manual mode.

Switching Between Modes

Switch to auto mode if you want to create and/or run play lists created in Pyris Player or you want to work with the Pyris software. Switch to manual mode if you want to load or change samples in a magazine, load or unload the reference pan in the reference cell, or open and close the sample holder cover.

To switch from auto to manual mode:

1. Press the **Reset** key on the robotic system keypad.

2. Press the **Manual** key on the keypad.

   The LED on the **Manual** key lights and the "–" symbol appears on the display window.

To switch from manual to auto mode:

1. Press the **Reset** key on the robotic system keypad.
2. Press the **Auto** key on the keypad.

   The LED on the **Auto** key lights and the letter "H" appears on the display window.

NOTE: The robotic system will not switch to auto mode if a manual operation is currently being performed. Wait until the manual operation is complete, then press the **Reset** and **Auto** keys.

NOTE: If you select Go To Load, Go To Temp, or Hold from the Control Panel while a play list is running, the current sample run will end and the playback of the play list ends.

DSC 7 Robotic System Controls

To perform manual operations such as opening and closing the sample holder cover, changing reference samples, or adding/removing samples from magazines, you can use the keypad and display on the robotic system while in manual mode. The function keys on the keypad are identified by the small LED on the key that lights when the key is active. The function of each key and what appears in the display window when that key is active are given below:

Key	**How to use it**	**Display**
Open Cover	Press this key to pneumatically open the sample holder cover in manual mode only. You cannot use this key while another manual operation is being performed or when the robot arm is above the sample holder.	O while the action is performed; – when the cover is open.
Close Cover	Press this key to pneumatically close the sample holder cover and seal it gas-tight, in manual mode only. You cannot use this key while another manual operation is being performed or when the robot arm is above the sample holder.	L while the action is performed; – when the cover is closed.
Manual	Press this key to switch the robotic system to manual mode. This activates the Open Cover, Close Cover, Change Sample, and Auto keys. You must press Reset first.	–
Auto	Press this key to switch the robotic system to auto mode. You must press the Reset key first.	H

Key	How to use it	Display
Change Sample	Press this key in manual mode only to rotate the sample tray to a specific tray position to change samples.	S after the key is pressed; S XX while the action is performed; XX is the entered position number.
Reset	Press this key before pressing the Auto key to switch to auto mode. The robot arm returns to its starting position when you press Reset. Press this key before pressing the Manual key to switch to manual mode.	–0
Numeric keys	Use these keys to select tray position numbers when using the Change Sample key. These keys are also used for special robot commands.	Selected tray position numbers appear on the display.
Enter	Use this key to enter numerical selections.	
CE	Use this key to clear numerical entries from the display window.	

Sample Handling for the DSC 7 Robotic System

Handling samples encompasses the following topics:

- **Preparing Samples**
- **Loading Samples for the First Time**
- **Changing Samples**

Preparing Samples

When preparing your samples for analysis in the DSC 7 Robotic System, take the following operating variables into consideration:

- **Sample size**
- **Sample atmosphere**
- **Temperature range**
- **Scanning rate**

These are discussed earlier for a DSC 7 without a robotic system attached. In addition to these considerations, the robotic system has specific requirements for the choice of sample pan and cover and for the method of sample encapsulation.

Sample Pans

Sample pans and covers of various capacities and wall thicknesses are available from Perkin Elmer. These pans and covers will resist the varying internal pressures of sample capsules without deformation. These items are listed in the **DSC Sample Pan Table** and discussed in the **Robotic System Sample Pans** section presented earlier.

> **CAUTION:** Never touch the sample or the sample pan and cover with your bare fingers. Contamination from human body oils and greases can affect your results. Always use tweezers or the Perkin Elmer Standard Suction Manipulator (P/N B014-3263) or the Special Suction Manipulator (P/N B014-2512).

Sample Encapsulation

The Universal Crimper Press (P/N B013-9005) is required for preparing samples for the robotic system. See the instructions in **How to Use a Universal Crimper Press** in the Pyris Multimedia Presentations Help for how to use the accessory to encapsulate your samples.

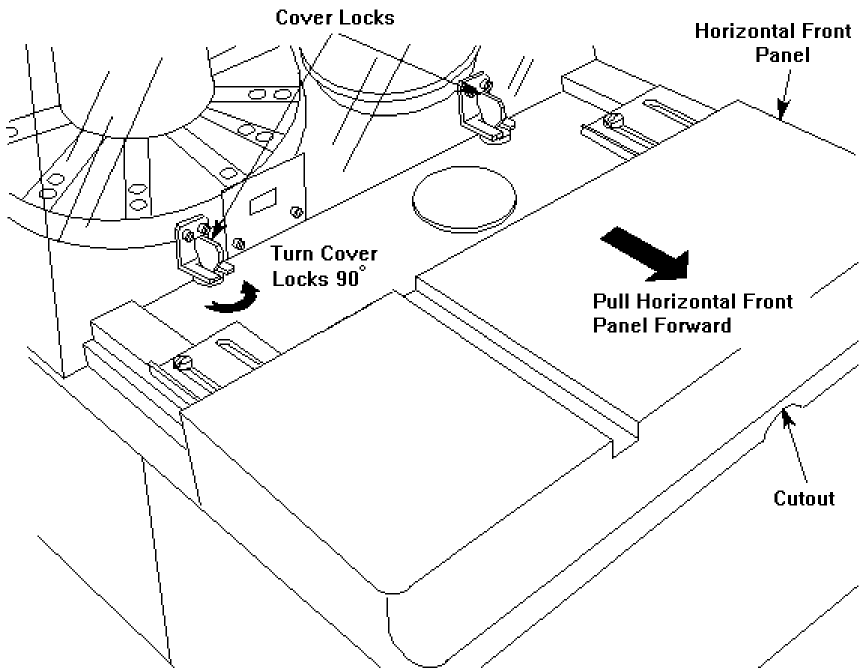
Loading Samples for the First Time

Use the procedure below to load samples into the sample tray if you have not used the robotic system yet.

1. Make sure the robotic system is turned on and the working gas is turned on and adjusted to the correct pressure.
2. The robotic system should be in manual mode (a dash “–“ should appear on the left-hand side of the display window and the light on the **Manual** key is lit). If it is not, press the **Reset** key and the **Manual** key to switch to manual mode.
3. Open the protective cover by carefully lifting it up and back.

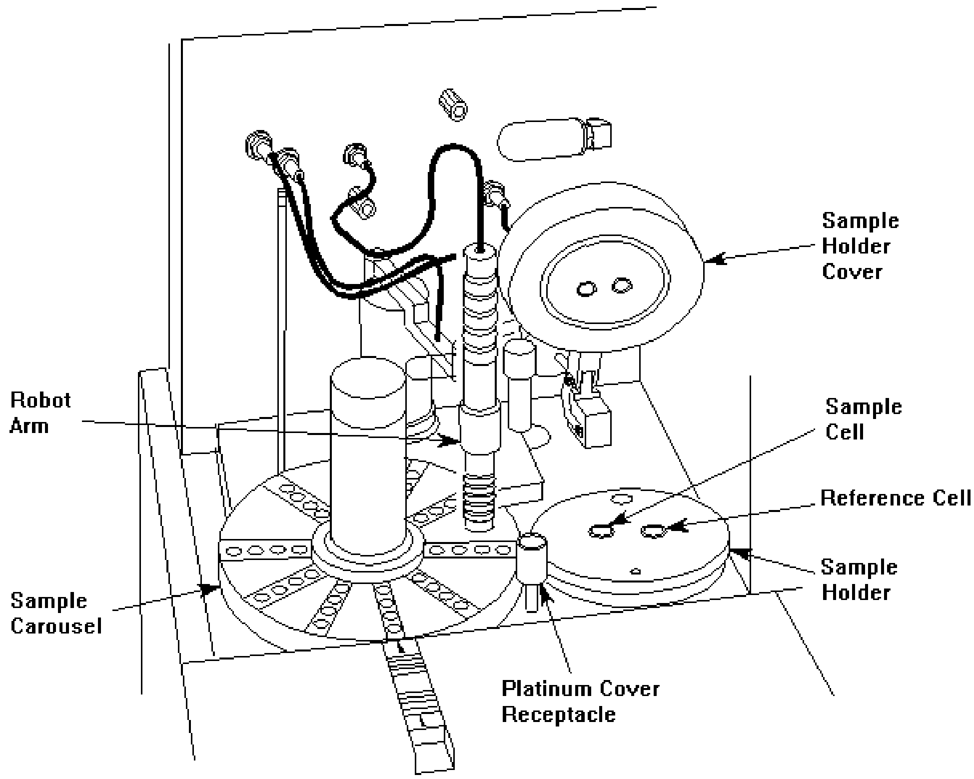
   If the protective plastic cover is locked, perform the following steps while referring to the figure seen by clicking here:

   a. Open the DSC 7 front panel to access the robotic system horizontal front panel.
   b. While holding the horizontal front panel by the cutout, carefully pull the panel straight forward until the two cover locks are completely exposed.
   c. Turn both locks 90° to release the cover.
   d. Carefully lift the cover up and back.



NOTE: If you are using a subambient cooling device such as the Intracooler, open the working space *before* you cool down the system. If the Intracooler is on and the system is already at subambient temperatures, use the sample changing procedure described in Changing Samples.

4. Press the **Open Cover** key to open the sample holder cover.
5. Using a suction manipulator (P/N B014-3263 or B014-2512) or tweezers, insert an empty, sealed sample capsule into the reference cell of the sample holder.



6. Cover the reference cell with a platinum cover (supplied with the robotic system). Make sure the sample cell of the sample holder is empty.
7. Place a platinum cover on the platinum cover receptacle.

NOTE: If you are going to perform high-precision specific heat, you must be very particular about the reference sample pan and the platinum lid used.

8. Prepare your samples as described in Preparing Samples and Sample Preparation.
9. Insert the magazines into their numbered positions in the sample tray.

CAUTION: The numbers on the magazines must correspond with the numbers on the sample tray.

10. Using a suction manipulator (P/N B014-3263 or B014-2512), insert the sealed sample capsules into the desired tray magazine locations.
11. Close the robotic system cover and lock it. Push the horizontal front panel back to its original position and close the DSC 7 front panel.

Preparing for Subambient Operation

If you plan to use the DSC 7 Robotic System for subambient applications, observe the following guidelines:

- Connect the cooling system you are using as described in the topic Subambient Operation and subtopics.

- To perform controlled measurements, the block temperature should be at least 30°C lower than the lowest measuring temperature. The lowest block temperature allowed for subambient operation is –100°C. This means that temperature measurements as low as –70°C are possible.
- Before turning on the cooling system, purge the robotic system working space. This prevents air humidity condensation on the sample holder. To purge the working space, first make sure that the operating gas is connected and adjusted to the correct pressure and the protective cover is closed and locked. Move the AUX Purge switch on the back of the robotic system to the On position. Purge the working space with dry operating gas (compressed air or nitrogen) for at least 15 minutes.
- Keep the robotic system protective cover closed and locked while operating your cooling system. Change samples using only the **Change Sample** key and slide-in magazine.
- Open the sample holder cover only when the temperature is above 20°C.
- For the DSC 7 Robotic System, you must set the End Condition of the method **Go To Temp** to 30°C in the Set End Condition section in the Program page. The load or unload step in a play list will occur when the sample or reference temperature is within plus or minus 5°C of the program temperature.

Changing or Adding Samples

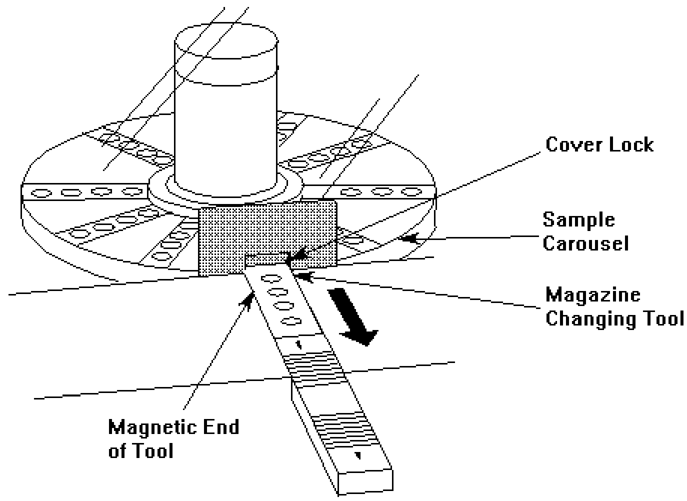
You may need to change samples in or add samples to the sample tray while a run is in progress. For example, you may want to run a play list that analyzes more than the maximum 48 samples that the tray can hold. However, to prevent humidity and dust from entering the robotic system's working space, or if the DSC 7 is in subambient mode, you should keep the protective cover closed and locked at all times. To access the sample tray to remove and add pans, the protective cover is equipped with a port through which magazines can be removed and inserted easily.

To change or add samples while a method step or a postrun data analysis step in the play list is being performed, you do not have to pause the play list. Use the Autosampler Control dialog box to select the tray position number that you want to access. The tray will rotate into position so that you can remove the magazine that contains the desired position. You may also use the keys on the robotic system keypad to perform this operation.

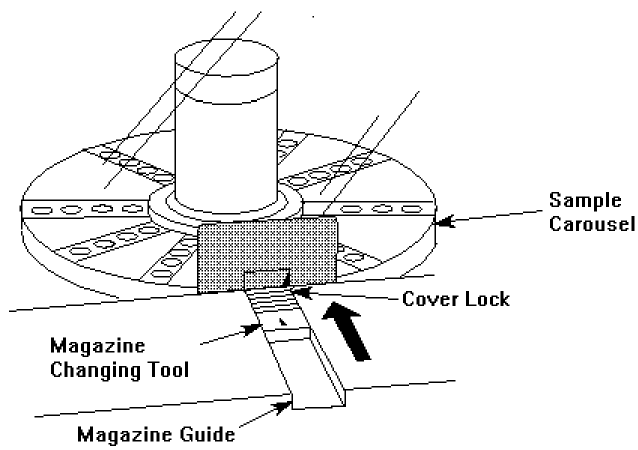
CAUTION: Perform this operation only during data acquisition, not when a Return Sample or a Load Sample is being executed in the play list.

Using the Autosampler Control dialog box, follow the procedure below to change or add samples during a run:

1. Click on the **Autosampler Control** button in the control panel.
2. In the Autosampler Control dialog box, enter the tray location in which you want to insert a sample pan or pans and click on the **Move Carousel to Location** button. The tray rotates so that the magazine containing that location is facing the insertion slot at the front of the analyzer.

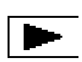


Removing a Magazine

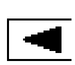


Inserting a Magazine

3. Position the magazine-changing tool in the magazine guide so that the magnetic end faces the port. The magnetic end of the magazine-changing tool looks like this:



4. Push the magazine-changing tool through the port until it contacts the magazine in the tray.
5. Pull the magazine-changing tool with the magazine attached out of the port.
6. Add sample pans to or remove sample pans from the magazine using a suction manipulator or tweezers.
7. Place the magazine in the magazine guide.
8. Position the magazine-changing tool in the magazine guide so that the nonmagnetic end faces the port. The nonmagnetic end looks like this:



9. Push the magazine back through the port.

10. When finished with one magazine, return to the Autosampler Control dialog box and either enter another location that is in another magazine to add or change samples or click on the **Close** button.

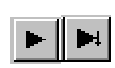
If you want to use the robotic system keypad to add or change samples during a run, follow the procedure below:

1. Put the robotic system in manual mode by pressing the **Reset** button and then the **Manual** button on the keypad. A dash should be displayed in the left-hand side of the display window and the light on the **Manual** key should be lit.
2. Press the **Change Sample** key.
3. Using the numeric keys on the keypad, type the number of the sample tray position that you want to change (1 – 48).

   The position number will appear on the display window. To clear the entry, press **CE**.
4. Press **Enter** on the keypad. The sample tray will rotate until the magazine containing the position number you selected or the empty magazine groove is directly behind the port.
5. Position the magazine-changing tool in the magazine guide so that the magnetic end faces the port.
6. Push the magazine-changing tool through the port until it contacts the magazine in the tray.
7. Pull the magazine-changing tool with the magazine attached out of the port.
8. Add or remove sample pans to the magazine using the suction manipulator or tweezers.
9. Position the magazine in the magazine guide.
10. Place the magazine-changing tool in the magazine guide so that the nonmagnetic end faces the port.
11. Push the magazine back through the port.
12. When finished, press **Reset** and **Auto** on the keypad to return control of the autosampler back to Pyris.

Running a Play List in DSC 7 Robotic System

Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items - Load Sample, Run Method, Return Sample - there is a Sample Group. This simplifies grouping like samples together (as you would have in a sample tray of an autosampler) that use the same test method and data analysis within a play list. A Sample Group consists of a Sample List and a Data Analysis List. A specific method is selected for the samples in the Sample List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization and calculations for automatic data analysis. Display curve allows the last run data set to be recalled. Playback of the play list begins by clicking on the **Start** or **Start at Current Step** button on the Player toolbar. They are the two leftmost buttons:



NOTE: If you select the **Start at Current Step** button, the focused item must be a main-level item, i.e., Prepare Sample, Sample Group, or Data Analysis. However, if in a Sample Group, you can start a play list if the current item is a Sample line.

Before starting a play list, perform the steps below:

1. Review the safety and warning notes in Warnings and Safety Practices.
2. Verify that the purge and pneumatic gas tubing for the DSC 7 and the robotic system and the tubing for a cooling device, if applicable, are properly connected.
3. Verify that the electrical and cable connections between the robotic system and the DSC 7 and all other cables are properly connected.
4. Turn on the power to the system components in the following order:
   - computer
   - robotic system
   - DSC 7
   - TAC
   - GSA 7 or TAGS if present
   - printer
5. Turn on the pneumatic and purge gases and adjust pressures.
6. Prepare your samples as described in Sample Preparation.
7. Load the samples into the sample tray and the reference cell of the sample holder as described in Loading Samples for the First Time. Place a platinum lid on the reference sample pan in the reference cell and on the platinum lid receptacle.
8. If using a subambient cooling device, follow the instructions in Operating the DSC 7 Using an Intracooler 1 or 2 or Operating the DSC 7 with Liquid Nitrogen.
9. Start up Pyris Software for Windows and either create a new play list or load an existing play list you wish to use to run your samples.
10. Make sure that the robotic system is in Auto mode. If it is not, press **Reset** and **Auto** on the keypad.
11. Start the play list.

NOTE: If you select Go To Temp, Go To Load, or Hold at Temp from the Control Panel while a play list is running, the current sample run and the playback of the play list end.

Running More Than 48 Samples

The DSC 7 Robotic System sample tray holds up to 48 samples at one time; however, you can run more than 48 samples by switching to manual mode, removing the sample magazines, changing samples, and reinserting the sample magazines with new samples.

1. Start the play list by clicking on the **Start at Top** button on the Player toolbar.

   Wait until a number of samples have been run. You will replace the samples already run with new samples.
2. Click on the **Autosampler** button on the Control Panel. The Autosampler Control dialog box is displayed.
3. Press the **Reset** and **Manual** keys on robotic system keypad. The system is now in manual mode.
4. In the RS Control Panel, enter the number of the first sample tray position to which you want to add a new sample in the Move Carousel to Location field. (The number appears on the display window.)

The tray rotates until the magazine containing the position number you entered is directly behind the port in the protective cover.

5. Use the magnetic end of the magazine-changing tool to remove the magazine from the tray. Change the samples in the magazine. Reinsert the magazine through the cover port using the other end of the tool.

CAUTION: Perform this procedure only during data acquisition, not while the robot is changing samples.

6. Repeat steps 4 and 5 to change more samples.
7. Press **Reset** and **Auto** on the keypad to switch back to auto mode.
8. Click on the **Close** button in the Autosampler Control dialog box. The play list will resume the run.

DSC 7 Robot Commands

If you want to use the DSC 7 as a standalone system, i.e., without the autosampler, or if you want to clear an error without opening the protective cover, you can use special robot commands given on the keypad. These special robot commands let you move the sample tray and robot arm without running any samples. To access the commands:

1. Make sure the robotic system is turned on; the operating gas is properly connected, turned on and adjusted to the correct pressure; and the protective cover is closed and locked.
2. Press the Help button located on the back of the autosampler.
3. Press the **Reset** key.
4. Press the **Auto** key.

   The robotic system will perform a brief self-diagnostic test. **– 0** will appear on the display window and the sample holder cover will open. You can now use the robotic system numeric keys to send robot commands as shown in the **Special Robot Commands Table**.

To exit the special robot commands:

1. Press the **Change Sample** key on the keypad. The robot arm moves to the position above the platinum cover receptacle.
2. Press **Change Sample** again. The robotic system executes its power-on routine and a dash (**–**) appears in the display window, indicating manual mode.

Load Sample Pan Using Robot Commands

The following example describes how to load a sample pan from tray position 15 to the sample holder using the special robot commands. Note that all keys pressed are on the robotic system keypad.

1. Press the Help button on the back of the robotic system.
2. Press **Reset** and **Auto**.

   The sample holder cover opens and the tray rotates until the robot arm is centered over position 1.

3. Press **6** three times to rotate the tray three magazines counterclockwise. The magazine with position numbers 13 – 16 will be next to the sample holder.

When you press a number on the robotic system keypad, the number appears on the display during movement. When a movement is completed, –0 appears on the display, indicating that you are in manual mode.

4. Press **5** twice to move the robot arm from tray position 13 to 15.
5. Press **1 4 1** to order the robot arm to move a short distance toward the sample pan, turn the vacuum on (picking up the sample pan in position 15), then move the arm back up.
6. Press **5** twice to move the robot arm (now holding the sample pan from position 15) to a position directly above the sample holder sample cell.
7. Press **0 4 0** to move the robot arm all the way down to the sample holder sample cell, turn the vacuum off (releasing the sample pan), then move the robot arm up.
8. Press **5** to move the robot arm to a position directly above the platinum cover receptacle.
9. Press **0 4 0** to move the robot arm all the way down to the platinum cover receptacle, turn the vacuum on (picking up the platinum cover), then move the arm back up.
10. Press **2** to move the robot arm (now holding the platinum cover) counterclockwise to a position directly above the sample holder sample cell.
11. Press **0 4 0** to move the robot arm all the way down to the sample holder sample cell, turn the vacuum off (releasing the platinum cover), then move the arm back up.
12. Press **2** twice to move the robot arm to tray position 15.
13. Press **Close Cover** to close the sample holder cover.

    Once the sample holder cover is closed, you cannot open it without first pressing **Reset** and **Auto**. This brings you back to step 3 above.

Remove Sample Pan Using Robot Commands

To remove the sample pan from the sample holder using special robot commands:

1. Press the Help button on the back of the robotic system.
2. Press **Reset** and **Auto.**
3. Press **5** four times to move the robot arm to a position above the sample cell.
4. Press **0 4 0** to move the robot arm all the way down to the sample holder sample cell, turn on the vacuum (picking up the platinum cover), then move the arm back up.
5. Press **5** to move the robot arm to the platinum cover receptacle.
6. Press **0 4 0** to move the robot arm all the way down to the platinum cover receptacle, turn the vacuum off (releasing the platinum cover), then move the arm back up.
7. Press **2** to move the robot arm counterclockwise to a position directly above the sample holder sample cell.
8. Press **0 4 0** to move the robot arm all the way down to the sample holder sample cell, turn on the vacuum (picking up the sample pan), then move the arm back up.
9. Press **2** twice to move the robot arm counterclockwise to a position above the sample tray.
10. Press **6** three times to rotate the sample tray counterclockwise. The magazine with position numbers 13 – 16 will be next to the sample holder.
11. Press **1 4 1** to move the robot arm a short distance down to position 15 in the magazine, turn the vacuum off (releasing the sample pan into position 15), then move the arm back up.
12. Press **Close Cover** to close the sample holder cover.

13. Press **Change Sample** twice to return to Manual mode.

DSC 7 Special Robot Commands Table

Key	Action
0	Move the robot arm down the maximum distance over the sample holder. Press again to move the robot arm up.
1	Move the robot arm down a short distance over the sample tray. Press again to move the robot arm up.
2	Move the robot arm counterclockwise to its next working cycle position.
3	Rotate the sample tray clockwise to the next magazine.
4	Turn the vacuum generator in the robot arm on or off, letting the robot arm deposit a sample capsule or a platinum cover.
5	Move the robot arm clockwise to the next tray position.
6	Rotate the sample tray counterclockwise to the next magazine.
7	Move the robot arm to the sample holder position, then turn the vacuum generator in the robot arm on to pick up the platinum cover.
8	Reposition the robot arm over the current tray position.
9	Reposition the sample tray at the current position.

Troubleshooting

When there is a problem with the DSC 7 Robotic System, an error code will appear on the display window and an error message will appear in the Pyris software. The error codes are described below.

WARNING: **If an error code appears that is not described here, contact a Perkin Elmer Service Engineer immediately. Do not try to solve the problem yourself.**

Two errors that occur often are

1. When the robotic system is first powered on, the tray moves so that sample 1 is at the cover lock and the robot arm is moved over the tray. Occasionally, if the robot arm started at the platinum cover receptacle, there may be too much friction in the linkage system to let the robot arm move. When this occurs, the robotic system reports an error and cannot continue. Either repeatedly power the robotic system off and on until the arm becomes unstuck or open the protective cover and carefully push the robot arm into movement.
2. After many cycles, the robot arm may fumble the platinum cover (and, less frequently, sample capsules). When this occurs, the robotic system reports an error and cannot continue. Open the protective cover and replace the platinum cover.

   If an error occurs during playback of a play list, the playback ends. If an error occurs during a method run in a play list, the playback stops and you have to stop the run by clicking on the **Stop Method** button in the Control Panel. Restart the run after correcting the problem. The History page in Pyris Player can be used to determine what samples were run prior to the error.

No Working Gas Pressure

Possible Causes	Solutions
The working gas is not connected.	1. Press Reset and Auto. 2. Connect the working gas to the Gas Inlet port on the RS. 3. Set the inlet pressure to 500 kPa (5 bar).
The inlet pressure is below 400 kPa (4 bar).	1. Press Reset and Auto. 2. Set the inlet pressure to 500 kPa.
The internal gas tube has a leak or has slipped from its nozzle.	1. Turn the robotic system off, disconnect the power cord, and remove the rear cover. 2. Check the internal gas tube for leaks; repair if necessary. 3. Check the position of the internal gas tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the robotic system. 6. Press Reset and Auto.
Switch for inlet pressure is defective.	Contact a Perkin Elmer Service Engineer.

Robot Arm Not in Raised Position

Possible Causes	Solutions
The upper pressure tube at the robot arm has slipped off or has a leak.	Tighten the upper pressure tube.
The upper knurled screw in the center of the lifting head is loose.	Tighten the upper knurled screw.
The switchover valve is hung up.	Turn the robotic system off and on (several times if necessary).
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.
The robot arm throttle is not adjusted properly.	Contact a Perkin Elmer Service Engineer.

Robot Arm Not in Lowered Position

Possible Causes	Solutions
The lower pressure tube at the robot arm has slipped off or has a leak.	Tighten the lower pressure tube.
The lower knurled screw in the center of the robot arm is loose.	Tighten the lower knurled screw.
The switchover valve is hung up.	Turn the robotic system off and on (several times if necessary).
The robot arm throttle is not adjusted properly.	Contact a Perkin Elmer Service Engineer.

Sample Cover Could Not Be Closed

Possible Causes	Solutions
The switchover valve is hung up.	Turn the robotic system off and on (several times if necessary).
The lifting cylinder gas tube has a leak or has slipped off.	1. Turn off the robotic system, disconnect the power cord, and remove the rear cover. 2. Check the lifting cylinder pressure tube for leaks; repair if necessary. 3. Check the position of the lifting cylinder pressure tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the robotic system. 6. Press Reset and Auto.
The lifting cylinder is defective.	Contact a Perkin Elmer Service Engineer.
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.

Sample Cover Closed But Not Sealed

Possible Causes	Solutions
An object is caught between the sample holder and the sample holder cover.	Inspect the sample holder and remove the object as follows: **NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and the method. Enter 50°C in Go To Temp field and click on Go To Temp button. Once the sample temperature has reached 50°C, you can perform the steps below: 1. Press Reset and Auto. 2. Press Open Cover. 3. Remove the object. 4. Press Close Cover.

	5. Make sure H is displayed on robotic system display. 6. Restart the playlist.
The sample holder cover O ring is defective.	Inspect the sample holder cover O ring and replace it if necessary.
The purge gas inlet for the sample holder is open and no gas pressure can build up.	Connect the gas tube for the sample holder purge to the gas supply or interconnect the gas lines.
The lifting cylinder gas tube has a leak or has slipped off.	1. Turn off the robotic system and remove the rear cover. 2. Check the lifting cylinder pressure tube for leaks; repair if necessary. 3. Check the position of the lifting cylinder pressure tube; correct if necessary. 4. Replace the rear cover. 5. Turn on the power to the robotic system. 6. Press Reset and Auto.
The sample holder is out of alignment.	Contact a Perkin Elmer Service Engineer.
The pressure switch is defective.	Contact a Perkin Elmer Service Engineer.

Robot Arm in Wrong Position

Possible Causes	**Solution**
The coding disk/light barrier is defective.	Contact a Perkin Elmer Service Engineer.

Tray Not in Correct Position

Possible Causes	**Solutions**
The lever gear/sample tray is blocked; it will not reach the end position of any particular movement within 5 min.	Check the lever gear/sample tray movement for obstructions and, if necessary, remove the obstruction. **NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and the method. Enter 50°C in Go To Temp field and click on Go To Temp button. Once the sample temperature has reached 50°C you can perform the procedure above.
The coding disk/light barrier for the level gear/sample tray is defective.	Contact a Perkin Elmer Service Engineer.

Sample/Platinum Cover Could Not Be Found

Possible Causes	Solutions
The sample pan or platinum cover in the sample holder cannot be picked up (i.e., it is stuck) by the robot arm.	**NOTE:** If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and the method. Enter 50°C in Go To Temp field and click on Go To Temp button. Once the sample temperature has reached 50°C you can perform the steps below: 1. Press Reset and Auto. 2. Open the protective cover and loosen the pan or cover, leaving it in the sample holder. Close the protective cover. 3. Click on Close Cover to close the sample holder cover. 4. Restart the playlist.
The sample pan or platinum cover is deformed or the sample pan cover was crimped asymmetrically, causing the robot arm to lose the sample pan.	1. Press Reset and Auto. 2. Open the protective cover and verify that the sample pan is deformed, then replace it with a newly encapsulated sample. Close the protective cover. 3. Restart the playlist.
The location in the magazine is empty.	1. Press Reset and Auto. 2. Open the protective cover and verify that the sample position is empty, then place an encapsulated sample in the magazine. Close the protective cover. 3. Alternatively, click on the Autosampler Control button and in the dialog box enter the empty position in the Select Carousel Position field and click on Move Carousel to Location. The tray will rotate to place the magazine with the empty position so you can remove it through the magazine port and insert a sample pan. Return the magazine back into the tray. Click on Close to close the dialog box. 4. Restart the playlist.
The platinum lid is dropped when unloading a sample.	1. Press Reset and Auto. 2. Open the protective cover and replace the lid on the sample holder. Close the protective cover. 3. Restart the playlist.
The suction tip on the robot arm is defective.	Replace the suction tip.

Final Position of Autosampler Not Achieved

Possible Causes	Solutions
The lever gear/sample tray is blocked; it will not reach the end position of any particular movement within 5 min.	Check the lever gear/sample tray movement for obstructions and, if necessary, remove the obstruction. **NOTE**: If data collection is in progress in subambient mode, in order to avoid frosting you will have to stop the play list and a method. Enter 50°C in Go To Temp field and click on Go To Temp button. Once the sample temperature has reached 50°C you can perform the procedure above.
The coding disk/light barrier for the level gear/sample tray is defective.	Contact a Perkin Elmer Service Engineer.

Sample Number/Tray Position Out of Range

Possible Causes	Solution
In the sample changing procedure, you entered a number greater than 48 in the Load Sample step in the playlist or in the Select Carousel Location field in the Autosampler Control dialog box.	Reenter a number less than or equal to 48 in the appropriate field.

Sample Position of Magazine Occupied

Possible Causes	Solution
The robotic system has found something (e.g., a sample capsule) located in a magazine position that should be empty.	1. Press the Reset and Auto. 2. In the Select Carousel Location field, enter the number of tray position that is occupied. 3. Click on Move Carousel to Location. The tray will rotate so that the magazine with the specified position faces the magazine port. 4. Using the magazine-changing tool, remove the magazine and remove the unwanted object. Reinsert the magazine. 5. Click on the Close button in the Autosampler Control dialog box. 6. Restart the playlist.

Sample Cell/Reference Cell Already Occupied

Possible Causes	Solution
The robotic system has found a sample pan in the sample cell in the sampler holder when it should be empty.	1. Press Reset and Auto. 2. Click on the Autosampler Control button in the Control Panel. 3. Enter an empty sample tray position in the

	Select Carousel Location field. 4. Click on Move Carousel to Location to have the pan deposited in the empty position. 5. Click on the Close button in the dialog box. 6. Restart the playlist.

No Platinum Cover Available

Possible Causes	**Solutions**
A sample that has already been run is still in the sample holder.	1. Press Reset and Auto. 2. Click on the Autosampler Control button in the Control Panel. 3. Enter an empty sample tray position in the Select Carousel Location field. 4. Click on the Move Carousel to Location field to have the pan deposited in the empty position. 5. Click on the Close button in the dialog box. 6. Restart the playlist.
During a run, the platinum cover was not placed in its receptacle.	1. Press Reset and Auto. 2. Open the protective cover and use the suction tip tool to place the platinum cover in its receptacle. 3. Restart the playlist.

Syntax Error

Possible Causes	**Solutions**
Transmission error.	Press Reset and Auto.

Command Not Recognized

Possible Causes	**Solutions**
Transmission error.	Press Reset and Auto.

Carrier Detect Signal Dropped

Possible Causes	**Solutions**
Transmission error.	Check the RS-232 cable.

Maintenance

The DSC 7 Robotic System does not require any special maintenance except to be kept clean. Observe the following guidelines when cleaning:

- The exterior surfaces of the robotic system are resistant to diluted acids but **not** to concentrated acids.
- The painted surfaces and protective cover are **not** resistant to organic solvents.

- To clean surfaces under the protective cover (the working space), use only a cloth dampened with clear water to which a small amount of commercial household cleaning agent was added. Wipe these areas dry with a soft, dust-free cloth or tissue.
- Remove any dust that has accumulated in the folds of the robot arm bellow or in the magazine groove of the sample tray with a medium soft brush.

Parts Provided

The parts provided with the DSC 7 Robotic System are as follows:

Description	Part No.	Quantity
DSC 7 Robotic System	B013-7100	1
RS 232 Cable	B014-3264	1
Power supply cable, Schuko-type plug, 220 V	B001-9800	1
Power supply cable, U.S. standard	B001-3775	1
Black gas hose, 4-m long, includes connectors for pressure-reducing valve	B014-7780	1
Sample holder parts kit	B013-8721	1
Set of 12 magazines	B014-2511	1
Magazine changer	B013-9120	1
Molding tool, upper part, for Sealing Press	B013-8773	1
Molding tool, lower part, for Sealing Press	B013-9036	1
Dust cover	B014-26531	1
Hexagonal stud driver, 2 mm	B001-8041	1
Hexagonal stud driver, 3 mm	B001-8043	1
Hexagonal stud driver, 4 mm	B001-8044	1
Fuses, 1A Slow Blow	B001-8622	10
Fuses, 1.6A	B003-9629	10
Fuses, 2.5A Slow Blow	B003-9024	10
Fuses, 0.16A	B003-8065	10

Chapter 3
Pyris 6 DSC

Overview

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

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

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

Furnace and Sensor Design

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

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

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

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

Display

An LCD display on the front panel of the Pyris 6 DSC allows you to see instrument temperature and status. Direct readout of system status tells you when the instrument is in one of four modes: standby, heating, cooling, or isothermal. Display information is available in English, French, German, Italian, Japanese, and Spanish.

Cooling

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

Cooling with liquid nitrogen gas is simple, fast, and inexpensive. It requires only a transfer line and a low-pressure LN2 dewar — no extra accessories are needed. For cooling with liquids, many commercial circulating coolers are available that can be interfaced to the Pyris 6 DSC.

Modular Construction

The Pyris 6 DSC offers state-of-the-art modular construction containing very few parts. As a result, instrument reliability is very high and serviceability is fast and easy in the rare event that it is required. In addition, the analyzer uses on-board "Flash" EPROM technology that allows new firmware to be downloaded conveniently from the PC.

WARNING: **Do not attempt to service the Pyris 6 DSC. Service must be performed by a Perkin Elmer Service Representative.**

Other topics related to the Pyris 6 DSC are

- **Safety Precautions**
- **Operating Variables and Sample Handling**
- **Calibration**
- **Subambient Operation**
- **Maintenance**
- **Part Numbers**
- **Autosampler**

Safety Precautions

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

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

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

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

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

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

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

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

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

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

Operating Variables and Sample Handling

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

- **Sample Preparation**
- **Sample Pans**
- **Sample Encapsulation**
- **Sample Size**
- **Sample Atmosphere**
- **Temperature Range**
- **Scanning Rate**
- **Sample Loading**

Sample Preparation

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

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

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

The Pyris 6 DSC analyzes both solid and liquid samples. Solid samples can be in film, powder, crystal, or granular form. Proper sample preparation that maximizes the contact surface between the pan and the sample will reduce any imbalance of the sample in the preformed sample pan and will result in maximum peak sharpness and resolution.

To learn how to prepare samples, see the topics **Preparing a Solid Polymer Sample, Preparing a Liquid Sample,** and **Preparing a Powdered Sample** for encapsulation in a sample pan.

The prepared samples are encapsulated in sample pans. Your choice of sample pan depends on the nature of the sample and the temperature range of interest. A summary of sample pans and sealers available from Perkin Elmer is given in **Sample Pans and Sealers** below.

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

After the sample has been prepared and placed in a sample pan, it must be encapsulated. The method of sample encapsulation most widely used is crimping the sample pan cover in place. The normal crimping procedure yields a tightly but not hermetically sealed sample. For a detailed discussion of how to encapsulate samples properly, see **Sample Encapsulation**.

If you do not use a reference sample in the reference side of the Pyris 6 DSC with the AS6 autosampler when performing a run on a sample, you will notice that the baseline slopes. This slope generally does not affect the data quality, just the appearance of the curve. You can correct the slope using Change Slope in the Pyris software. Click on the **Change Slope** button on the Rescale Tools toolbar to access the Change Slope dialog box. Make sure to click on the Align Endpoints box.

Sample Pans and Sealers

After preparing the sample, it must be placed into a sample pan which will then be placed in the sample holder for analysis. The following types of sample pans are available for the Pyris 6 DSC:

Part No.	**Description**	**Quantity**
0219-0041	Standard aluminum sample pans and covers (for standard nonvolatile samples)	400 each
0219-0042	Standard gold sample pans and covers (for high-temperature nonvolatile samples, coal, and soil samples)	10 each
0319-0024	Standard platinum pans and covers (high-temperature nonvolatile samples that react with gold)	4 each
0319-0025	Standard graphite pans and covers (high-temperature nonvolatile samples that react with metal pans)	4 each
0319-0026	Standard copper pans and covers (oxidative stability testing of polymers and other specialized applications)	200 each
0219-0048	Standard sample pan crimper press (sealer for standard sample pans)	
B013-9005	Universal crimper press (with the aid of the proper sealing inserts, this press can seal the standard aluminum pans, O-ring-sealed large-volume stainless-steel pans, and the 20- µL volatile sample pans)	
B014-4637	Sealing insert for volatile sample pans (use with universal crimper press)	
B050-5340	Sealing insert for volatile sample pans (use with universal crimper press)	
B050-8921	Sealing insert for standard aluminum pans (use with universal crimper press)	
0219-0061	Volatile sample pan sealer	
0219-0062	Aluminum volatile sample pans and covers (used with standard volatile samples to pressures as high as 3 atm)	400 each
0219-0080	Gold volatile pans and covers (volatile samples that react with aluminum)	10 each

Part No.	Description	Quantity
0319-0021	Large-volume stainless-steel sealer and capsule kit (samples that are volatile in the temperature range of interest, such as dilute aqueous solutions and some curing reactions; pressure studies as high as 24 atm)	
0319-0218	Large volume stainless-steel sample capsules (same application as 0319-0021)	20 capsules
0990-8467	Quick press (requires spacer die, P/N 0319-1047) (sealer for large-volume stainless-steel capsules)	
B018-2864	High-pressure capsule sealing tool (sealer for stainless-steel, gold, or titanium high-pressure capsules)	
B018-2901	Stainless-steel high-pressure capsules (for use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials)	5 capsules, 20 copper seals
B018-2902	Gold-plated high-pressure capsules (see B018-2901)	5 capsules, 20 gold-plated copper seals
B018-2903	Titanium high-pressure capsules (see B018-2901)	5 capsules, 20 titanium seals
B018-2904	Replacement copper seals (see B018-2901)	10 each
B018-2905	Replacement gold-plated copper seals (see B018-2901)	10 each
B018-2906	Replacement titanium seals (see B018-2901)	10 each
B519-0180	Aluminum oxide pans and covers (oxidation of metals, inorganics, and minerals)	6 each

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

Standard Sample Pans

Standard sample pans come in three different types:

- **Aluminum Sample Pans**
- **Copper Sample Pans**
- **Gold Sample Pans**

In the standard sample pan, the sample is completely enclosed in a highly conductive capsule and distributed in a thin layer such that the internal resistance in the sample itself is very small. The sample pan material and the sample holder design have very small thermal resistance. This contributes to superior thermal conductivity and temperature control between the sample and the sample pan. Because of the high thermal conductivity of aluminum, copper, and gold, heat transfer is maximized. This means that the sample size can be minimized. Because of their high temperature capabilities, platinum and gold can be used to 725°C.

CAUTION: Never exceed 600°C when using aluminum sample pans. Since aluminum melts at 660°C, an aluminum sample pan will alloy with and destroy the sample holder.

The metal standards (indium, tin, lead, and zinc) must be encapsulated in aluminum pans because they will alloy with gold, copper, or platinum pans.

Aluminum Sample Pans (P/N 0219-0041)

Aluminum sample pans are used with nonvolatile solid samples such as plastics and polymers. They are used routinely for applications such as polymer melting, crystallization, glass transitions of polymers, thermoplastics, and thermosets. The pans are crimped but not hermetically sealed. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Gold Sample Pans (P/N 0219-0042)

Gold sample pans are used for any nonmetal, i.e., ceramics, coal, soils, or minerals. They can also be used for any material that reacts with aluminum or which must be heated over 600°C. These pans are crimped with the Standard Sample Pan Crimper Press and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

Copper Sample Pans (P/N 0319-0026)

Copper sample pans can be used for samples that have transitions that occur above the temperature range of the aluminum pans. Typical applications for the use of copper sample pans include antioxidant testing, thermoset and thermoplastic glass transition, and melting point determinations. These pans are crimped with the Standard Sample Pan Crimper Press (P/N 0219-0048) and may also be crimped using the Universal Crimper Press (P/N B013-9005) with the appropriate sealing insert.

See the topics on how to **Crimp the Sample Pan by Hand** or **Using the Standard Sample Pan Crimper** in the Pyris Multimedia Presentations Help.

Specialty Sample Pans

There are two specialty sample pans for use with a Pyris 6 DSC:

Graphite Sample Pans (P/N 0319-0025)

Graphite sample pans should be used in an inert atmosphere. They are used primarily with metals, but not for oxidation studies. They have extremely good heat transfer properties that yield sharp transition curves. A crimper is not required.

Platinum Sample Pans (P/N 0319-0024)

Use platinum sample pans and covers when gold and graphite pans and covers are not suitable. These pans are used to analyze mineral, soil, and ceramic samples. They are also used to allow a chemical reaction where platinum acts as a catalyst. Platinum sample pans are easy to clean and are reusable. A crimper is not required.

Volatile Sample Pans

Volatile sample pans are used with volatile solid or liquid samples which exert significant vapor pressure at the temperature of interest. They are available in both aluminum (P/N 0219-0062) and gold (P/N 0219-0080). The benefits of the volatile sample pan are as follows:

- They can withstand an internal pressure of 2 atm (30 psi).
- Aqueous solutions can be scanned up to and through 100°C to observe solute behavior.
- The heats of fusion of materials that sublime can be determined can be determined accurately.
- The effect of an enclosed atmosphere on thermal behavior of a sample can be observed.
- Capsules have an effective volume of 20 µL.
- Gold pans are available for samples requiring temperatures above 600°C.
- Covers with 50-µL-pinhole lids are available [Vapor Pressure Sample Pan Kit (P/N N519-0788)] for measurement of boiling points, heats of vaporization, and sublimation temperatures.

The major applications for this type of pan are purity analyses (i.e., pharmaceuticals, melting of lipids or liquid crystals) and phase transitions, heat of vaporization, and boiling points.

See the topic **Seal a Volatile Sample Pan Using a Volatile Sample Sealer** in the Pyris Multimedia Presentations Help.

Large-Volume O-Ring-Sealed Stainless-Steel Pans

Large-volume O-ring-sealed stainless-steel sample pans (P/N 0319-0218) contain the sample in a sealed environment throughout an experiment. The Viton O-ring allows formation of a seal which suppresses the vaporization of a solvent or contains a volatile reaction product, thereby eliminating the interfering effects of the heat of vaporization. The benefits of this type of sample pan are

- sealed capsules prevent any mass loss
- capsules can withstand an internal pressure of 24 atm
- high internal pressure capability allows water samples to be heated higher than 100°C
- capacity of 60 µL
- large sample capacity yields higher sensitivity
- operating range of –40°C to 300°C unless otherwise limited by sample vapor pressure

The large-volume O-ring-sealed stainless-steel sample pan is used in the study of aqueous biological solutions (such as protein in water) where the dilution requires large sample sizes and water vaporization must be suppressed. They are also used in epoxy and phenolic curing and vulcanization of rubber where the loss of volatiles can otherwise make interpretation difficult.

See the topic **Seal a Large-Volume O-Ring Stainless-Steel Sample Pan** in the Pyris Multimedia Presentations Help.

NOTE: A large-volume O-ring-sealed stainless-steel sample pan can be sealed with a **universal crimper press** with optional sealing insert (P/N B050-5340) or with a **quick press** with the quick press spacer (P/N 0319-1047).

Large-Volume Stainless-Steel Capsule Kit (P/N 0319-0021)

This kit contains the quick press (P/N 0990-8467), the quick press spacer die (P/N 0319-1047), guard ring inserts (1 pair) (P/N 0319-0236), and 20 pans, covers, and O-rings (P/N 0319-0218).

High-Pressure Capsules

For DSC runs where high pressure is expected in the capsule, we recommend that you use reusable high-pressure capsules. The reusable high-pressure capsules and sealer are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used. The capsules are safe to handle because of a bursting disk on the cover of the capsule that allows the sample to escape if the pressure should exceed 150 atm.

There are three high-pressure capsules available:

Stainless-Steel High-Pressure Capsules (P/N B018-2901)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number contains 5 capsules and 20 copper seals.

Gold-Plated High-Pressure Capsules (P/N B018-2902)

For use in DSC runs where high pressure is expected in the capsule, e.g., explosive materials. This part number consists of 5 capsules and 20 gold-plated copper seals.

Titanium High-Pressure Capsules (P/N B018-2903)

For use in DSC runs where high-pressure is expected in the capsule, e.g., explosive materials. Part number consists of 5 capsules and 20 titanium seals.

Some of the benefits of using the high-pressure capsule are

- permits suppression of the endothermic signal resulting from the volatilization of sample material or from the volatilization or decomposition of reaction by-products
- permits the study of explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used
- availability in stainless steel, gold-plated steel, and titanium
- can withstand an internal pressure of 150 atm maximum
- broad temperature range, subambient to 400°C
- reusable after sealing
- used in hazard testing

The high-pressure capsules are sealed with the high-pressure capsule sealing tool (P/N B018-2864).

See the topic **Use the High-Pressure Capsule Sealing Tool** in the Pyris Multimedia Presentations Help.

Robotic System Sample Pans

The robotic system sample pans are used to prepare sample materials for the robotic system for the Pyris 6 DSC. Pans are available in sealed or vented versions. These pans can also be used for nonrobotic systems. The Universal Crimper Press (P/N B013-9005) provides high-quality sample crimping for the robotic system sample pans.

Below is a list of robotic system pans available:

1-bar (100 kPa) maximum internal pressure

Part No.	Capacity	Thickness	Description
B014-3015	10 µL	0.1 mm	sealed aluminum pans (400)
B014-3016	30 µL	0.1 mm	sealed aluminum pans (400)
B014-3017	50 µL	0.1 mm	sealed aluminum pans (400)
B014-3018	30 µL	0.1 mm	vented aluminum pans (400)
B014-3019	50 µL	0.1 mm	vented aluminum pans (400)
B014-3024	50 µL	0.1 mm	sealed gold pans (10)

2 bar (200 kPa) maximum internal pressure

B014-3020	25 µL	0.15 mm	sealed aluminum pans (400)
B014-3021	50 µL	0.15 mm	sealed aluminum pans (400)

See the topic **Crimp the Robotic System Sample Pan Using the Universal Crimper Press** in the Pyris Multimedia Presentations Help.

Robotic System Sample Pan Covers

B014-3003	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (400)
B014-3004	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (400)
B014-3040	0.1 mm	aluminum cover used for all 0.1-mm-thick aluminum sample pan types (2000 covers)
B014-3050	0.15 mm	aluminum cover used for all 0.15-mm-thick aluminum sample pan types (800)

Starter Kits

B014-6340	Aluminum Sample Pan Starter Kit. Contains 50 of each type of robotic system aluminum sample pan and 350 sample pan covers.
B014-3030	Aluminum Sample Pan Starter Kit. Contains 400 of each type of robotic system aluminum sample pan and 2000 sample pan covers.

Pan and Cover Kit (2 atm, 200 kPa)

B016-9320	Aluminum sample pans and covers, 30-µL internal volume (400 pans and covers)
B016-9321	Aluminum sample pans and covers, 50-µL internal volume (400 pans and covers)
B700-1015	Aluminum sample pan cover with centered 2.5-mm hole. Designed for OIT measurements and for experiments that require an open sample pan for maximum exposure of the sample to the gaseous environment. Can be used with 30- or 50-µL Robotic System aluminum sample pan bottoms (400 lids)
B700-1014	Aluminum sample pan cover with centered 0.05-mm hole. Designed for vapor pressure and boiling point measurements. Can be used with 10-, 30-, or 50-µL Robotic System aluminum sample pans (400 lids)

Sample Encapsulation

The standard aluminum sample pans and covers provided with the DSC are suitable for most thermal analysis applications. The most common method of sample encapsulation is crimping the sample pan cover in place. The normal procedure yields a tightly, but not hermetically, sealed pan. When maximum contact of the sample with the atmosphere is necessary, perforate the sample pan cover with tweezers before crimping or use a cover punched from a 60/100 mesh screen.

For materials that emit volatile byproducts or need to be hermetically sealed, additional sample pans are available.

See the topic **How to Encapsulate Samples** in the Pyris Multimedia Presentations Help.

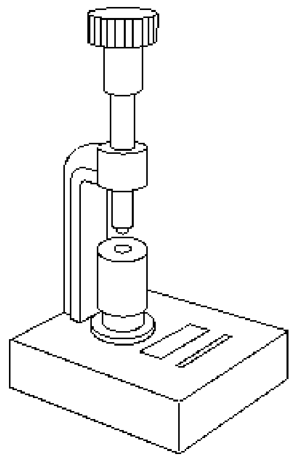
Crimpers

The following table lists the crimpers available for the sample pans used with a DSC for most thermal analysis applications.

Crimper	Use	Part No.
Standard sample pan crimper press	Crimps covers on Standard Sample Pans of aluminum, gold, and copper. Has a replaceable crimper head	0219-0048
Volatile sample sealer	Used with Volatile Sample Pans	0219-0061
High-pressure capsule sealing tool	Used with High-Pressure Capsules	B018-2864
Universal crimper press	Used with the Large-Volume O-Ring-Sealed Stainless-Steel Pans and Volatile Sample Pans. This is accomplished by using appropriate sealing insert.	B013-9005
Quick Press	Used with Large-Volume O-Ring-Sealed Stainless-Steel Pans	0990-8467

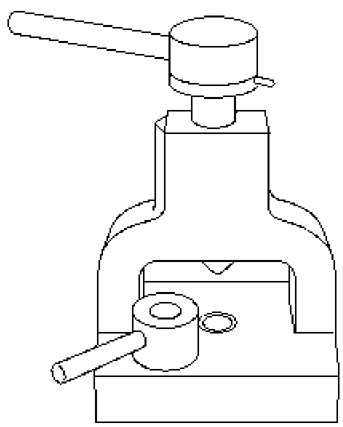
Standard Sample Pan Crimper Press

The Standard Sample Pan Crimper Press (P/N 0219-0048) is used to crimp covers on standard sample pans of aluminum, gold, and copper for use with the Pyris 6 DSC. Samples are crimped in pans but are not hermetically sealed. The design incorporates a replaceable crimper head.



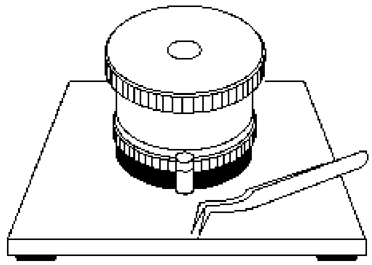
Volatile Sample Sealer

The Volatile Sample Sealer (P/N 0219-0061) is used to seal volatile sample pans. The pans and sealer are used with volatile solid or liquid samples that exert significant vapor pressure in the temperature range of interest. For example, aqueous solutions can be scanned up to and through 100°C to observe solute behavior. The heats of fusion of materials which sublime (e.g., camphor) can be determined accurately using these sample pans. Additionally, the effect of an enclosed atmosphere (e.g., water vapor evolved in dehydration below 100°C) on thermal behavior of a sample can be observed.



High-Pressure Capsule Sealing Tool

The high-pressure capsule sealing tool (P/N B018-2864) is used to seal the reusable high-pressure capsules. Those capsules are used to suppress the endothermic signal resulting from volatilization of sample material or from the volatilization or decomposition of reaction byproducts. High-pressure capsules can also be used to study explosive materials and can be used in any situation where the advantage of a self-generating atmosphere is to be used.



The capsules that can be sealed by this tool are

- **stainless-steel high-pressure capsules**
- **gold-plated stainless-steel high-pressure capsules**
- **titanium high-pressure capsules**

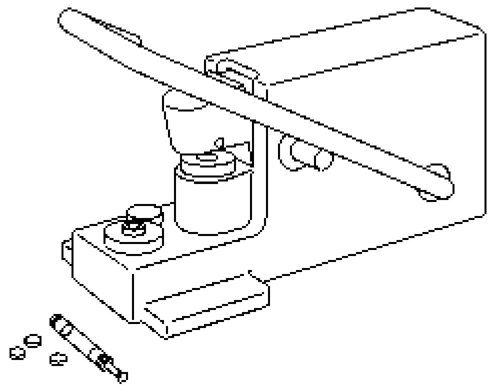
Vapor Pressure Sample Pan Kit (P/N N519-0788)

This kit includes 100 volatile aluminum pans and 100 pierced covers. A 50-µm-diameter hole is centered in the cover. Use this kit for more reproducible measurements of boiling points, heats of vaporization, and sublimation temperatures. Vapor pressure studies can be conducted when using these pans and covers with the high-pressure DSC.

Universal Crimper Press

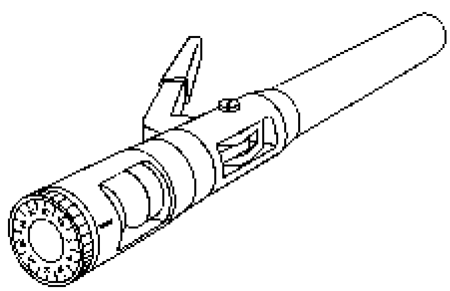
The Universal Crimper Press (P/N B013-9005) is used for sealing all of the robotic system sample pan types as well as most of the other Perkin Elmer DSC sample pans when used with the appropriate optional sealing insert. Other pans that can be crimped by a universal crimper press are listed in the table below:

Part No.	DSC Sample Pan Type
B050-8921	Standard Sample Pans
B050-5340	Large-Volume O-Ring Sealed Stainless-Steel Sample Pans
B014-4637	Volatile Sample Pans
B013-9033	Robotic System Sample Pans (included with the Universal Crimper Press)
B017-4929	12.5-L, 2-atm aluminum sample pans



Quick Press

The Quick Press (P/N 0990-8467) is used to crimp large-volume O-ring sealed stainless-steel sample pans (P/N 0319-0218) which are used with samples that vaporize or contain a volatile reaction product in the temperature range of interest. Application areas include the study of dilute aqueous solutions or curing reactions at temperatures below 300°C (e.g., phenolic or epoxy resins).



Sample Size

The quantity of sample that can be analyzed by the DSC is limited only by the volume of the sample pan that you use. However, the sample size in conjunction with the scanning rate and the sensitivity (Y range) will affect the quality of the results.

With the DSC, running a large sample at a fast scanning rate will improve the usable sensitivity. Running a small sample at a slow scanning rate will improve the resulting peak resolution. Most samples run on the DSC analyzer will be in the 0.5–30-mg range.

NOTE: Use small samples of 1 mg or less for decomposition studies and other analyses in which the sample may tend to contaminate the sample holders or in which the behavior of the sample has not previously been investigated. Also, use a flow through cover in such analyses.

Sample Atmosphere

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

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

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

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

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

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

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

Temperature Range

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

Scanning Rate

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

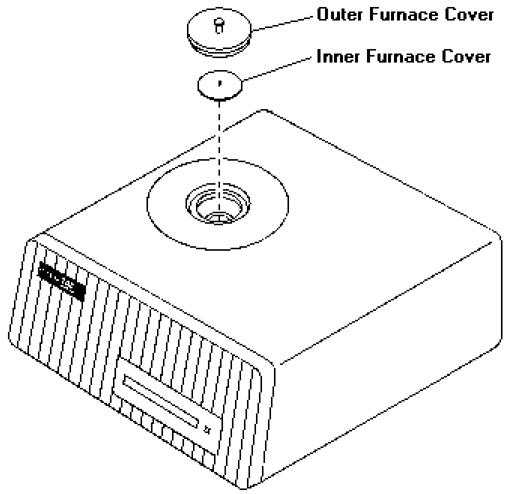
Generally, slower heating rates improve the peak resolution while faster heating rates improve the apparent sensitivity. Heating rates greater than 40°C/min are generally used for rapidly heating or cooling the Pyris 6 DSC to the beginning temperature of the next segment or to selected isothermal temperatures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

Sample Loading

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

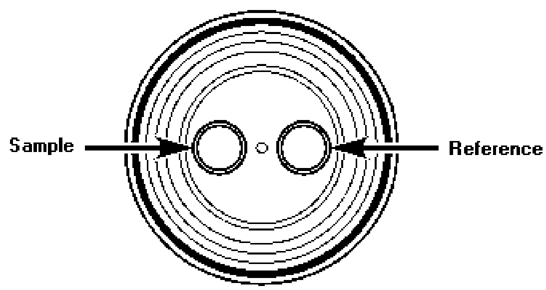
You load the sample into the Pyris 6 DSC as follows:

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



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

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



Calibration

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

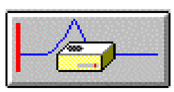
We recommend that the temperature calibration and heat flow calibration be checked using the precrimped samples of indium and zinc before obtaining data. [The Spares and Accessories Kit contains crimped indium/zinc calibrating reference materials (Part No. N519-0762) for use in temperature calibration of the Pyris 6 DSC.] We also recommend that you perform calibrations in the following order:

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

Baseline Correction

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

To correct the baseline, perform the following steps:

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

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

Temperature Calibration

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

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

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

To calibrate temperature of the Pyris 6 DSC:

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

After you have done these steps, perform the following three steps to complete the temperature calibration of the Pyris 6 DSC:

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

Perform Indium and Zinc Runs

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

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

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

Perform a Peak Area with Onset Calculation

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

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

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

Perform a Temperature Calibration

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

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

Heat Flow Calibration

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

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

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

The measured ΔH value for indium is used to calibrate the heat flow of the Pyris 6 DSC. If you just performed the temperature calibration on a data file, the data file should still be displayed. To perform the heat flow calibration, perform the following steps:

1. With either the Instrument Viewer or the Method Editor as the active window, select **Calibrate** from the View menu. The Calibrate dialog box is displayed.
2. Select the Heat Flow tab.
3. Enter the name of the reference material you used.
4. Enter the expected ΔH for the reference material used (e.g., 28.45 for indium).
5. Enter the measured ΔH result.
6. Enter the weight of the reference material used.
7. Enter the calibration method used.
8. Select the **Save and Apply** button in the Calibration window to send the new calibration value to the analyzer and save the current calibration file.
9. Select **Close** to close the Calibration window and begin using the new calibration values.

Subambient Operation

The Pyris 6 DSC can be operated in the subambient mode. The temperature range of subambient mode is –120°C to 200°C. Cold gaseous nitrogen serves as the coolant and nitrogen is used as the purge gas to the sample area. It is also possible to use helium as a purge gas. There are two Pyris 6 DSC Subambient Kits available:

- Pyris 6 DSC Subambient Kit with Dewar (P/N N520-0032) includes the fittings, tubing, insulation, regulator, and liquid nitrogen dewar.
- Pyris 6 DSC Subambient Kit without Dewar (P/N N520-0033) includes the fittings, tubing, and insulation, but does not contain the regulator or liquid nitrogen dewar.

WARNING: **If you supply your own dewar and regulator, be sure that they meet the following requirements:**

The liquid nitrogen dewar (or tank) must have an outlet that provides a constant flow of cold *gaseous* nitrogen, not liquid nitrogen.

You must be able to maintain the dewar at constant pressure of 6.9 – 69 kPa (1 – 10 psi).

Pressurization may be accomplished via an external gas pressure regulator or by internal means (i.e., heating devices).

Adequate safety precautions, such as rupture disks, must be included in the dewar.

An air regulator that can handle up to 345 kPa (50 psi) and has a pressure gauge with a range of 0 – 414 kPa (0 – 60 psi) must be used to pressurize the dewar.

Subambient operation of the Pyris 6 DSC is covered in the following topics:

- **Precautions for Subambient Operation**
- **Parts Recommended for Subambient Operation**
- **Operating the Pyris 6 DSC in Subambient Temperature Range**
- **Parts Provided in Subambient Kits**
- **Installing the Pyris 6 DSC Subambient Kit**

Precautions for Subambient Operation

Performance of the Pyris 6 DSC in the subambient mode should be similar to that in ambient mode with respect to noise, baseline linearity, baseline repeatability, resolution, etc. To achieve this level of performance while using cold gaseous nitrogen as a coolant, the following requirements must be met:

- The nitrogen purge gas must be exceptionally dry [use of a filter dryer accessory is recommended]. If the purge gas is not dry, condensation may occur on the walls of the furnace and affect baseline performance.
- The use of cold gaseous nitrogen as a coolant will require recalibration of both temperature and calorimetric sensitivity which will be affected by a few degrees and a few percent, respectively.
- Always use extreme caution when working with liquid nitrogen. It can cause severe burns. Protective gloves must be worn when touching any metal surface that has been in contact with or in the vicinity of liquid nitrogen.
- Use protective gloves when handling samples and when opening the furnace since these areas get extremely cold.
- Always use tweezers when lifting off the outer and inner furnace covers since they may be extremely cold.

- Use of cold gaseous nitrogen as a coolant requires that the instrument be placed in a well-ventilated area.
- Always check that the temperature of the analyzer is at least at 30°C before removing the outer and inner furnace covers.
- Always leave the Pyris 6 DSC on overnight at a furnace temperature between 50°C and 100°C. This will help prevent moisture buildup in the furnace.
- Make sure the pressure of the liquid nitrogen tank is between 6.9 and 20.7 kPa (1 and 3 psi) since pressures above 20.7 kPa (3 psi) can cause spillover of liquid nitrogen into the heat exchanger of the analyzer. If spillover occurs, it is possible that there will be excess noise in your data.

Parts Recommended for Subambient Operation

The following parts are used to connect the Pyris 6 DSC to a liquid nitrogen tank. We suggest the suitable parts for setting up and operating at subambient levels. A kit containing all the necessary hardware to connect the analyzer to a liquid nitrogen tank is available from Perkin Elmer. Contact your local Sales Representative.

Contact your local specialty gas supplier or local Perkin Elmer Representative for recommendations on the type of liquid nitrogen tank and transfer line suitable for your laboratory. The tank needs to provide a pressure from 6.9 to 20.7 kPa (1 – 3 psi).

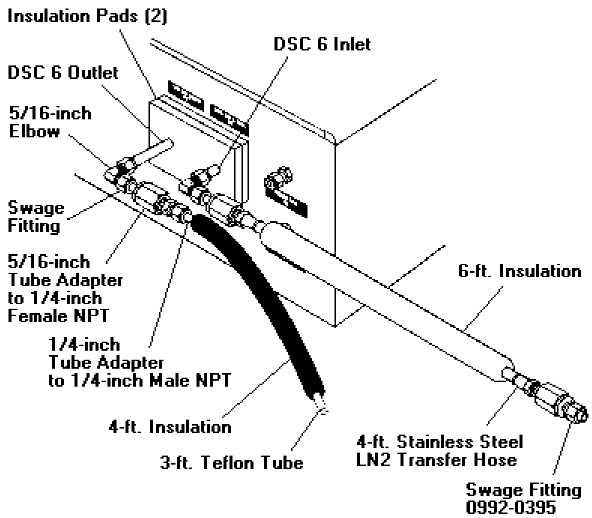
We recommend that you use the following items to set up the analyzer for subambient operation:

A **filter dryer accessory** with **all** purge gases to eliminate moisture from the lines.

An insulated liquid nitrogen transfer line of adequate length to connect the liquid nitrogen dewar to the analyzer.

The fittings below are brass Swagelok fittings and are suggested as one way of setting up the analyzer for subambient operation:

1. Two 5/16-in. elbows
2. Two 5/16-in. tube adapters to 1/4-in. male NPT connectors
3. Two 5/16-in. tube adapters to 1/4-in. female NPT connectors
4. Insulation for the connections
5. Teflon tubing



NOTE: Once the Swagelok fittings are in place, you cannot remove them to switch back to ambient operation. You can easily switch from one configuration to the other by removing or adding the 1/4-in. NPT to 5/16-in. tube adapter to the end of the connections for the Cooling Liquid In.

Operating the Pyris 6 DSC in Subambient Temperature Range

NOTE: Use protective gloves when turning the valves on the pressurized tank of liquid nitrogen.

WARNING: **When starting the liquid nitrogen cooling system, turn on the house or tank supply of dry gas before opening the "Liquid In" valve on the dewar. When shutting down the system, close the "Liquid In" valve before shutting off the house or tank supply of dry gas. This will help to prevent liquid nitrogen from backing up into the gas line to the pressure regulator.**

1. Perform a temperature calibration after the Pyris 6 DSC has equilibrated. Suitable subambient calibration reference materials are listed in **Calibration Reference Materials for Subambient Operation**. Volatile sample pans (P/N 0219-0062) should be used for all liquids and organic solids to prevent vaporization and condensation in the furnace.

   CAUTION: Always load samples into the Pyris 6 DSC furnace at room temperature (30°C) or higher. Never open the furnace covers when the furnace is below room temperature since this can cause condensation in the furnace.

2. Load your sample and reference material into the Pyris 6 DSC.
3. Perform your experiment, observing the following precautions:

- When operating at subambient temperatures, it is recommended that the instrument be left on at all times and the temperature of the Pyris 6 DSC furnace be left at 50°C overnight. Leave the sample area purge gas flowing.
- The samples used in calibration must be of 99.9% minimum purity as even small levels of impurity can grossly influence the results.
- A small amount of frost buildup may be noted around openings of the inner and outer furnace covers. This is normal.

NOTES: The materials listed in Calibration Reference Materials, if used for calibration, must be of 99.9% minimum purity as even small levels of impurity can affect the temperature and/or energy of the transition.

1. The above samples must be encapsulated in volatile sample pans.

2. If the peaks are not sharp, the sample may be impure, and the temperatures you measure may not be correct. Use a higher purity sample, or check the purity of your sample by an alternate technique such as gas chromatography.

Parts Provided in Pyris 6 DSC Subambient Kits

The parts listed below are found in P/N N520-0034 which is contained in both the Pyris 6 DSC Subambient Kit with Dewar (P/N N520-0032) and the Pyris 6 DSC Subambient Kit without Dewar (P/N N520-0033). The Subambient Kit with Dewar contains additional parts which are listed in Parts Provided in Pyris 6 DSC Subambient Kit with Dewar.

Part No.	Description	Quantity
0992-0372	Fitting – 5/16-in. tube adapter to 1/4-in. male NPT	1
0992-0371	Fitting – 5/16-in. tube adapter to 1/4-in. female NPT	3
0992-0146	Fitting – 5/16-in. tube elbow	2
N519-0081	Hose assembly – DSC 6 LN2 transfer line	1
N519-2149	Insulation pads for the DSC 6	2
0992-0044	SS hose clamp – 1 7/8 in. / 1/2 in.	2
0992-0396	Fitting – 1/4-in. male NPT connector to 5/16-in. TBG	1
0250-6240	Tubing (insulation) – ½-in. i.d. x 3/8-in. wall	4 ft
0250-8059	Teflon tubing – 1/4-in. i.d. x 5/16-in. o.d.	4 ft
0993-6077	Installation Guide	1
0990-8216	Teflon pipe tape – 3/8-in. x 0.003-in. thick	1
0250-7948	Tubing (insulation) – 7/8-in. i.d.	6 ft

Parts Provided in Pyris 6 DSC Subambient Kit with Dewar

The parts listed below are additional parts found in the Pyris 6 DSC Subambient Kit with Dewar (P/N N520-0032).

Part No.	Description	Quantity
N520-1020	Dewar DSC 6 LN2	1

The following parts are for the regulator:

0990-7085	Air Regulator, 345 kPa (50 psi)	1
0990-7113	Pressure Gauge, 0 – 414 kPa (0 – 60 psi)	1
0992-0237	Nipple (brass hex.) 1/8-in. NPT 3-in. long	1
0990-3666	Plug (brass pipe) 1/8-in. 27 MPT	1
0990-3428	Fitting connector (brass) 1/8-in. male NPT to 1/4-in. TBG	1
0250-6515	Tygon tube (black round) 1/4-in. o.d. x 1/8-in. i.d.	10 ft
0990-3199	Nut (brass) 1/4-in. TBG	2
0990-3103	Ferrule back (brass) 1/4-in. TBG	2
0990-3104	Ferrule front (brass) 1/4-in. TBG	2
0990-3094	Insert (A1) 1/4-in. o.d. x 1/8-in. i.d. TBG	2
0992-0397	Reducing Union 1/2-in. TBG to 1/4-in. TBG	1
0990-8235	Black electrical tape – 3/4 in. wide	1

Installing the Pyris 6 DSC Subambient Kit

Once you have checked all of the parts in your Pyris 6 DSC Subambient Kit with Dewar or without Dewar, you are ready to install your subambient kit. The procedure outlined in the following topics is the same for both subambient kits.

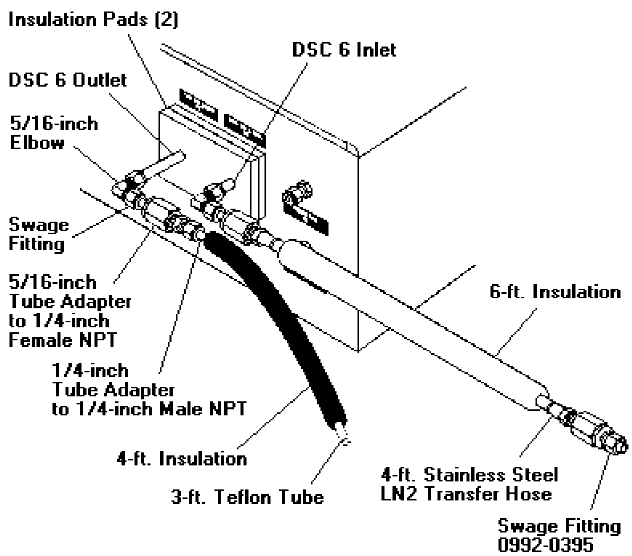
- **Preparing the Pyris 6 DSC for Subambient Operation**
- **Assembling the Regulator**
- **Connecting the Pyris 6 DSC to the Dewar**

Preparing the Pyris 6 DSC for Subambient Operation

NOTE: The steps to prepare the Pyris 6 DSC for ambient operation are in the section **Preparing the Pyris 6 DSC for Ambient Operation.**

To help in the preparation of the Pyris 6 DSC for subambient operation by performing the steps below, study the figure below showing the fittings attached to the back of the analyzer:

Fittings Attached to Back of Pyris 6 DSC for Subambient Operation



CAUTION: It is extremely important that you make sure that all water or other liquid coolant is removed from the heat exchanger before you connect the cold gaseous nitrogen to the analyzer.

Before attaching the Swagelok fittings, attach an air line or the purge gas line to the Cooling Liquid In connector and blow out all water or other liquid coolant used to operate the analyzer in the ambient mode from the heat exchanger.

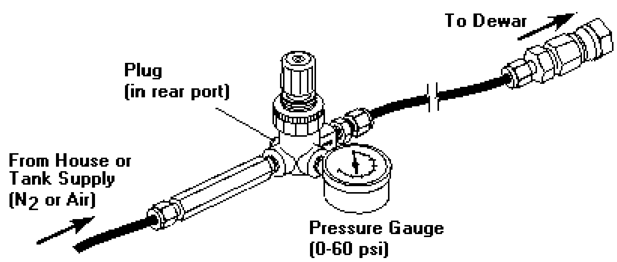
1. Place both insulation pads (P/N N519-2149) over the tubes labeled Cooling Liquid Out and Cooling Liquid In so that they are flush with the back of the analyzer.
2. Swage one of the 5/16-in. elbows (P/N 0992-0146) to the tube labeled Cooling Liquid Out.
3. Swage the other 5/16-in. elbow (P/N 0992-0146) to the tube labeled Cooling Liquid In.
4. Swage a 5/16-in. tube to 1/4-in. female NPT adapter (P/N 0992-0371) to the elbow connections on both cooling tubes.
5. Wrap Teflon tape (P/N 0990-8216) over the threads of the liquid nitrogen transfer line (P/N N519-0081). Connect the transfer line to the 1/4-in. female NPT adapter connected to the tube labeled Cooling Liquid In.
6. Wrap Teflon tape on the threads of the 1/4-in. tube to 1/4-in. male NPT adapter (P/N 0992-0372). Connect it to the female fitting on the end of the tube labeled Cooling Liquid Out.
7. Attach the 4-ft long Teflon tubing (P/N 0250-8059) to the Cooling Liquid Out tube fitting so that the cold gaseous nitrogen exiting the analyzer does not empty on to the benchtop. (If necessary, extend the length of this line so that it vents the N2 gas into a suitable fume hood or other safety container.)
8. Swage the last 5/16-in. tube to 1/4-in. female NPT adapter (P/N 0992-0371) to the other end of the LN2 transfer line.
9. Swage the 1/4-in. male NPT to 5/16-in. tube fitting (P/N 0992-0396) on to the adapter placed at the end of the transfer line in step 8. (This will connect to the dewar.)

10. Place the 4-ft-long insulation tubing (P/N 0250-6240) over the Teflon tube and hose clamp (on Cooling Liquid Out) and work it all the way to the DSC 6 insulation pads. The Swagelok fittings and elbow should be covered with insulation. If necessary, use electrical tape (P/N 0990-8235) to close the ends of the insulation for a tight fit.
11. Place the 6-ft-long black insulation tube (P/N 0250-7948) over the transfer line and work it all the way to the DSC 6 insulation pads. The Swagelok fittings and elbow should be covered with the insulation. If necessary, use electrical tape to close the ends of the insulation for a tight fit.

The next step in installing the Pyris 6 DSC Subambient Kit is **Assembling the Regulator**.

> **NOTE:** Once the Swagelok fittings are in place, you cannot remove them to switch back to ambient operation. You can easily switch from one configuration to the other by simply removing or adding the 1/4-in. NPT to 5/16-in. tube adapter to the end of the connections for the Cooling Liquid In.

Assembling the Regulator



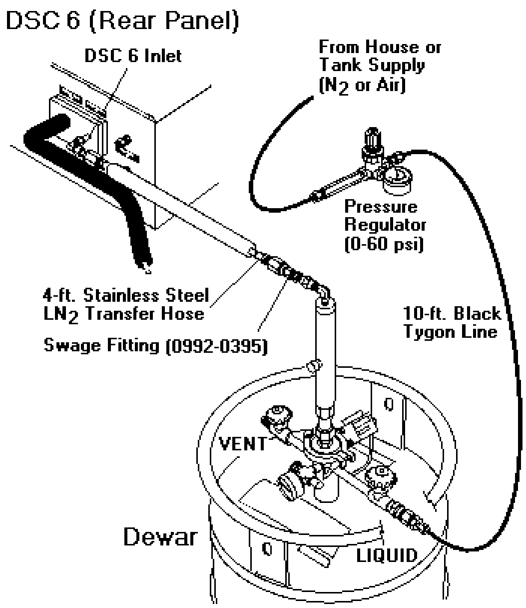
1. Remove the regulator and retaining ring (P/N 0990-7085) and plug (P/N 0990-3666) from the packaging.
2. Place the retaining ring over the top portion of the regulator (i.e., push/pull knob) and screw it onto the threads.

   The regulator has four openings. It also has an arrow stamped on one side of it. This arrow indicates the direction of the flow.
3. Position the regulator so that the arrow is facing you. The arrow should be stamped on the opening to your right (i.e., at the 3 o'clock position).
4. Wrap the threads of one of the plugs with Teflon tape (P/N 0990-8216) and then screw the plug (P/N 0990-3666) into the end of the regulator that is at the 12 o'clock position.
5. Wrap the threads of the pressure gauge (P/N 0990-7113) with Teflon tape and then screw the gauge into the regulator so that it is directly opposite the plug (i.e., the 6 o'clock position).
6. Wrap the threads of the nipple (P/N 0992-0237) with Teflon tape and then screw the nipple into the opening so that is to the left of the gauge (i.e., the 9 o'clock position).
7. Wrap the threads of the 1/8-in. male NPT to 1/4-in. tube fitting (P/N 0990-3428) with Teflon tape and then screw the fitting to the remaining opening of the regulator. (This should be the opening that has the arrow stamped on it.)
8. Remove the 1/4-in. swage nut from the fitting used in step 7. Retain the front and back ferrules inside the swage nut.

9. Place the 1/4-in. nut and front and back ferrules over one end of the black Tygon tubing (P/N 0250-6515).
10. Place an insert (P/N 0990-3094 from N520-0032) into the black Tygon tubing and swage the tubing to the 1/8-in. male NPT to 1/4-in. tube fitting installed in step 7.
11. Swage a 1/4-in. nut with ferrules (P/N 0990-3199, 0990-3103, 0990-3104 from N520-0032) and insert (P/N 0990-3094) using the 1/2-in. to 1/4-in. reducing union (P/N 0992-0397 from N520-0032) to the opposite end of the black Tygon tubing.
12. Remove the swaged tubing from the 1/2-in. reducing union and put the tubing aside.
13. Place the 1/2-in. nut and rear and front ferrules from the reducing union over the stainless steel tube that extends from the dewar (labeled LIQUID).
14. Place the reducing union inside of the 1/2-in. nut installed in step 13, making sure that it is all the way inside the nut. Swage the reducing union to the stainless steel tube.
15. Connect the swaged black Tygon tubing from the regulator to the 1/4-in. end of the reducing union on the dewar.

The regulator is now assembled and is attached to the dewar. Now you can connect the Pyris 6 DSC to the dewar.

Connecting the Pyris 6 DSC to the Dewar



First, the pickup line must be installed in the dewar.

1. Insert the tube removed from the dewar box into the top of the tank and tighten the nut to secure it to the tank.
2. Wrap Teflon sealant tape on the Swagelok fitting (P/N 0992-3095).
3. Place the fitting on the end of the pickup line and tighten.
4. Remove the nut and ferrules off of the end of the pickup line and put the nut and ferrules on the end of the transfer line.

5. Swage the end of the transfer line to the pickup line.

Now, the final connections are made:

1. Connect the inlet side of the regulator to either the house or tank supply of nitrogen or air.
2. Pull the knob of the regulator out and set the pressure on the regulator (by turning the knob) until you have a constant flow of **cold gaseous nitrogen** exiting the exhaust line of the dewar at a pressure between 6.9 and 69 kPa (1 – 10 psi).
3. Press the locking knob down once the pressure is set.

This concludes the installation procedure for the Pyris 6 DSC Subambient Kits.

Once you have connected the Pyris 6 DSC to the cooling supply, you will need to **connect the purge gas supply**, if not already connected.

Preparing the Pyris 6 DSC for Ambient Operation

You can set up the Pyris 6 DSC for ambient operation two different ways, depending on whether you intend to use the analyzer in subambient mode in the future.

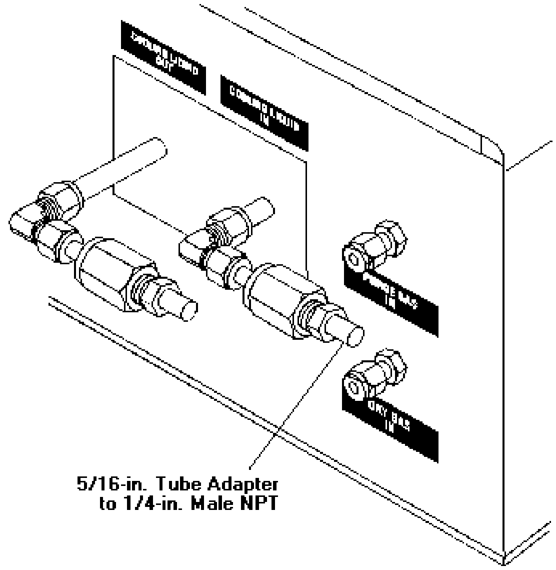
To set up the Pyris 6 DSC for **ambient operation only**, see the full installation instructions given in the online Installation Help.

1. The cooling inlet and outlet are located at the rear of the analyzer and are marked **Cooling Liquid In** and **Cooling Liquid Out**. In order to connect the cooling supply to the Pyris 6 DSC, connect a suitable tubing to the inlet and outlet, e.g., the Tygon tubing (P/N 0250-6519) supplied with the analyzer.
2. Attach the stainless steel hose clamps (P/N 0992-0044) around the tubing and the inlet and outlet tubes.
3. If using tap water, connect the inlet to the tap and the outlet to a suitable drain. If using a water circulator, connect the inlet of the Pyris 6 DSC to the outlet of the circulator, and the outlet of the Pyris 6 DSC to the inlet of the circulator.

CAUTION: The heat exchanger of the Pyris 6 DSC will NOTaccept pressure. Therefore, flow regulation should be done before the liquid enters the Pyris 6 DSC.

CAUTION: It is essential to maintain a very constant flow rate of approximately 10 – 30 L/h. The liquid should be free from air bubbles. Changes in the liquid flow rate or the presence of air bubbles will negatively influence the quality of the measurement signal.

To convert the Pyris 6 DSC **from subambient operation to ambient operation,** you first have to setup the Pyris 6 DSC for subambient operation. Once the Swagelok fittings for subambient operation are attached to the Pyris 6 DSC, you cannot remove them. To convert from subambient to ambient, add the 1/4-in. NPT to 5/16-in. tube adapter to the end of the connections for the Cooling Liquid In.



After you have connected the Pyris 6 DSC to the cooling supply for ambient operation, you will need to **connect the purge gas supply**.

Connect Purge Gas Supply to the Pyris 6 DSC

After you have connected the cooling supply to the Pyris 6 DSC for either ambient or subambient operation, you need to connect your purge gas supply and dry gas supply, if needed. The gas connections of the Pyris 6 DSC are suited for 1/8-in. Teflon or stainless steel tubing. The connections are the standard Swagelok type. The purge gas pressure should be approximately 1 atm, and the purge rate should be controlled by a needle valve and a flow meter. The procedure for connecting the purge gas supply consists of the following steps:

Connect the Purge Gas Line to the Gas Supply

Filter Dryer Installation

Thermal Analysis Gas Station (TAGS) Installation

Connect the Purge Gas and Dry Gas Lines to the Pyris 6 DSC

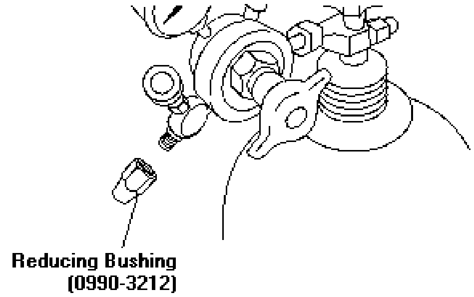
WARNING: **When making connections, first turn the fitting until it is fingertight, then turn it an additional 1/2 to 3/4 turn with a wrench. Use a second wrench to keep the connection at the Pyris 6 DSC stationary. Turn preswaged fittings 1/8 turn past fingertight. DO NOT OVERTIGHTEN.**

Connect the Purge Gas Line to the Gas Supply

Perform the following procedure to make the connections at your purge gas supply cylinder or cylinders if you intend to use a **Thermal Analysis Gas Station (TAGS)**:

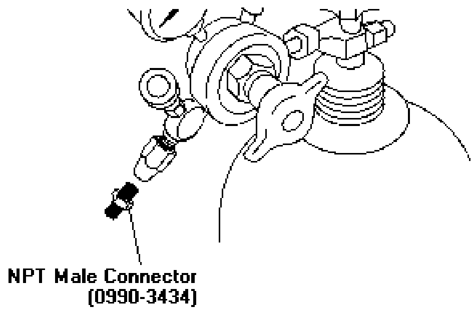
NOTE: The gas regulators, bushings, and connectors described in this topic must be supplied by the user. These items are **not** included with the Pyris 6 DSC.

1. Wrap the threads of the shutoff valve fitting with Teflon tape, going around twice. Be sure to start wrapping one or two threads in from the end of the fitting so that the full width of the tape is on the threads.
2. Connect a reducing bushing to the taped shutoff valve fitting.

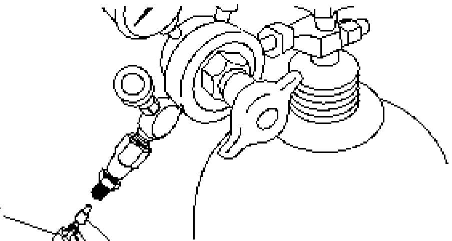


3. Remove the nuts from the 1/8-in. NPT male connector. Wrap the threads of the connector that goes into the bushing (installed in step 2) with Teflon tape, as described in step 1.

   Do not wrap the threads of the 1/8-in. side of the male connector.
4. Connect the taped end of the 1/8-in. NPT male connector to the reducing bushing.



5. If you are installing a filter dryer in the purge gas line, skip the remainder of this topic and go to **Filter Dryer Installation**.
6. Place a 1/8-in. nut and rear and front ferrules over one end of the 1/8-in. Teflon tubing provided.
7. Connect the 1/8-in. Teflon tubing, nut, and ferrules to the end of the male connector:



If you are going to use a TAGS, prepare each supply tank as described above. Then c**onnect the purge gas tanks to the TAGS**.

If you are not using a TAGS, you are now ready to **connect the purge gas and dry gas lines to the Pyris 6 DSC**.

Filter Dryer Installation

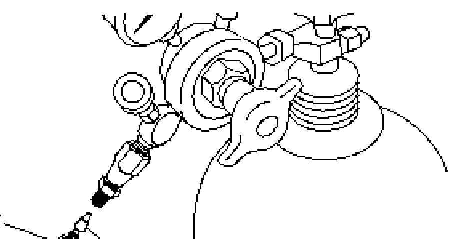
If you are going to use a Filter Dryer Accessory (P/N 0319-0039), install it in the purge gas line as described in the following procedure:

NOTE: The Filter Dryer Accessory is optional and may be purchased from Perkin Elmer. Contact your local Perkin Elmer Sales Representative.

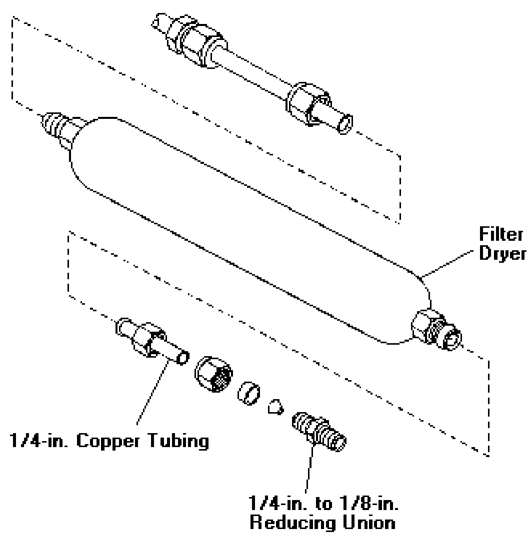
If you have a Drierite Compressed Air Filter Drier (P/N 0992-3454), its installation instructions follow these instructions.

NOTE: If you are going to use a TAGS, you must install a filter dryer for each of the purge gases you will attach to the TAGS. The filter dryer is installed between the gas tank and the TAGS.

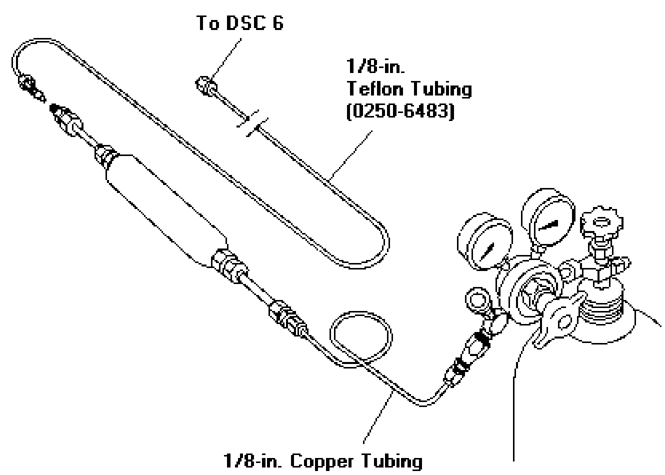
1. Connect one end of the 1/8-in. copper tubing supplied with the Filter Dryer Accessory to the NPT male connector which was installed on the regulator shutoff valve.



2. Remove the outer plugs from the ends of the filter dryer and discard the copper caps covering the filter dryer orifices.
3. Remove the nuts from the ends of the filter dryer. Place a nut over the flared end of each 4-in.-long 1/4-in. copper tubing (supplied with the Filter Dryer Accessory).
4. Connect the flared end of each piece of 1/4-in. copper tubing to the end fittings of the filter dryer. Uses two wrenches to tighten the connections.
5. Connect the 1/4-in. to 1/8-in. reducing unions (supplied with the Filter Dryer Accessory) to the free ends of the 1/4-in. copper tubing on both ends of the filter dryer using the 1/4-in. nuts and ferrules provided. Use two wrenches to tighten the connections: one wrench to hold the nut on the tubing and the second wrench to tighten the reducing union.



6. Connect the 1/8-in. Teflon tubing to one reducing union using the 1/8-in. nut and ferrules provided. This tubing will attach to the Pyris 6 DSC or to the TAGS.
7. Connect the 1/8-in. copper tubing, previously connected to the gas supply, to the remaining union on the filter dryer using the 1/8-in. nut and ferrules provided.



Now you can connect the purge gas line to the Pyris 6 DSC as described in **Connect the Purge Gas and Dry Gas Lines to the Pyris 6 DSC**.

If you are going to use a TAGS, then after installing a filter dryer for each of the purge gases you need, connect the purge gas line to the TAGS as described in **Thermal Analysis Gas Station (TAGS) Installation**.

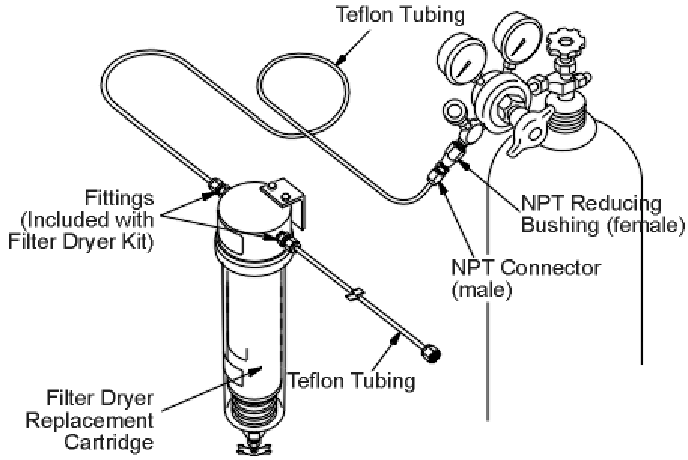
Drierite Filter Dryer Installation

If you are going to install the Drierite Compressed Air Drier Model 207 (P/N N537-0103), follow the instructions below.

WARNING: **Failure or improper selections or improper use of this product can cause death, personal injury, and property damage. Maximum working pressure is 125 psig.**

Read all warnings and safety precautions before installing this dryer. Install the dryer in an upright position as near to the point of use as practical.

1. Mount the bracket that comes with the dryer (attached) in the desired location.
2. Attach the aluminum head to the bracket with ¼-20 bolts provided.
3. With the nut and ferrules from the 1/8-in. NPT male connector that you installed on the gas supply, swage the end of a piece of 1/8.-in. Teflon tubing.
4. Connect the 1/8-in. Teflon tubing with nut to the 1/8-in. NPT male connector installed on the regulator shutoff valve on the gas cylinder.
5. With the nut and ferrules from the ¼-in. NPT to 1/8-in. tubing male connector (P/N 0990-3087) that is provided with the dryer, swage the other end of the Teflon tubing that is attached to the gas supply.
6. Wrap Teflon tape around the ¼-in. end of the male connector.
7. Connect the taped end of the connector to the IN connector on the dryer head.
8. Connect the purge gas supply to the inlet side of the aluminum head by connecting the 1/8-in. Teflon tubing to the IN connector.
9. Wrap Teflon tape around the threads of the male end of the other ¼-in. NPT to 1/8-in. tubing connector provided with the filter dryer.
10. Remove the 1/8-in. nut and ferrules from the connector and connect them to the end of another piece of Teflon tubing.
11. Connect the taped end of the connector to the OUT connector on the dryer head.
12. Connect the 1/8-in. Teflon tubing to the OUT connector on the dryer.



You can now connect the purge gas line to the Pyris 6 DSC as described in **Connect the Purge Gas and Dry Gas Lines to the Pyris 6 DSC**.

If you are going to use a TAGS, then after installing a filter dryer for each of the purge gases you need, connect the purge gas line to the TAGS as described in **Connecting a TAGS to a Pyris 6 DSC**.

Connecting a TAGS to a Pyris 6 DSC

The Thermal Analysis Gas Station allows you to use up to four gases for the purge gas. You can switch gases in the gas program of the method. This topic discusses how to connect the gas lines to the TAGS and Pyris 6 DSC.

Preparing the Laboratory for TAGS

The TAGS will accept the same laboratory conditions suited for the Pyris 6 DSC. Some precautions are

- Do not place the TAGS in direct sunlight or close to heating and cooling units.
- The temperature of the area should be between 10°C and 35°C.
- Relative humidity should be between 20% and 80% and noncondensing.
- The TAGS requires little bench space. The electrical power consumption is only 8 VA max. Therefore, it can be connected directly to the instrument's power line. The supply must be smooth, clean, and free of transient voltages over 40 V. The operating frequency is 50 – 60 Hz.

Unpacking the TAGS

The TAGS is shipped in one box and is surrounded by foam on all sides. To unpack the TAGS, follow the steps below:

1. Remove the power cable, the serial cables, and the manual from the box.
2. Remove the foam insert from the box.
3. Remove the upper foam piece.
4. Remove the TAGS from the box.
5. Remove all remaining packing material.

Installing the TAGS

Setting the Correct Voltage

Upon delivery, the TAGS is set at a voltage of 220/240 V. Changing the input voltage should be done at the AC inlet plug.

1. Remove the fuse holder with a small screwdriver by gently prying it out of the compartment. Once the holder is out far enough to grasp, slide the fuse holder out of the slot.
2. Rotate the fuse holder 180°.
3. Remove the fuse from the 220-V position (100 mA Slow Blow) and insert a 200 mA Slow Blow fuse for 110 V (it goes into the right side when "110 – 120 V" is in the upright position.
4. Reinsert the fuse holder.
5. Make sure that the arrow next to the voltage label is pointing at the white line below the fuse holder.

Connecting the Gases

The procedure below assumes that there are four gas supplies for the purge gas and that the gas supplies (and filter dryers, if used) are already connected.

1. Connect a length of 1/8-in. Teflon tubing from the Gas A supply to the input #1 connector on the rear of the TAGS.
2. Connect a length of 1/8-in. Teflon tubing from the Gas B supply to the input #2 connector.
3. Connect a length of 1/8-in. Teflon tubing from the Gas C supply to the input #3 connector.
4. Connect a length of 1/8-in. Teflon tubing from the Gas D supply to the input #4 connector.
5. Connect a length of 1/8-in. Teflon tubing to the OUT connector on the TAGS using a Swagelok fitting.

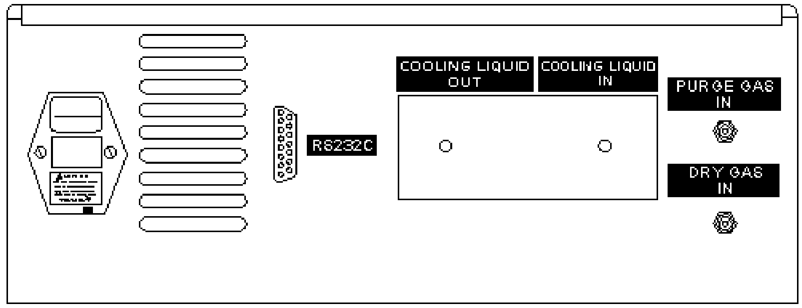
NOTE: When making the connections, first turn the fitting until it is fingertight, then turn it an additional 1/2 to 3/4 turn with a wrench. Use a second wrench to keep the connection at the TAGS stationary. Turn preswaged fittings 1/8 of a turn past fingertight. Do not overtighten.

Now you are ready to connect the purge gas(es) and dry gas to the Pyris 6 DSC.

Connect the Purge Gas and Dry Gas Lines to the Pyris 6 DSC

Perform the following procedure to connect your purge gas and dry gas lines to the Pyris 6 DSC regardless of the other components in the purge gas line. The Pyris 6 DSC purge gas and dry gas connectors are mounted to the back of the analyzer.

NOTE: Dry gas is necessary only if you are going to run the Pyrsi 6 DSC in subambient mode.

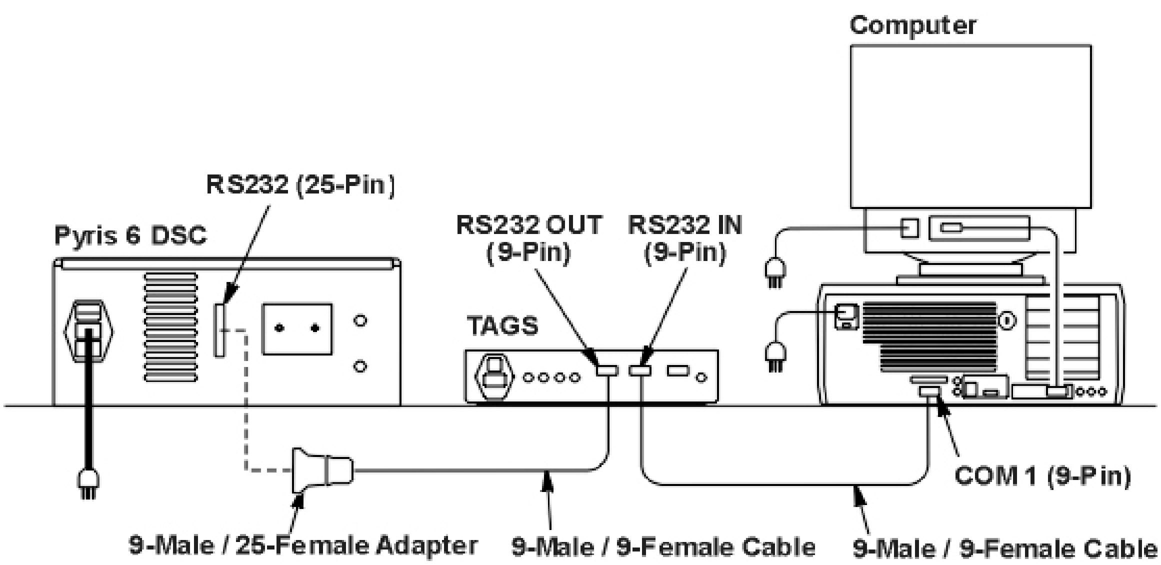


1. Place the 1/8-in. Teflon tubing from your purge gas line or TAGS onto the inlet labeled Purge Gas In.
2. Place the 1/8-in. Teflon tubing from your dry gas line onto the inlet labeled Dry Gas In.

After finishing the connection of purge gas(es) and dry gas supplies, you are now have to connect the Pyris 6 DSC, computer, and TAGS, if used.

Connecting the Pyris 6 DSC, TAGS, and Computer

1. Attach one of the two 9 male/9 female RS232 cables provided with the TAGS to COM1 or COM2 on the computer.
2. Attach the other end of that cable to the **RS232 IN** connector on the TAGS.
3. Connect the other 9 male/9 female cable to the **RS232 OUT** connector on the TAGS.
4. Connect the 9 male/25 female AT adapter to the other end of that second cable.
5. Connect the 25 female end of the adapter to the **RS232** connector on the Pyris 6 DSC.



Startup of the Pyris 6 DSC Thermal Analysis System

After installing the cooling supply and purge gas and dry gas supplies, start the Pyris 6 DSC Thermal Analysis System as follows:

1. Power up the computer, TAGS, if installed, Pyris 6 DSC, and the printer.

2. Turn on the purge gas(es), dry gas, and cooling device.
3. Start Windows NT or Windows 95 on the computer.
4. After "Main Menu" on the TAGS display has stopped blinking, click on the Pyris Manager icon in the Pyris Software for Windows group or select Pyris Manager from the Program menu in the **Start** button menu.
5. Select **Configure** from the Pyris Manager panel.
6. From the Pyris Configuration dialog box, select Pyris 6 DSC.
7. Click on the **Edit** button.
8. Under Accessories, click on the TAGS box.
9. Click on **Close** in each dialog box to return to the Pyris Manager.

NOTE: The TAGS will display REMOTE which means that the Pyris software controls the TAGS.

10. Select Pyris 6 DSC from the Pyris Manager to enter the Pyris 6 DSC Application.
11. Select **Preferences** from the Tools menu.
12. Select the Purge Gases tab.
13. In the Purge Gases page, select the gases and the initial flow rates for the gases attached to the TAGS. Gas A is connected to gas input 1, Gas B to 2, etc.
14. Click on **OK** to save the settings.

You are now able to use the TAGS to switch the purge gases as indicated in a method's gas program.

Calibration Reference Materials for Subambient Operation

Very-high-purity (>99.999%) organic materials can be used to calibrate the temperature when operating the DSC in the temperature range from –170°C. See the table below for a list of recommended subambient calibration standards that can be used with your DSC. Normally, indium is the reference material that is recommended for energy calibration for all operating conditions.

Substance	**Transition**	**Transition Temp. (°C)**	**Transition Energy (J/g)**
Cyclopentane	Crystal	–151.16	69.45(a)
Cyclopentane	Crystal	–135.06	4.94(a)
Cyclohexane	Crystal	–87.06	79.58(b)
Cyclohexane	Melt	6.54	31.25(b)
n-Heptane	Melt	–90.56	140.16(c)
n-Octane	Melt	–56.76	182.0(c)
n-Decane	Melt	–29.66	202.09(c)
N-Dodecane	Melt	–9.65	216.73(c)
n-Octadecane	Melt	28.24	241.42(d)

Hexatriacontane	Crystal(*)	72.14	18.74(d)
Hexatriacontane	Crystal	73.84	60.25(d)
Hexatriacontane	Melt	75.94	175.31(d)
P-Nitrotoluene	Melt	51.64	

(*) Shows some thermal history dependence
(a) Aston, J.G. et al., *JAC*, **65**, 341 (1943)
(b) Aston, J.G. et al., *JAC*, **65**, 1035 (1943)
(c) Finke, H.L. et al., *JAC*, **76**, 33 (1954)
(d) Schaerer, A.A. et al., *JAC*, **77**, 2017 (1955)

1. The materials listed here, if used for calibration, must be of 99.999% minimum purity as even small levels of impurity can affect the temperature and/or energy of the transition.
2. These substances must be encapsulated in volatile sample pans. To improve peak resolution, place a small piece of aluminum foil over your sample before sealing the pan.
3. If the peaks are not sharp (as in indium), the sample may be impure and the temperatures you measure may not be correct. Use a higher purity sample or check the purity of the sample by an alternate technique such as gas chromatography.

Maintenance

The Pyris 6 DSC needs little routine maintenance other than proper treatment as a sensitive electromechanical device.

Since the **sensor disk** is the hottest component in the furnace area, it will usually remain clean and free of condensates. However, samples may ooze out of sample pans or be accidentally spilled onto the sensor disk. Such materials should be removed at low temperatures whenever possible by using tweezers or moistened cotton swabs.

Solvents should not be placed directly on the sensor. Moisten a cotton swab or a tissue with solvent; then clean the sensor or upper portion of the furnace wall with the swab.

Any residual sample that has carbonized may remain on the sensor disk after it has been cleaned with a solvent. To remove the residue, gently scrub the sensor with a fiberglass or nylon brush. Be careful not to exert excessive force when scrubbing the sensor since it can be damaged. The sensor must be as clean and flat as possible for proper operation.

To avoid contaminating the **furnace**, use a purge gas at proper flow rates. If the furnace becomes contaminated, clean the furnace as soon as possible.

Part Numbers

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Below is a list of the part numbers that are in the **Spares kit (P/N N520-0031)**.

Part No.	**Description**	**Quantity**
0319-0033	Indium calibrating reference material	1
0319-0036	Zinc calibrating reference material	1
0990-8400	Tweezers	1
0991-9184	Pan	4
N519-0762	Kit – crimped indium/zinc calibrating reference materials	1
N520-2014	Label – Serial	1
N520-2015	Label – Nameplate back	1
N520-2016	Nameplate	1
N520-2017	Label – Power entry 230 V	1
N539-0123	Aluminum pans and covers	pkg. of 100

Also included with the Pyris 6 DSC are

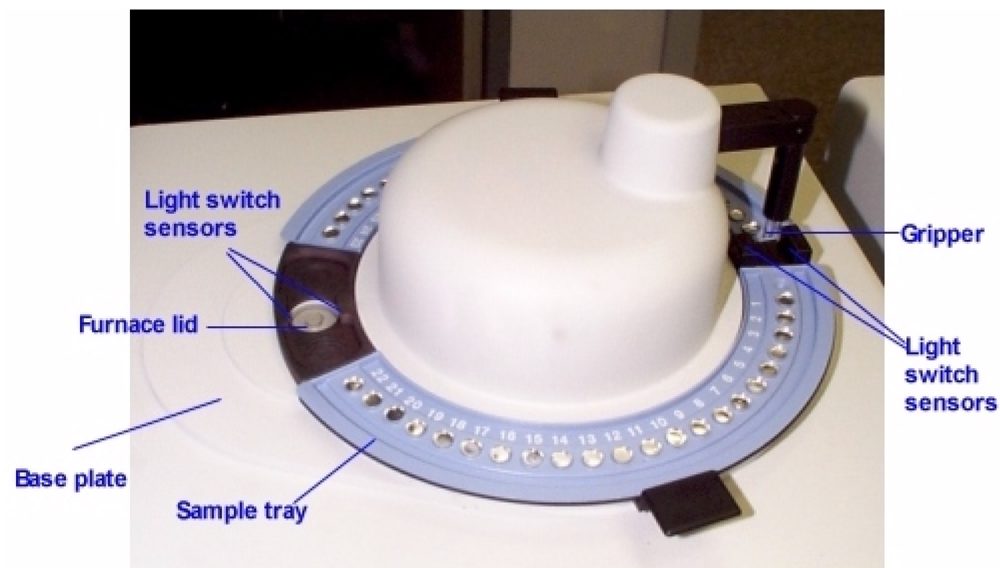
1. Tygon tubing (P/N 0250-6519) for connection of liquid coolant
2. hose clamps (P/N 0992-0044)
3. Teflon tubing (P/N 0250-8059) for purge gas connection
4. RS-232 cable for communicating to the computer
5. Power cable

AS 6 Autosampler

The AS 6 autosampler accessory brings automation capability to the Pyris 6 DSC when used in conjunction with the Pyris Software for Windows Player feature. The autosampler comes in an upgrade kit and must be installed by a service engineer. In the future the autosampler will be factory installed.

- The autosampler is powered by the power supply of the parent instrument.
- The part of the autosampler that manipulates the sample pan is called the gripper. It comprises three fingers, 120° apart from each other. They extend out of the end of the gripper housing. For a more detailed description of the gripper device, see **Gripper Description and Alignment** below.
- There are only two ranges of motion for the gripper: the gripper arm moves up and down and rotates. This allows the gripper to access each sample pan in the trays, transfer a pan from the tray, through the furnace access hole in the base plate, to the furnace, and then return it to its position on the tray. Movement in these two directions also allows the gripper a sufficient range of motions to access the furnace lid(s) and move it onto and off of the furnace access hole.
- The gripper moves from position to position by a motor/encoder type system.

- For the Pyris 6 DSC, you can use the existing robotic sample pans sealed with the Universal crimper press. All other standard sample pans (sealed and vented) can also be used.
- The AS 6 communicates with the computer via the parent instrument's connection to the computer via the RS232 port.
- The parent instrument communicates with the AS 6 via a I2C bus.
- The autosampler operates over the full temperature range of the Pyris 6 DSC.
- The unique three-finger gripper mechanism uses the power of "memory metal," i.e., nickel titanium, to open and close with minimal moving parts for highly reproducible and dependable operation. When power is supplied to the metal it shrinks and the fingers open; when power is not supplied, the metal expands and the fingers close.
- The autosampler operates with a feedback system from the instrument. When a sample pan touches the sensor at the bottom of the furnace, the autosampler senses this and rises up out of the furnace.
- The light switch sensors by the furnace hole and at the home position detect the presence of a sample pan or furnace lid being placed in the furnace hole. If the autosampler is supposed to be removing a lid, for example, and the gripper fails to pick up the lid, it is detected by the sensors and error message is detected.
- The autosampler is fully automated and controlled by the software. The two-piece sample tray holds up to 45 samples for efficient unattended operation.



One tray holds 22 samples and the other 23 samples. They were designed with the different number of locations so that the user could not mistakenly place one tray in the wrong position. The trays have have recesses on the bottom which catch positioning pins in the base plate. The positioning pins for one tray are located in different places than those for the other tray, which also prevents one tray from being inserted into the the wrong location. The trays can be removed independently so that you can load one with fresh samples while the autosampler runs samples in the other.You could also add samples to the tray "in use" to replace samples that are completed by pausing the play list.

The Pyris Player feature allows for creation of sequences or play lists to operate the autosampler and instrument. During an autosampler sequence, you can perform multiple experiments, curve optimization, calculations, and printing. With Pyris Player, you can create Sample Groups that group similar analyses together to be run in any order.

The following topics discuss the AS 6 autosampler in more detail:

- **Safety Precautions**
- **How the Autosampler Works**
- **Gripper Description and Alignment**
- **Sample Preparation and Encapsulation**
- **Sample Pans**
- **Running a Play List**
- **Troubleshooting**
- **Maintenance**
- **Part Numbers**
- **Install a Reference Pan into a Pyris 6 DSC**

Safety Precautions for the AS 6

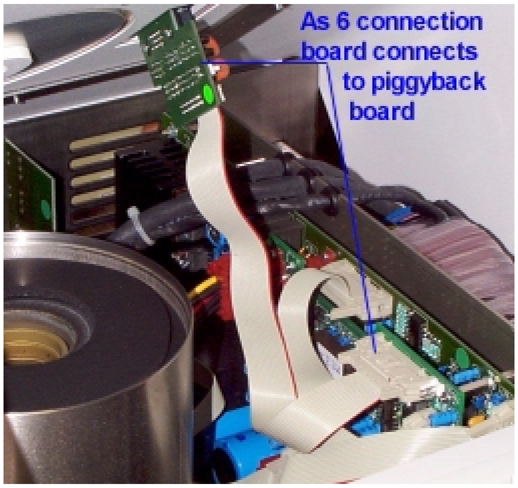
The safety precautions to be followed when using the AS 6 autosampler are the same as those for the parent instrument: the Pyris 6 DSC. In addition, the following two precautions should be kept in mind:

WARNING: To remove the furnace lid(s) of the parent instrument, always use tweezers. The furnace lid(s) might be hot.

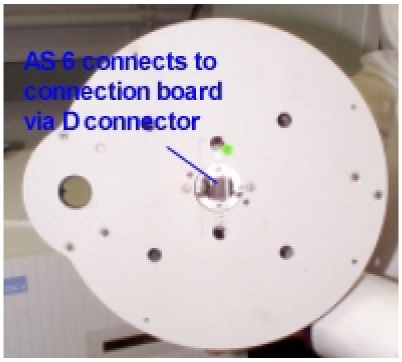
CAUTION: Before powering up the parent instrument, make sure that the AS 6 is properly attached to the base plate on the parent instrument.

How the AS 6 Autosampler Works

When a Pyris 6 DSC is upgraded with an AS 6 autosampler, the service engineer installs an AS 6 controller board piggyback style on to the parent instrument's controller board. There also is a connection board that connects the autosampler to the AS 6 controller board.



The autosampler connects to the connection board via a D connector.



The AS 6 uses a two-stepper motor design and moves from position to position by a motor/encoder type system. Upon installation, the service engineer calibrates and aligns the gripper with the lid(s), sample pans in locations 12 and 33, and the furnace vertical position. An alignment wizard is provided for the user to align the gripper at locations 12 and 33.

The autosampler has to be configured into the Pyris 6 DSC system via the Pyris Configuration dialog box in order for the software to recognize it.

The AS 6 autosampler is computer controlled and fully automated. Information between the host computer with Pyris software and the AS 6 is sent via the parent instrument's connection to the computer via the RS232 port. Light switch sensors by the furnace access hole and the home position of the grippers detect the presence or absence of a furnace lid or sample pan. An error message is generated and displayed in the software and on the LCD of the parent instrument if the sensors detect the presence of an object when there should not be one and vice versa.

NOTE: The instrument cannot detect if a sample pan is present in the furnace from a previous power on period. If you turn off the instrument with a sample pan in the furnace, you must remove the pan before loading another sample during the following power on session.

In general, you place samples into sample pans and load the pans in the sample trays. The sample trays are then placed on the base plate on the parent instrument. At the beginning of a run, the gripper arm is up all the way and is over the home position. The fingers are closed. The upper and lower furnace lids are on the lid holders in the furnace access hole. The gripper device is instructed via a command in the Pyris software to begin a session.

Autosampler Work Cycle

When you begin a sample run by starting a play list, the autosampler performs a typical work cycle. If you use the Sample Group instead of a regular play list, many steps are automatically added to the play list by the software. These additional steps are seen when you select the View History page. For example, if you wish to run two of more samples, you have to include a Return Sample line after the Start Method line for each sample before a Load Sample. When using Sample Group, the software adds that Return Sample line automatically when the play list is played back.

A typical work cycle for the autosampler on a Pyris 6 DSC and used with a play list is as follows:

Before the start of a play list, the gripper should be over the home position and there should be no sample pan in the furnace. The lower furnace lid and the upper lid should be in place.

1. Start the play list. Your play list may have an explicit **Load Sample** command, or, if using a Sample Group, the entry Sample List implies Load Sample. Upon reaching this line in the list, the gripper swings around to remove the upper lid and place it on the ring.
2. The gripper then removes the lower lid and places it on the ring.
3. The gripper retrieves the sample pan and places it in the furnace.
4. The lower lid is placed within the furnace.
5. The upper lid is placed on the furnace.
6. The gripper returns to the home position.
7. The method runs.
8. At the end of the run, the system removes the upper lid and places it on the ring.
9. The system removes the lower lid and places it on the ring.
10. The sample pan is removed from the furnace and returned to its place in the tray.
11. The system places the lower lid back into the furnace.
12. The upper lid is placed back on the lid holder.
13. Gripper goes to the home position.
14. The data analysis list for the sample is performed.
15. If there is another sample in the list, the work cycle, starting with step 1, begins again.

Gripper Description and Alignment

Description

The AS 6 autosampler comprises a base plate, two removable sample pan trays that sit on the ground plate (the bottom of the assembly), and a gripper assembly. The gripper assembly consists of an elongated gripper housing with the gripper fingers extending out from the end of the housing.

Inside the housing, at the bottom of the arm, there is a spool that can move up and down. The spool has three vertical grooves on its outside that hold the gripping fingers in place and spaced equidistantly. There is a receiving rod in the center of the spool. The rod is used to pull the spool up.

The gripping fingers are movable between an open position and a closed position for gripping and releasing the sample pan or furnace lid. Above the spool, near the bend of the gripper assembly arm, there is a fixed piece that holds the top of the gripper fingers and keeps them aligned properly during operation.

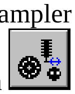
A shape memory metal wire, a nickel–titanium alloy of 0.15 mm, is used to move the spool between the upper and the lower positions. When heat provided by voltage (about 4 V) is applied to the wire, it causes the temperature of the metal to rise. The metal then undergoes a phase transition and shrinks. This pulls up on the rod which raises the spool from the lower to the upper position. When the spool is in the upper position, the gripper fingers move from the closed position to the open position.

Each gripping finger has a bushing at its end. Each bushing has a recess in which the lip of the furnace lid can be placed when the fingers close around it. This is important since the lid is heavy compared with the sample pans. The bushings are not needed to manipulate the pans; friction alone is sufficient to lift a pan.

The gripper assembly moves in both a rotational direction and a vertical direction and from position to position in the sample tray by a number of stepper motors. A lead screw in the center of the assembly (under the dome) is connected to a gear wheel that is driven by a stepper motor. This screw connects to the gripper arm and moves it up and down.

There are two LEDs (light-emitting diodes) connected to the autosampler. One is positioned above the furnace of the parent instrument and the other is on the opposite side of the ground plate. These LEDs work in conjunction with sensors positioned opposite them that allow the autosampler to determine if a sample pan is in the gripper fingers or whether an error as occurred. The LED by the furnace determines if the gripper fingers contain a sample pan or a furnace lid. The LED by the zero position allows the autosampler to determine of the gripper fingers have released a pan or if the pan has remained in their grip.

Gripper Alignment

The autosampler is aligned by the service engineer when it is installed. However, if you install a reference pan into the Pyris 6 DSC or if the gripper does not grasp the sample pans correctly, you should perform a gripper alignment procedure. Access the procedure from the DSC6/TGA6 Autosampler Control dialog box which is displayed by clicking on the **Autosampler Control** button  on the control panel. Click on **Align Gripper** to open the gripper alignment wizard.

1. The first wizard informs you that the upper furnace lid should be in place on top of the furnace access hole for either parent instrument. The lower furnace lid should be in its place on the ring. It is not necessary to have sample pans in positions 12 and 33. If you wish to use the previous calibration's values as a start for this calibration, click on the radio button. (The calibration values are stored in the firmware on the AS 6 controller board.) Otherwise, the default values will be used to start. Click on **Next** to continue.
2. The gripper moves from the home position to location 12. The buttons in the next wizard are used to move the gripper with respect to the dimple of location 12 so that the fingers are even around the dimple. You also have to adjust the height of the fingers above the tray surface. There should be only 0.1 mm between the tray and the finger. Use the buttons on the wizard to move the gripper up and down and left and right. When satisfied with the positioning, click on **Next**. The gripper swings around to position 33.
3. Do the same thing for position 33 as you did for 12. Click on **Next**.
4. The last wizard screen is displayed. Click in **Finish** to exit from the Align Gripper wizard and return to the Autosampler Control dialog box.

Realigning the Pyris 6 DSC with AS 6

When the Pyris 6 DSC with the AS 6 is installed, or when an instrument is upgraded with the AS 6, the service engineer aligns the base plate with respect to the furnace sensor. He then tightens the base plate in place. He then aligns the furnace lid holder with respect to the furnace sensor. The lower furnace lid holder is not fixed in place so occasionally it may need to be realigned. This is also done if the gripper is blocked in its descent into the furnace.

1. Remove the upper furnace lid.
2. Remove the lower furnace lid.
3. Insert the furnace lid holder alignment tool (see below) to align the lower furnace lid holder. Make sure that it is all the way down.
4. Look down into the tool and make sure that the furnace is centered.

5. Carefully remove the furnace lid holder alignment tool without disturbing the lower furnace lid holder; it is not fixed in place.
6. After realigning the lower furnace lid holder, you should perform the Align Gripper procedure.

Sample Preparation and Encapsulation

Sample preparation and encapsulation for Pyris 6 DSC with AS 6 is basically the same as that for the Pyris 6 DSC alone. See the **Sample Preparation** and **Sample Encapsulation** sections above.

Sample Pans

The types of sample pans that you can use for the Pyris 6 DSC with autosampler are all sizes of the standard robotics sample pans that are available. The Pyris 6 DSC is shipped with a Sample Pan starter kit (P/N B014-6340). The sample pans must be sealed using the Universal Crimper press which comes with the Pyris 6 DSC + AS 6 lab systems. The capacities for the sample pans are 10, 25, 30, and 50 µL. See the **Sample Pans and Sealers** section above for a full list of sample pans you can use with the autosampler.

Running a Play List with the AS 6

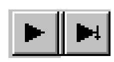
Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items — Load Sample, Run Method, Return Sample — there is also a Sample Group. The Sample Group simplifies grouping like samples together (as you would have in a sample tray of the autosampler). These like samples use the same test method and data analysis as part of the Sample Group. A Sample Group consists of a

Sample List and a Data Analysis List. A specific method is selected for the samples in the Sample List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization, and calculations for automatic data analysis.

Before starting a run, perform the steps below:

1. Review the safety and warning notes for the Pyris 6 DSC.
2. Verify that the purge gas, if you are using it, and cooling device tubes are properly connected at the rear of the instrument.
3. Turn on the gas and adjust the pressure. Turn on the chiller and adjust according to its instructions.
4. Verify that the electrical and cable connections between the computer and the instrument and other components of the system are properly connected.
5. Turn on the power to the system components.
6. Prepare your samples. Weigh the samples before placing them in the sample pans and crimping the pans.
7. Start Pyris Software for Windows and click on the Pyris 6 DSC button in the Pyris Manager.

   Click on the **Pyris Player** button on the toolbar: . Either open an existing play list and edit it accordingly or create a new one. You have to enter the weights into the Sample Details area of the Edit Step: Sample section.
8. Save the play list by selecting **Save Player** from the File menu.
9. Start playback of the play list by clicking on the **Start at Top** button or the **Start at Current Step** button on the Player toolbar. These are the first two buttons on the toolbar:



NOTE: If you select the **Start at Current Step** button, the focused item must be a main-level item, i.e., Prepare Sample, Data Analysis, or Sample Group. However, if you are in a Sample Group, you can start a play list from a Sample line in the Sample List.

NOTE: If you select Go To Load, Go To Temp, or Hold at Temp from the control panel while a play list is running, the current sample run will end and the playback of the play list ends.

Troubleshooting

When the AS 6 autosampler malfunctions, in most cases it will generate an error message which is sent to the computer and is displayed in the Pyris software. The troubleshooting list below should be used when the AS 6 malfunctions and there is no error message displayed in Pyris but an error message is displayed on the Pyris 6 DSC. There are two kinds of error message:

- LCD error messages shown on the parent instrument's LCD. An error caused by the AS 6 results in the error message ERR_DUE_TO_AS6 to be displayed.
- RS232 error messages that are enabled after initialization of the remote control, e.g., after starting the Instrument Application.

To continue normal operation, power off the instrument and power it back on after 10 seconds. Depending on the error, normal operation may or may not be possible. If normal operation can not be resumed, the problem has to be resolved by a service engineer.

Error message on LCD and RS232	**Error**	**Possible Cause**	**Corrective Action**
ERR_NO_12VOLT	No +12V power supply available (stepper motor power supply)	Overload condition in switched power supplies 12-V power supply not OK	Check stepper motor wires Replace the AS 6 assembly Replace controller PCB
ERR_HOR_STEP_BLOCKED	Horizontal stepper motor blocked	Blocked gear wheels Blocked by external object	Check gear wheels Remove external object
ERR_VER_STEP_BLOCKED	Vertical stepper motor blocked	Incorrect calibration Blocked gear wheels Blocked by external object Inner lid of Pyris 6 DSC misplaced	Recalibrate the AS 6 Check gear wheels Remove external object Replace Pyris 6 DSC inner lid
ERR_DUE_TO_PARENT	Parent instrument has entered fatal error state that results in the AS 6 also entering error state	Error occurred in parent instrument	Check parent instrument
ERR_GRP_WIRE_BROKEN	Gripper wire is broken or no AS 6 mechanics connected	Gripper wire is broken AS 6 connector not connected AS 6 cable not OK	Replace gripper assembly Connect AS 6 connector Check AS 6 cable
ERR_GRP_NO_SAMPLE	Gripper contains no sample when entering furnace. Light switch in state CST_FURN_PUT_SAMPLE during an automatic calibration	There was no sample pan at the reserved position on the sample tray	Place a sample on position 0 of the sample tray
ERR_GRP_NOT_EMPTY	Gripper still holds sample while moving OUT of furnace during state CST_FURN_PUT_SAMPLE or in state CST_PERFORM_TEST	Sample sticks to gripper fingers	Clean gripper tips Use clean sample pans
ERR_LIGHT_SWITCH	Light switches are not functioning or are blocked when the gripper is in raised position	Light switch is blocked Light switch in not OK AS 6 cable is not OK	Remove object that blocks light switch Replace AS 6 mechanics Check/replace AS 6 connection board
ERR_MICRO_SWITCH	Microswitch is activated when gripper is NOT in reset state	Microswitch is not OK Microswitch wiring not OK AS 6 cable not OK	Replace gripper assembly Check microswitch wiring Check/replace AS 6 connection board

ERR_FURN_NOT_EMPTY	Furnace still contains a sample while trying to load a new one. This error can be generated only after loading at least one sample after power on, as the instrument cannot detect if a sample is present in the furnace from a previous power on period.	Failure picking sample out of furnace. Incorrect horizontal baseplate adjustment. Incorrect horizontal calibration AS 6 run was interrupted after putting a sample in furnace and restarting.	Check if gripper fingers are bent. Replace gripper assembly. Adjust baseplate Recalibrate the AS 6 Reset parent instrument and setup link again
ERR_NOT_AT_SENS	Sensor position not reached during autocalibrating the sensor position	Vertical stepper missed steps because it was blocked while running at slow speed. (During slow gripper speed, the stepper blocked detection is disabled.)	Check the Pyris 6 DSC lower lid position.
ERR_NO_ZERO_DETECT	Zero position is not detected.	No detection of the gripper fingers because of direct sunlight at the light switches. Gripper assembly displaced.	Do not place instrument in direct sunlight. Align gripper assembly

Error Messages on the Pyris 6 DSC

Error message on Pyris 6 DSC LCD and RS232	**Error**	**Possible Cause**	**Corrective Action**
ERR_DUE_TO_AS6	AS 6 entered fatal error state. This caused Pyris 6 DSC to enter fatal error state.	Refer to AS 6 error message.	Refer to AS 6 troubleshooting.
ERR_AS6_RESPONS	Timeout on command. Response from command transmitted to AS 6.	I2C connection cable defective.	Replace I2C cable.
ERR_AS6_I2C_WD	No link between Pyris 6 DSC and AS 6 via I2C.	I2C connection cable defective.	Replace I2C cable.

AS 6 Autosampler Maintenance

The AS 6 autosampler case is painted metal. The exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution.

The bushings of the gripper fingers should also be kept clean. Use a Q-tip dampened with a mild detergent and water solution to carefully wipe them clean. Use the same method to clean the two sample trays.

WARNING: **There are no user-serviceable parts inside the autosampler. Refer all servicing to a qualified Perkin Elmer service engineer.**

Part Numbers

Part Number	Description
N537-0736	Pyris 6 DSC + AS 6 Lab System 17, 100 V
N537-0737	Pyris 6 DSC + AS 6 Lab System 18, 115 V
N537-0738	Pyris 6 DSC + AS 6 Lab System 19, 220 V
N520-3120	AS 6 Update Kit for Pyris 6 DSC

Pyris 6 DSC Lab System 17 (100 V) P/N N537-0736

This kit includes the Pyris 6 DSC, the AS 6 autosampler, Pyris Software for Windows, sample pan starter kit (B014-6340) with 50 of each type of pan and 350 lids, and the Universal Crimper press. Service installation is required.

Pyris 6 DSC Lab System 18 (115 V) P/N N537-0737

This kit includes the Pyris 6 DSC, the AS 6 autosampler, Pyris Software for Windows, sample pan starter kit (B014-6340) with 50 of each type of pan and 350 lids, chiller circulating cooling system, and the Universal Crimper press. Service installation is required.

Pyris 6 DSC Lab System 19 (220 V) P/N N537-0738

This kit includes the Pyris 6 DSC, the AS 6 autosampler, Pyris Software for Windows, sample pan starter kit (B014-6340) with 50 of each type of pan and 350 lids, chiller circulating cooling system, and the Universal Crimper press. Service installation is required.

AS 6 Upgrade Kit P/N N520-3120

If you have a Pyris 6 DSC and order an AS 6 autosampler, the service engineer will upgrade your instrument using this kit. It contains the following items:

General AS 6 Parts

Name	Description
AS 6 assembly and sample trays	Autosampler assembly for use with the Pyris 6 DSC including sample tray 1 – 22 and tray 23 – 45.
AS 6 main control board	Circuit board to be service installed onto the main circuit board inside the parent instrument in order to connect the AS 6 to the instrument.
AS 6 connector circuit board plus attached ribbon cable	Circuit board to be service installed underneath and on to the instrument's cabinet (outer cover) to connect the AS 6 to the AS 6 main control circuit board.
Instrument cover	Outer cover for the parent instrument
Instrument front panel	New front panel for the Pyris 6 DSC to reflect the new color. This upgrades older existing models.
PCB mounting screws (3)	Mounts the AS 6 main circuit board to the parent instrument's main controller board.

Cover screws (4)	Attaches the instrument cover to the instrument. They are the new color.
Base plate mounting profile	Metal plate (U-shaped) that mounts under the instrument cover. Used to attach the AS 6 base plate to the instrument. Includes three screws.
Cable I2C	Ribbon jumper cable to connect the AS 6 PCB to the instrument's PCB communications channel.
Cable RS232	Ribbon cable to connect the AS 6 RS232 port to the instrument's RS232 port mounted on the inside back panel.
Download connector	25-pin D shell connector used only when downloading firmware for the AS 6. It connects to the RS232 port on the back of the instrument.

Pyris 6 DSC Parts

Name	**Description**
Pyris 6 DSC + AS 6 base plate + lid holders + lids	Base plate mounted by 3 screws to the instrument cover. It must be aligned with the furnace for proper operation.
Pyris 6 DSC + AS 6 base plate alignment tool	Tool to adjust the base plate with respect to the sensor. This tool is also used to position the inner furnace lid.
Inner/lower furnace lid holder mounting tool	Used to fix the inner/lower furnace lid holder in the Pyris 6 DSC. Can also fix M2 hexagonal screw.
Pyris 6 DSC firmware upgrade	Main firmware to operate the AS 6 with the Pyris 6 DSC.

Install a Reference Pan in a Pyris 6 DSC with AS 6

When running samples with the Pyris 6 DSC, it is recommended that a reference capsule be used in the furnace. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated. Because the AS 6 autosampler and its base plate cover access to the reference pan area, they have to be removed first. The procedure to do this and place a reference pan inside the Pyris 6 DSC is as follows:

1. Make sure that the Pyris 6 DSC power is off.
2. Remove both sample trays.
3. Remove the four screws that attach the autosampler to the base plate.

> **NOTE:** Never remove the screws from the plate that is directly attached to the analyzer's cover. This plate is positioned with respect to the furnace. The positioning is performed by the service engineer upon installation.

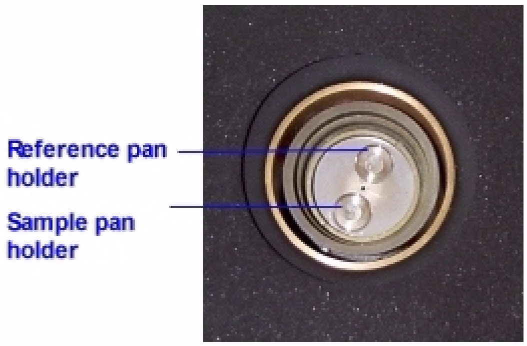
4. Place the tips of your fingers in the indents in the sides of the base plate and lift the autosampler to disengage the D connector and the two standoffs on the base plate.
5. Remove the upper furnace lid and the base plate lid holder.

Base plate lid holder with the upper furnace lid removed.

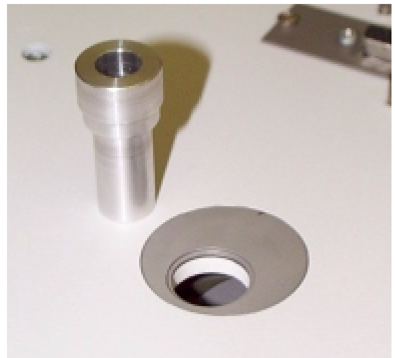
6. Remove the lower furnace lid and the lower furnace lid holder.

Lower furnace lid holder with lower lid removed.

This exposes the reference pan position. The sample pan position is the one closer to the side of the instrument. The reference pan position is closer to the center of the instrument.



7. Place a reference pan on the reference position.
8. Replace the lower furnace lid holder.
9. Replace the base plate lid holder.
10. Insert the furnace lid holder alignment tool into the base plate lid holder to position the lower furnace lid holder properly with respect to the upper lid holder. Make sure that the tool is all the way down. Look down into the tool to make sure that the furnace is centered in the tool.



11. Carefully remove the alignment tool without disturbing the lower furnace lid holder.
12. Place the autosampler on top of the base plate, aligning the two holes near the D connector that engage the two standoffs on the base plate. Gently push the autosampler down to snap in place.
13. Attach the autosampler to the base plate with the four screws.
14. Place the sample trays on the autosampler with tray 1 – 22 toward the front and tray 23 – 45 on the rear.

After placing a reference pan within the Pyris 6 DSC and replacing the autosampler, you should run through the gripper alignment procedure to make sure that the gripper aligns with the sample pans at locations 12 and 33.

Chapter 4
Pyris 6 TGA

Overview

The Perkin Elmer Pyris 6 TGA Thermogravimetric Analyzer features reliability, robustness, low cost of operation, and ease of use, all of which make it an outstanding analyzer for quality control laboratories. It has been developed to be compatible with the Pyris 6 DSC.

The Pyris 6 TGA is controlled by Pyris Software for Windows on the computer to which the analyzer is connected. The information it provides is essential to industries such as plastics and polymers; automotive; semiconductors and electronics; adhesives; paints and coatings; fuels; ceramics, clays, and soil; food; pharmaceuticals; and medical devices and equipment. The Pyris 6 TGA measures the weight of a substance as a function of its temperature or the time. Weight changes can be measured accurately, generating information on various processes in a sample such as dehydration, degradation, oxidation, etc. Also, since measurements can be performed in a well-defined atmosphere, the behavior of a sample in atmospheres other than air can be studied easily. The Pyris 6 TGA can be used for component analysis (weight %), evaluation of stabilizers, loss on drying, proximate analysis, lubricating oil analysis, and oxidation studies.

The Pyris 6 TGA has an integrated cooling system. You can use various types of cooling liquids with temperatures higher than 0°C (noncondensing). It is recommended that normal tap water be used as the cooling agent. During operation, the cooling liquid must flow through the heat exchanger at a maximum flow rate of 100 mL/min (6 L/h). The cooling liquid should be at a temperature such that there is no condensation inside the analyzer.

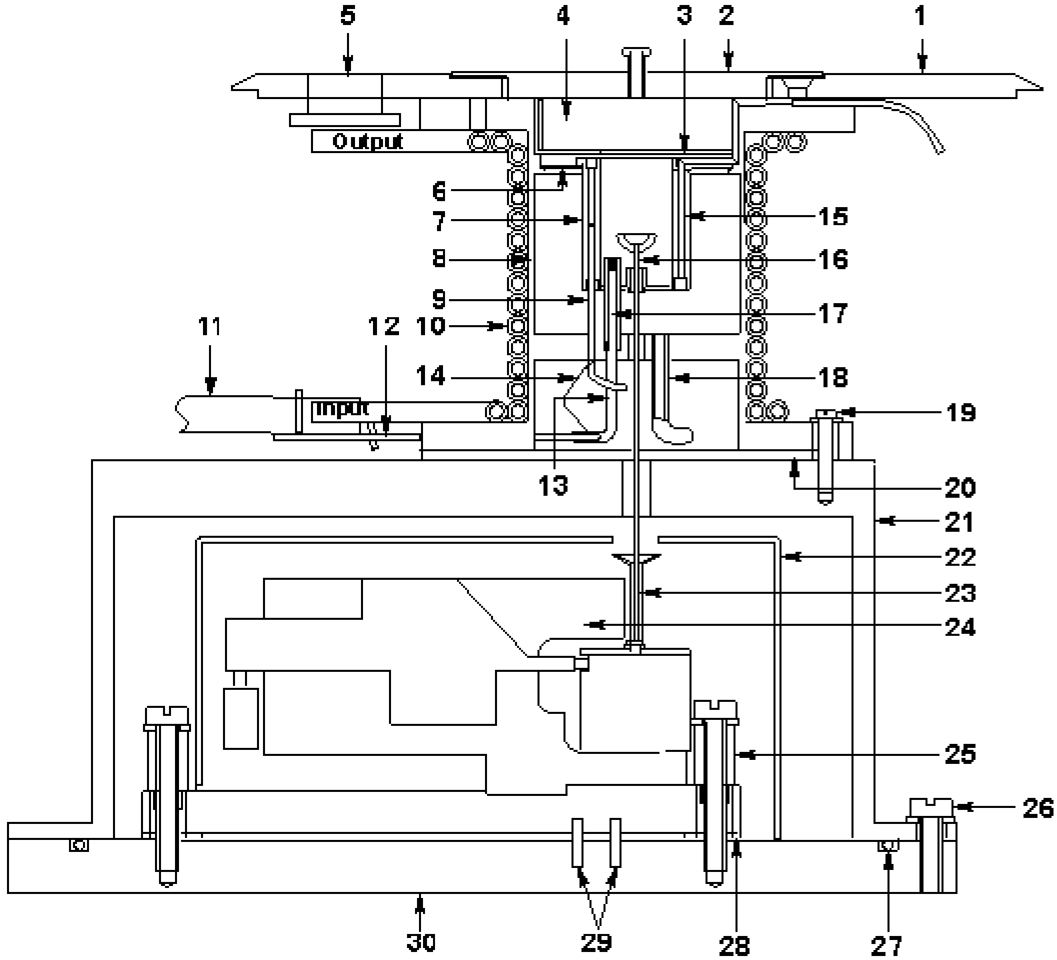
A unique feature of the Pyris 6 TGA is its top-loading microbalance. The sample pan is placed onto the sample holder which connects to the microbalance via a very thin rod. The top-loading balance has many advantages over a hangdown wire design. With this design, however, placement of the sample in the sample pan and of the sample pan in the sample holder is very important. The microbalance is very sensitive to sample positioning.

The furnace has a large isothermal zone. The sample position in the Pyris 6 TGA is approximately in the middle of the isothermal zone. This increases reproducibility of temperature measurements.

The rapid cooling rate of the furnace is achieved by mounting the furnace in a liquid-cooled jacket. To reduce the heating power at high temperatures, the cooling jacket is nickel plated. Also, there is forced air cooling. A small air pump is mounted inside the instrument and activated by clicking on the **Cooling Air** button on the control panel. Liquid cooling is also used to keep the temperature of the balance housing constant. The cooling jacket is mounted on and thermally insulated from the balance housing. Temperature gradients are eliminated as much as possible.

See the cross-sectional view of the Pyris 6 TGA below:

Cross-Sectional View of the Pyris 6 TGA



1. Cover plate	11. Furnace power cable	21. Balance case
2. Lid	12. PCB	22. Balance cover
3. Furnace cover	13. Reaction gas input	23. Sample holder fitting
4. Alsiflex paper	14. Platinum furnace leads	24. Balance
5. Spirit level	15. Sample thermocouple	25. Balance attachment
6. Furnace suspension	16. Sample holder	26. Screw
7. Furnace	17. Alumina tube	27. Isolation strip
8. Cooling jacket	18. Cooling air input	28. Sealant
9. Furnace thermocouple	19. Screw	29. Guidance pins
10. Cooling helix	20. Isolation	30. Base plate

For more information on the Pyris 6 TGA, see the topics below:

- **Features**
- **Safety Precautions**
- **Leveling**
- **Sample Handling**
- **Calibration**
- **Maintenance**
- **Part Numbers**
- **Autosampler**

Features of the Pyris 6 TGA

To see why the Pyris 6 TGA is so versatile, see each of the subtopics on the features of the instrument.

Microbalance

The microbalance of the Pyris 6 TGA is a top-loading balance with a maximum sensitivity of 5 µg. A top-loading balance has some advantages over a hangdown balance: It is less fragile, easier to operate, and allows the instrument to be smaller. The sensitivity of the top-loading balance is equal to the hangdown wire balance. The difference between the two is that with the hangdown balance, the sensitivity for sample positioning is less. The center of gravity positions itself exactly below the mounting point of the hangdown wire to the balance arm. Placement of the sample pan and sample on the top-loading balance requires more attention.

The Pyris 6 TGA top-loading balance has an accuracy of better than 0.2% of the total weight of the sample and the sample pan for sample weights greater than 20 mg. The balance is surrounded by a rectangular thin nickel-plated copper housing. This housing eliminates temperature gradients as much as possible. This housing is then surrounded by thick-walled stainless steel housing. This housing permits a large heat capacity.

Sample Holder

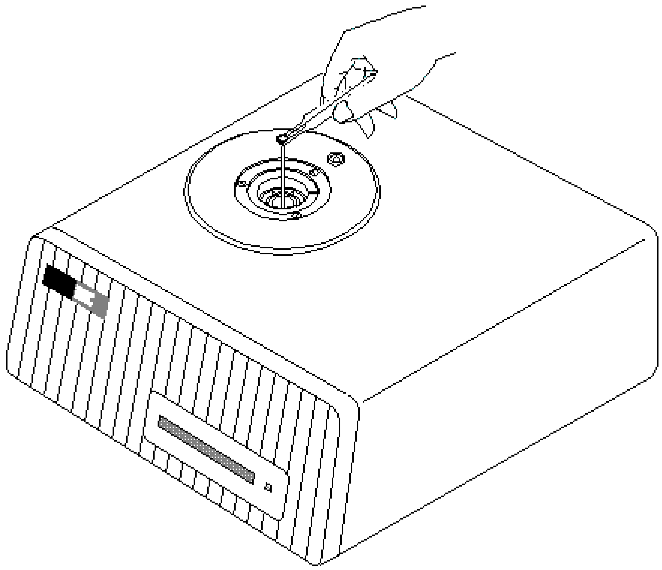
The sample holder is attached to an alumina rod of low thermal conductivity. This reduces heat flow toward the microbalance. The shaped sample holder is also made of alumina, a very hard, temperature-resistant ceramic material. The sample pan used is shaped to fit into the holder snugly.

Removing a Sample Holder

The Pyris 6 TGA sample holder should be removed by using the tweezers supplied with the instrument. Gently place the tweezers on the rim of the sample holder and slowly pull it up. Remember that the holder is attached to a long, thin ceramic rod. Lift the holder vertically until the rod has cleared the bottom of the furnace.

Installing a New Sample Holder

Carefully grasp the cup part of the sample holder with the supplied tweezers. Position the end of the rod over the center hole at the bottom of the furnace.



Insert the rod into the furnace until there is a slight resistance. Carefully move the sample holder down another 10 mm until it is resting in the microbalance fitting and can go no further. The tip of the sample holder rod actually goes into the hole on the microbalance.

DO NOT FORCE THE SAMPLE HOLDER INTO POSITION.

Removing a Broken Sample Holder

Even though the sample holder stem is quite sturdy, it can break if excessive forces are applied to it. The rod contains a wire thread inside that runs from the base of the cup to the tip of the rod. Therefore, if the rod should break or the cup breaks off of the rod, the pieces can be removed from the analyzer by grasping the cup (if still attached) or the wire with the tweezers supplied with the TGA 6 (P/N 0990-8400) and lifting gently upward.

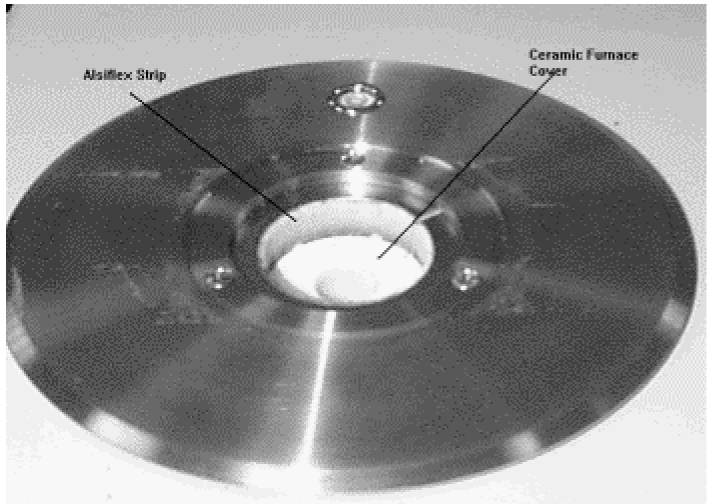
A broken sample holder cannot be repaired and should be replaced.

Furnace

The Pyris 6 TGA furnace is made of alumina and is heated by vertically wound Kanthal wires. These alloys have a melting temperature of 1500°C so it is easy to maintain the furnace at 1000°C. The wire is wound through holes in the furnace wall. The furnace thermocouple is mounted in the furnace wall.

To help keep material from falling into the furnace a ceramic furnace cover is provided. To help keep the furnace wall clean, Alsiflex strips are provided. A strip of this felt-like substance is to be inserted along the furnace wall.

Alsiflex Placement



Cooling

For quality control applications, speed is important. The cooling speed of the furnace must be as high as possible. The Pyris 6 TGA incorporates a built-in cooling device called the cooling jacket. The jacket is a copper coil that is wrapped around the furnace. It uses a simple in–out design in which normal tap water can be circulated. To reduce the heating power at high temperatures, the cooling jacket has been nickel plated.

In addition to the cooling jacket, the Pyris 6 TGA uses forced air cooling. A small air pump is mounted inside the instrument and is activated by clicking on the **Cooling Air** button on the control panel.

The liquid cooling is also used to keep the temperature of the balance housing constant. This is done by mounting the cooling jacket onto the balance housing, with a thermal insulation disk between the two pieces. Thus, temperature gradients (which lead to drift) are eliminated as much as possible. Both the isothermal drift and the dynamic drift of the Pyris 6 TGA are low.

Gases

The Pyris 6 TGA requires the use of a balance protective gas. This must be a **dry** gas, preferably nitrogen. It should flow at a rate of between 60 and 100 cc/min.

You can also choose to use a reactive or sample purge gas. The sample purge gas enters directly into the sample/furnace area. Since the furnace and sample holder are ceramic, practically all gases are possible for use but it should not be corrosive. A corrosive purge gas decreases the lifetime of the system. The gas used should not be reactive with the materials used in the construction of the Pyris 6 TGA. A size 1A cylinder equipped with a suitable regulator is recommended. Complete instructions on installing the purge gases and how to connect them to the Pyris 6 TGA are given in the online Installation Help.

A recommended flow rate for the sample purge gas is between 60 and 100 cc/min. **The sample purge gas should be in the furnace only.**

NOTE: The balance protective gas flow rate should be equal to or greater than the sample purge gas.

If you wish to use more than one sample purge gas, you can use a Thermal Analysis Gas Station (TAGS). This accessory allows you to switch between four different gases.

Heating

The basic temperature range of the Pyris 6 TGA is from ambient to 1000°C. Since the system is liquid-cooled, it is possible, in principle, to begin a run at subambient temperatures. However, condensation of water vapor on the cooling tubes around the furnace could occur and should be avoided. Reducing condensation is the user’s responsibility.

The heating rates are from 0.1 to 100°C/min up to 1000°C. The heating rate can be set in 0.1°C/min increments. In general, lower heating rates lead to better separation of transitions. For most experiments, a heating rate of 20°C/min is optimal. The free heating rate of the Pyris 6 TGA furnace has been limited to 200°C/min.

Safety Precautions

The following precautions must be observed when using the Pyris 6 TGA:

- Never turn the computer off until the following message appears:

  It’s now safe to shut off your computer.

- Never press the **Reset** button on the computer if the Pyris software appears to malfunction. Press the **Ctrl–Alt–Del** keys simultaneously and select Task Manager. From the Task Manager, close the Pyris software.
- Never remove the outer instrument cover of the Pyris 6 TGA without shutting the instrument down and disconnecting its power cord from the power source.
- Before connecting the Pyris 6 TGA to the power outlet, check the voltage setting and fuse.
- The Pyris 6 TGA requires a good earth ground that is common to the earth ground of the computer.
- Do not disconnect cables or tubes from the Pyris 6 TGA while the instrument is on.
- Only high-quality gas should be used for the balance protective gas. This must a dry gas, preferably nitrogen. The recommended flow rate is between 60 and 100 cc/min.
- Always observe the proper startup and shutdown procedures with the Pyris 6 TGA and all related instruments.
- Do not operate the instrument in a cold room. The ambient temperature and the temperature of the instrument should be between 10°C and 30°C.

WARNING: **To remove the furnace lid, always use tweezers; the furnace might be hot.**

WARNING: **Do not touch the inside of the furnace; it might be hot and the sample holder may be damaged.**

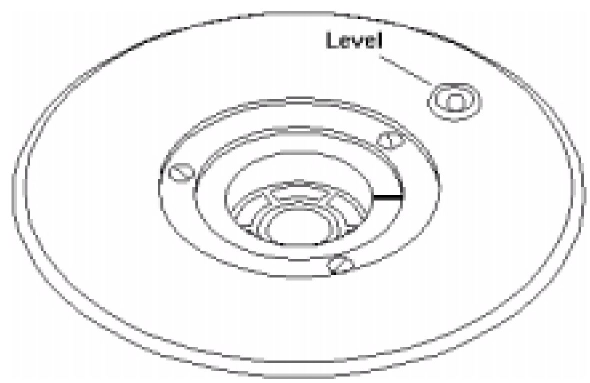
WARNING: **Always ensure that there is adequate ventilation when operating the Pyris 6 TGA. Operate the analyzer in a fume hood when running samples that give off toxic gases as the reaction gases escape through the furnace lids.**

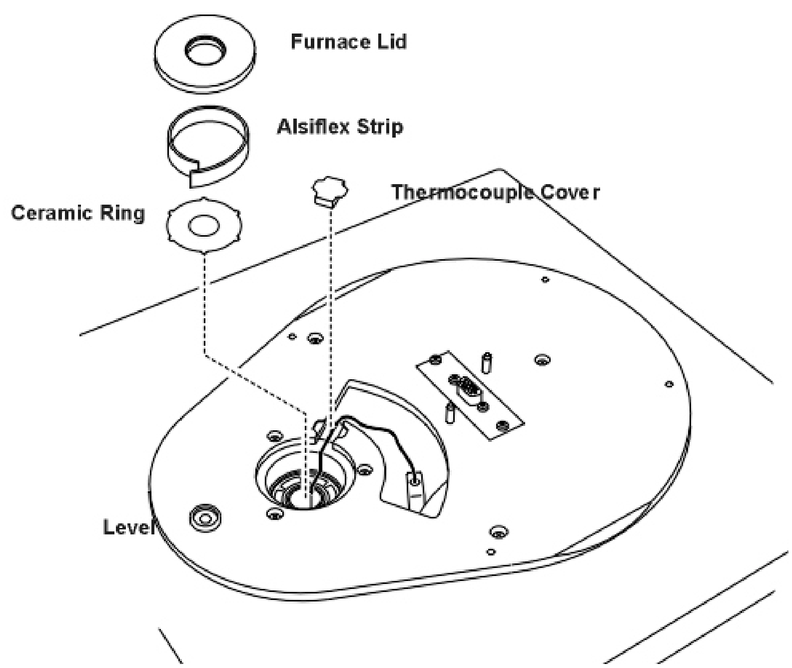
CAUTION: Do not expose the sample holder to mechanical stress. If no external cooling is applied, do not operate the Pyris 6 TGA above 50°C.

It is recommended that a reactive or sample purge gas be present. Any gas can be used as long as it is not corrosive. The recommended flow rate is 60 – 100 cc/min.

Leveling the Pyris 6 TGA

1. Locate the level mounted either on the furnace plate on top of the analyzer for a regular Pyris 6 TGA or on the autosampler base plate.





2. Lift the analyzer up at the front and turn each foot until it is screwed all the way into the bottom of the analyzer.

NOTE: The analyzer is heavy. When lifting it along the front edge, be sure your hand is firmly underneath it and can support the analyzer while you adjust the feet.

3. Position the analyzer in the exact spot on the bench where you will be using it.
4. Check whether the analyzer rocks on the bench or if one foot is not touching the bench. If so, lift the analyzer and slightly turn the foot to extend it. Lower the analyzer to check that it does not rock. Once all of the feet are touching the bench, make sure that the analyzer is steady.
5. Look at the level to see if the bubble is centered. If not, readjust the feet as in step 4 until the bubble is centered in the level. The Pyris 6 TGA is level when the bubble is centered inside the ring.
6. Once the analyzer appears to be level, make sure all of the feet are in contact with the bench and that the analyzer does not rock.

Sample Handling

The Pyris 6 TGA measures the change in weight of a sample as a function of temperature or time. The materials and techniques used to obtain data with a Pyris 6 TGA are discussed below.

Sample Preparation

The thermogravimetric analyzer analyzes solid samples in powder, crystal, or granular form. Although quantitative accuracy will remain the same regardless of sample shape, the qualitative appearance of a run may be affected by the sample configuration. Proper sample preparation that maximizes the contact surface between the pan and the sample will reduce any imbalance of the sample in the preformed sample pan and will result in maximum peak sharpness (DTGA) and resolution.

The best sample form for optimum performance is powder or fine granules. Solid materials can be sliced into small pieces with a razor or knife.

If you are using the AS 6 autosampler with the Pyris 6 TGA, the sample size should be approximately 40 mg.

Sample Pans

The preferred sample pan is the self-centering, thin-walled, ceramic sample pan provided with the instrument. The sample centers itself in the pan and the pan centers itself on the sample holder. Three sample pans are provided in the Spares kit.

Other sample pans can also be used. The only requirement that the pans must meet is that they do not react or melt within the temperature range of interest and that the sample does not form alloys with the material of the sample pan.

Sample Atmosphere

It is important that the Pyris 6 TGA be able to operate in various gas atmospheres. The change from one atmosphere to another should be quick. For this, the Pyris 6 TGA has two gas inlets. One is for the balance protective gas. This should be a dry inert gas that flows through the microbalance chamber. This keeps the environment of the balance constant, prevents absorption or desorption of vapors, and protects the balance against gaseous products evolving from the samples. The balance protective gas should have a flow rate of between 60 and 100 cc/min. Since the equilibration time of the analyzer is significant, it is recommended that the balance protective gas be present at all times.

You can control the atmosphere in which the sample is run by using a sample reactive or purge gas to displace or introduce reactive gases into the sample furnace. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another, always check the temperature calibration. Flow rates of between 60 and 100 cc/min are recommended. These flow rates will be realized by inlet purge gas rates of 20 – 30 psi.

The reaction gas enters the furnace/sample area directly, just below the sample, and flows via the furnace wall to the sample. Thus, dead volume can be low resulting in a small gas change time constant. The time constant depends on the flow rate.

NOTE: The flow rate for the balance protective gas should be equal to or greater than that for the sample purge gas.

The gas atmosphere should be pure (99.9% minimum), especially if you use nitrogen. There should be no trace of oxygen as this could lead to unwanted reactions. **The gas must be dry.** A size 1A cylinder equipped with a suitable regulator is recommended.

The degradation byproducts from the sample leave the instrument directly via a small hole in the furnace cover. These byproducts could be harmful so use adequate suction such a fume hood. There is some condensation of less volatile products on the cold spots of the analyzer: the lower side of the cover and the upper inner side of the cooling jacket. These spots can be cleaned easily with a suitable solvent. Condensation may also occur on the top inner side of the cooling jacket. To prevent this, use the porous ceramic furnace cover to absorb the less volatile byproducts. When necessary, this ring should be changed.

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

Sample Loading

Use the following procedure to load samples or remove samples:

1. Using tweezers, remove the stainless steel cover off of the top of the analyzer. Place it on the stainless steel ring surrounding the opening to the furnace.
2. Place the sample inside the sample pan.
3. With tweezers, carefully place the sample pan with sample onto the sample holder and make sure that the pan is centered.
4. Replace the lid.

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. It can be used with the all analyzers. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Connecting a TAGS to a Pyris 6 TGA

The Thermal Analysis Gas Station allows you to use up to four gases for the purge gas. You can switch gases in the gas program of the method. How to connect the gas lines to the TAGS and Pyris 6 TGA is described below.

Preparing the Laboratory for TAGS

The TAGS will accept the same laboratory conditions suited for the Pyris 6 TGA. Some precautions are

- Do not place the TAGS in direct sunlight or close to heating and cooling units.
- The temperature of the area should be between 10°C and 35°C.
- Relative humidity should be between 20% and 80% and noncondensing.
- The TAGS requires little bench space. The electrical power consumption is only 8 VA max. Therefore, it can be connected directly to the instrument's power line. The supply must be smooth, clean, and free of transient voltages over 40 V. The operating frequency is 50 – 60 Hz.

Unpacking the TAGS

The TAGS is shipped in one box and is surrounded by foam on all sides. To unpack the TAGS, follow the steps below:

1. Remove the power cable, the serial cables, and the manual from the box.
2. Remove the foam insert from the box.
3. Remove the upper foam piece.
4. Remove the TAGS from the box.
5. Remove all remaining packing material.

Installing the TAGS

Setting the Correct Voltage

Upon delivery, the TAGS is set at a voltage of 220/240 V. Changing the input voltage should be done at the AC inlet plug.

1. Remove the fuse holder with a small screwdriver by gently prying it out of the compartment. Once the holder is out far enough to grasp, slide the fuse holder out of the slot.
2. Rotate the fuse holder 180°.
3. Remove the fuse from the 220-V position (100 mA Slow Blow) and insert a 200 mA Slow Blow fuse for 110 V (it goes into the right side when "110 – 120 V" is in the upright position.
4. Reinsert the fuse holder.
5. Make sure that the arrow next to the voltage label is pointing at the white line below the fuse holder.

Connecting the Gases

The procedure below assumes that there are four gas supplies for the purge gas and that the gas supplies (and **filter dryers**, if used) are already connected.

1. Connect a length of 1/8-in. Teflon tubing from the Gas A supply to the input #1 connector on the rear of the TAGS.

2. Connect a length of 1/8-in. Teflon tubing from the Gas B supply to the input #2 connector.
3. Connect a length of 1/8-in. Teflon tubing from the Gas C supply to the input #3 connector.
4. Connect a length of 1/8-in. Teflon tubing from the Gas D supply to the input #4 connector.
5. Connect a length of 1/8-in. Teflon tubing to the OUT connector on the TAGS using a Swagelok fitting.
6. Connect the other end of the tubing from the OUT connector to the **Sample Purge In** connector on the Pyris 6 TGA.

Connecting the Pyris 6 TGA, TAGS, and Computer

1. Attach one of the two 9 male/9 female RS232 cables provided with the TAGS to COM1 or COM2 of the computer.
2. Attach the other end of that cable to the **RS232 IN** connector on the TAGS.
3. Connect the other 9 male/9 female cable to the **RS232 OUT** connector on the TAGS.
4. Connect the 9 male/25 female AT adapter to the other end of that cable.
5. Connect the 25 female end of the adapter to the **RS232** connector on the Pyris 6 TGA.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

If you are going to use a purge gas or a balance protective gas, you may want to use a filter dryer. Perkin Elmer provides the Drierite Compressed Air Filter Dryer Accessory for use with its thermal analysis instruments.

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 0.25-in. pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

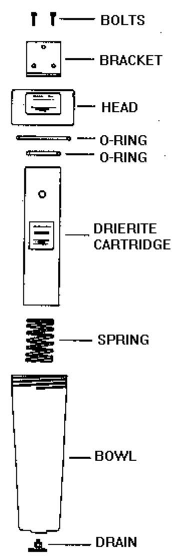
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

> **CAUTION:** Read all warnings and safety precautions before installing this drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4" pipe fitting using Teflon tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4" pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

> **NOTE:** If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2.5-in. o.d. O-ring on top of cartridge.
8. Place the 4-in. o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD SOAP AND WATER. DO NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

Calibration

The furnace, temperature, and weight are calibrated by the service engineer upon installation. The Pyris 6 TGA's calibration should remain unchanged for quite some time, provided that there are no changes in the instrument's operating conditions. Even when the system is shut off, the calibration values are stored so that the next time the instrument is turned on, it will still be calibrated. Some of the conditions that could change either the temperature or the weight calibration of the Pyris 6 TGA and require recalibration are as follows:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
- If the purge gas type or purge gas flow rate changes, the calibration should be checked for highest accuracy.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new sample holder is installed, the system should be recalibrated.
- If a new sample thermocouple is installed or if the existing thermocouple has been disturbed, you should perform a temperature calibration.
- If the analyzer is moved or releveled, you should perform a weight calibration.

There are three calibration routines for the Pyris 6 TGA. They should be performed in the following order:

- **Furnace Calibration**
- **Temperature Calibration**
- **Weight Calibration**

Furnace Calibration

NOTE: The Furnace calibration must be performed **before** the Temperature calibration for a Pyris 6 TGA.

Before performing the furnace calibration of the Pyris 6 TGA, make sure that the thermocouple is functioning properly and that the sample holder is empty. To verify thermocouple operation, program the temperature to 100°C. Enter 100 in the Go To Temp entry field of the control panel and click on the **Go To Temp** button.

Check that the current sample temperature displayed in the Status Panel is at or below the intended minimum calibration temperature. The easiest way to accomplish this is to click on the **Go To Load** button on the control panel.

When selecting minimum and maximum temperatures for the furnace calibration, select the limits so that they encompass the temperature range in which you plan to operate.

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
2. Select the Furnace tab in the Calibration window.
3. In the Minimum field, enter a minimum temperature at which the calibration will begin. This should be below your normal operating region.
4. In the Maximum field, enter a maximum temperature at which the calibration will end. This should be above your normal operating region.

NOTE: Maximum temperature minus minimum temperature must be greater than 100°C or the furnace calibration will not be performed.

5. Select the **Begin Calibration** button.

   A message stating that the system defaults will be used for all temperature calibrations if you perform a furnace calibration is displayed. If you wish to continue, click on **OK**. The Furnace calibration begins immediately.

6. The Furnace Calibration dialog box is displayed showing the approximate time remaining in the calibration. You can stop the calibration, which takes about an hour, by clicking on the **Stop Calibration** button.
7. When the calibration routine is finished, select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.
8. Select the Temperature Calibration tab or the **Close** button to close the Calibration window and begin using the new calibration values.

Temperature Calibration

NOTE: You must perform a Furnace calibration **before** a Temperature calibration for the Pyris 6 TGA.

To calibrate the temperature of the Pyris 6 TGA, perform the following steps:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu. The Calibration window is displayed.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu (this step is optional).
3. Select the **Save and Apply** button in the Calibration window to send the default values to the analyzer and save the current calibration file. The Save As dialog box appears. Select **OK** to save the default file.
4. Select **Close** to close the Calibration window.

The Pyris 6 TGA is calibrated by performing two runs each using two or three of the four magnetic standards provided: alumel, perkalloy, nickel, and iron. These are supplied with the instrument. The runs should use the same conditions under which you would run your samples. This temperature calibration uses the Curie transition of the materials, i.e., the point at which the magnetic properties disappear.

The first run of each reference material should use the lower scanning rate (e.g., 5°C/min) and the second run the higher scanning rate (e.g., 50°C/min). You must run at least two reference materials but you can use all three if desired. Therefore, you will do from four to six runs in order to perform a temperature calibration.

After you have completed the steps above, go on to the following steps to complete the temperature calibration:

5. From the Method Editor, select Open Method from the File menu and double click on Alumel.t6m, Perkall.t6m, Nickel.t6m, or Iron.t6m, depending on the sample you are going to run.
6. Place an empty sample pan on the sample holder that is already in place in the furnace.
7. Place the furnace lid back on the analyzer.

8. Click on the **Zero Weight** button on the control panel. The weight of the empty sample pan is displayed in the Zero field on the Sample Info page.
9. Remove the furnace lid. Without removing the pan, place 1 – 2 mm (10 – 20 mg) of reference material in the sample pan.
10. Place the furnace lid on the analyzer, position the magnet over the center of the lid, and click on the **Sample Weight** button on the control panel. The weight appears in the Weight field in the Sample Info page.
11. Fill in the other information on the Sample Info page.
12. To run Curie points, you must run the reference material under the same experimental conditions that you will run your samples. Check the purge gas type, coolants used, and the scanning rate. The first run of a reference material is performed at a scanning rate of 5°C/min.
13. Start the run by clicking on the **Start Method** button in the control panel.
14. When the run is finished, remove the sample pan and repeat steps 5 through 13 for the same reference material but at a scanning rate of 50°C/min.
15. After the second run for one reference material, repeat steps 5 through 14 for the other reference material(s) you choose to use.
16. For each data file collected, open the Data Analysis window, select the data file, and calculate the Offset value by performing an Onset calculation at the end of the Curie point transition and note the Onset result.
17. When all runs are complete and Onset calculations have been performed, select the Calibrate command in the **View** menu. The Calibration window appears.
18. Select the Temperature Calibration tab. Enter the Operator name. Enter the names of the reference materials used, the expected Onset values (provided in the documentation accompanying the reference materials), and the measured Onset results at the two scanning rates for each reference material.
19. Enter the scanning rates used in the Rates 1 and 2 fields and click on the appropriate radio button to indicate which references to use for the calibration: 1 and 2 or 1, 2, and 3.
20. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the current calibration file.
21. Select **Close** to close the Calibration window and begin using the new calibration values.

Weight Calibration

To perform a weight calibration of the Pyris 6 TGA:

1. While in Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Enter the weight of the reference material in the Reference Weight box. A 100-mg calibration weight is supplied with the instrument.
4. Click on **Begin Calibration**.
5. To prepare for a zero weight reading, remove any sample in the sample crucible and place the crucible into the furnace and replace the furnace cover. Click on **OK** in the dialog box.
6. When the zero weight reading is stable in the Read Zero field, click on **OK** in the dialog box.

7. Place the reference weight into the crucible. You can either first remove the crucible from the furnace and place the weight in it or place the weight with the crucible in place. Replace the furnace cover. Click on **OK** in the dialog box.
8. When the weight reading is stable in the Read Value field, click on **OK** in the dialog box. The calibration is complete.
9. Select **Save and Apply** to send the new calibration value to the analyzer and save the calibration file.

Maintenance

The Pyris 6 TGA Thermogravimetric Analyzer needs little routine maintenance other than giving it the proper treatment of a sensitive electromechanical device. Avoid contamination of the furnace by always using a balance protective gas and a purge gas. If the furnace should become contaminated, clean it as soon as possible.

Maintenance includes the following procedures:

- **Cleaning the Furnace and Sample Holder**
- **Cleaning the Cover**
- **Changing the Sample Thermocouple**
- **Replacing the Sample Thermocouple for an AS 6 Autosampler System**

Cleaning the Furnace and Sample Holder

When the furnace and sample holder have become contaminated with reaction products, the best method of cleaning them is to heat the furnace up to 1000°C in an oxygen atmosphere for approximately 10 minutes. This should remove all products that can be oxidized.

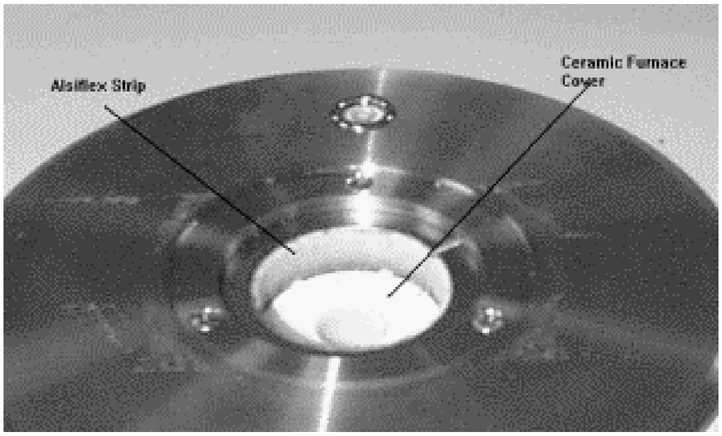
CAUTION: Be careful not to touch the sample thermocouple.

If the sample holder becomes contaminated, remove it from the furnace and clean it gently with a small brush.

When a sample has dropped into the furnace, remove the sample holder first, if present, and then remove the sample material, preferably with a vacuum.

Cleaning the Cover

The reaction byproducts from the samples run inside the Pyris 6 TGA furnace condensate on cold spots of the instrument: the bottom side of the cover and the top inner part of the cooling jacket. The bottom side of the cover can be cleaned easily with a tissue and some solvent. This should be done on a regular basis. The top inner side of the cooling jacket can be cleaned in a similar manner. To keep the top of the cooling jacket clean, a ring of porous ceramic material is provided and should be positioned as seen in the figure below. The ring should be replaced when necessary.

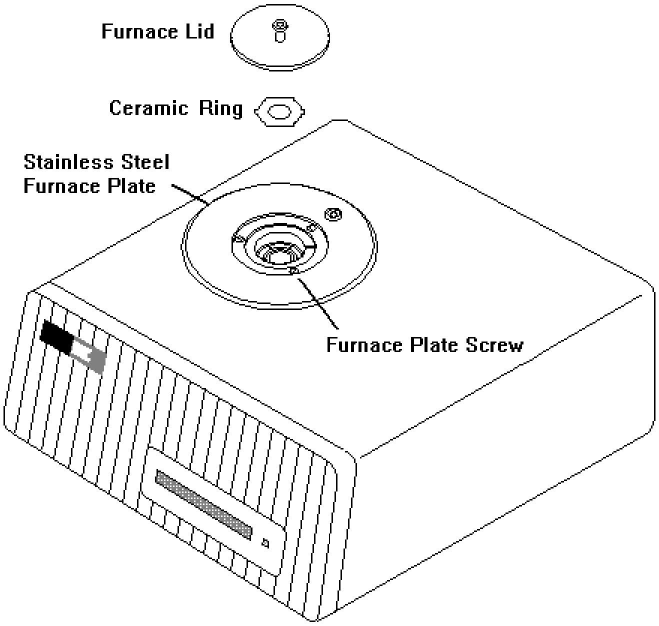


Changing the Sample Thermocouple

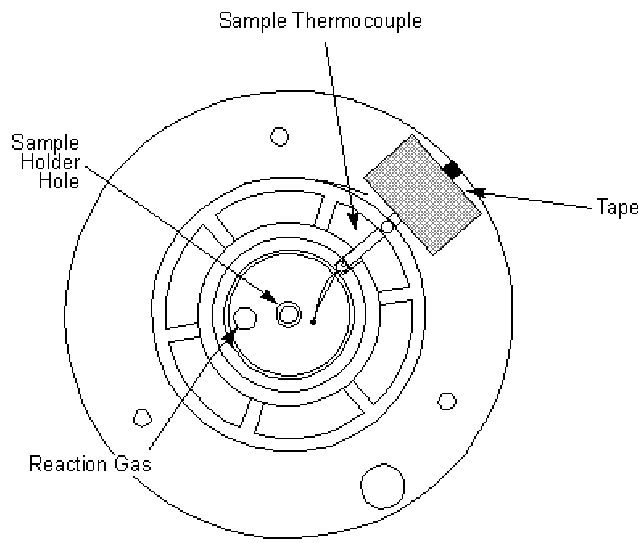
The sample thermocouple has a limited lifetime because of the range of temperatures and the high maximum temperature it withstands. Therefore, the Pyris 6 TGA is designed so that a user can change the sample thermocouple.

To remove the sample thermocouple, perform the following procedure:

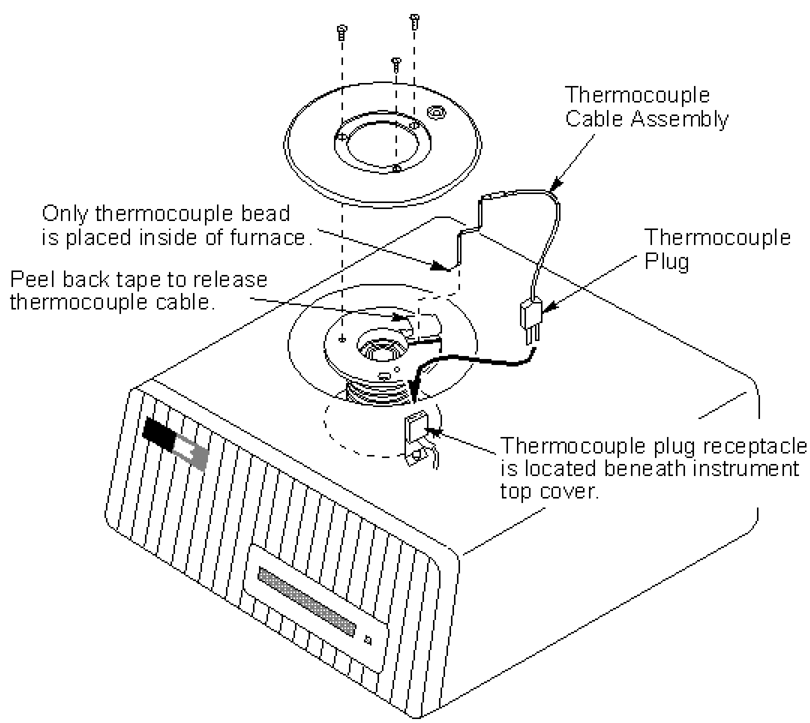
1. Power off the analyzer and the remove the power cord.
2. Remove the furnace lid.
3. Remove the porous ceramic ring.
4. Remove the sample holder.
5. Remove the three screws of the top stainless steel furnace plate and then remove the plate.



6. Lift the tape off of the top of the furnace to free the thermocouple.



7. Unplug the yellow thermocouple plug.
8. While holding the plug, gently lift the thermocouple up and out of the furnace. Be careful not to bend the thermocouple. Note how the thermocouple is positioned in the furnace wall.



To install a new sample thermocouple, perform the following steps:

1. While holding the new thermocouple by its yellow plug, gently lower it into the furnace. Make sure that the lower vertical ceramic bead goes into the slit in the furnace wall. The two wires at the end of the thermocouple should be inside the furnace, near the center hole, but not over it. The horizontal bead at the top of the thermocouple should lie inside the groove at the top of the cooling jacket.
2. Connect the thermocouple plug with the connector at the side of the jacket.

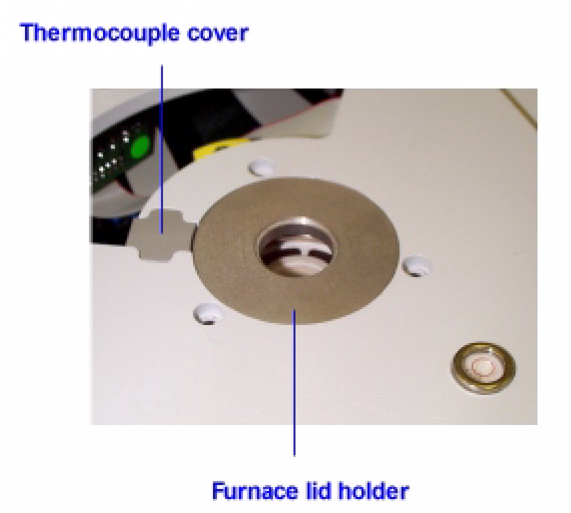
3. Replace the tape covering the thermocouple at the top of the cooling jacket.
4. Replace the stainless steel furnace plate and replace the three screws.
5. Position the sample holder in the furnace.
6. Reposition the porous ceramic ring.
7. Place the furnace lid back on top of the analyzer.
8. Reconnect the power cord and power up the analyzer.
9. Perform a temperature calibration.

Replace the Sample Thermocouple for an AS 6 System

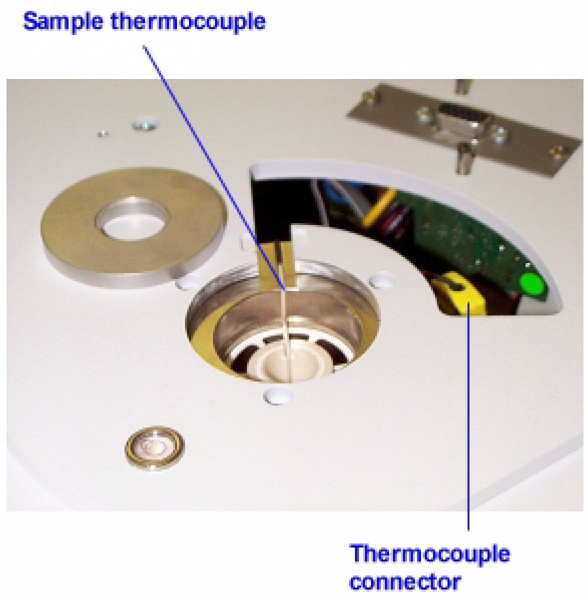
1. Power off the Pyris 6 TGA.
2. Remove both sample trays.
3. Remove the four screws that attach the autosampler to the base plate.

NOTE: Never remove the screws from the plate that is directly attached to the analyzer's cover. This plate is positioned with respect to the furnace. The positioning is performed by the service engineer upon installation.

4. Place your fingers in the indents in the sides of the base plate and lift the autosampler up and off of the instrument.
5. Remove the furnace lid and the lid holder.
6. Remove the base plate thermocouple cover.



7. Remove the alumina furnace cover disk.
8. Remove the alsiflex strip.
9. Remove the sample holder.
10. Remove the thermocouple by disconnecting the thermocouple connector and gently pulling the thermocouple out of the furnace.



11. Install the new thermocouple and replace the sample holder, alumina furnace cover disk, and the alsiflex strip.
12. Replace the lid holder and furnace lid.
13. Replace the base plate thermocouple cover.
14. Place the autosampler on top of the base plate, aligning the screw holes.
15. Attach the autosampler to the base plate with the four screws.
16. Place the sample trays on the autosampler with tray 1 – 22 toward the front and tray 23 – 45 on the rear.

After installing a new sample thermocouple, you will have to perform the **gripper alignment procedure**.

Part Numbers

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Pyris 6 TGA Spares Kit (P/N 0250-3000)

Part No.	Description	Quantity
0154-1498	H restrictor	2
0219-0071 includes:	Calibration Standards Kit	1
0998-8015	Alumel calibration standard	1
N519-0869	Nickel calibration standard	1
0998-8018	Iron calibration standard	1
N519-0616	Perkalloy calibration standard	1
0990-9608	Calibration Specification card	1
0990-8400	Tweezers – flat forceps	1
N519-0275	Magnet	1
N520-0040	Sample pans	1
N520-3020	Sample holder	2 (+1 installed)
0990-3906	Female connector 1/8 in. to 1/4 in.	2
0992-0044	Hose clamps	4
N520-0042	Calibration Weight	1
—	Ceramic furnace ring	1
—	Fuse 3.15 A Slow Blow	1
—	Fuse 6.3 A Slow Blow	2
—	Alsiflex strips	5

Also included with the Pyris 6 TGA package are

Tygon tubing (P/N 0250-6519) (5 m) for connection of liquid coolant

Teflon tubing (P/N 0250-6483) (6 m) for purge gas connection

RS-232 cable for communicating with the computer

Power cable

Male RJ45 to DB9 modem cable (P/N 0941-0042)

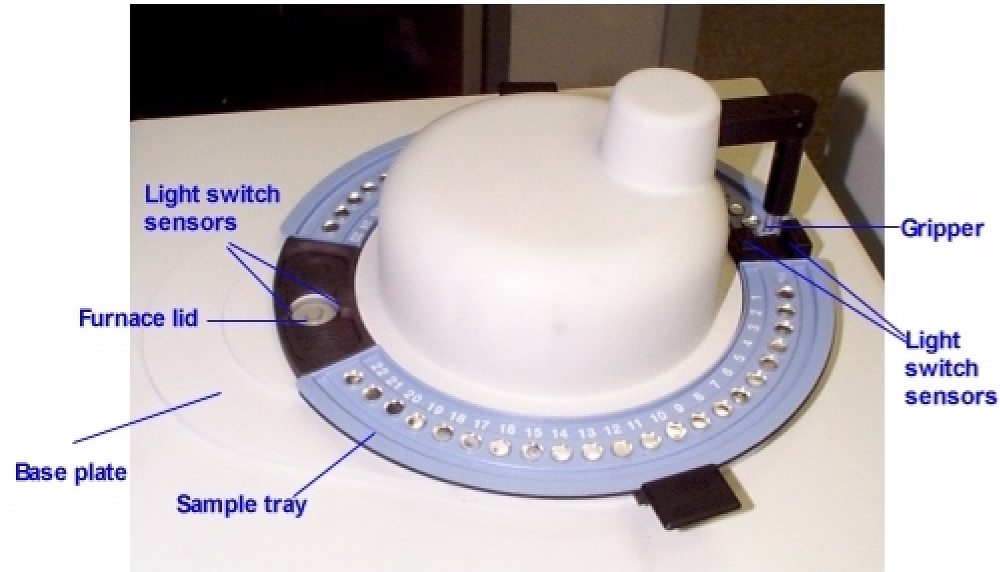
Nickel-plated 5/16-in. tubing (2 pieces) for use on Swageloks on the Cooling Liquid In and Out connections

Calibration Data Sheet

AS 6 Autosampler

The AS 6 autosampler accessory brings automation capability to the Pyris 6 TGA when used in conjunction with the Pyris Software for Windows Player feature. The autosampler comes in an upgrade kit and must be installed by a service engineer. In the future the autosampler will be factory installed.

- The autosampler is powered by the power supply of the Pyris 6 TGA.
- The part of the autosampler that manipulates the sample pan is called the gripper. It comprises three fingers, 120° apart from each other. They extend out of the end of the gripper housing. For a more detailed description of the gripper device, see **Gripper Description and Alignment** below.
- There are only two ranges of motion for the gripper: the gripper arm moves up and down and rotates. This allows the gripper to access each sample pan in the trays, transfer a pan from the tray, through the furnace access hole in the base plate, to the furnace, and then return it to its position on the tray. Movement in these two directions also allows the gripper a sufficient range of motions to access the furnace lid(s) and move it onto and off of the furnace access hole.
- The gripper moves from position to position by a motor/encoder type system.
- The AS 6 communicates with the computer via the Pyris 6 TGA's connection to the computer via the RS232 port.
- The Pyris 6 TGA communicates with the AS 6 via a I2C bus.
- The unique three-finger gripper mechanism uses the power of "memory metal," i.e., nickel titanium, to open and close with minimal moving parts for highly reproducible and dependable operation. When power is supplied to the metal it shrinks and the fingers open; when power is not supplied, the metal expands and the fingers close.
- The autosampler operates with a feedback system from the instrument. When a sample pan touches the sensor at the bottom of the furnace, the autosampler senses this and rises up out of the furnace.
- The light switch sensors by the furnace hole and at the home position detect the presence of a sample pan or furnace lid being placed in the furnace hole. If the autosampler is supposed to be removing a lid, for example, and the gripper fails to pick up the lid, it is detected by the sensors and error message is detected.
- The autosampler is fully automated and controlled by the software. The two-piece sample tray holds up to 45 samples for efficient unattended operation.



One tray holds 22 samples and the other 23 samples. They were designed with the different number of locations so that the user could not mistakenly place one tray in the wrong position. The trays have have recesses on the bottom which catch positioning pins in the base plate. The positioning pins for one tray are located in different places than those for the other tray, which also prevents one tray from being inserted into the the wrong location. The trays can be removed independently so that you can load one with fresh samples while the autosampler runs samples in the other.You could also add samples to the tray "in use" to replace samples that are completed by pausing the play list.

- The Pyris Player feature allows for creation of sequences or play lists to operate the autosampler and instrument. During an autosampler sequence, you can perform multiple experiments, curve optimization, calculations, and printing. With Pyris Player, you can create Sample Groups that group similar analyses together to be run in any order.

The following topics discuss the AS 6 autosampler in more detail:

- **Safety Precautions**
- **How the Autosampler Works**
- **Gripper Description and Alignment**
- **Sample Preparation and Encapsulation**
- **Sample Pans**
- **Running a Play List**
- **Troubleshooting**
- **Maintenance**
- **Part Numbers**

Safety Precautions for the AS 6

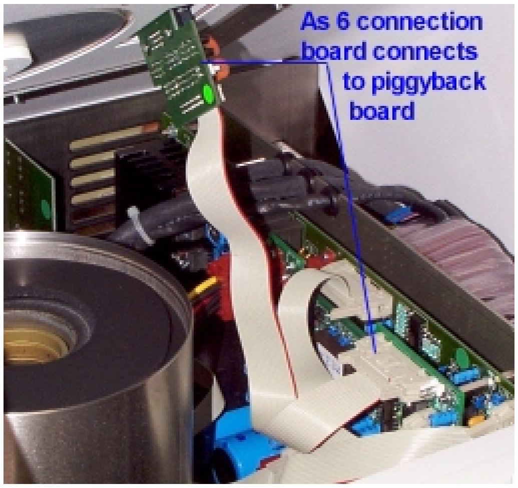
The safety precautions to be followed when using the AS 6 autosampler are the same as those for the parent instrument: Pyris 6 DSC or the Pyris 6 TGA. In addition, the following two precautions should be kept in mind:

WARNING: To remove the furnace lid(s) of the parent instrument, always use tweezers. The furnace lid(s) might be hot.

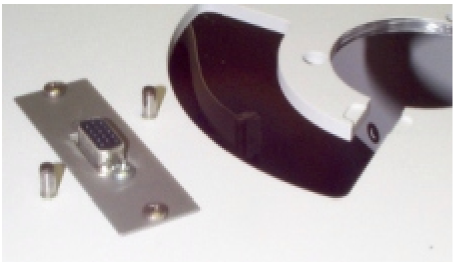
CAUTION: Before powering up the parent instrument, make sure that the AS 6 is properly attached to the base plate on the parent instrument.

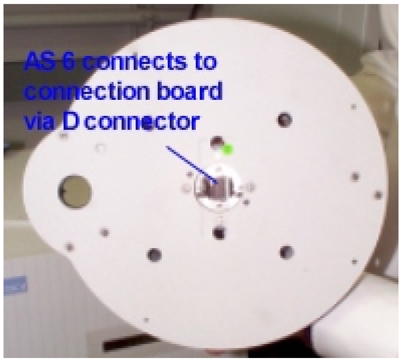
How the AS 6 Autosampler Works

When a Pyris 6 TGA is upgraded with an AS 6 autosampler, the service engineer installs an AS 6 controller board piggyback style on to the parent instrument's controller board. There also is a connection board that connects the autosampler to the AS 6 controller board.



The autosampler connects to the connection board via a D connector.





The AS 6 uses a two-stepper motor design and moves from position to position by a motor/encoder type system. Upon installation, the service engineer calibrates and aligns the gripper with the lid(s), sample pans in locations 12 and 33, and the furnace vertical position. An alignment wizard is provided for the user to align the gripper at locations 12 and 33.

The autosampler has to be configured into the Pyris 6 TGA system via the Pyris Configuration dialog box in order for the software to recognize it.

The AS 6 autosampler is computer controlled and fully automated. Information between the host computer with Pyris software and the AS 6 is sent via the parent instrument's connection to the computer via the RS232 port. Light switch sensors by the furnace access hole and the home position of the grippers detect the presence or absence of a furnace lid or sample pan. An error message is generated and displayed in the software and on the LCD of the parent instrument if the sensors detect the presence of an object when there should not be one and vice versa.

NOTE: The instrument cannot detect if a sample pan is present in the furnace from a previous power-on period. If you turn off the instrument with a sample pan in the furnace, you must remove the pan before loading another sample during the following power-on session.

In general, the user loads samples into sample pans and places the pans into the positions in the sample trays. The sample trays are then placed on the base plate on the parent instrument. At the beginning of a run, the gripper arm is up all the way and positioned over the home position. The fingers are in the closed position. The furnace lids are on the lid holders in the furnace access hole. The gripper device is instructed via a command in the Pyris software to begin a session.

Autosampler Work Cycle

When you begin a sample run by starting a play list, the autosampler performs a typical work cycle. If you use the Sample Group instead of a regular play list, many steps are automatically added to the play list by the software. These additional steps are seen when you select View History. For example, if you wish to run two of more samples, you have to include a Return Sample line after the Start Method line for each sample before a Load Sample. When using Sample Group, the software adds that Return Sample line automatically when the play list is played back.

A typical work cycle for the autosampler on a **Pyris 6 TGA** and used with a play list is as follows:

Before the start of a play list, the gripper should be over the home position and there should be no sample pan in the furnace. The furnace lid should be in place on the lid holder over the furnace.

1. Start the play list. Your play list may have an explicit **Load Sample** command, or, if using a Sample Group, the entry Sample List implies Load Sample. Upon reaching this line in the list, the gripper swings around to remove the furnace lid and place it on the ring.
2. The gripper retrieves the specified sample pan and places it in the furnace.
3. The furnace lid is placed on the lid holder.
4. The gripper returns to the home position.
5. The method runs.
6. At the end of the run, and after the end condition temperature is achieved, the system removes the furnace lid and places it on the ring.
7. The sample pan is removed from the furnace and returned to its place in the tray.
8. The furnace lid is placed back on the lid holder.
9. Gripper goes to the home position.
10. The data analysis list for the sample is performed, e.g., the curve from the run is displayed.

11. If there is another sample in the list, the work cycle, starting with step 1, begins again.

Gripper Description and Alignment

Description

The AS 6 autosampler comprises a base plate, two removable sample pan trays that sit on the ground plate (the bottom of the assembly), and a gripper assembly. The gripper assembly consists of an elongated gripper housing with the gripper fingers extending out from the end of the housing.

Inside the housing, at the bottom of the arm, there is a spool that can move up and down. The spool has three vertical grooves on its outside that hold the gripping fingers in place and spaced equidistantly. There is a receiving rod in the center of the spool. The rod is used to pull the spool up.

The gripping fingers are movable between an open position and a closed position for gripping and releasing the sample pan or furnace lid. Above the spool, near the bend of the gripper assembly arm, there is a fixed piece that holds the top of the gripper fingers and keeps them aligned properly during operation.

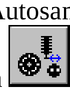
A shape memory metal wire, a nickel–titanium alloy of 0.15 mm, is used to move the spool between the upper and the lower positions. When heat provided by voltage (about 4 V) is applied to the wire, it causes the temperature of the metal to rise. The metal then undergoes a phase transition and shrinks. This pulls up on the rod which raises the spool from the lower to the upper position. When the spool is in the upper position, the gripper fingers move from the closed position to the open position.

Each gripping finger has a bushing at its end. Each bushing has a recess in which the lip of the furnace lid can be placed when the fingers close around it. This is important since the lid is heavy compared with the sample pans. The bushings are not needed to manipulate the pans; friction alone is sufficient to lift a pan.

The gripper assembly moves in both a rotational direction and a vertical direction and from position to position in the sample tray by a number of stepper motors. A lead screw in the center of the assembly (under the dome) is connected to a gear wheel that is driven by a stepper motor. This screw connects to the gripper arm and moves it up and down.

There are two LEDs (light-emitting diodes) connected to the autosampler. One is positioned above the furnace of the parent instrument and the other is on the opposite side of the ground plate. These LEDs work in conjunction with sensors positioned opposite them that allow the autosampler to determine if a sample pan is in the gripper fingers or whether an error as occurred. The LED by the furnace determines if the gripper fingers contain a sample pan or a furnace lid. The LED by the zero position allows the autosampler to determine of the gripper fingers have released a pan or if the pan has remained in their grip.

Gripper Alignment

The autosampler is aligned by the service engineer when it is installed. However, if you **change the sample thermocouple** of the Pyris 6 TGA, or if the gripper does not grasp the sample pans correctly, you should perform a gripper alignment procedure. Access the procedure from the DSC6/TGA6 Autosampler Control dialog box which is displayed by clicking on the **Autosampler Control** button  on the control panel. Click on **Align Gripper** to open the gripper alignment wizard.

1. The first wizard informs you that the upper furnace lid should be in place on top of the furnace access hole for either parent instrument. It is not necessary to have sample pans in

positions 12 and 33. If you wish to use the previous calibration's values as a start for this calibration, click on the radio button. (The calibration values are stored in the firmware on the AS 6 controller board.) Otherwise, the default values will be used to start. Click on **Next** to continue.

2. The gripper moves from the home position to location 12. The buttons in the next wizard are used to move the gripper with respect to the dimple of location 12 so that the fingers are even around the dimple. You also have to adjust the height of the fingers above the tray surface. There should be only 0.1 mm between the tray and the finger. Use the buttons on the wizard to move the gripper up and down and left and right. When satisfied with the positioning, click on **Next**. The gripper swings around to position 33.
3. Do the same thing for position 33 as you did for 12. Click on **Next**.
4. The last wizard screen is displayed. Click in **Finish** to exit from the Align Gripper wizard and return to the Autosampler Control dialog box.

Sample Preparation

Sample preparation for the Pyris 6 TGA with an AS 6 is basically the same as for the parent instrument alone.

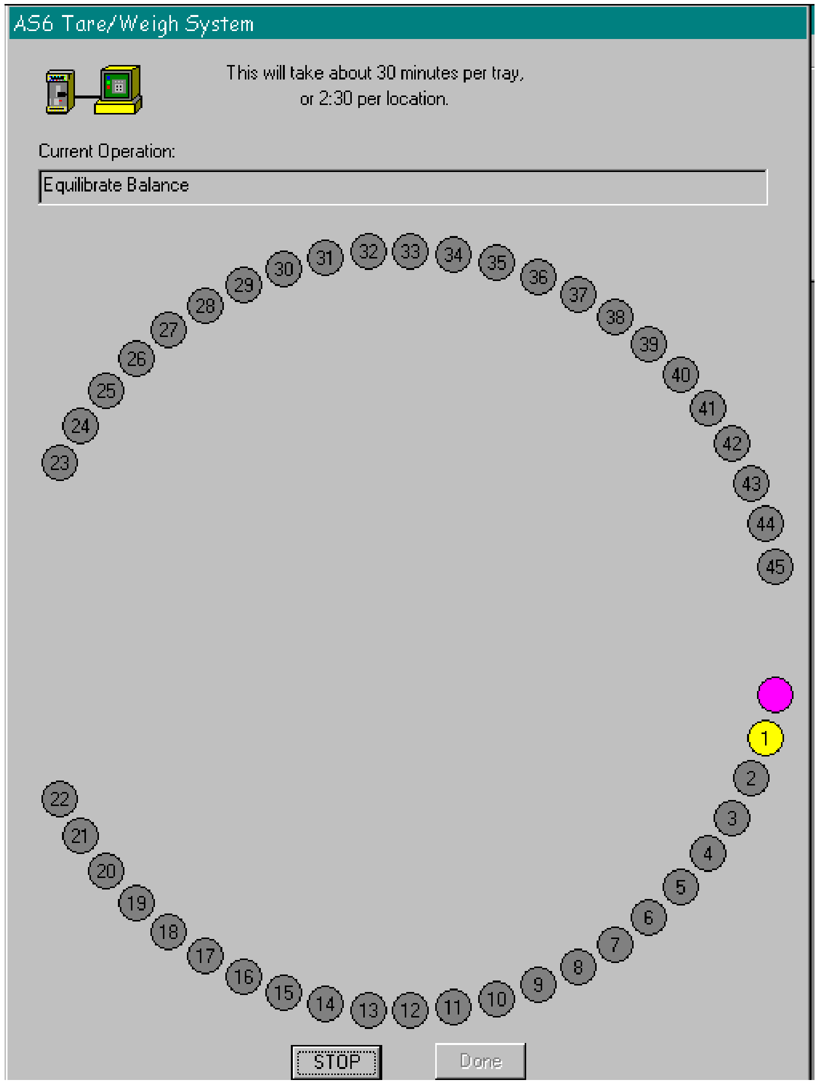
Sample Pans

The only sample pan that can be used with the Pyris 6 TGA is the ceramic crucible that is shipped with the Pyris 6 TGA + AS 6 lab systems.

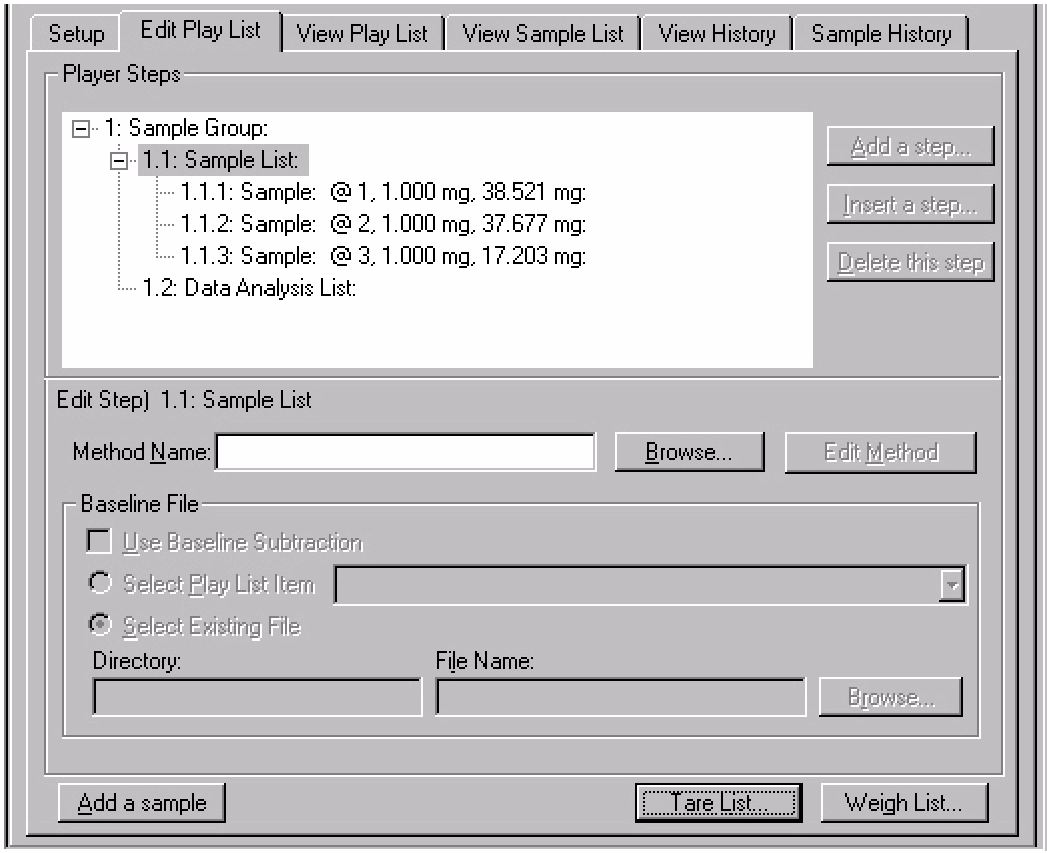
Sample Loading

Before loading the sample into the crucibles for the Pyris 6 TGA, you will have to tare the crucibles, i.e., have the system weigh them so the weight of the crucible is not included in the data. Load the empty crucibles into the sample trays. You can do this with the trays off of the autosampler or while they are still in place. After filling the locations with crucibles, carefully place the trays back onto the autosampler, making sure that the two "buttons" on the bottom side of each tray engage the holes in the autosampler. If you keep the trays on the autosampler, load the sample into the crucible while it is off the tray and then use tweezers to place the crucible onto the tray.

You can tare all of the crucibles in the tray using the Pyris Player Tare All feature. To do this your play list must contain a Sample Group. If you are creating a new play list, select Sample Group as the first entry. If you are using an existing play list, it should have a Sample Group in it in order to use the tare feature. In a new play list, with Sample Group highlighted, click on the **Tare All** button to display the Advanced Tare Options dialog box. If you choose **This Group Only; Populate from Tray**, the Sample List is filled with a line for each position in the trays that contains a crucible and the tare weight of the crucible is included. You can also add sample lines yourself and then select **This Group Only; Sample List Items Only** from the dialog box. Only the crucibles in the positions that you selected for your Sample List will be tared. When you click on the **OK** button in the Advanced Tare Options dialog box, the AS6 Tare/Weigh System screen appears:



After taring, the Sample List will now display the tare weights of the crucibles:



Once the empty crucibles are tared, remove each crucible and load the prepared sample into each one. Return the crucible to the correct position in the tray. Now the system can weigh all the samples before running the play list, or you can have each sample weighed at the beginning of its run. With Sample List highlighted, select the **Weigh List** button. The AS6 Tare/Weigh System dialog box appears. The system automatically starts the program to weigh each sample in the list. After the last sample, if there was a missing sample encountered, a message is displayed:

> **The following samples could not be weighed. It is possible that the crucibles are empty.**

A list of samples follows. Click on the dialog box's **Close** button to clear the box.

Click on the **Done** button in the AS6 Tare/Weigh System window. The Sample List will now display the weights of each sample.

For efficient use of the autosampler and to increase sample throughput, you can use remove one tray after the samples have been run and replace the crucibles with empty ones while the samples in the other tray are being run.

Running a Play List with the AS 6

Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items — Load Sample, Run Method, Return Sample — there is also a Sample Group. The Sample Group simplifies grouping like samples together (as you would have in a sample tray of the autosampler). These like samples use the same test method and data analysis as part of the Sample Group. A Sample Group consists of a Sample List and a Data Analysis List. A specific method is selected for the samples in the Sample

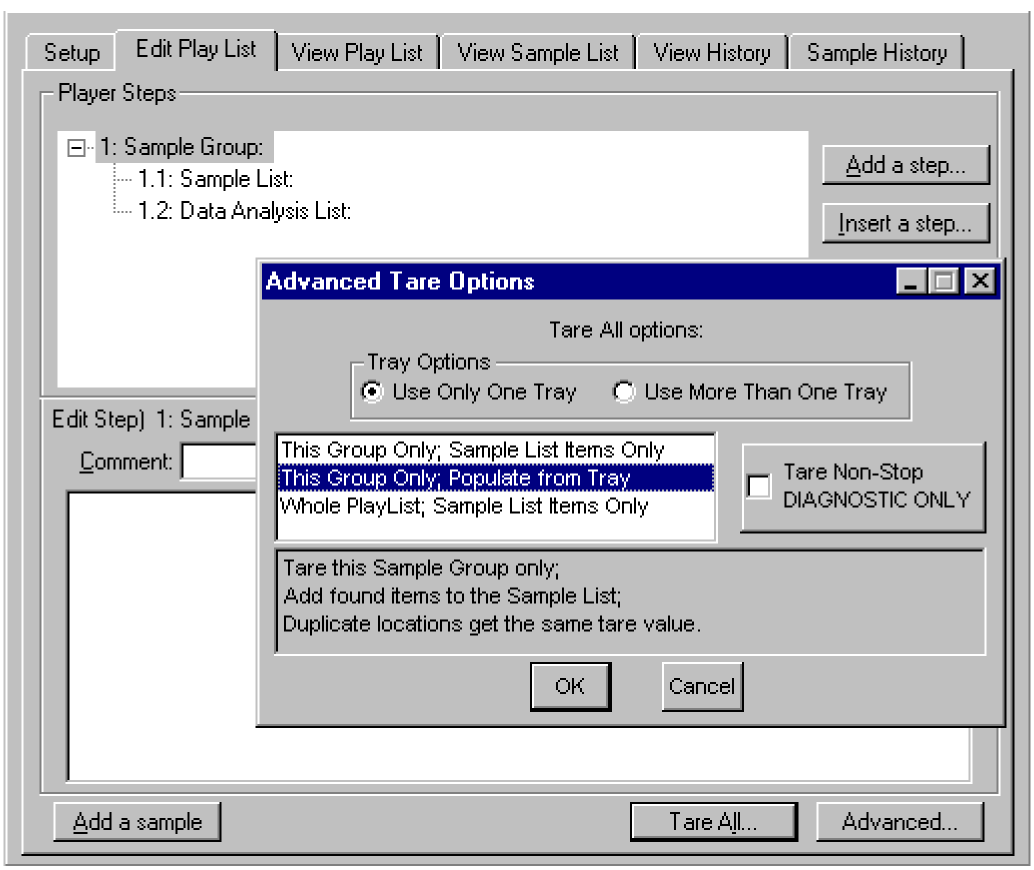
List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization, and calculations for automatic data analysis.

Before starting a run, perform the steps below:

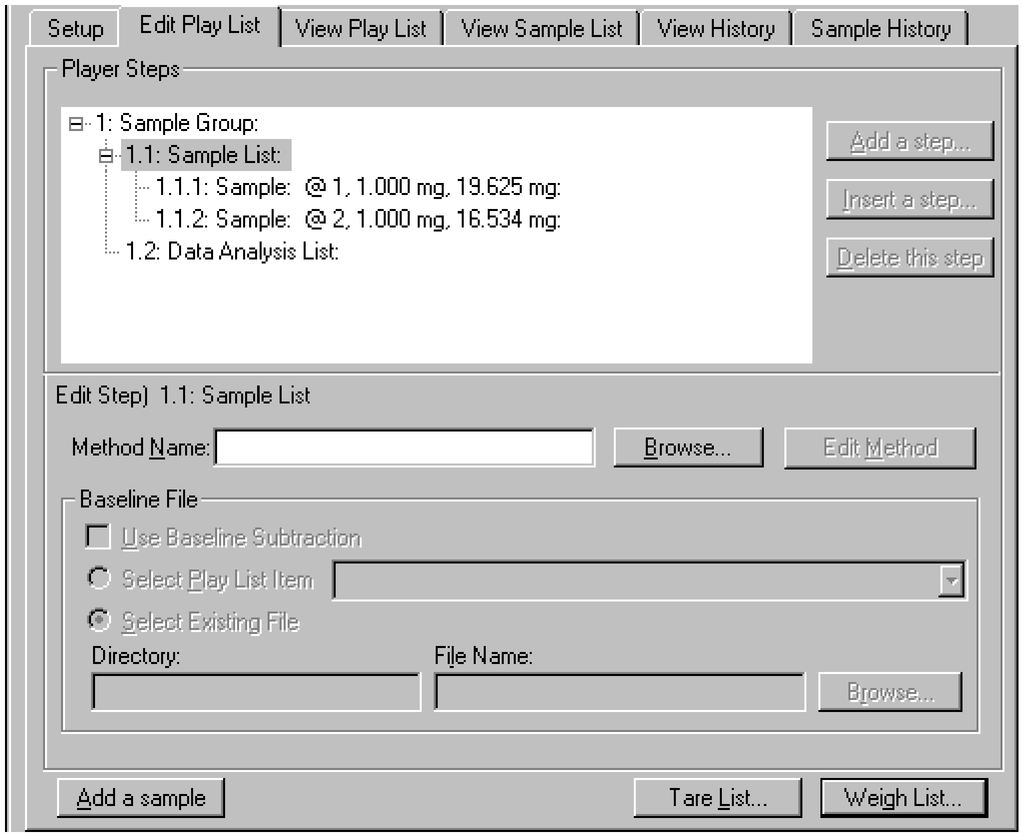
1. Review the safety and warning notes for the Pyris 6 TGA.
2. Verify that the purge gas, if you are using it, and cooling device tubes are properly connected at the rear of the instrument.
3. Turn on the gas and adjust the pressure. Turn on the chiller and adjust according to its instructions.
4. Verify that the electrical and cable connections between the computer and the instrument and other components of the system are properly connected.
5. Turn on the power to the system components.
6. Prepare your samples.
7. Start Pyris Software for Windows and click on the parent instrument's button in the Pyris Manager. Click on the **Pyris Player** button on the toolbar: . Either open an existing play list or create a new one.

   There are many ways to use the play list with the autosampler. A quick way to create a play list for a **Pyris 6 TGA** using the Sample Group feature is given below:

   Load empty sample pans into the locations you want to use in the sample tray. Create a new play list that contains only a Sample Group. Click on the **Tare All** button. In the Advanced Tare Options dialog box, select This Group Only; Populate from Tray.

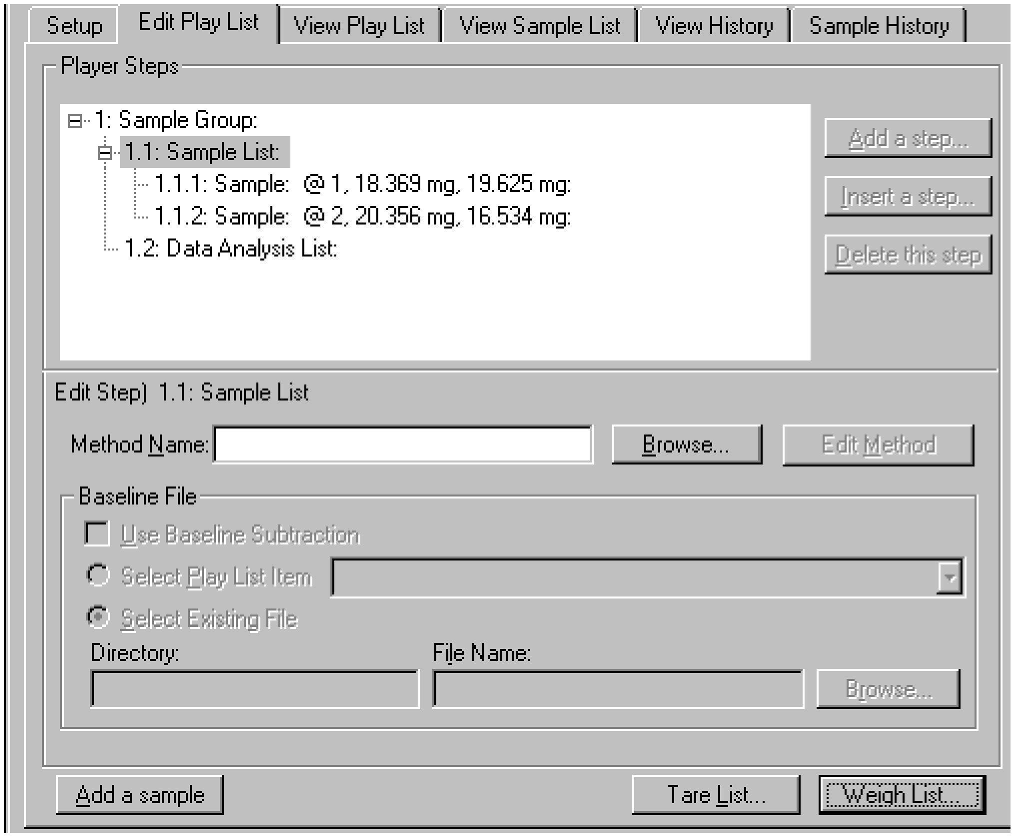


Click **OK** and the AS6 Tare/Weigh System dialog box appears. The AS 6 begins the procedure of taring the sample pans present on the sample trays. As it finds and tares each pan, it populates the Sample List and includes the tare weight for the sample pan in the Sample line. When taring is complete, click on the **Done** button. The Sample List will be filled in.



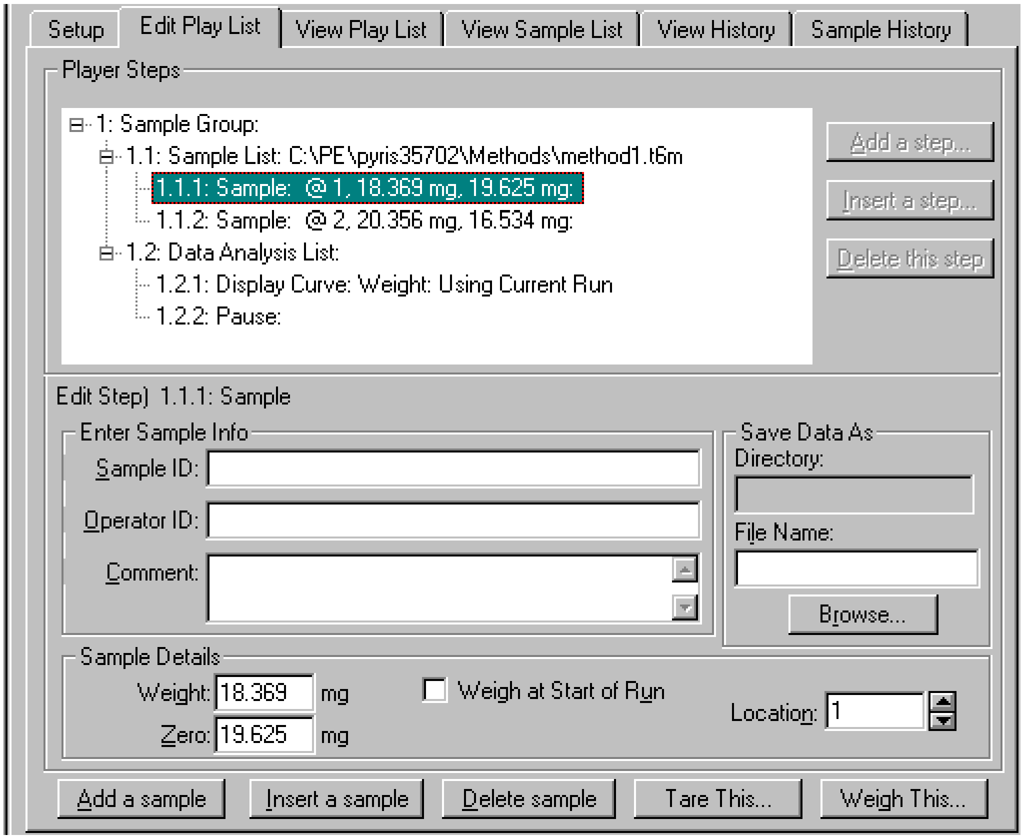
Remove the sample trays from the autosampler or leave them in place to load the crucibles for the Pyris 6 TGA onto the trays. If you removed the trays, carefully return them to the autosampler and make sure that the two knobs on the bottom of each tray engage the holes in the autosampler plate.

Now you can have the system weigh each sample in the list. You can also choose to have each sample weighed right before the run starts. To weigh all the samples before starting the play list, highlight the Sample List line. A message informing you that a method has not been selected is displayed. You can select the method after the samples have been weighed. Click on **Weigh List**. The AS6 Tare/Weigh System screen appears and the system begins to weigh the samples listed in the Sample List. When finished, the weights are displayed, along with the tare weights, in each Sample line.

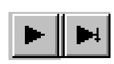


Next you need to add some items to the Data Analysis List. If you have not entered a method for the Sample List, however, you cannot fill in the Data Analysis List. To enter a method, highlight the Sample List line. Type in the name of the method in the Method Name field or click on the **Browse** button and find and select the method you want to use. Edit the method's program and initial state parameters by clicking on the **Edit Method** button. When finished editing, close the Method Editor by clicking on the window's **Close** button in the upper-right-hand corner.

Click on the Data Analysis List line and then **Add a step**. Select Display Curve. The default selection in the Edit Step area is Use Current Run. After adding the display of a curve, addition items are listed in the Player Step Options box when you add another step.



8. Save the play list by selecting Save Player from the File menu.
9. Start playback of the play list by clicking on the **Start at Top** button or the **Start at Current Item** button on the Player toolbar. These are the first two buttons on the toolbar:



NOTE: If you select the Start at Current Item button, the focused item must be a main-level item, i.e., Prepare Sample, Data Analysis, or Sample Group. However, if you are in a Sample Group, you can start a play list from a Sample line in the Sample List.

NOTE: If you select Go To Load, Go To Temp, or Hold at Temp from the control panel while a play list is running, the current sample run will end and the playback of the play list ends.

Troubleshooting

When the AS 6 autosampler malfunctions, in most cases it will generate an error message which is sent to the computer and is displayed in the Pyris software. The troubleshooting list below should be used when the AS 6 malfunctions and there is no error message displayed in Pyris but an error message is displayed on the parent instrument. There are two kinds of error message:

- LCD error messages shown on the parent instrument's LCD. An error caused by the AS 6 results in the error message ERR_DUE_TO_AS6 to be displayed.
- RS232 error messages that are enabled after initialization of the remote control, e.g., after starting the parent instrument's Application software.

To continue normal operation with the parent instrument, power off the instrument and power it back on after 10 seconds. Depending on the error, normal operation may or may not be possible. If normal operation can not be resumed, the problem has to be resolved by a service engineer.

Error message on LCD and RS232	**Error**	**Possible Cause**	**Corrective Action**
ERR_NO_12VOLT	No +12V power supply available (stepper motor power supply)	Overload condition in switched power supplies 12-V power supply not OK	Check stepper motor wires Replace the AS 6 assembly Replace controller PCB
ERR_HOR_STEP_BLOCKED	Horizontal stepper motor blocked	Blocked gear wheels Blocked by external object	Check gear wheels Remove external object
ERR_VER_STEP_BLOCKED	Vertical stepper motor blocked	Incorrect calibration Blocked gear wheels Blocked by external object	Recalibrate the AS 6 Check gear wheels Remove external object
ERR_DUE_TO_PARENT	Parent instrument has entered fatal error state that results in the AS 6 also entering error state	Error occurred in parent instrument	Check parent instrument
ERR_GRP_WIRE_BROKEN	Gripper wire is broken or no AS 6 mechanics connected	Gripper wire is broken AS 6 connector not connected AS 6 cable not OK	Replace gripper assembly Connect AS 6 connector Check AS 6 cable
ERR_GRP_NO_SAMPLE	Gripper contains no sample when entering furnace. Light switch in state CST_FURN_PUT_SAMPLE during an automatic calibration	There was no sample pan at the reserved position on the sample tray	Place a sample on position 0 of the sample tray
ERR_GRP_NOT_EMPTY	Gripper still holds sample while moving OUT of furnace during state CST_FURN_PUT_SAMPLE or in state CST_PERFORM_TEST	Sample sticks to gripper fingers	Clean gripper tips Use clean sample pans
ERR_LIGHT_SWITCH	Light switches are not functioning or are blocked when the gripper is in raised position	Light switch is blocked Light switch in not OK AS 6 cable is not OK	Remove object that blocks light switch Replace AS 6 mechanics Check/replace AS 6 connection board
ERR_MICRO_SWITCH	Microswitch is activated when gripper is NOT in reset state	Microswitch is not OK Microswitch wiring not OK AS 6 cable not OK	Replace gripper assembly Check microswitch wiring Check/replace AS 6 connection board

Error message on LCD and RS232	Error	Possible Cause	Corrective Action
ERR_FURN_NOT_EMPTY	Furnace still contains a sample while trying to load a new one. This error can be generated only after loading at least one sample after power on, as the instrument cannot detect if a sample is present in the furnace from a previous power on period.	Failure picking sample out of furnace. Incorrect horizontal baseplate adjustment. Incorrect horizontal calibration AS 6 run was interrupted after putting a sample in furnace and restarting.	Check if gripper fingers are bent. Replace gripper assembly. Adjust baseplate Recalibrate the AS 6 Reset parent instrument and setup link again
ERR_NO_ZERO_DETECT	Zero position is not detected.	No detection of the gripper fingers because of direct sunlight at the light switches. Gripper assembly displaced.	Do not place instrument in direct sunlight. Align gripper assembly

Error Messages on the Pyris 6 TGA

Error message on Pyris 6 TGA LCD and RS232	Error	Possible Cause	Corrective Action
ERR_DUE_TO_AS6	AS 6 entered fatal error state. This caused Pyris 6 TGA to enter fatal error state.	Refer to the AS 6 error message.	Refer to the AS 6 troubleshooting.
ERR_AS6_RESPONS	Timeout on command. Response from command transmitted to AS 6.	I2C connection cable defective.	Replace I2C cable.
ERR_AS6_I2C_WD	No link between Pyris 6 TGA and AS 6 via I2C.	I2C connection cable defective.	Replace I2C cable.

AS 6 Autosampler Maintanence

The AS 6 autosampler case is painted metal. The exterior surface may be cleaned with a soft cloth, dampened with a mild detergent and water solution.

The bushings of the gripper fingers should also be kept clean. Use a Q-tip dampened with a mild detergent and water solution to carefully wipe them clean. Use the same method to clean the two sample trays.

WARNING: There are no user-serviceable parts inside the autosampler. Refer all servicing to a qualified Perkin Elmer service engineer.

Part Numbers

Part Number	Description
N537-0739	Pyris 6 TGA + AS 6 Lab System 20, 100 V
N537-0740	Pyris 6 TGA + AS 6 Lab System 21, 115 V
N537-0741	Pyris 6 TGA + AS 6 Lab System 22, 220 V
N520-3121	AS 6 Upgrade Kit for Pyris 6 TGA

Pyris 6 TGA Lab System 20 (100 V) P/N 537-0739

This kit includes the Pyris 6 TGA, the AS 6 autosampler, Pyris Software for Windows, and the 45 sample pan starter kit. Service installation is required.

Pyris 6 TGA Lab System 21 (115 V) Part Number N537-0740

This kit includes the Pyris 6 TGA, Pyris Software for Windows, the AS 6 autosampler, 45 sample pan starter kit, and chiller.

Pyris 6 TGA Lab System 22 (220 V) Part Number N537-0741

This kit includes the Pyris 6 TGA, Pyris Software for Windows, AS 6 autosampler, 45 sample pan starter kit, and chiller.

AS 6 Upgrade Kit Part Number N520-3121

If you have a Pyris 6 TGA and order an AS 6 autosampler, the service engineer will upgrade your instrument using this kit. It contains the following items:

General AS 6 Parts

Name	Description
AS 6 assembly and sample trays	Autosampler assembly for use with the Pyris 6 TGA including sample tray 1 – 22 and tray 23 – 45.
AS 6 main control board	Circuit board to be service installed onto the main circuit board inside the parent instrument in order to connect the AS 6 to the instrument.
AS 6 connector circuit board plus attached ribbon cable	Circuit board to be service installed underneath and on to the instrument's cabinet (outer cover) to connect the AS 6 to the AS 6 main control circuit board.
Instrument cover	Outer cover for the parent instrument
PCB mounting screws (3)	Mounts the AS 6 main circuit board to the parent instrument's main controller board.
Cover screws (4)	Attaches the instrument cover to the instrument. They are the new color.
Base plate mounting profile	Metal plate (U-shaped) that mounts under the instrument cover. Used to attach the AS 6 base plate to the instrument. Includes three screws.

Cable I2C	Ribbon jumper cable to connect the AS 6 PCB to the instrument's PCB communications channel.
Cable RS232	Ribbon cable to connect the AS 6 RS232 port to the instrument's RS232 port mounted on the inside back panel.
Download connector	25-pin D shell connector used only when downloading firmware for the AS 6. It connects to the RS232 port on the back of the instrument.

Pyris 6 TGA Parts

Name	Description
Pyris 6 TGA + AS 6 base plate + lid holder + lid + thermocouple cover	Base plate mounted by 3 screws to the instrument cover. It must be aligned to the furnace for proper operation.
Pyris 6 TGA + AS 6 base plate alignment tool	Tool to adjust the base plate with respect to the furnace.
Pyris 6 TGA firmware upgrade	Main firmware to operate the AS 6 with the Pyris 6 TGA.

Chapter 5
Pyris 1 TGA

Overview

Thermogravimetry is a branch of thermal analysis that examines the mass change of a sample as function of temperature or time as it is subjected to a controlled temperature program in a controlled atmosphere. Not all thermal events cause a change in the sample mass (e.g., melting, crystallization, glass transition), but there are some important exceptions such as desorption, absorption, oxidation, decomposition. Thermogravimetry characterizes the decomposition and thermal stability of materials under a variety of conditions and examines the kinetics of the physico-chemical processes occurring in the sample.

The Pyris 1 TGA is a computer-controlled analyzer that has been designed to take advantage of the enhanced features of Pyris Software for Windows. It is made up of two major components: a **microbalance** and a **furnace** element. There are two furnace options available: standard and high temperature. Most standard polymer applications are routinely performed with the standard furnace. The high temperature furnace is used for applications such as ceramics, metals, and geological studies.

The Pyris 1 TGA Thermogravimetric Analyzer is a new analyzer from Perkin Elmer. Its foundation is the TGS-2 and the TGA 7 instruments. Some of the new features of the Pyris 1 TGA are

- Faster furnace cooldown with the cooling fan above the furnace instead of underneath. This creates positive upward ventilation of the furnace during cooldown.
- Quick cool is a new furnace cooling system that incorporates a Teflon tube within the Tygon exhaust tube. The inner tube provides better air flow to the furnace while it is in the Cool position. It provides for faster cooldown times and increased throughput.
- A light on the sample loading tray, the sample illuminator, helps you load the stirrup onto the hangdown wire.
- Under the lower utility tray you can store items such as sample pans or reference samples.
- Built-in control panel has a two-line display that shows the temperature and the ordinate signal value. The buttons on the control panel can be used to raise, lower, or bring the furnace to the cool position. The menu items seen the two-line display can be used to control the antistatic device, the upper fan, the autosampler, or the furnace (e.g., clean).
- The sample gas interface has been repositioned for easier access.
- A direct reaction gas injection port on the optional furnace tube gets the reaction gas directly into the furnace tube.

- Tightly controlled balance environment gives the Pyris 1 TGA long-term ordinate stability. The weighing balance is housed in a thermostat-controlled enclosure that eliminates thermal gradient effects.
- The balance mechanism can be adjusted horizontally and vertically using external adjustment screws.
- Anticonvection iris prevents decomposition products and heat from the furnace from backstreaming into the balance chamber.
- The balance chamber dome has a new clamp down mechanism.
- The "quick release" furnace tube makes furnace tube cleanup easy. The furnace tube is attached to the furnace base by a clamp.
- Temperature control of the segmented furnace is optimized by the heater/sensor furnace technology. The platinum heater element is also the temperature sensor.
- The removable quartz furnace chamber sleeve regulates furnace convection by reducing the effective furnace volume. The reduced volume also enhances gas switching time because there is less furnace volume to displace.
- The antistatic device creates an ion stream which is an invisible curtain of charged particles that surrounds the sample loading area. This minimizes static cling between the sample pan and the furnace wall during sample loading and static drift during analysis. Loading fine, powdered, or static-sensitive samples is no longer a problem. The device can be controlled from the control panel in the software or the control panel on the instrument.
- Quasi-DTA signal enables the analyst to choose the calibration technique that is compatible with ISO 9000 by calibrating temperature with Curie point reference materials or with certified melting point reference materials.

In addition to these new features, there is now an autosampler designed for the R&D laboratory and the quality control/quality assurance environment. The autosampler increases the speed of analysis by being able to hold up to 20 samples ready for analysis. The autosampler enables samples to be prepared and run during the same shift. Using the Pyris Player feature of Pyris Software for Windows, you can create play lists that will run the samples in the autosampler tray and analyze the data automatically.

The features of the Pyris 1 TGA and autosampler are in presented in the following topics:

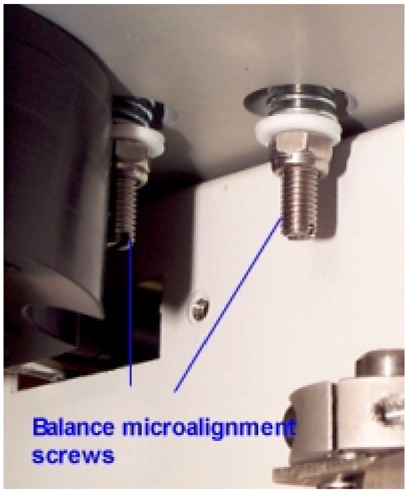
- **Instrument Overview**
- **Safety Precautions**
- **Leveling the Pyris 1 TGA**
- **Calibration**
- **Operating Variables**
- **Connecting Gas Supplies**
- **Maintenance**
- **Part Numbers**
- **Technical Specifications**
- **Updating the Firmware**
- **Autosampler**

Microbalance

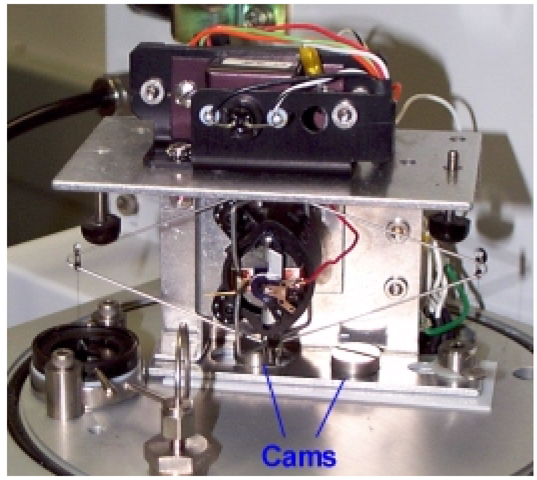
The microbalance of the Pyris 1 TGA operates as a high gain electromechanical servo system that permits detection of weight changes as small as 0.1 µg, with a maximum capacity of of 1300 mg. The null balance design uses a servo-controlled torque motor to automatically compensate for weight changes in the sample material. When a sample is placed in a sample pan and the pan is on the hangdown wire, the beam that supports the sample pan deflects. A beam position detector measures the deflection with an optical sensor and used current to return the beam to its original position. The amount of current necessary to maintain the system in the "null" state is directly proportional to the weight change in the sample. The current is amplified and filtered and then displayed in the display panel of the instrument in mg.

There are a few new features of this balance over the TGA 7:

- With a new design layout, noise on the balance weight signal is reduced enough to allow a 26-mg range in addition to the 130- and 1300-mg ranges.
- The capillary tubes through which the platinum hangdown ribbons go now have a flared top to help stabilize the ribbon. The ribbon has a bead at the top that seats down in the flare.
- The balance is under constant temperature control instead of being temperature compensated with a thermistor. The balance is held at a constant 42°C by the TGA balance temperature control board which is mounted on the terminal block side of the balance assembly.
- You can adjust the position of the balance mechanism, and therefore the position of the hangdown wire, by using the X-Y microalignment system. This system allows you to move the balance by using the two adjustment screws underneath the balance plate so you do not have to disturb the balance by removing the dome. The left screw (the one closest to the antistatic device) adjusts the vertical or back and forth position and the right screw adjusts the horizontal or left to right position.



The balance can also be adjusted within the mechanism by **removing the dome** and placing a screwdriver down from the top into the mechanism to the two cams on the plate:

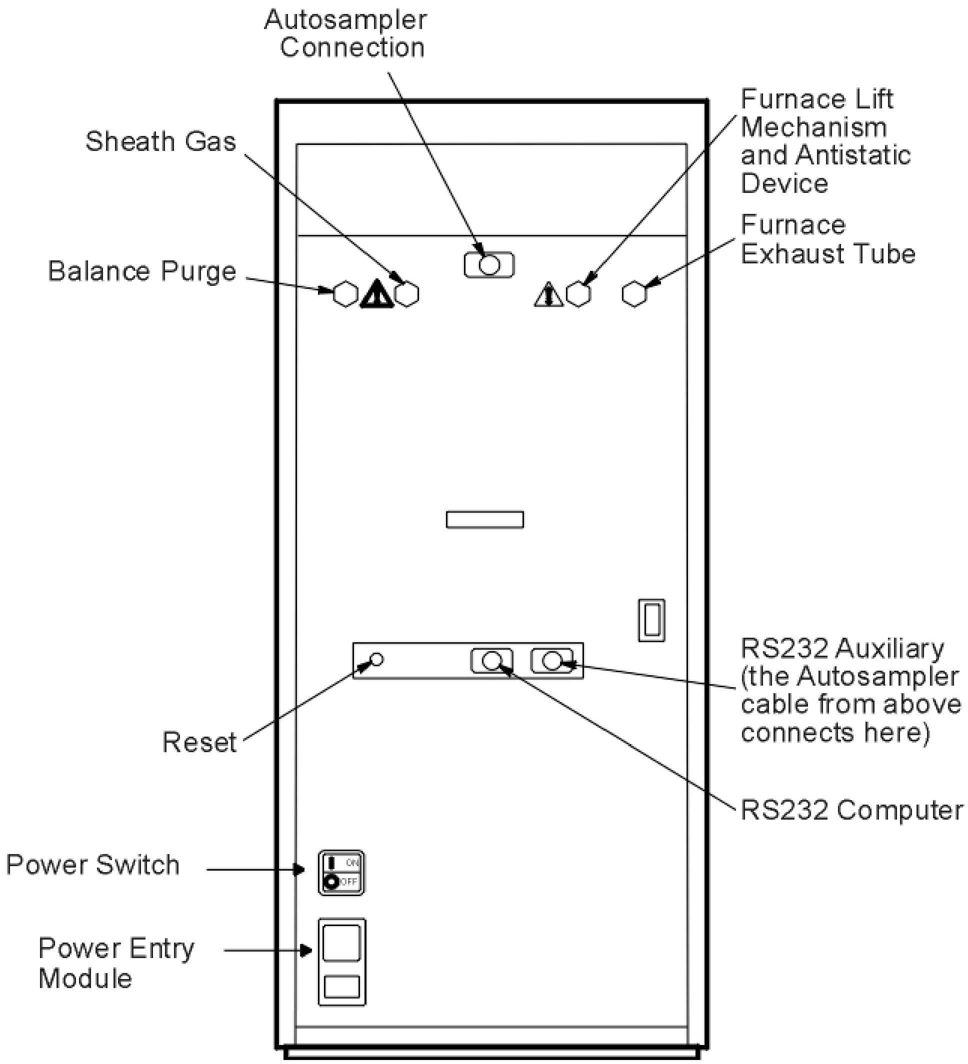


The balance slides on the white Teflon piece between the balance foot and the base plate. The left cam (as you face the balance) adjusts the vertical position and the right cam adjusts the horizontal position.

See the topic **Adjusting the Balance Mechanism** below.

Rear Panel

The rear panel of the Pyris 1 TGA contains the following connections and controls:



The **Reset** button reinitializes the instrument as if you turned it on and then off. If the system seems to hang up, it is recommended that you press **Ctrl - Alt - Del** on the computer to select the Task Manager. At the Task Manager select Pyris Software for Windows as the task to end. Restart the Pyris software to reinitialize the instrument.

The Power Entry Module contains a popout drawer that contains two fuses. If a fuse should blow, you can easily **change the fuse**.

The N2 or compressed air used for the furnace lift mechanism is also used for the **antistatic device**.

Changing a Fuse in the Pyris 1 TGA

To change a fuse in the power entry module of the Pyris 1 TGA:

1. Make sure that the instrument is switched OFF.

2. Disconnect the power cord from instrument.
3. Remove the fuse drawer by pressing up on the small tab below the fuse drawer. Use a small flat blade screwdriver to press the tab up. The drawer should pop out partially.

1. Remove the fuse drawer. There should be two 10.0 A fuses inside.
2. Remove the defective fuse and replace it with a new fuse from the Spares kit (P/N 0998-1753).
3. Replace the fuse drawer. Push the drawer back into place.
4. Reconnect the power cord and power on the instrument.

Control Panel

The top cover of the Pyris 1 TGA has a control panel containing 9 buttons or keys and a LED display:

Panel Display

- The Pyris 1 TGA control panel is a 2-line, 20-character-per-line vacuum fluorescent display. When the instrument is powered on, the display reads:

PERKIN ELMER
PYRIS 1 TGA

After bootup, the nonrun default items are displayed:

37.19C –22.508mg

. . .

- The top line displays real-time temperature in °C and the weight signal in mg. The bottom line shows the Menu symbol (. . .). This symbol indicates that the **Menu** button can be pressed to access special menus.
- When a run is in progress, the top line will display the temperature and weight values along with the ↕ symbol which represents the green **Scroll** buttons:

and the status line changes messages indicating what is going on, e.g.,

02 Heat to 300C . . .

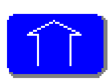
This shows that step 2 of the method program is the current step.

- At the default display, press the **Menu** button to get the first item in the Main Menu: TGA OPERATION. Press the **Scroll Up** button to scroll through the Main Menu. The other three items in the Main Menu are SELECT LANGUAGE, FIRMWARE VERSION, and AS OPERATION. In a submenu item such as Furnace in TGA OPERATION, press the **Menu** button to see the first of the Furnace menu items.
- Display of the ↕ symbol means that you can use the **Scroll** buttons to scroll through menu items on the display. For example, there are two items in the Furnace menu: FURNACE TEMPERATURE and FURNACE MOVEMENT. If you press the **Scroll Up** arrow at the FURNACE TEMPERATURE display, you get to FURNACE MOVEMENT.

Control Keys

Below the LED display on the control panel are the control keys that you can use to interact with the Pyris 1 TGA. The instrument ships with key labels in six languages. Affix the label appropriate for your lab.

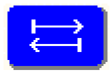
Raise Furnace



This button on the Pyris 1 TGA control panel moves the furnace into the Raised position. It is equivalent to the **Raise Furnace** button on the Pyris Software for Windows' control panel. If the furnace is in the Cool position, pressing the Raise Furnace button makes the furnace swing out to the Lowered position and then raises it up to engage the ball joint. If your analyzer has an autosampler, make sure that it is in the Safe position first before pressing the Raise Furnace button.

Lower Furnace

This button on the Pyris 1 TGA control panel moves the furnace into the Lowered position. It is equivalent to the **Lower Furnace** button on the Pyris Software for Windows' control panel. If the furnace is in the Cool position, pressing the Lower Furnace button makes the furnace swing out to the Lowered position. If the furnace is in the Raised position, Lower Furnace brings the furnace down.

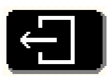
Cool Furnace 

This button on the Pyris 1 TGA control panel will lower the furnace to the Lowered position and then into the Cool position if in the Raised position, or will swing the furnace into to the Cool position if in the Lowered position. Pressing this button is equivalent to clicking on the Cool Furnace button on the Pyris Software for Windows' control panel.

Stop 

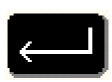
When you select this button during a run, the run terminates immediately and a Run Stopped message appears on the bottom line of the display. The final method step number flashes on the default display.

Within a special menu, Stop can be used to terminate a particular action, e.g., stop the furnace cleaning procedure.

Exit 

This key is used in four ways:

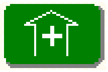
1. Exit a secondary menu display without performing any action and return to the display from which you came, i.e., the parent user-selected display.
2. Exit a user-selected display without performing any action and return to the default display.
3. Exit a secondary special menu without performing any action and return to the parent special menu.
4. Exit a special menu without performing any action and return to the default display.

Enter 

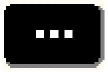
1. Press Enter to move between the default display and user-selected displays.
2. Press Enter to move between the default display and special menus.
3. Within secondary menus, Enter can be used to accept a condition, start an action, or enter a lower-level submenu.

Scroll Down 

This button on the Pyris 1 TGA control panel is used to scroll through menu items. After pressing the Menu button at the default display to access the first Main Menu item, TGA OPERATION, repeatedly pressing the Scroll Down button scrolls through the Main Menu. This includes TGA OPERATION, SELECT LANGUAGE, FIRMWARE VERSION, and AS OPERATION. The items are displayed in the opposite order as that of Scroll Up. If the ↕ symbol appears in the display, it indicates that you can use the Scroll Down button.

Scroll Up 

This button on the Pyris 1 TGA control panel is used to scroll through menu items. After pressing the Menu button at the default display to access the first Main Menu item, TGA OPERATION, repeatedly pressing the Scroll Up button scrolls through the Main Menu. This includes TGA OPERATION, SELECT LANGUAGE, FIRMWARE VERSION, and AS OPERATION. If the ↕ symbol appears in the display, it indicates that you can use the Scroll Up button.

Menu 

From the default display on the analyzer's control panel:

22C –120.00mg

. . .

the **Menu** button is used to access the four items that the Main menu comprises:

- TGA OPERATION
- FIRMWARE VERSION
- SELECT LANGUAGE
- AS OPERATION

It is also used to access items in submenus. The **Menu** button is functional whenever the Menu symbol (**. . .**) is displayed on the bottom line of the control display. The action that results from pressing **Menu** depends on what is currently displayed.

TGA OPERATION

After accessing the Main Menu item TGA OPERATION, press the **Menu** button again to access the TGA OPERATION menu. Use the **Scroll Up** or **Scroll Down** button to scroll through the TGA OPERATION menu, which consists of the following items:

Antistatic Device

Access the Antistatic Device item on the TGA OPERATION menu by pressing the **Menu** button when TGA OPERATION is displayed. With Antistatic Device displayed on the control panel, press the **Menu** button to display:

AutoMode Disabled ↕

Enable AutoMode? ↵

If you wish to enable the automode feature of the antistatic device, press the **Enter** button. While in automode, the antistatic device is ON while the furnace travels up to the ball joint. Before it reaches the ball joint, the furnace stops for about 15 seconds while the antistatic device sprays the ball joint area. The furnace then continues traveling and the antistatic device turns OFF when the furnace reaches the joint. It is also ON when the furnace travels to and from the Cooling position. It is OFF when the furnace is in the Cooling position. If you wish to see the other item on the Antistatic Device menu, press the **Scroll Up** button to display:

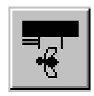
Antistatic Off ↕
Turn On? ↵

If you want to turn the antistatic device ON, press the **Enter** button. When the status of the antistatic device is ON, rather than AutoMode Enabled, the antistatic device travels all the way up to the ball joint without stopping. It stays on while the furnace is all the way up and after it has been lowered to the Lowered position. It remains on for 10 minutes and then shuts off. The status of the Antistatic Device is displayed in the Status Panel in the software and on the instrument's control panel.

If you press the **Enter** button at either item above, the response is accepted and you exit the Main Menu and return to the default display. If you do not want to press the **Enter** button and want to get out of the Antistatic Device menu, press the **Exit** button. You return to the default display.

NOTE: The antistatic device must be ON when using the autosampler.

The antistatic device can also be controlled from the software's control panel via the **Antistatic Device** button:



Clean Furnace

To access the Clean Furnace item in the TGA OPERATION menu, press the **Scroll Up** button at the Antistatic Device display. The display will change to

CLEAN FURNACE
Ready to Clean ↵

If you wish to clean the furnace at this time, press the **Enter** button. The furnace swings to the Cooling position as the temperature goes up to 950°C. The furnace is in the Cooling position for cleaning so the fan above the furnace can pull out any fumes that may be created. They would exit through the hole at the top of the instrument. This should be positioned beneath a fume hood if necessary.

If you do not want to clean the furnace at this time, press the **Exit** button to return to the default display.

Fan Mode

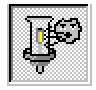
To access Fan Mode in the TGA OPERATION menu, press the **Scroll Up** button twice when Antistatic Device is displayed. When FAN MODE is displayed, press the **Menu** button to display the following:

Fan Is in AutoMode
Turn AutoMode Off? ↵

If the fan is off, the display will prompt if you would like to turn AutoMode on. The upper fan of the Pyris 1 TGA blows on the outside of the furnace tube when it is in the Raised position. The fan creates a gradient for the furnace to help it attain ambient temperature. The temperature gradient is needed in order for the motor to stay on and the furnace to run. Press the **Enter** button to turn AutoMode on or off. If you do not want to turn the fan's automode on or off at this time, press the **Exit** button to return to the default display.

NOTE: The upper fan is NOT used when using an autosampler.

The upper fan can also be controlled from the software's control panel via the **Upper Fan** button:



Furnace

Access the Furnace option on the TGA OPERATION menu by pressing the **Scroll Up** button three times when Antistatic Device is displayed. Once FURNACE is displayed, press the **Menu** button to display to first of two items in the FURNACE menu:

FURNACE TEMP ↕
HOLD WHEN LOWERING? ↵

Press the **Enter** button if you want to have the furnace temperature hold at its current temperature while the furnace is going to the Lowered position. You may need to lower the furnace by pressing the **Lower Furnace** button. The furnace may need to be down for a short time before you send it to the Raised position. Holding the furnace temperature when lowering saves time. After pressing the **Enter** button, you are returned to the default display.

Press the **Scroll Up** or **Scroll Down** button at the FURNACE TEMP display to see the other item in the FURNACE menu:

FURNACE MOVEMENT ↕
INHIBIT MOVEMENT? ↵

Press the **Enter** button if you want the movement of the furnace to be inhibited, i.e., it will not move if you press the **Raise Furnace** button or the **Lower Furnace** button on the control panel. It also will not move if you try to start a play list from the software. If you press **Enter**, you return to the default display. If you do not want to choose either of these items, press the **Exit** button at either item.

FIRMWARE VERSION

To access the firmware version of the Pyris 1 TGA, at the default display on the control panel press the **Menu** button; TGA OPERATION is displayed. Then press the **Scroll Up** button. The current version of the firmware in the analyzer is displayed. You can also check the firmware version in the Pyris 1 TGA Configuration dialog box. Select Configure Analyzer from the **Start Pyris** button menu. Highlight Pyris 1 TGA from the list of analyzers, then click on **Edit**. The Pyris 1 TGA Configuration screen appears.

SELECT LANGUAGE

AT the default display, press the **Menu** button; TGA OPERATION is displayed. Press **Scroll Up** twice to display

22C –120mg ↕
SELECT LANGUAGE . . .

Press the **Menu** button to display the first item in the Select Language menu. Use the Scroll buttons to scroll through the rest of the list of languages available to use on the control panel display:

- English
- French
- German
- Italian

- Japanese
- Spanish

AS OPERATION

To access the AS OPERATION item on the control panel display's Main Menu, press the **Menu** button at the default display; then press the **Scroll Up** button three times. The display will appear similar to the following:

22C –120mg ↕
AS OPERATION . . .

Press the **Menu** button to access the AS OPERATION menu. The display changes to

AS OPERATION ↕

Load Sample . . .

Press the **Menu** button to display

Load Sample ↕

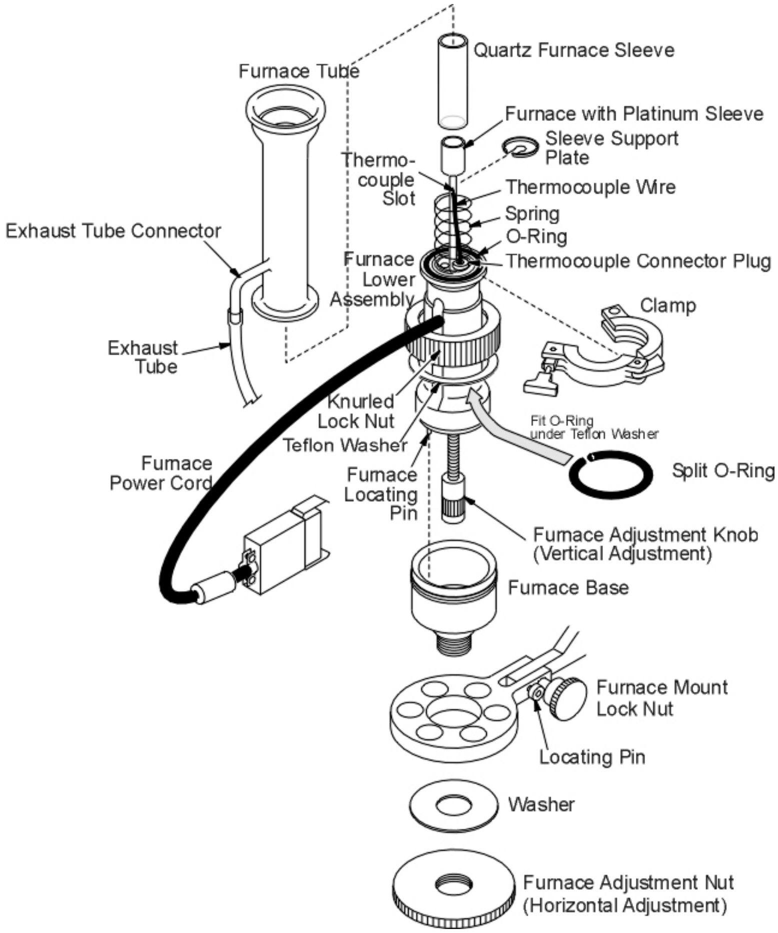
From Position X ↵

Use the **Scroll** buttons to select the position from which to load a sample from the autosampler. Once you have selected the position, press the **Enter** button. You return to the default display as the sample is loaded. If you do not want to load a sample, press the **Exit** button to return to the default display. This is another way to load a sample; you could also click on the **Autosampler Control** button in the software's control panel to display the Autosampler Control dialog box.

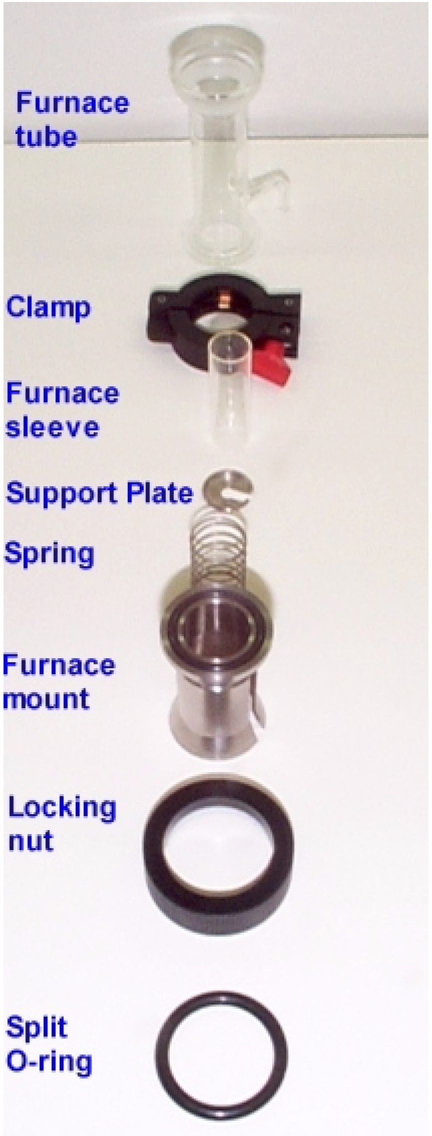
Standard Furnace

The standard furnace allows operation of the Pyris 1 TGA from subambient to 1000°C. The furnace uses a microfurnace that has a low thermal mass for quick cooling and equilibration. The platinum heater element acts as a resistance temperature detector under tight feedback control. The furnace detects its own temperature and supplies power to heat the sample. Accurate program temperature control reduces overshoot to 0.2°C at 100°C/minute which results in stepwise analysis precision. The sample temperature is sensed by a chromel–alumel thermocouple.

Parts of the Standard Furnace



The photo below shows the parts of the furnace that you would have to remove to replace the furnace or clean the furnace tube.



Standard Furnce Tube

Part Number N537-0459

Clamp

Part Number 0992-3447

Pyrex Furnace Insert

Part Number N537-6556

Support Plate

Part Number N537-6522

Spring

Part Number N537-6521

Furnace Mount

Part Number N537-6540

Locking Nut

Part Number N537-6562

Split O-Ring

Part Number 0990-2131

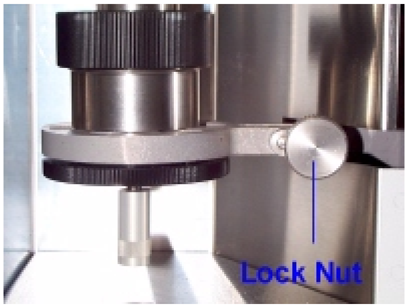
Adjusting the Standard Furnace

When your Pyris 1 TGA with standard furnace and **no autosampler** is installed by a service engineer, he performs a series of adjustments in order to optimize its performance. If you need to remove the furnace and/or furnace tube, perhaps to clean it, you will have to perform these steps to ensure proper operation of the instrument. These include:

- **Aligning the Standard Furnace**
- **Adjusting the Height of the Furnace**
- **Centering the Sample Pan**
- **Adjusting the Balance Mechanism**
- **Adjusting the Convection Iris**

Aligning the Standard Furnace

1. Power on the Pyris 1 TGA. The furnace will go to the Cooling position.
2. Press the **Lower Furnace** button on the instrument's control panel. The furnace will swing out to the Lowered position.
3. Remove the lock nut from the furnace mounting assembly and swing the furnace assembly down so that it is at an angle with the instrument.

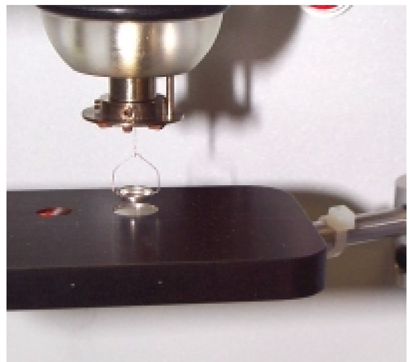


4. Look down into the furnace tube and see if the furnace is exactly centered within the tube. If it is not, loosen the clamp and move the quartz tube around until the furnace is centered. Tighten the clamp.
5. Raise the furnace assembly back up and reinsert the lock nut into the furnace mounting assembly and tighten.
6. Turn off the antistatic device by pressing the **Menu** button on the instrument's control panel three times. At the Antistatic Device display, press the **Scroll Up** button to display the status of the antistatic device. If it is ON, press the **Enter** button to respond Yes to Turn Off? If it is already OFF, press the **Exit** button to return to the default display.
7. Raise the furnace by pressing the **Raise Furnace** button on the control panel. If the furnace is properly aligned, the furnace tube should move smoothly into position around the upper ball joint. You may also want to use the sample loading platform to stop the furnace from rising so that you can make minor adjustments as the furnace tube meets the ball joint.

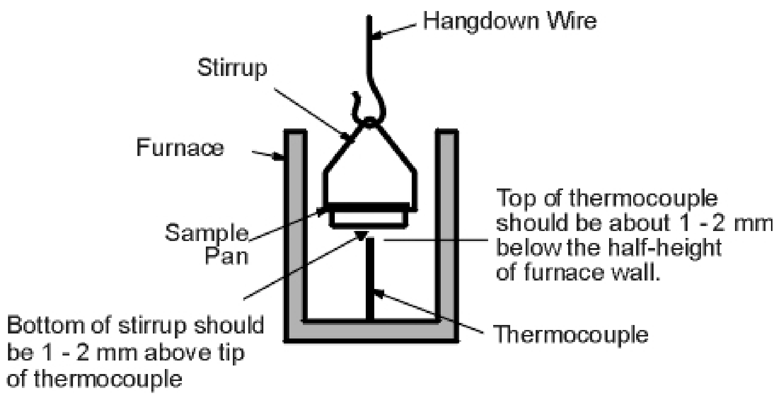
   The sample loading platform can be used to control the movement of the furnace while it is ascending. When the platform is in the safe position (to the right), the furnace moves up toward the ball joint. When the platform is not in the safe position, the furnace will not move. Move the platform back and forth to stop and start the motion of the furnace in small increments.
8. To adjust the horizontal position of the furnace tube, slightly loosen the furnace adjustment nut at the base of the furnace mounting assembly.
9. Press the **Lower Furnace** button on the instrument's control panel.
10. Press the **Raise Furnace** button on the instrument's control panel.
11. As the furnace gets close to the ball joint, position the furnace assembly so that the furnace tube moves smoothly onto the ball joint. When the tube and the ball joint appear to be aligned, allow the furnace to go all the way up by pushing the sample loading platform into the safe position and leaving it there.
12. Adjust the furnace assembly manually until it appears that the furnace is aligned correctly (i.e., the furnace tube is perpendicular to the table top). Lock the furnace assembly into position by tightening the furnace adjustment nut at the base of the furnace mounting assembly.

Adjusting the Height of the Furnace

1. Bring the furnace to the Lowered position by pressing the **Lower Furnace** button on the instrument's control panel.
2. Place a stirrup onto the sample loading platform. Place a sample pan in the stirrup. Hang the stirrup with sample pan onto the hangdown wire by using the sample loading platform to guide the stirrup onto the hangdown wire and gently lower the platform so the stirrup hangs from the wire.



3. Press the **Raise Furnace** button.
4. Check the position of the stirrup with respect to the top of the thermocouple. The bottom of the stirrup should be approximately 1 – 2 mm above the tip of the thermocouple. The tip of the thermocouple should be 1 – 2 mm below the halfway point of the furnace wall.



5. Adjust the height of the furnace using the furnace height adjustment knob located below the furnace adjustment ring. Turn the knob clockwise to raise the furnace and counterclockwise to lower the furnace.
6. Make sure that the sample pan hangs in the center of the furnace. It is important that the sample pan does not touch the side wall of the furnace or the tip of the thermocouple.

Centering the Sample Pan

If the sample pan is not centered, do the following:

1. Press the **Lower Furnace** button.
2. Check the status of the antistatic device, either on the Status Panel of the Pyris software, if it is on, or by going into the instrument's menu. Press the Menu button three times. At the

Antistatic Device display, press the **Scroll Up** button; the status of the antistatic device is displayed. If it is not Auto Enabled, press the **Enter** button to enable the device. Press the **Exit** button to return to the default display.

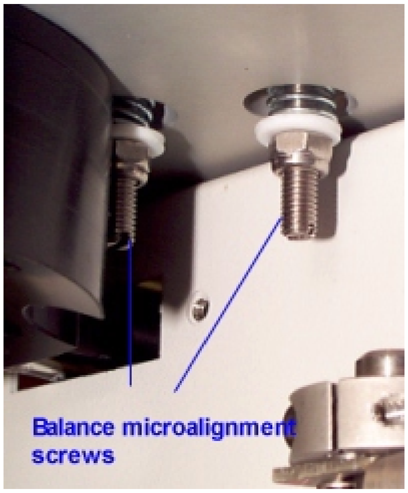
3. Press the **Raise Furnace** button. The furnace will stop below the ball joint for about 15 seconds. It will then finish its trip and engage the ball joint.

4. Loosen the furnace adjustment ring and carefully move the furnace so that the sample pan is in the center of the furnace. When it is centered, tighten the ring.

CAUTION: If the sample pan cannot be centered in the furnace using the adjustment procedure above, then the balance mechanism has to be adjusted.

Adjusting the Balance Mechanism

If the hangdown wire cannot be centered by moving the furnace using the furnace adjustment ring, you will have to adjust the position of the balance, and therefore the position of the hangdown wire, using the two X-Y microbalance adjustment screws.



1. If not already, enable the antistatic device automode. Press the **Menu** button three times. At the Antistatic Device display, press the **Scroll Up** button; the status of the antistatic device is displayed. If it is not Auto Enabled, press the **Enter** button to enable the device. DO NOT press the **Exit** button yet.

2. Make sure that the upper fan is off; otherwise, the hangdown wire will swing. Press the **Scroll Up** button twice. With the Fan mode displayed, press the **Menu** button to see the status of the fan. If Automode is ON, turn it OFF by pressing the **Enter** button. Press the **Exit** button to return to the default display.

   Before proceeding, you may want to check that the **antistatic device is operating properly**.

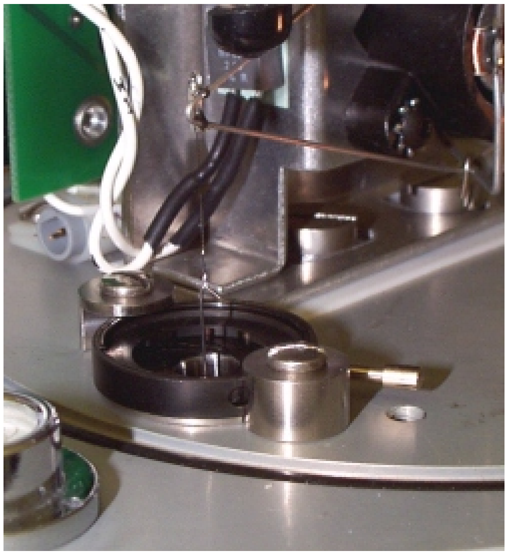
3. Press the **Raise Furnace** button.

4. If the hangdown wire needs to be centered, carefully use a screwdriver to adjust the position of the balance, and, therefore, the position of the hangdown wire. The left-hand screw (that closest to the antistatic device) adjusts the Y position and the right-hand screw adjusts the X position.

Once the sample pan is centered within the furnace tube, it is important that the hangdown wire not touch the anticonvection iris when it is closed. The iris prevents reaction gases from rising into the balance chamber from the furnace. After adjusting the balance mechanism, the hangdown wire may no longer be centered within the iris.

Adjusting the Convection Iris

1. Lift the top cover of the analyzer up to access the balance dome.
2. Remove the balance dome by turning the locking devices and pulling the arms out of the groove in the dome. Lift the dome straight up and away from the balance. The iris is now exposed.
3. Open the iris all the way by pushing the arm clockwise.
4. Loosen the screws on the two stands that hold the iris in place.
5. Move the iris around until the hangdown wire is centered in the iris.



6. When the hangdown wire is centered, tighten the screws on the stands. Do not overtighten; the iris arm will not be able to move.
7. Close the iris as far as possible without it touching the hangdown wire by turning the lever counterclockwise.
8. Replace the dome back onto the balance plate and lock in place.
9. Lower the access cover.

Check the Antistatic Device Operation

When the antistatic device is in AutoMode, i.e., AutoMode is enabled, and you press the **Raise Furnace** button, the antistatic device turns on (the indicator light illuminates) and the furnace travels up toward the ball joint. It should stop about an inch from the ball joint and remain there for about 15 seconds. During this time, the antistatic device emits an ion spray to reduce any static around the ball joint. The furnace should then continue up and the antistatic device should turn off.

If the furnace travels up in a halting manner or does not continue to go up all the way and goes back down, the antistatic device's gas needs to be adjusted. When the gas flow is adjusted properly, you should be able to hear it slightly. If the valve that controls the antistatic device is closed too much, the furnace does not go up all the way; it goes half way and then travels back down. There is too much backflow in this case.

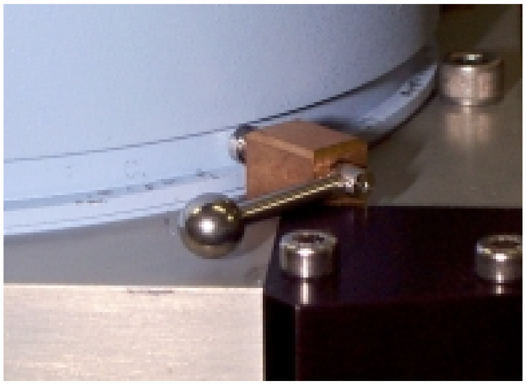
To adjust the antistatic device's gas flow, perform the following steps:

1. Remove the cover by removing the four screws (two on the bottom of each side) and slide the cover up and off.
2. On the left-hand side on the top of the instrument you will see four solenoids. The one closest to the front of the instrument controls the antistatic device. Turn the frit on that solenoid to adjust the gas flow.

3. Press the **Raise Furnace** button again and observe how the furnace travels up.
4. Continue to do this until the furnace travels correctly.
5. Replace the cover on the analyzer.

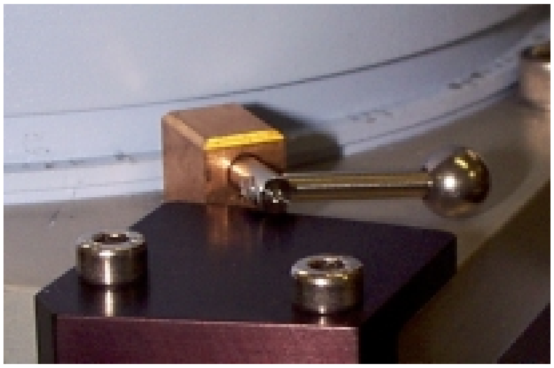
Removing the Balance Dome

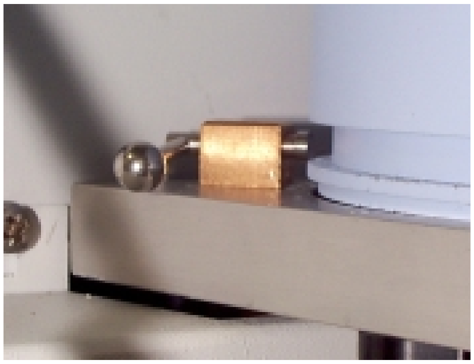
The balance dome is held in place by two L-shaped locking mechanisms. They each have a ball on one end to help you lock and unlock them and a cylinder on the other end that fits into the dome's groove and holds the dome down. Flip the locks over to loosen them and then pull out the lock's arm from the groove on the dome. Grasp the dome with two hands and carefully lift it straight up until it clears the balance mechanism.



Front locking mechanism in the locked position.

Back locking mechanism in locked position.





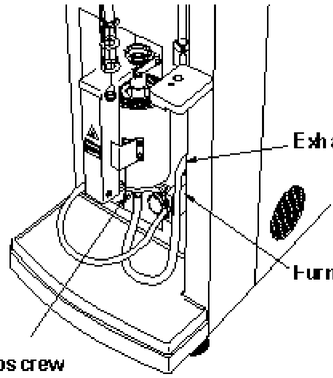
Front locking mechanism in open position. **Back locking mechanism in locked position.**

High Temperature Furnace

The high temperature furnace allows operation of the Pyris 1 TGA from 50°C to 1500°C. This furnace uses platinum / 30% rhodium heating element and can be operated at scanning rates of 0.1°C/min to 50°C/min. The high temperature furnace provides excellent temperature control and precision (±5°C) for even the most demanding applications. A sensitive platinum/10% rhodium – platinum thermocouple in close proximity to the sample measures the sample temperature during an analysis.

Adjusting the High Temperature Furnace

1. Power on the analyzer.
2. Press the **Lower Furnace** button on the instrument's control panel. The furnace is sent to the Lowered position if not there already.
3. To adjust the horizontal position of the furnace, slightly loosen the three thumbscrews that lock the furnace mounting assembly in place.



4. Raise the furnace by pressing the **Raise Furnace** button on the instrument's control panel. If properly aligned, the furnace tube should move smoothly into position around the upper ball joint. You may want to use the sample loading platform to stop the furnace from rising so that you can make minor adjustments as the furnace tube meets the ball joint.

   The sample loading platform can be used as an aid to control the movement of the furnace while it is rising. When the platform is in the safe position, the furnace moves up toward the ball joint. When the platform is not in the safe position, the furnace will not move.

5. Press the **Lower Furnace** button.
6. Press the **Raise Furnace** button.

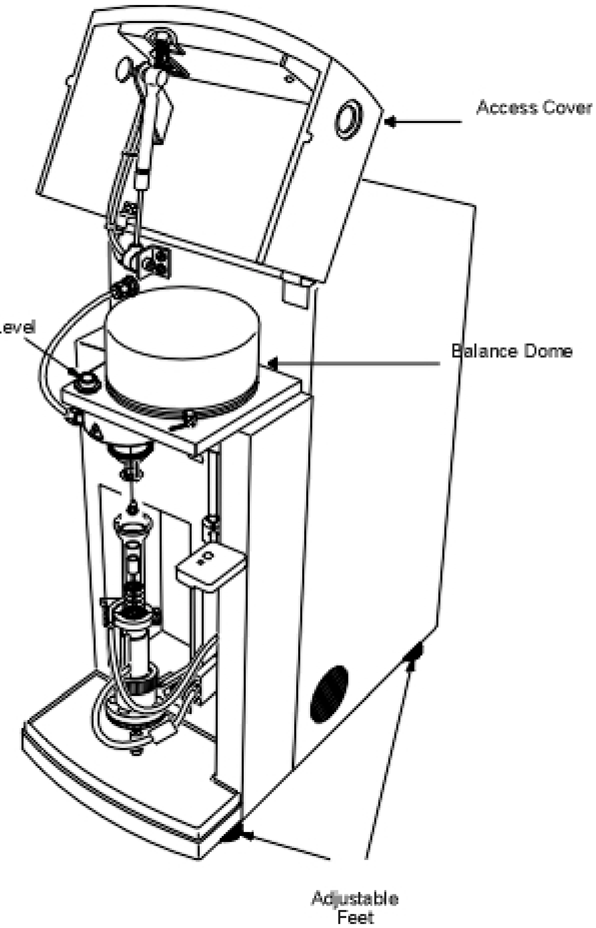
NOTE: If the furnace does not move when you press the Raise Furnace button, you may have to increase the pressure to the furnace lift mechanism. If the furnace moves too quickly, decrease the pressure slightly. Do not exceed a pressure of 241.5 kPa (35 psi).

7. As the furnace tube gets close to the upper ball joint, position the furnace assembly so that the furnace tube will move smoothly onto the ball joint. When the tube and ball joint appear to be aligned, allow the furnace to go all the way up by putting the sample loading platform in the safe position and leaving it there.
8. Adjust the furnace assembly by hand until it appears that the furnace is aligned correctly (i.e., the tube is at a right angle to the table top).
9. Lock the furnace assembly into position using the three thumbscrews at the base of the furnace mounting assembly.

Leveling the Pyris 1 TGA

Before you start using your Pyris 1 TGA for analysis, you must make sure that it is level.

1. Lift the access cover of the instrument to expose the balance dome.
2. Locate the level mounted in front of the balance chamber on the left-hand side.
3. Locate the adjustable feet.



4. Turn each foot counterclockwise until it is screwed all the way into the bottom of the analyzer.
5. Position the analyzer in the exact spot on the bench where you will be using it.
6. Check to see if the analyzer rocks on the bench or if one foot is not touching the bench. Starting with that foot, turn the foot clockwise until it is touching the bench. Once all the feet are touching the bench, make sure that the analyzer is steady.
7. Look at the level to see if the bubble is in the center. If it is not, slowly turn the knobs of the adjustable feet until the bubble is centered in the level. The Pyris 1 TGA is level when the bubbled is centered inside the ring.
8. Once the analyzer appears to be level, make sure all of the feet are in contact with the bench and that the analyzer does not rock.

Safety Precautions

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

The following precautions must be observed when using the Pyris 1 TGA Thermogravimetric Analyzer:

- Never turn the computer off until the following message appears:

  It is now safe to shut off your computer.

- Never press the Reset button on the computer if the Pyris software appears to malfunction. Press the **Ctrl–Alt–Del** keys simultaneously and select the Task Manager. From the Task Manager, close the Pyris software.
- Never remove the outer instrument cover without shutting down the instrument and disconnecting its power cord from the power source.
- Only high-quality purge gases should be used. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for the removal of any moisture from the purge gases.
- Always observe the proper startup and shutdown procedures with the Pyris 1 TGA and all related instruments.
- Always maintain a positive purge flow (40 – 60 cc/min) through the balance chamber at all times. An inert gas such as nitrogen or argon is recommended as a balance purge.
- Do not raise the furnace when the Pyrex furnace tube has been removed. This will cause severe damage to the furnace and other components of the analyzer.
- Prior to performing any experiment or calibration procedure, be sure that the thermocouple currently installed is functioning properly. The thermocouple can be checked by programming the Pyris 1 TGA to 100°C and checking the temperature displayed in the status panel. The temperature should be close to 100°C if the instrument is calibrated.
- To prolong the life of the standard and high temperature furnaces, it is recommended that the automatic furnace cleaning procedure be performed routinely such as every 5 to 10 runs. Pyrolysis products should be burned off after every analysis that involves pyrolysis of a sample in the absence of air or oxygen.
- When the Pyris 1 TGA standard or high temperature furnace is heating, keep the protective plastic shield in the down position. This is for **nonautosampler** instruments. This will protect you from accidentally touching the furnace when it is hot.
- Do not attempt to move the Pyris 1 TGA while it is on. Wait until the power is off and the furnace cooling fan has stopped spinning before moving the instrument.
- Do not remove cables from the analyzer while it is on.
- Do not touch the opening on the furnace lift mechanism or insert objects into the opening.
- Do not use hydrogen gas with the Pyris 1 TGA since it is highly explosive.

See the following topics relating to safety and warnings about the Pyris 1 TGA

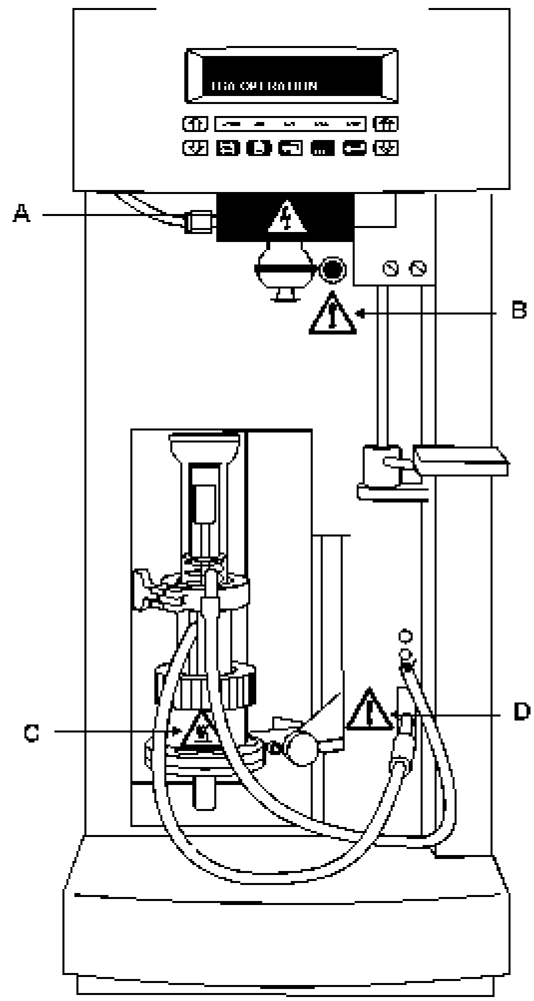
Warnings and Labels on the Pyris 1 TGA

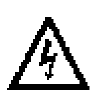
Environmental Requirements

Technical Specifications

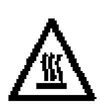
Warnings and Labels on the Pyris 1 TGA

The labels shown below appear on the front of the instrument:



A  WARNING: **Antistatic Device: To reduce the risk of electrical shock, do not place fingers or objects into holes.**

Avertissement: **Pour réduire les risques d'électrocution, ne pas mettre de doigts ou d'objets dans les trous.**

B  WARNING: **Hot Surface: Use care when working around this area to avoid being burned by hot components.**

Avertissement: **Surface chaude.**

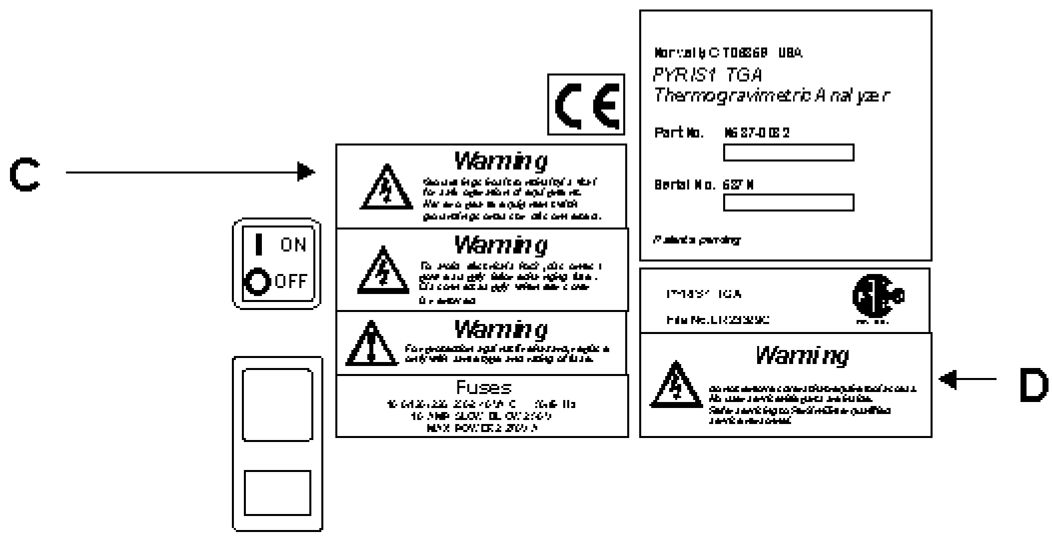
C  WARNING: **The antistatic device light is illuminated when the device is active. The furnace may be moving up or down. Do not place fingers or objects between the furnace and the device or underneath the device.**

Avertissement: **L'indicateur du dispositif anti-statique est allumé quand ce dernier est en marche. Au même moment, le four pourrait aussi être en montée ou en descente. Evitez de placer un doigt ou un autre objet entre le four et le dispositif anti-statique ou en-dessous de ce dernier.**

D WARNING: **Furnace exhaust tube connection. Use pinch clamp to secure tube to fitting.**

Connector is used for furnace power cable.

The following labels are affixed to the rear of the instrument as shown in the figures below:



A WARNING: **Use nitrogen or oxygen for Balance Purge and Sheath Gas. Gas flow for each shall not exceed 100 cc/min.**

Avertissement: **Employez de l'azote ou de l'oxygène comme gaz d'épuration pour la balance. Le débit de gaz ne doit pas dépasser 100 cc/min.**

B **WARNING:** **Compressed nitrogen or air pressure for furnace lift mechanism shall not exceed 240 kPa (35 psig).**

Avertissement: **La pression de l'air ou de l'azote comprimé utilisé pour le méchanisme d'élèvement du four ne doit pas dépasser 240 kPa (35 psig).**

C **WARNING:** **Grounding circuit continuity is vital for safe operation of equipment. Never operate equipment with grounding conductor disconnected.**

Avertissement: **La continuité du circuit de mise à la terre est essentielle pour le bon fonctionnement de l'appareil. N'utilisez jamais l'appareil lorsque le câble de mise à la terre est débranché.**

WARNING: **To avoid electrical shock, disconnect power supply before changing fuse. Disconnect supply whenever cover is removed.**

Avertissement: **Afin d'eviter les risques d'électrocution, mettez l'appareil hors tension avant de changer le fusible. Mettez l'appareil hors tension chaque fois que vous retirez le couvercle.**

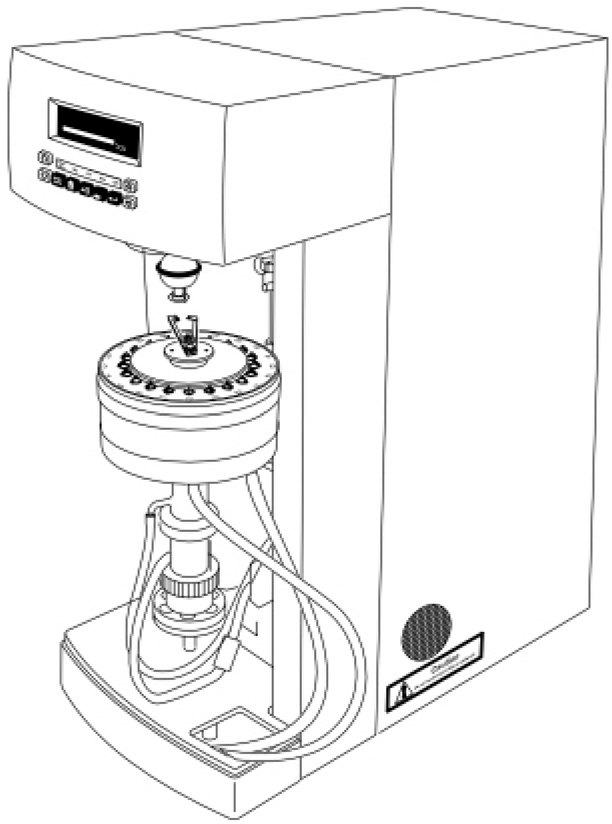
WARNING: **For protection against fire hazard, replace only with same type and rating of fuse.**

Avertissement: **Pour éviter tout risque d'incendie, ne remplacez les fusibles que par d'autres du même type et de la même puissance.**

D **WARNING:** **To reduce the chance of electrical shock, do not remove covers that require tool access. No user serviceable parts are inside. Refer servicing to Perkin Elmer qualified service personnel.**

Avertissement: **Pour réduire les risques d'electrocution, ne retirez pas les couvercles don't l'ouverture nécessite des outils. Ils ne protègent aucune pièce réparable par l'utilisareur. Contactez le personnel qualifié de Perkin Elmer.**

The label shown below appears on the right side of the instrument:

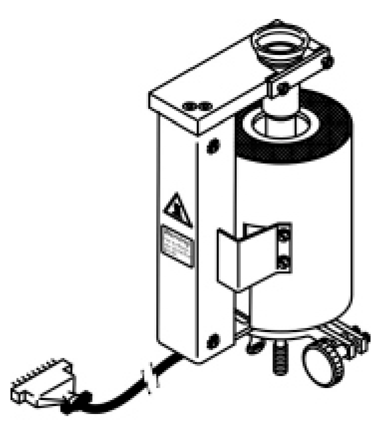


CAUTION: Do not restrict air intake or exhaust.

Attention: N'obstruez oas l'arrivée ou l'évacuation de l'air.

This instrument requires clearance around this opening to allow proper air flow.

The following label appears on the Pyris 1 TGA high temperature furnace:



WARNING: **Hot surfaces; use protective gloves.**

Avertissement: **Très chaud. Utilisez des gants de protection.**

The label on the visor indicates the following warning:

WARNING: **The Pyris 1 TGA uses high temperatures for sample processing. The protective cover, when fitted on the instrument, should be lowered to provide protection for the operator from contact with high temperature components and movements of the furnace assembly during normal operation.**

Avertissement: **Le Pyris 1 TGA emploie des températures élevées pour traiter les prises d'essai. Le couvercle de protection, lorsqu'il est monté sur l'appareil, doit être abaissé de façon à empêcher l'utilisateur de toucher des parties à température élevée, ou le four en cours de mouvement, lors d'un emploi normal de l'appareil.**

Environmental Requirements

The Pyris 1 TGA has been designed for indoor use only and should not be operated in an explosive environment.

The Pyris 1 TGA operates most efficiently under the following conditions:

- Ambient temperature range of 10°C – 35°C (50°F – 95°F). The instrument will remain safe when operated between 5°C and 40°C (41°F to 104°F).
- Ambient relative humidity between 20% and 80% without condensation.

- Altitude no more than 2000 m.
- Clean area free of dust, smoke, vibration, and corrosive fumes.
- On a bench area out of direct sunlight.
- Away from heating or cooling units or ducts.
- The instrument must be positioned so the power switch on the rear panel can be easily reached by the operator.
- There must be an adequate and stable power source for all system components.
- The Pyris 1 TGA is a highly sensitive, precision laboratory instrument. The environment in which it is used must be free of radio frequency interference sources which may affect the performance of the instrument. If performance is affected by RF fields, the instrument should be reoriented or relocated, or separated by a greater distance from the interfering source. Consult Perkin Elmer for additional assistance.

WARNING: If the heating of materials could lead to the liberation of hazardous gases, the use of a fume extraction system will be required.

WARNING: There may be possible hazards of explosion, implosion, or the release of toxic or flammable gases arising from the materials being heated.

WARNING: Do not mount the instrument of a surface of flammable material.

CAUTION: Before using any cleaning or decontamination method, except that recommended in this guide, the user should check with Perkin Elmer that the proposed method will not damage the instrument.

Hazardous Chemical Warnings

Before using any chemicals or solvents with the instrument, you should be thoroughly familiar with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage, and disposal. These recommendations are normally provided in the material safety data sheets (MSDS) supplied with the solvents.

WARNING: Some chemicals used with this instrument may be hazardous or may become hazardous after completion of an analysis. The responsible body (e.g., Lab Manager) must take the necessary precautions to ensure that the surrounding workplace and instrument operators are not exposed to hazardous levels of toxic substances as defined in the applicable Material Safety Data Sheets or OSHA, ACGIH, or COSHH documents. Venting for fumes and disposal of waste must be in accordance with all national, state, and local health and safety regulations and laws.

Some definitions of terms used above are

- **Responsible body:** "Individual or group responsible for the use and maintenance of equipment, and for ensuring that operators are adequately trained" [per IEC 1010-1, Amendment 2].
- **Operator:** "Person operating equipment for its intended purpose" [per IEC 1010-1, Amendment 2].
- **OSHA:** Occupational Safety and Health Administration (United States)
- **ACGIH:** American Conference of Governmental Industrial Hygienists
- **COSHH:** Control of Substances Hazardous to Health (United Kingdom)

Installation Category

This instrument is able to withstand transient overvoltage according to Installation Category II as defined in IEC 1010-1.

Pollution Degree

This instrument will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in IEC 1010-1.

Storage Conditions

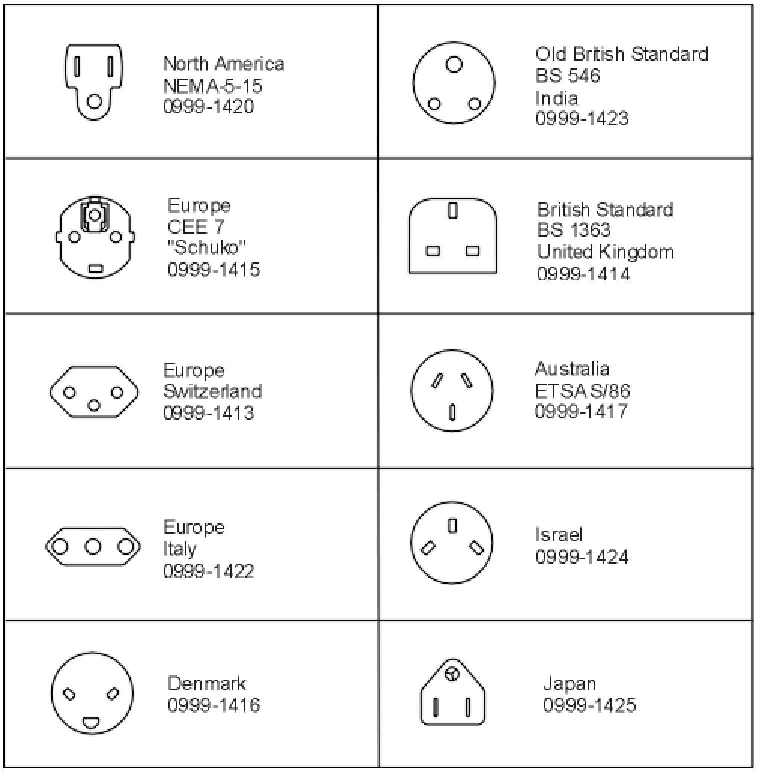
The Pyris 1 TGA may be stored under the following conditions:

- ambient temperature from –20°C to +60°C (–4°F to +140°F)
- ambient relative humidity from 20% to 80% noncondensing
- altitude in the range of 0 – 12,000 m

Electrical Safety Guidelines

Service on electrical components should be performed only by a qualified Perkin Elmer service engineer.

Be sure the power cord is the correct one for your laboratory. The Pyris 1 TGA power cord must be rated at or better than 100/120 VAC at 15 A or 150 AC at 10 A. The line cord used must meet the National Safety Agency's guidelines for the particular country.



Maximum Power: 2270 VA

Frequency: 50/60 Hz (±1%)

Fuses

NOTE: Service on electrical components should be performed only by a qualified Perkin Elmer service engineer. The fuses in the power entry module can be changed by the user.

The following fuses are in the power entry module to protect the analyzer:

100/120/220/230/240 VAC at 50/60 Hz: 10 A, Slow Blow, 3AB (P/N 0998-1753).

Use the 240-V selection on the power entry module for 230/240 V nominals.

Jumper SP1 on the main board must be changed according to the voltage:

100/120 VAC	Jumper 1–2
220/230/240 VAC	Jumper 1–3

The following fuses are on the main board:

P/N 0998-1617	**F107:** 5-A, Slow Blow, 3AG, 250 V
P/N 0998-1611	**F102, F103, F106:** 1 A, Slow Blow, 3AG, 250 V
P/N 0998-1614	**F104, F105:** 2 A, Slow Blow, 3AG, 250 V

The following fuse is mounted on the chassis in the top of the instrument to protect the antistatic device:

P/N 0998-1619 **F3:** 0.375 A, Slow Blow, 3AG, 250 V

Dimensions and Weight

The physical dimensions of the Pyris 1 TGA are as follows:

Weight

Pyris 1 TGA with standard furnace and autosampler	39.9 kg (88 lb)
Pyris 1 TGA with standard furnace and without autosampler	37.4 kg (82.5 lb)
Autosampler	2.5 kg (5.5 lb)
Standard furnace	0.45 kg (1 lb)
High temperature furnace	1.35 kg (3 lb)

Height: 67.3 cm (26.5 in.)

Width: 27.9 cm (11 in.)

Depth: 55.8 cm (22 in.)

Operating Variables

In thermogravimetric analysis, the object of an experiment is to record the change in weight of a sample as function of temperature or time using a constant heating rate or a more complex temperature program. Below are some techniques that can be used to obtain the most accurate data in a minimum amount of time with a Pyris 1 TGA.

Sample Preparation

The Pyris 1 TGA can analyze solid or liquid samples. Solid samples can be in the form of film, crystal, or grains. In some cases, you may want to chop or grind the sample to create a large surface area that is exposed to the purge gas atmosphere. In other cases, larger chunks or sections of sample may be placed directly into the sample pan or crucible for analysis.

Sample Size

The size of sample that can be analyzed ranges from <1 mg to 400 – 500 mg. In most cases, the typical sample size for TGA analysis is 2 – 50 mg. The type of transition or reaction that you expect to occur in the sample should dictate the amount of sample used. For example, in the case of polymer decomposition studies, sample sizes of 2 – 15 mg are recommended since very large weight loss values are associated with this type of experiment. If there will be a very small weight loss such as with water loss or solvent evaporation, larger sample sizes should be used. It is not uncommon to use samples as large as 40 – 50 mg.

Temperature Range

The temperature used for your experiment will depend on the type of samples to be run as well as the specific application performed. The Pyris 1 TGA has two furnaces. This permits operation over a broad temperature range. The standard furnace operates from subambient to 1030°C with a temperature precision of ± 2°C. This furnace as broad applications in the polymer, pharmaceutical, and organic chemical industries. For higher-temperature applications such as ceramics and metals, a high-temperature furnace is available. This furnace can operate from 50°C to 1500°C with a temperature precision of ± 5°C. The autosampler can be used with the standard furnace.

Because of the unique design of the Pyris 1 TGA, you can perform controlled heating or cooling experiments as well as constant temperature experiments as a function of time. You can even perform multiple-step experiments using any combination of heating, cooling, or isothermal segments, thereby allowing the performance of virtually any thermogravimetric application. In addition, there is also an **AutoStepwise Scan** available in the method program. This scan heats the sample rapidly between weight loss regions, and slows down or stops heating during rapid weight loss regions.

AutoStepwise Scan

Pyris Software for Windows' AutoStepwise Scan feature uses programmable criteria to automatically determine the start and end points of a weight loss. It also switches between various heating rates or isothermal steps in order to optimize the TGA analysis.

Autostepwise thermogravimetric analysis is a technique in which thermogravimetric reactions (such as vaporization causing weight losses) may be studied more accurately by reducing the scan rate in a temperature program or holding the temperature constant when such a reaction is detected. The weight loss is detected by monitoring the rate of weight loss during a temperature scan. If the rate of weight loss is greater than a preset value, the instrument will either continue to scan the temperature at a preset reduced rate (stepwise scan) or hold the temperature constant. If during this reduced rate segment the rate of weight loss is less than a preset value, then the normal scan is resumed.

In the Method Editor Program Page, the user can enter the criteria for an autostepwise TGA temperature program. The data items collected will be the same as for a standard TGA: sample temperature, program temperature, and sample weight. Autostepwise parameters can be used in conjunction with only a temperature scan, not an isotherm.

The entrance and exit criteria during an autostepwise run is performed by the TAC board inside of the Pyris 1 TGA. This prevents a lengthy time delay between detection or a reaction and reduction of the scan rate. The instrument's firmware provides "alerts" that are synchronized with the data stream so that the proper data segments can be generated.

It is possible, and generally the case, that more than one extra segment is generated as a result of a change in the rate of weight loss. This occurs as a result of the following:

1. The instrument detects a weight loss rate greater than the entrance criterion and causes an autostepwise segment to be generated.
2. After a period of time the rate of weight loss drops below the exit criterion and the original temperature scan rate is restored.
3. The rate of weight loss may again go over the entrance criterion and *another* autostepwise segment is generated.

An autostepwise segment in a method consists of the original starting and ending temperatures for the segment, the original scan rate for the segment, and the complete set of entrance and exit criteria.It is this single segment that may be expanded at run time into several scans or scans and isotherms. Methods using autostepwise segments may be recalled for reuse. However, **only** the original single segment (scanning segment before runtime) is displayed in the Method Editor.

Autostepwise for Pyris 1 TGA

1. It is possible to have more than one scanning segment with defined and used stepwise parameters.
2. There is no limit on the number of generated segments within a stepwise segment (i.e., the generated segments do not count toward the final total number of segments).
3. Criteria are sent to the Pyris 1 TGA as mg/min.
4. The criteria for each segment may be entirely different.

NOTE: You can change the final temperature of the original scan during data collection.

Scanning Rate

Scanning rates range from 0.1°C/min to 200°C/min for the standard furnace and from 0.1°C/min to 50°C/min for the high-temperature furnace. The exact scanning rate used will depend on the experiment and the end result you are trying to achieve. For example, most TGA experiments are performed at heating rates of 5°C/min to 50°C/min. However, there may be times when you want to heat or cool rapidly to a selected temperature and then hold isothermally or scan a controlled rate. In such cases, very fast heating or cooling rates (i.e., 100°C/min or 200°C/min) are typically used to quickly increase or decrease the sample temperature.

Sample Atmosphere

WARNING: Due to the highly explosive nature of hydrogen, it is recommended that it not be used with this instrument.

The atmosphere to which the sample is exposed is carefully controlled by the selection of the **sample purge or sheath gas** and flow rate. Recommended sheath gases are nitrogen, argon, helium, carbon dioxide, air, oxygen, or other inert or reactive gases, as long as they do not react with the furnace materials, thermocouple, or other analyzer components in which gases come into contact. Analyses are done at normal pressure or at reduced pressures.

In addition, sheath gases may be switched at any point during an analysis by using a **Gas Selector Accessory** or a **Thermal Analysis Gas Station**. A portion of an experiment (e.g., the pyrolysis of the polymer portion of a sample) may be performed in an inert gas such as nitrogen. The purge gas may then be switched to an active gas such as air or oxygen to selectively oxidize the other components in the sample such as carbon black.

Purge and Reactive Gases

The Pyris 1 TGA has connections for two purge gases: balance chamber and sample area (called the sheath gas). The balance purge and sheath gas lines connect at the rear of the instrument (click here for instructions on connections). In addition, there are connections for the **furnace lift pneumatic gas**, which also serves as the antistatic device gas, and the exhaust gas from the furnace tube.

The balance chamber **must** be purged; the sheath gas to purge the sample/furnace area is optional. The balance purge gas supply flows through the balance chamber, through the iris and down the center of the furnace tube into the furnace. Some escapes into the ball joint area from 3 pin holes in the convection tube just underneath the balance plate. The rest goes out of the furnace tube through an exhaust line attached to the tube. Note that the exhaust line from the furnace tube to the instrument contains a piece of Teflon tubing inside. This is the "quick cool" line. When the furnace is in the Cool position, air blows through the tubing to help cool down the furnace more quickly. The recommended balance purge gas is an inert gas such as nitrogen or argon with a minimum of purity of 99.9%. The gas must be dry. The recommended flow rate for the balance purge gas is 40 – 60 mL/min. These rates must be realized by an output pressure of 27,000 – 41,400 Pa (4 – 6 psi) on the output side of the regulator when using the type A flow restrictor.

The sheath gas can be nitrogen, argon, helium, carbon dioxide, air, oxygen, or other inert or reactive gases.

> **NOTE**: The gas used should **not** be reactive with the antistatic device or with the materials used in the construction of the Pyris 1 TGA, including Pyrex, stainless steel, and platinum.

A size 1A cylinder equipped with a suitable regulator is recommended.

The sheath gas purges the furnace area. This gas bypasses the balance chamber area and enters directly above the sample/furnace area. It flows down the outside of the quartz insert and purges the area between the insert and the glass furnace tube. It then flows out the same exhaust line as the balance purge. This design allows you to rapidly switch the sample atmosphere without having to purge the entire balance chamber. You will always have the controlled purge through the balance chamber, protecting the balance components at all times. The flow rate of the sheath gas must be less than the balance purge flow rate at all times. If the sheath gas flow rate is greater than the balance flow rate, decomposition products may be deposited in the balance chamber and may eventually cause damage to the microbalance. The recommended flow rate for the sheath gas is 20 – 35 mL/min. This rate will be realized by an output pressure of 13,800 – 24,150 Pa (2 – 3.5 psi) on the output side of the regulator when using the type A flow restrictor.

The chamber sleeve or liner regulates furnace convection by reducing the furnace chamber area. Gas switching time is substantially reduced as a result of the smaller furnace area volume. The time to purge the area of ambient gases (remove 99% of oxygen) and replace the volume with an inert purge gas is less than 3 minutes. It takes 10 minutes to achieve a 99.99% oxygen-free environment.

If you want to use more than one sheath gas, you can use either a GSA 7 Gas Selector Accessory for two gases or a Thermal Analysis Gas Station (TAGS) for four gases.

See the section **Connecting Gas Supplies** that follows for more information.

Reactive Gas

In addition to purge gases, you can also use an optional reactive gas in the sample area. A special furnace tube with a reaction gas injection port is available. This accessory allows the reaction gas to couple the sample specimen to the purge or sheath gas.

Furnace Lift Mechanism Gas

Use either compressed air or nitrogen for the furnace lift mechanism gas. This gas is also used for the antistatic device. The recommended pressure is between 103.5 and 207.0 kPa (15 – 30 psi). We recommend that the tank regulators used for the pneumatic supply have a maximum output of 690 kPa (100 psi) or less, since the use of higher output regulators may damage the gas supply cylinder.

> **NOTE:** At initial setup, we recommend using the lowest pressure and then adjusting the pressure accordingly so that the furnace moves at a slow, steady rate. If the pressure is set too high, the furnace may move too quickly and slam into the ball joint.

Connecting Gas Supplies

When the Pyris 1 TGA was installed by the service engineer, he should have connected the gases necessary to operate your instrument, i.e., balance purge gas, sheath gas, and furnace lift mechanism and antistatic device gas. However, you may need to set one of these gases up yourself. You can use individual gas supplies or the same gas source for both the balance purge and the sheath gas, and even for the furnace lift mechanism by using a serial pressure gas setup (P/N N519-0462).

Connect the Purge and Sheath Gas Lines to the Gas Supply

To connect the balance purge and sheath gases, using separate sources, you will need parts included in the Spares Kit (P/N N537-0474).

1. Connect suitable connectors to the gas supplies that will be used to purge the system (you are responsible for supplying the regulators). The regulators should be equipped with shutoff valves at the outlet. The shutoff valves should have a 1/4-in. NPT male thread on the outlet side.
2. Tape the threads of the outlet fitting on both shutoff valves with the thread sealant tape provided (P/N 0990-8134).
3. Connect a H restrictor (P/N 0154-1498) to the taped shutoff valve fitting on each gas supply cylinder. The H restrictor maintains a flow of 1 cc/min for every pound of pressure in the purge lines for nitrogen, air, or oxygen. When using helium with this restrictor, the flow rate is substantially faster.
4. Tape the threads on the outlet side of the H restrictors with thread sealand tape.
5. Connect the end of a female connector (P/N 0990-3196) to the taped male end of the H restrictor on both cylinders.

   If you want to **install a filter dryer** into the purge gas or sheath gas line, click here. When finished, return here.

   If you want to **install a GSA 7 gas selector accessory** or a **TAGS** gas selector accessory in the sheath gas line, do so now and then return here. See the respective subsections below on how to install a GSA 7 or a TAGS.
6. Place a 1/8-in. nut and rear and front ferrules over one end of the 1/8-in. Teflon tubing provided (P/N 0250-6493).
7. Connect the 1/8-in. Teflon tubing and nut to the end to the end of the NPT male connector on the gas supply.
8. Repeat steps 5 – 7 for the other gas line.

You can now connect the purge and sheath gas lines to the Pyris 1 TGA if not already done with installing a GSA 7 or TAGS.

Connecting the Balance Purge Line

1. Place a 1/8-in. nut and rear and front ferrules over the other end of the 1/8-in. Teflon tubing which is connected to the gas cylinder that is to be used for the balance purge.
2. Connect the end of the Teflon tubing to the fitting labeled **Balance Purge** located on the back of the Pyris 1 TGA. Tighten the nut fingertight and then, using the wrench provided (P/N 0990-7222), tighten 1/4 turn past fingertight.

Connecting the Sheath Gas Line

1. Place a 1/8-in. nut and rear and front ferrules over the other end of the 1/8-in. Teflon tubing which is connected to the gas cylinder that is to be used for the sheath gas.
2. Connect the end of the Teflon tubing to the fitting labeled Sheath Gas located on the back of the Pyris 1 TGA. Tighten the nut fingertight and then, using the wrench provided, tighten 1/4 turn past fingertight.

Install the Furnace Lift Mechanism Gas Supply

The Pyris 1 TGA has an automatic furnace movement feature that uses compressed air or nitrogen at a recommended pressure between 103.5 and 207.0 kPa (15 – 30 psi). We recommend that the tank regulators used for the pneumatic supply have a maximum output of 690 kPa (100 psi) or less, since the use of higher output regulators may damage the gas supply cylinder.

NOTE: The gas supply used for the furnace lift mechanism is also used for the antistatic device.

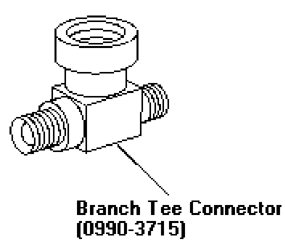
NOTE: At initial setup, we recommend using the lowest pressure and then adjusting the pressure accordingly so that the furnace moves at a slow, steady rate. If the pressure is set too high, the furnace may move too quickly and slam into the ball joint.

CAUTION: Never use pressure greater than 240 kPa (35 psi); this will damage the cylinder.

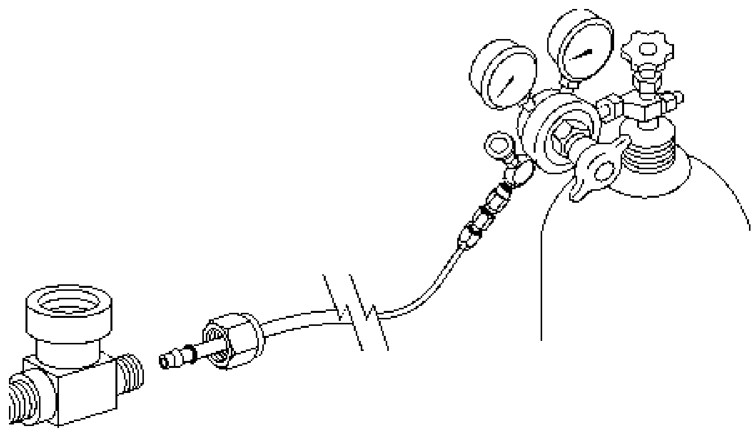
Use the procedure below and the parts included in the Spares Kit to connect the furnace lift mechanism gas supply to the Pyris 1 TGA.

NOTE: In newer systems, the parts are already assembled when the instrument is attached. You will just have to attach it to the gas supply.

1. Connect a suitable pressure regulator to a pressurized tank or house line of compressed air or nitrogen. (We recommend compressed air.) The regulator should have a maximum output rating of 100 psi. Connect a suitable length of Tygon tubing to the pressure regulator so that it reaches the lift mechanism when it is attached to the instrument.
2. Remove the branch tee connector (P/N 0990-3715) from the Spares Kit. This connector has two 1/4-in. male tube fittings and a 1/4-in. female fitting at a right angle.



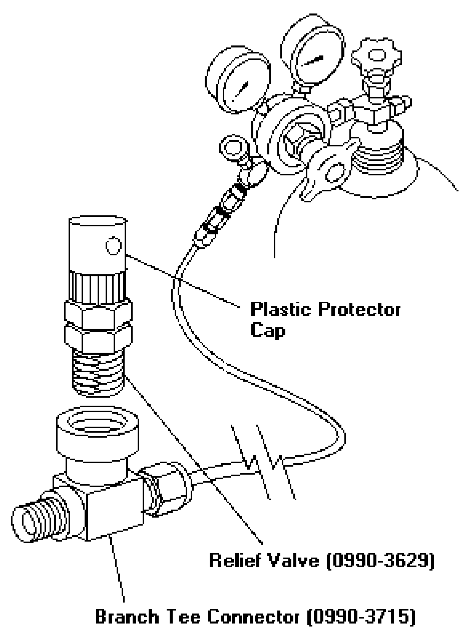
3. Connect one end of the branch tee connector to the pressure regulator at your pressure tank or house line.



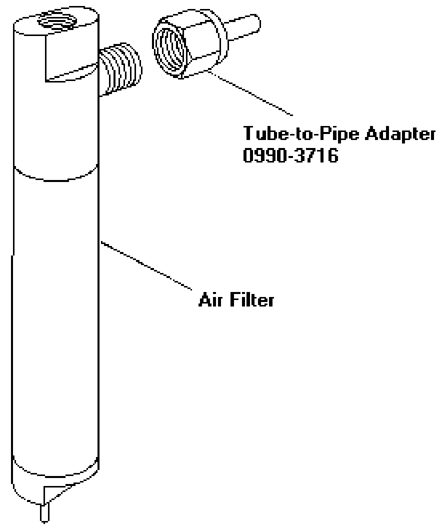
4. Remove the pressure relief valve (P/N 0990-3629) from the kit. One end of this valve has a plastic protector cap and the other end has a 1/4-in. male fitting.

   CAUTION: Do not remove the plastic cap.

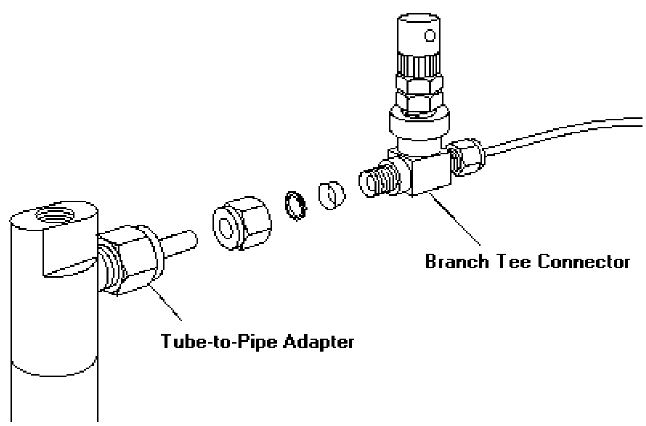
5. Tape the threads on the male end with thread sealant tape (P/N 0990-8134).
6. Connect the relief valve to the female fitting on the branch tee connector.



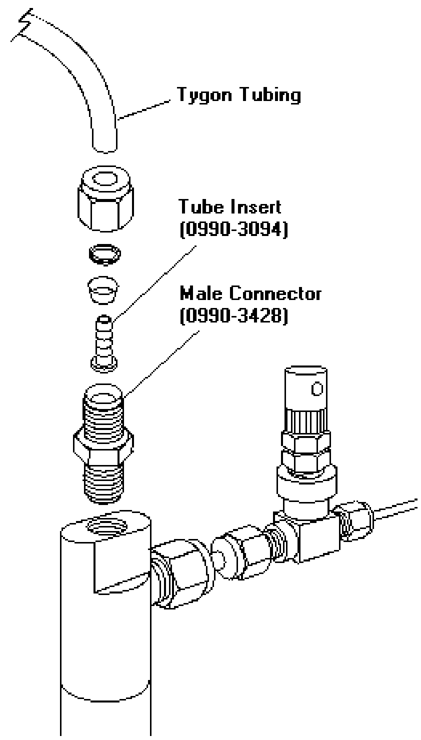
7. Remove the air filter (P/N 0990-3630) from the Spares Kit. The air filter has a 1/8-in. male fitting on the side, a 1/8-in. female fitting on one end, and a small pressure bleed needle at the other end. Tape the male fitting with thread sealant tape.
8. Remove the tube-to-pipe adapter (P/N 0990-3716) from the kit. This adapter has a 1/8-in. female fitting at one end and a straight tube at the other end. Connect the female end of the adapter to the male fitting on the air filter.



9. Remove the nut and two ferrules from the free end of the branch tee connector and slide them over the tube end of the adapter.
10. Insert the tube end of the adapter into the branch tee connector and tighten the nut 1/2 turn past fingertight with a wrench.



11. Remove the male connector (P/N 0990-3428) from the kit. Tape the threads of the connector with thread sealant tape.
12. Connect the male connector to the female end of the air filter.
13. Remove the Tygon tubing (P/N 0250-6518) from the Spares Kit and cut the tubing to the correct length needed to reach the Pyris 1 TGA.
14. Place a tube insert (P/N 0990-3094) in each end of the tubing. The tube inserts prevent the Tygon tubing from being pinched closed when the end of the tubing is secured.
15. Remove the nut and two brass ferrules from the male connector that you connected to the female end of the air filter.
16. Connect one end of the Tygon tubing to the male connector using the nut and ferrules.



17. Connect the other end of the Tygon tubing to the fitting labeled N2/Air on the back of the Pyris 1 TGA using the nut and ferrules located on the analyzer. Tighten the nuts 1/2 turn past fingertight using a wrench.

For more recent Pyris 1 TGA's, the furnace lift mechanism (P/N N537-0334) comes assembled and attached to the rear panel of the instrument as seen in the photo below. All you need to do is attach the piece of Tygon tubing that is provided in the Spares kit from the open end of the tee connector on the assembly to the gas tank or house line. Connect a suitable pressure regulator to the pressurized tank or house line of compressed air or nitrogen. (We recommend compressed air.) The regulator should have a maximum output rating of 100 psi.

Connecting Gases Using a Common Source

Instead of using two or more gas cylinders containing the same gas or two house lines of the same gas, you can use one source of gas for both or all end uses, e.g., balance purge and sheath gas for the Pyris 1 TGA. You can do this by installing gauges in series off of the regulator on the source. These gauges are P/N N519-0462. Detailed instructions on how to put two or more gauges together prior to installing them on the regulator are included with the gauges. You should also install the connectors for the tubing to the outlets of the gauges before attaching the gauges to the source. The cylinder or house line should be prepared with a regulator. Attach the series of gauges to the outlet connector on the regulator with the arrows on the gauges — which indicate the direction of the gas flow — pointing away from the source.

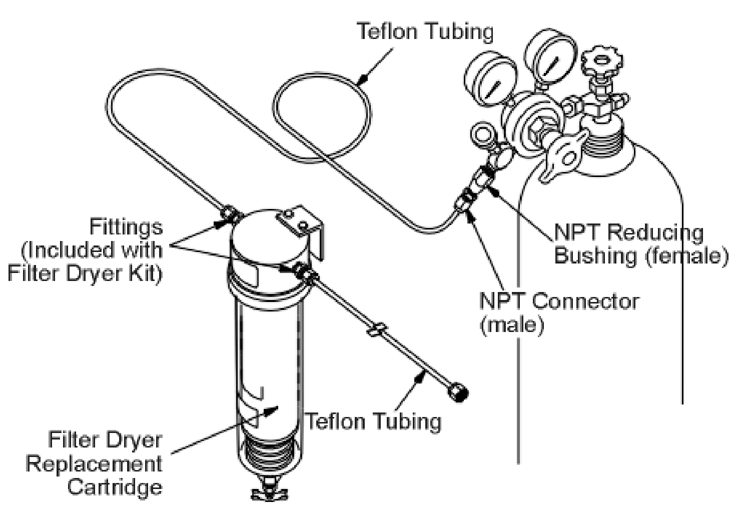
Installing a Filter Dryer for Pyris 1 TGA Gas Supplies

To install a Drierite Compressed Air Drier Model 207 (P/N 0992-3453) into the balance purge gas or sheath gas line, perform the following steps:

WARNING: FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL INJURY, AND PROPERTY DAMAGE. Maximum working pressure is 125 psig.

Read all warnings and safety precautions before installing this dryer. Install the dryer in an upright position as near to the point of use as practical.

1. Mount the bracket that comes with the dryer in the desired location.
2. Attach the aluminum head of the dryer to the bracket with 1/4-20 bolts provided.
3. With the nut and ferrules from the NPT male connector (P/N 0990-3434) that was attached to the reducing bushing on the gas supply, swage the end of a piece of 1/8-in. Teflon tubing.
4. Connect the 1/8-in. Teflon tubing with nut to the male connector installed on the regulator shutoff valve of the gas cylinder.
5. With the nut and ferrules from one of the male 1/4-in. NPT to 1/8-in. tubing connectors (P/N 0990-3087) that is provided with the dryer, swage the other end of the piece of 1/8-in. Teflon tubing attached to the gas supply.
6. Wrap Teflon tape around the threads of the connector.
7. Connect the connector to the IN connector on the dryer.
8. Connect the gas supply to the inlet side of the aluminum head by connecting the swaged 1/8-in. Teflon tubing to the IN connector.
9. Wrap Teflon tape around the threads of the OUT connector on the dryer.
10. With the nut and ferrules from the other 1/4-in. NPT to 1/8-in. tubing connector (P/N 0990-3150), swage the end of a piece of 1/4-in. Tygon tubing.
11. Wrap Teflon tape around the threads of the connector.
12. Connect the connector to the OUT connector on the air dryer.



13. The cartridge (P/N 0992-3453) must be punctured in both ends before use:
    a. Close the gas supply valve.
    b. Release air pressure in the cartridge by opening the condensate drain valve.
    c. Unscrew and remove the bowl by turning counterclockwise by hand (use no tools).
    d. Remove the cartridge.
    e. Puncture both ends with an opener or sharp instrument.
    f. Place the cartridge back in the bowl on the spring support with the indication window toward the top.
    g. Place the 2-1/2-in. o.d. O-ring on top of the cartridge.
    h. Place the 4-in. o.d. O-ring inside the aluminum head. The O-rings should be clean and dry. Use no grease.
    i. Secure the bole to the head by screwing clockwise until contact with the O-ring is made. Handtighten only; do not overtighten.
    j. Close the condensate drain valve.
    k. Open the gas supply valve.

Return to **Connecting the Gas Supplies to the Pyris 1 TGA** if you are installing gas supplies.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the rear of the Pyris 1 TGA at the 9-pin connector on the right-hand side and labeled **Gas Selector Accessory**.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

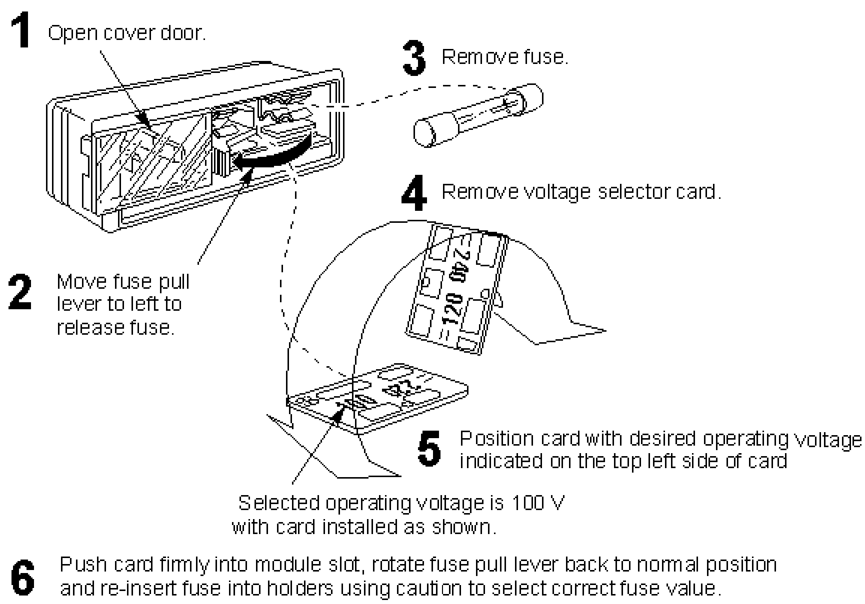
WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

Connecting a GSA 7 to a Pyris 1 TGA

The Gas Selector Accessory GSA 7 allows you to use up to two gases for the sheath gas. You can switch gases in the Gas Program of the Method. This topic discusses how to connect the gas lines to the GSA 7 and Pyris 1 TGA.

Before installing a GSA 7 Gas Selector Accessory into a sheath gas line, make sure that it is set at the correct voltage. If it is not, change the input voltage at the AC inlet plug.

1. Open the cover door of the inlet plug at the back of the GSA 7.
2. Rotate the fuse-pull to the left and remove the fuse.
3. Remove the voltage selection card by pulling it out with needle-nosed pliers. Grasp the card at the center of the card.
4. Select the operating voltage by orienting the voltage selection card to position the desired voltage on the top left side.
5. Using the pliers, insert the card back into the module slot and push it firmly into place.
6. Rotate the fuse-pull back into its normal position.
7. Reinsert the fuse into the fuse holders, making sure to select the correct fuse value:
   - 100/120 V, use a 3 AG (0.375 A) 250 V Slow Blow fuse (P/N 0998-1619)
   - 220/240 V, use a 3 AG (0.187 A) 250 V Slow Blow fuse (P/N 0999-1683)



Connecting the Gases

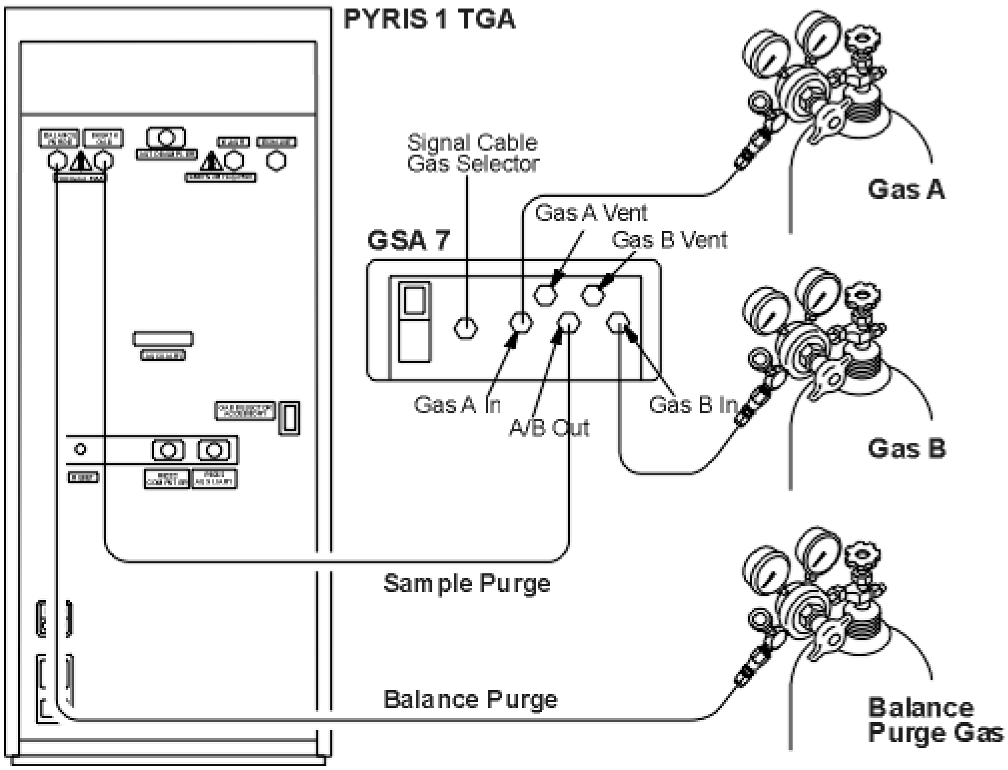
This procedure assumes that you are using two gas supplies, Gas A and Gas B, and that the gas supplies (and filter dryers, if used) are already connected. See figure below for connections.

1. Connect a length of 1/8-in. Teflon tubing between Gas A and the GAS A IN connector located on the back of the GSA 7.
2. Connect a length of 1/8-in. Teflon tubing between Gas B and the GAS B IN connector.
3. Connect a length of 1/8-in. Teflon tubing to the A/B OUT connector using a Swagelok fitting.

4. Run a length of 1/8-in. Teflon tubing between the GAS A VENT and GAS B VENT connectors to a fume hood or other suitable container.

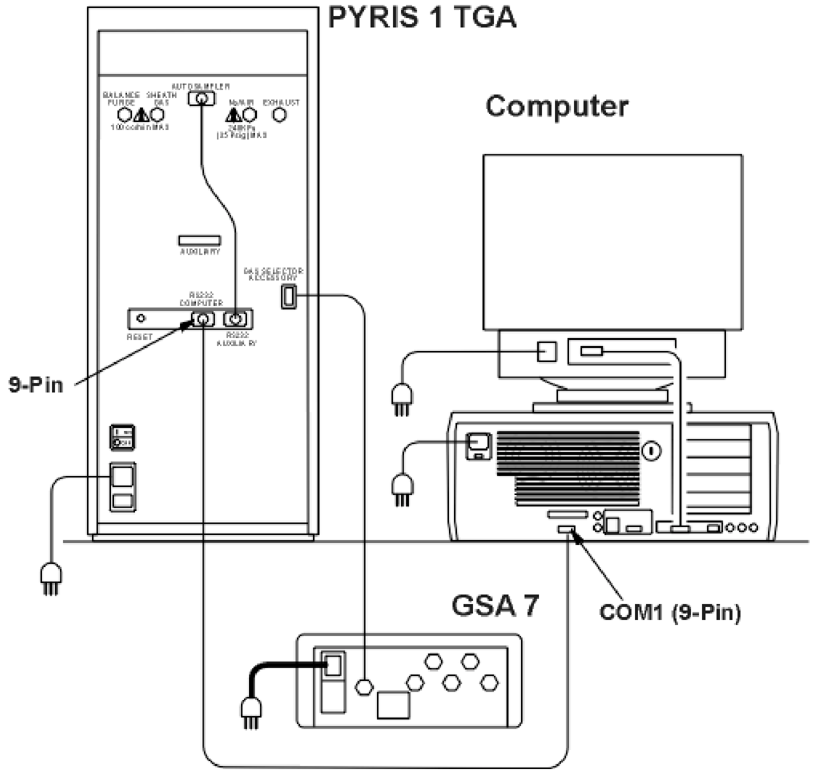
WARNING: Do not submerge the tubing in water or other liquids.

5. Connect the other end of the 1/8-in. Teflon tubing which is connected to the A/B OUT to the **Sheath Gas** connector on the Pyris 1 TGA.



Connecting the Pyris 1 TGA, GSA 7, and Computer

1. Remove the spacers from the other end of the signal cable that is attached to the GSA 7 and attach it to the **Gas Selector Accessory** connector on the Pyris 1 TGA.
2. Attach one end of the RS232 cable (N822-1178) to the RS232 Computer port on the rear of the Pyris 1 TGA.
3. Attach the other end of the RS232 cable to a COM port on the computer.



TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Preparing the Laboratory for TAGS

The TAGS will accept the same laboratory conditions suited for the Pyris 1 TGA. Some precautions are

- Do not place the TAGS in direct sunlight or close to heating and cooling units.
- The temperature of the area should be between 10°C and 35°C.
- Relative humidity should be between 20% and 80% and noncondensing.
- The TAGS requires little bench space. The electrical power consumption is only 8 VA max. Therefore, it can be connected directly to the instrument's power line. The supply must be smooth, clean, and free of transient voltages over 40 V. The operating frequency is 50 – 60 Hz.

Unpacking the TAGS

The TAGS is shipped in one box and is surrounded by foam on all sides. To unpack the TAGS, follow the steps below:

1. Remove the power cable, the serial cables, and the manual from the box.
2. Remove the foam insert from the box.
3. Remove the upper foam piece.
4. Remove the TAGS from the box.
5. Remove all remaining packing material.

Installing the TAGS

Setting the Correct Voltage

Upon delivery, the TAGS is set at a voltage of 220/240 V. Changing the input voltage should be done at the AC inlet plug.

1. Remove the fuse holder with a small screwdriver by gently prying it out of the compartment. Once the holder is out far enough to grasp, slide the fuse holder out of the slot.
2. Rotate the fuse holder 180°.
3. Remove the fuse from the 220-V position (100 mA Slow Blow) and insert a 200 mA Slow Blow fuse for 110 V (it goes into the right side when "110 – 120 V" is in the upright position.
4. Reinsert the fuse holder.
5. Make sure that the arrow next to the voltage label is pointing at the white line below the fuse holder.

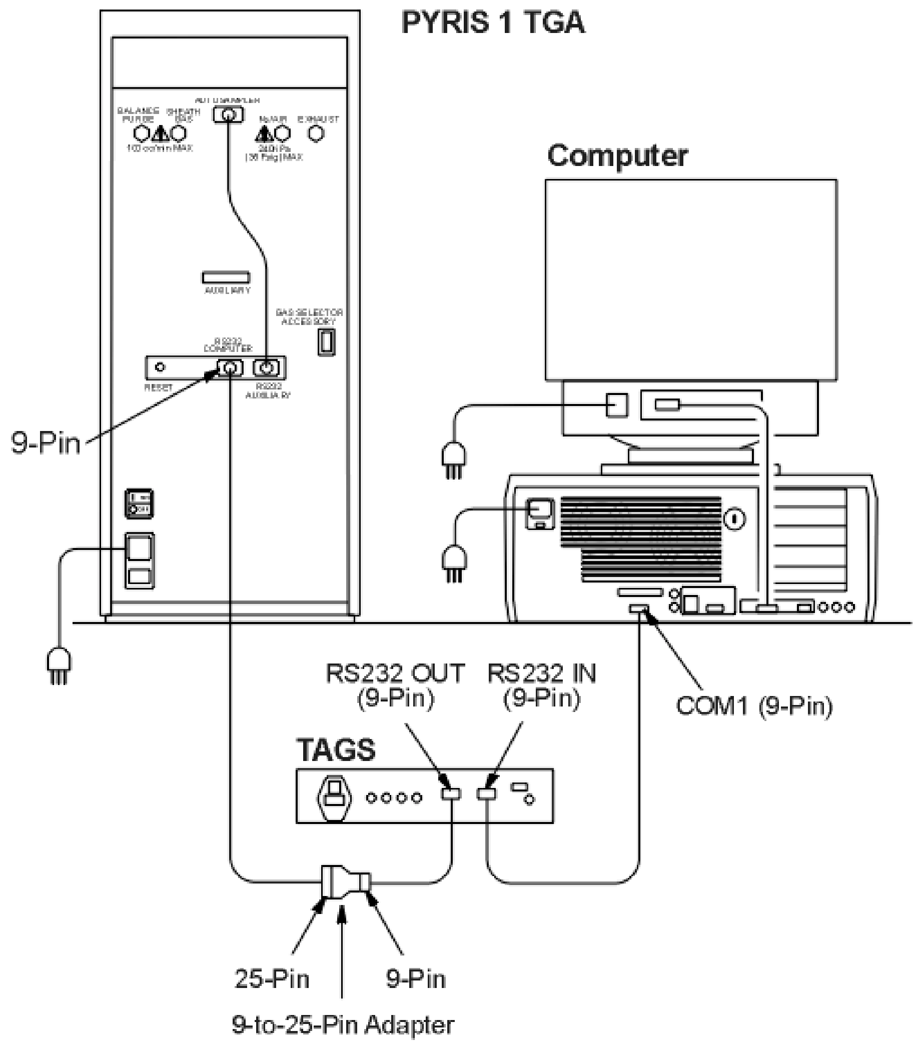
Connecting the Gases

The procedure below assumes that there are four gas supplies for the sheath gas and that the gas supplies (and filter dryers, if used) are already connected.

1. Connect a length of 1/8-in. Teflon tubing from the Gas A supply to the input #1 connector on the rear of the TAGS.
2. Connect a length of 1/8-in. Teflon tubing from the Gas B supply to the input #2 connector.
3. Connect a length of 1/8-in. Teflon tubing from the Gas C supply to the input #3 connector.
4. Connect a length of 1/8-in. Teflon tubing from the Gas D supply to the input #4 connector.
5. Connect a length of 1/8-in. Teflon tubing to the OUT connector on the TAGS using a Swagelok fitting.
6. Connect the other end of the tubing from the OUT connector to the **Sheath Gas** connector on the Pyris 1 TGA.

Connecting the Pyris 1 TGA, TAGS, and Computer

1. Attach one of the two 9 male/9 female RS232 cables to a COM port on the computer.
2. Attach the other end of that cable to the RS232 IN connector on the TAGS.
3. Connect the other 9 male/9 female cable to the RS232 OUT connector on the TAGS.
4. Connect the AT adapter (9 male/25 female) to the other end of that second cable.
5. Attach the 25-pin end of the last RS232 cable (9 male/25 male) to the adapter.
6. Connect the 9-pin end of the cable to the RS232 Computer connector on the rear of the Pyris 1 TGA.



Calibration

There are three calibration routines for the Pyris 1 TGA. All calibrations should be performed upon installation of the instrument. Periodic calibration checks will be necessary to verify accurate calibration.

The Pyris 1 TGA's calibration should remain unchanged for some time, provided that there are no changes in the instrument's operating conditions. Even when the system is shut off, the calibration values are stored so that the next time the instrument is turned on, it will still be calibrated. Some of the conditions that could change either the temperature or the weight calibration and require recalibration are

- If the operating temperature range of your experiments changes, recalibration may be necessary. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
- If the purge gas type or purge gas flow is changed, the calibration should be checked for highest accuracy.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new thermocouple is installed or if the position of the thermocouple changes, you should perform the temperature calibration again.

- If the analyzer is moved or releveled, you should perform the weight calibration again.

There are three calibration routines that should be performed for a Pyris 1 TGA with a standard furnace with or without an autosampler:

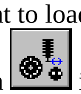
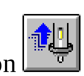
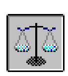
- **Temperature**
- **Weight**
- **Furnace**

The Weight and Furnace routines should be performed for the Pyris 1 TGA with a high temperature furnace.

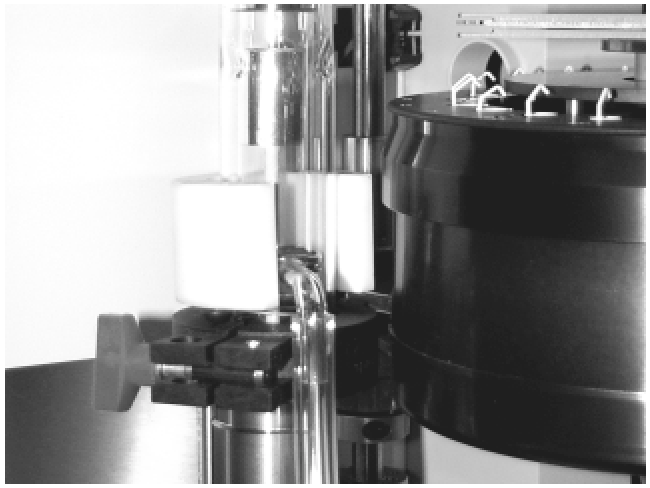
> **NOTE:** It is possible to calibrate the high temperature Pyris 1 TGA for temperature. The procedure for temperature calibration can be followed but you will need to supply a magnet that will work with the high temperature furnace and you will have to determine where to place the magnet.

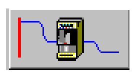
Calibrate Temperature

The temperature calibration is performed for a Pyris 1 TGA with a standard furnace, with or without an autosampler. Two runs are performed using the magnetic standards of nickel and iron, which are supplied in the Spares kit. The runs should use the same conditions under which you would run your samples. The temperature calibration uses the Curie transition of the materials, i.e., the point at which the magnetic properties disappear.

1. From the Method Editor, select Open Method from the File menu. Double click on the NickCal.tg1m method file. (The default location for the file is Program Files\Pyris\Methods.) The nickel calibration method is displayed.
2. Select the Sample Info tab and fill in the Sample ID, Operator ID, Comment, and File Name if desired.
3. Cut a small piece of nickel approximately 1 – 2 mm long. For a nonautosampler TGA, place the sample in the sample pan and place the pan in the stirrup hanging on the hangdown wire.
4. For an autosampler system, you want to load the sample into the crucible in position 1. Click on the **Autosampler Control** button  in the Pyris 1 TGA control panel. Enter 11 in the Select Carousel Position and click on Rotate Tray. This will cause the tray to rotate so that position 1 is facing out and you can access it. You can either remove the crucible, place the sample it it, and load the crucible into position 1 or just drop the sample into crucible 1 while it is in the tray. Click on **Load Sample**. The tray will move to the Load position and crucible 1 will be loaded onto the hangdown wire. Click on the **OK** button to close the dialog box.
5. Click on the **Raise Furnace** button  in the control panel.
6. Click on the **Zero Weight** button  in the control panel. The analyzer reads the weight of the sample which is entered into the Zero field of the Enter Sample Weight box in the Method Editor.
7. For a standard system, place the magnet onto the sample loading platform and move the platform next to the furnace tube. Adjust the height of the sample loading platform so that you get a weight reading between 3 and 6 mg. The apparent reading is a result of the magnetism of the sample. As the sample loses magnetism, the apparent weight reading decreases.

For an autosampler system, first observe the orientation of the crucible before positioning the magnet (P/N N537-0466) around the furnace tube, as seen below. Rotate the magnet to obtain the maximum weight reading, seen in the Status Panel, but also try to obtain the same crucible orientation as before placement of the magnet. It is less likely that the crucible will rotate when going through a Curie transition.



8. After weight reading (displayed in the Status Panel if Weight is selected to be displayed) has stabilized, click on the **Sample Weight** button  in the control panel. The weight is entered into the Weight field of the Enter Sample Weight area of the Sample Info page.
9. Click on the **Start Method** button  on the control panel.
10. For an autosampler system, after the run is complete, click on the **Lower Furnace** button .
11. For an autosampler system, click on the **Autosampler Control** button. Select Unload Sample to return the crucible to carousel position 1.
12. Repeat steps 1 – 8 for a standard system or steps 1 – 10 for an autosampler system for iron using the IronCal.t1gm method file.

Calculating the Onset Value

Since the curve from the last run is probably still displayed in the Data Analysis window, calculate the onset value for iron first. Then repeat the steps for nickel.

1. Be sure that the curve is displayed with the following axes:

   X = Temperature (change by selecting Rescale X from the Display menu and then select Temperature in the dialog box)

   Y = Weight (change by selecting Weight from the Curves menu)
2. Select Derivative from the Math menu. The first derivative of the curve is calculated.
3. Click on the sample curve so the derivative curve goes to the background.
4. Select Onset from the Calc menu.

5. Move the two red **X** marks on the curve. Position the left limit **X** at the inflection point of the first derivative curve and place the right limit **X** past the inflection point on the level part of the curve.
6. Click on the **Calculate** button in the Onset dialog box. The Adjust Tangents box appears.
7. Adjust the tangents of each point until they cross.
8. Click on the Calculate button in the Adjust Tangents dialog box. The onset temperature is calculated for the sample and displayed on the screen (Onset X).
9. Note the Onset value.
10. Repeat steps 1 – 9 for nickel.
11. In the Method Editor, select Calibrate from the View menu.
12. Click on the Temperature tab in the Calibration window. Enter the name of each reference material; the Expected Onset values, and the Measured Onset values. Click in the check box in the Use column for each reference material used in the calibration.
13. Click on **Save and Apply** to send the calibration values to the analyzer and save the current calibration file.
14. Select **Close** to exit the Calibration window.

Calibrate Weight

In order to perform the weight calibration for a Pyris 1 TGA, you will need the 100-mg calibration weight (P/N 0990-7222) in the Spares kit.

1. While in the Method Editor, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Fill in the Operator ID field if you wish.
4. Enter the weight of the calibration weight in the Ref. Weight field.
5. Select **Begin Calibration.**

   A dialog box appears with instructions to remove anything in the sample pan and then move the loading tray away from the sample pan. For the autosampler system, remove any sample from the crucible in position 1 in the sample tray. The autosampler should be in the Safe position.
6. Follow the instructions in the dialog box, then click on **OK**. The analyzer is tared for a zero reading.

   For the autosampler system, after accepting the reading, the crucible is unloaded and the furnace is lowered. After approximately 30 seconds, the autosampler rotates the tray about 180° so that position 1 is easily accessible.
7. When prompted, place the calibration weight in the sample pan, make sure that the loading tray is away from the sample pan and click on the **OK** button. The weight of the calibration weight is read and displayed in the dialog box. Click on **OK** to accept the reading.

   For the autosampler, place the reference weight in the crucible in position 1 of the sample tray and click on **OK**. The crucible at position 1 is then loaded onto the hangdown wire and the weight of the calibration weight is read and displayed. Click on **OK** to accept the reading. The furnace goes to the Cool position and the crucible is unloaded to position 1 of the tray.
8. The just measured value is automatically displayed in the Measured field. Click on the **Save and Apply** button to send the new calibration value to the analyzer and save the calibration file.

9. Click on the **Close** button to exit the Calibration window.

Calibrate Furnace

The Pyris 1 TGA furnace calibration takes approximately 1 hour.

1. If necessary, from the Method Editor, select Calibrate from the View menu.
2. Place an empty sample pan in the stirrup at the end of the hangdown wire.

   For an autosampler system, place an empty crucible at the end of the hangdown wire. Place it there manually with tweezers while the autosampler is in the Safe position, or use the Autosampler Control dialog box to load the sample from a specified location.
3. Enter the minimum and maximum temperatures between which the furnace will be calibrated (the defaults are 100°C and 900°C).
4. Click on **Begin Calibration**. An estimate of the amount of time needed to perform the calibration is displayed and it begins to count down.
5. When the calibration is complete, click on **Save and Apply** to send the new calibration value to the analyzer and save the file.
6. Click on the **Close** button to exit the Calibration window.

Calibration Reference Materials

Your company may have to comply with ISO 9000. Perkin Elmer Thermal Analysis calibration materials are ISO 9000 compatible. They are called Reference Materials. ISO has two classifications for calibration materials: Reference Materials and Certified Reference Materials.

Reference Materials (RMs) comply with ISO for instrumentation calibration. They are available from Perkin Elmer and Perkin Elmer provides documentation with each RM that cross references the material's lot code. This declaration should be filed with all other material certificates and documents in your ISO file.

ISO Guide 30 defines a RM as a

> "Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials."

Certified Reference Materials (CRMs) are traceable to national or international standards through an unbroken chain of comparisons. Not every calibration material is a CRM. CRMs can be very expensive and are not available for all thermal analysis techniques.

If you are not sure what material to use, then check with your Quality Assurance Department and discuss the differences between RMs and CRMs. If you find that you need CRMs, you may order them from the National Institute of Standards and Technology (301 975-6776).

Magnetic Calibration Reference Materials are used to perform the Curie point temperature calibration on the standard furnace. They are available from PE XPRESS in kit form and separately. We recommend that you use fresh reference materials each time you calibrate your instrument. This way you can avoid the effects of hysteresis and oxidation on the Curie point of the reference materials. The transition temperature for each of the magnetic standards is indicated in the documentation included with the reference material.

Part No.	Reference Material	Magnetic Transition Temperature
0219-0071	Kit of 5 reference materials including alumel, nickel, Perkalloy, iron, and Hisat-50	
0998-8015	Three 2-in. pieces of 99.99% alumel wire	163°C
N519-0869	Three 2-in. pieces of 99.99% nickel wire	354°C
N519-0616	Three 2-in. pieces of Perkalloy wire	596°C
0998-8017	Three 2-in. pieces of 99.99% iron wire	780°C

Maintenance

The Pyris 1 TGA needs little routine maintenance other than proper treatment as a sensitive electronic device. Occasionally, the furnace or furnace tube may become coated with sample residue and require cleaning, or furnace components and other accessories, such as hangdown wires and thermocouples, may need to be replaced.

NOTE: The Pyris 1 TGA case is painted metal. The exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution.

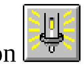
Maintenance of the Pyris 1 TGA includes the following procedures:

- **Cleaning the Furnace**
- **Cleaning the Standard Furnace Tube**
- **Cleaning the Sample Pan**
- **Standard Furnace Maintenance**
- **Standard Furnace Tube Maintenance**
- **Standard Furnace Hangdown Wire Maintenance**
- **High Temperature Furnace Maintenance**
- **High Temperature Furnace Tube Maintenance**
- **High Temperature Furnace Oven Thermocouple Maintenance**
- **High Temperature Furnace Sample Thermocouple Maintenance**
- **High Temperature Furnace Hangdown Wire Maintenance**

Cleaning the Furnace

An automatic procedure for cleaning the Pyris 1 TGA standard and high temperature furnaces is included in the Pyris software. The procedure involves lowering the furnace to expose it to the air and then heating it to approximately 900°C to burn off any materials coated onto the furnace surfaces.

WARNING: **Since the furnace will be programmed to 900°C, make certain that the protective plastic visor on the Pyris 1 TGA is in the down position. If you have a Pyris 1 TGA with an autosampler, make sure that the autosampler is in the SAFE position. DO NOT touch the furnace during this procedure.**

1. Start up the Pyris software and make sure the Control Panel is displayed.
2. Select the **Clean Furnace** icon  from the Control Panel.
3. The furnace will move to the lowered position and programmed to 900°C. When the cleaning procedure is complete, the furnace will move to the cool position and cool down to 30°C.

NOTE: To abort the furnace cleaning procedure at any time, select the **Cool Furnace** icon.

NOTE: You can also access the Clean Furnace procedure for the Pyris 1 TGA from the instrument's control display menu.

Cleaning the Standard Furnace Tube

In order to clean the furnace tube, you must first remove the tube from the furnace. Clean the furnace tube using **one** of the following methods:

1. Soak and scrub the furnace tube in a strong detergent (e.g., alconox).
2. Soak the furnace tube in a strong solvent such as cleaning solution (e.g., concentrated H_2SO_4 and K_2CrO_7).

WARNING: **Concentrated H_2SO_4 and K_2CrO_7 is a very strong, caustic acid solution. Wear protective gloves and safety glasses and perform this procedure in a fume hood.**

3. Dip the furnace tube in a dilute hydrogen fluoride (HF) solution (10% solution, 5 min maximum).

WARNING: **Hydrogen fluoride is a dangerous skin irritant. Wear protective gloves and safety glasses and perform this procedure in a fume hood.**

4. Rotate the furnace tube over a Bunsen burner (heat up slowly to avoid cracking).

After cleaning, reinstall the furnace tube.

Cleaning the Sample Pan

The platinum sample pans used with both the standard and high temperature furnaces in the Pyris 1 TGA and the ceramic crucible used with the Pyris 1 TGA autosampler can be cleaned with one of the following techniques:

1. Place the sample pan on the hangdown wire, raise the furnace, and, using air or oxygen as a sheath gas, heat the instrument to 950°C to burn off any material coated on the pans. After the pans cool, scrape off any residue or tap out any ash residue.
2. Carefully flame the pan over a Bunsen burner.

Note that you can use a disposable aluminum or stainless steel liner in the ceramic crucibles for the Pyris 1 TGA autosampler for easy cleanup.

Standard Furnace Maintenance

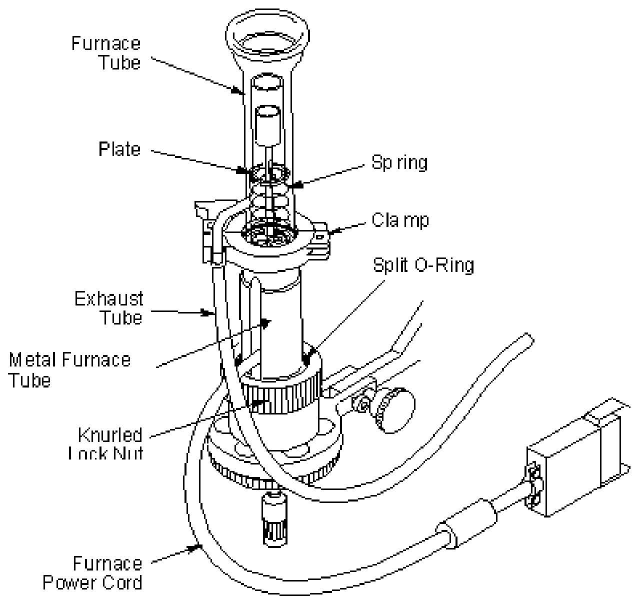
Standard furnace maintenance consists of the following procedures:

- Removing the Standard Furnace
- Remove and Install the Thermocouple
- Replacing the Standard Furnace
- Adjusting the Standard Furnace (No Autosampler)
- Adjusting the Standard Furnace (Autosampler System)

See the subtopic **Standard Furnace** in the **Overview** section for a diagram of the parts of the standard furnace.

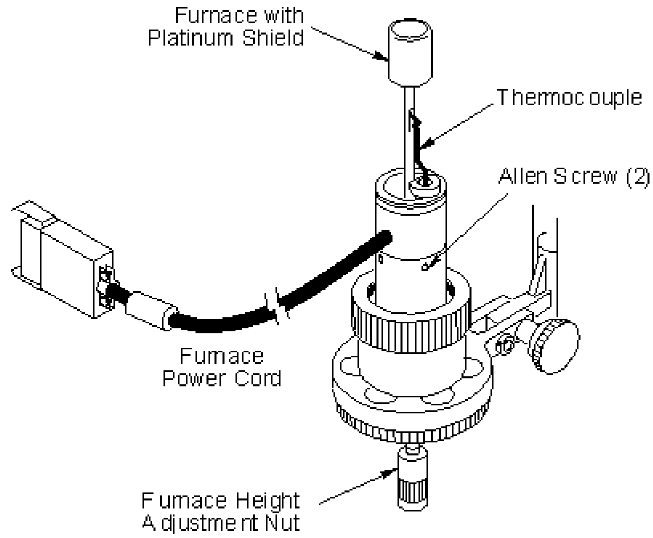
Removing the Standard Furnace

WARNING: **Before performing this procedure, shut down the system and remove the line power. Make sure the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.**

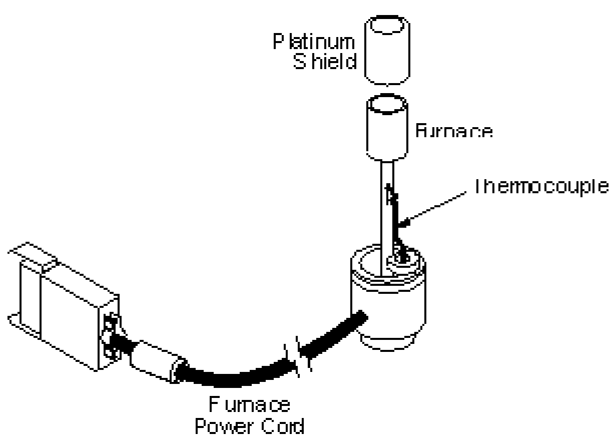


1. Disconnect the furnace power cord from its connector on the front of the Pyris 1 TGA. To disconnect the cord, squeeze the silver locking tabs and gently pull forward on the connector.
2. Loosen and remove the clamp holding the standard Pyrex furnace tube to the base of the furnace assembly.
3. Remove the standard Pyrex furnace tube. You do not have to disconnect the Tygon exhaust tube from the furnace tube. Lay the furnace tube on the bench top.
4. Remove the inner Pyrex furnace lining.
5. Remove the slotted ring or plate sitting on the spring.

6. Remove the spring.
7. Loosen the large knurled nut (furnace locking nut) and lift it up.
8. Remove the split O-ring that is underneath the nut.
9. Lift the furnace assembly off of the mounting.
10. While holding the furnace assembly in both hands, use your thumbs to gently push the metal furnace tube off. There will be some tension until the piece is approximately 0.5 in off of the assembly. Make sure that the piece comes off straight, otherwise you may damage the furnace.



11. Using a 0.050-in. hex wrench (P/N 0990-7236), loosen the three Allen screws on the furnace base that hold the furnace in place.
12. Remove the furnace by lifting it straight up and off of the base.
13. Remove the platinum furnace shield by sliding it off. Do not deform the shield.



If you plan to use the thermocouple from the removed furnace on the new furnace, remove it as described in **Remove and Install the Thermocouple in a Standard Furnace** below.

Remove and Install the Thermocouple in a Standard Furnace

To replace a thermocouple in a standard furnace of the Pyris 1 TGA, follow the procedure below:

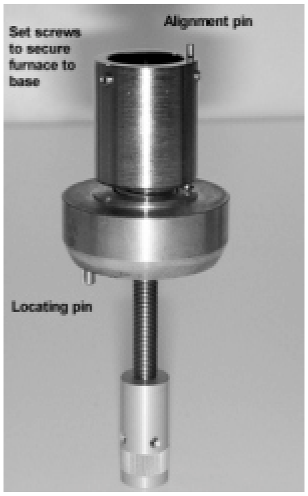
1. Disconnect the plastic thermocouple connector plug from the base of the furnace by gently pulling up on the connector plug.

   CAUTION: Do not bend the thermocouple severely as this may damage it. If the thermocouple appears old, worn, or broken, replace it (P/N 0319-1253).

2. Remove the tip of the thermocouple from inside the furnace by pulling the thermocouple straight down toward the bottom of the furnace.
3. Take the new thermocouple and, while looking inside the furnace, feed the tip of the new thermocouple through the thermocouple slot in the furnace stem until it is approximately 1 or 2 mm below the half-height of the furnace wall.
4. Center the thermocouple using tweezers.
5. Plug the plastic thermocouple connector plug on the base of the thermocouple into the base of the furnace. There are two small pins in the furnace base where the thermocouple connects. Be careful not to bend or break the thermocouple pins in the furnace base.

Replacing the Standard Furnace

1. Slide the platinum furnace shield over the furnace
2. Place the furnace on the furnace base. Note that there is a locating pin on the mounting assembly that fits into the hole on the bottom of the furnace to assist alignment.



3. Replace the metal furnace tube base over the furnace. Position it so that the cutout at the base allows the furnace electrical connection to exit without being bent or pinched. Gently push it down until it is fully engaged and seated down on the brass base.
4. Place the furnace assembly onto the mounting assembly.
5. Place the knurled furnace locking nut over the furnace.
6. Place the split O-ring underneath the knurled furnace locking nut.
7. Straighten the furnace and tighten the knurled nut.

8. Place the spring over the furnace and until it sits in the top of the metal furnace base. make sure that the top of the spring is straight.
9. Place the slotted plate on top of the spring. Make sure the plate is straight.
10. Place the quartz insert over the furnace and seat it on the plate.
11. Place the Pyrex furnace tube over the furnace so that it rests on the O-ring on the top of the furnace tube base. Position the exhaust tube so that it is left of center when the furnace is out of the cooling position.
12. Place the clamp around the base of the furnace tube and top of the furnace tube base. The red handle should be placed so that it is facing the cooling housing. The exhaust tube should be to the right of the clamp, as seen in the left-hand photo below. When the furnace swings into the Cooling position, the clamp will not touch the frame of the analyzer. Note the position of the exhaust tube and clamp when the furnace is in the Cooling position in the right-hand photo below.

Furnace in the lowered position

Furnace in the cooling position

13. Plug in the furnace power cord to its connector on the front of the Pyris 1 TGA. There are male and female guides on the connector that allow the connector to go on in only one way.

After installing the standard furnace, you will have to adjust it with respect to the ball joint, hangdown wire, and sample pan.

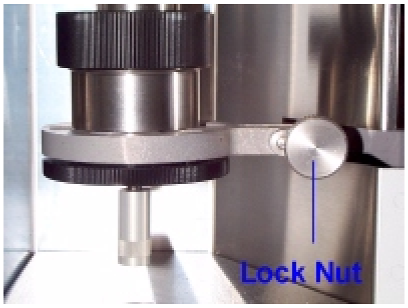
Adjusting the Standard Furnace

If you need to remove the furnace and/or furnace tube, perhaps to clean it, you will have to perform the following steps to ensure proper operation of the instrument:

- **Aligning the Standard Furnace**
- **Adjusting the Height of the Furnace**
- **Centering the Sample Pan**
- **Adjusting the Balance Mechanism**
- **Adjusting the Convection Iris**

Aligning the Standard Furnace

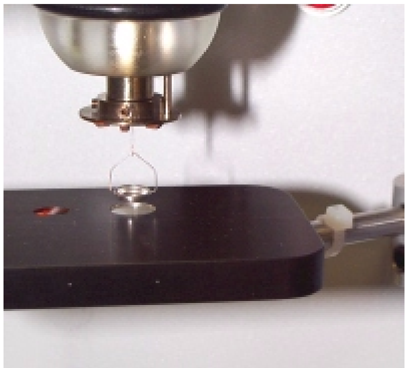
1. Power on the Pyris 1 TGA. The furnace will go to the Cooling position.
2. Press the **Lower Furnace** button on the instrument's control panel. The furnace will swing out to the Lowered position.
3. Remove the lock nut from the furnace mounting assembly and swing the furnace assembly down so that it is at an angle with the instrument.



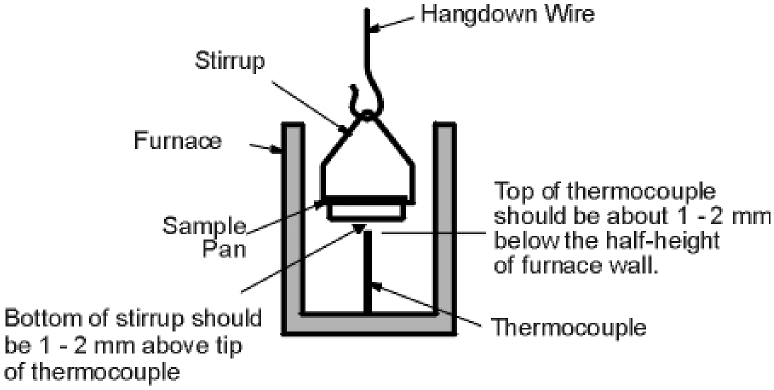
4. Look down into the furnace tube and see if the furnace is exactly centered within the tube. If it is not, loosen the clamp and move the quartz tube around until the furnace is centered. Tighten the clamp.
5. Raise the furnace assembly back up and reinsert the lock nut into the furnace mounting assembly and tighten.
6. Turn off the antistatic device by pressing the **Menu** button on the instrument's control panel three times. At the Antistatic Device display, press the **Scroll Up** button to display the status of the antistatic device. If it is ON, press the **Enter** button to respond Yes to Turn Off? If it is already OFF, press the **Exit** button to return to the default display.
7. Raise the furnace by pressing the **Raise Furnace** button on the control panel. If the furnace is properly aligned, the furnace tube should move smoothly into position around the upper ball joint. You may also want to use the sample loading platform to stop the furnace from rising so that you can make minor adjustments as the furnace tube meets the ball joint.
8. The sample loading platform can be used to control the movement of the furnace while it is ascending. When the platform is in the safe position (to the right), the furnace moves up toward the ball joint. When the platform is not in the safe position, the furnace will not move. Move the platform back and forth to stop and start the motion of the furnace in small increments.
9. To adjust the horizontal position of the furnace tube, slightly loosen the furnace adjustment nut at the base of the furnace mounting assembly.
10. Press the **Lower Furnace** button on the instrument's control panel.
11. Press the **Raise Furnace** button on the instrument's control panel.
12. As the furnace gets close to the ball joint, position the furnace assembly so that the furnace tube moves smoothly onto the ball joint. When the tube and the ball joint appear to be aligned, allow the furnace to go all the way up by pushing the sample loading platform into the safe position and leaving it there.
13. Adjust the furnace assembly manually until it appears that the furnace is aligned correctly (i.e., the furnace tube is perpendicular to the table top). Lock the furnace assembly into position by tightening the furnace adjustment nut at the base of the furnace mounting assembly.

Adjusting the Height of the Furnace

1. Bring the furnace to the Lowered position by pressing the **Lower Furnace** button on the instrument's control panel.
2. Place a stirrup onto the sample loading platform. Place a sample pan in the stirrup. Hang the stirrup with sample pan onto the hangdown wire by using the sample loading platform to guide the stirrup onto the hangdown wire and gently lower the platform so the stirrup hangs from the wire.



3. Press the **Raise Furnace** button.
4. Check the position of the stirrup with respect to the top of the thermocouple. The bottom of the stirrup should be approximately 1 – 2 mm above the tip of the thermocouple. The tip of the thermocouple should be 1 – 2 mm below the halfway point of the furnace wall.



5. Adjust the height of the furnace using the furnace height adjustment knob located below the furnace adjustment ring. Turn the knob clockwise to raise the furnace and counterclockwise to lower the furnace.
6. Make sure that the sample pan hangs in the center of the furnace. It is important that the sample pan does not touch the side wall of the furnace or the tip of the thermocouple.

Centering the Sample Pan

If the sample pan is not centered, do the following:

1. Press the **Lower Furnace** button.
2. Check the status of the antistatic device, either on the Status Panel of the Pyris software, if it is on, or by going into the instrument's menu. Press the Menu button three times. At the

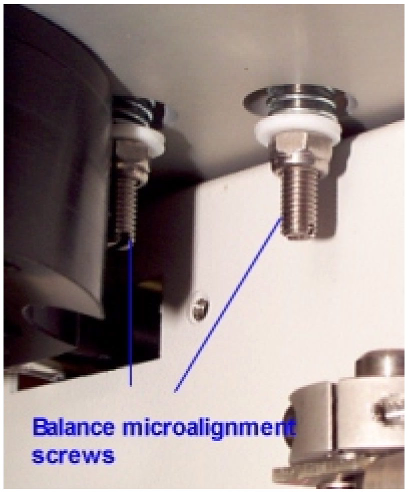
Antistatic Device display, press the **Scroll Up** button; the status of the antistatic device is displayed. If it is not Auto Enabled, press the **Enter** button to enable the device. Press the **Exit** button to return to the default display.

3. Press the **Raise Furnace** button. The furnace will stop below the ball joint for about 15 seconds. It will then finish its trip and engage the ball joint.
4. Loosen the furnace adjustment ring and carefully move the furnace so that the sample pan is in the center of the furnace. When it is centered, tighten the ring.

CAUTION: If the sample pan cannot be centered in the furnace using the adjustment procedure above, then the balance mechanism has to be adjusted.

Adjusting the Balance Mechanism

If the hangdown wire cannot be centered by moving the furnace using the furnace adjustment ring, you will have to adjust the position of the balance, and therefore the position of the hangdown wire, using the two X-Y microbalance adjustment screws.



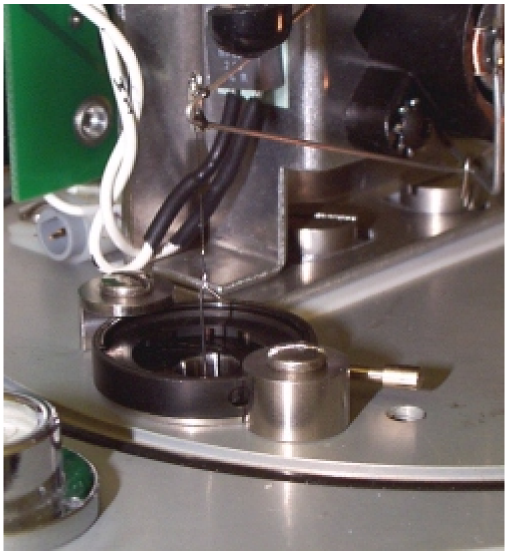
1. If not already, enable the antistatic device automode. Press the **Menu** button three times. At the Antistatic Device display, press the **Scroll Up** button; the status of the antistatic device is displayed. If it is not Auto Enabled, press the **Enter** button to enable the device. DO NOT press the **Exit** button yet.
2. Make sure that the upper fan is off; otherwise, the hangdown wire will swing. Press the **Scroll Up** button twice. With the Fan mode displayed, press the **Menu** button to see the status of the fan. If Automode is ON, turn it OFF by pressing the **Enter** button. Press the **Exit** button to return to the default display.

   Before proceeding, you may want to check that the **antistatic device is operating properly**.
3. Press the **Raise Furnace** button.
4. If the hangdown wire needs to be centered, carefully use a screwdriver to adjust the position of the balance, and, therefore, the position of the hangdown wire. The left-hand screw (that closest to the antistatic device) adjusts the Y position and the right-hand screw adjusts the X position.

Once the sample pan is centered within the furnace tube, it is important that the hangdown wire not touch the anticonvection iris when it is closed. The iris prevents reaction gases from rising into the balance chamber from the furnace. After adjusting the balance mechanism, the hangdown wire may no longer be centered within the iris.

Adjusting the Convection Iris

1. Lift the top cover of the analyzer up to access the balance dome.
2. Remove the balance dome by turning the locking devices and pulling the arms out of the groove in the dome. Lift the dome straight up and away from the balance. The iris is now exposed.
3. Open the iris all the way by pushing the arm clockwise.
4. Loosen the screws on the two stands that hold the iris in place.
5. Move the iris around until the hangdown wire is centered in the iris.



6. When the hangdown wire is centered, tighten the screws on the stands. Do not overtighten; the iris arm will not be able to move.
7. Close the iris as far as possible without it touching the hangdown wire by turning the lever counterclockwise.
8. Replace the dome back onto the balance plate and lock in place.
9. Lower the access cover.

Standard Furnace Tube Maintenance

Maintenance of the standard furnace tube consists of the following procedures:

- **Removing the Standard Furnace Tube**
- **Cleaning the Standard Furnace Tube**
- **Replacing the Standard Furnace Tube**

Removing the Standard Furnace Tube

1. Make sure that the furnace is in the lowered position.
2. Remove the exhaust line from the furnace tube side arm.
3. Remove the clamp around the base of the furnace tube.
4. Remove the glassware.

CAUTION: Do not pull the furnace tube very quickly or at an angle to avoid breaking the thin stem on the furnace.

Replacing the Standard Furnace Tube

This procedure is performed after removing the standard furnace tube.

1. Carefully place the tube over the furnace and place on the O-ring at the top of the metal furnace base. Position the furnace tube so that the exhaust tube connector is left of center while the furnace is in the lowered position. The exhaust tube connector should be able to go into the cool position without hitting the side of the analyzer.
2. Place the clamp around the furnace tube bottom and the top of the furnace base and tighten. Make sure that the red piece is just to the left of the exhaust tube.

3. Replace the exhaust tube to the furnace side arm.

   Note the positioning of the clamp and exhaust tube with respect to the autosampler when the furnace is in the lowered position, and with respect to the wall of the cooling area with the furnace in the cool position:

Furnace in lowered position

Furnace in cooling position

Standard Furnace Hangdown Wire Maintenance

There are three hangdown wires associated with the standard furnace: a quartz (P/N N537-0488) and a nichrome (P/N N537-0490) wire with the special feature for use with the autosampler, and a quartz wire (P/N N537-0487) for the Pyris 1 TGA without autosampler. There is also an optional nichrome hangdown wire for the analyzer without the autosampler (P/N 0250-7639 in N537-0470). For a nonautosampler system, a platinum stirrup hangs from the wire and supports a platinum sample pan. For an autosampler system, a ceramic crucible hangs directly on the hangdown wire. The high temperature furnace uses a platinum hangdown wire (N519-0276) upon which a stirrup is placed to hold the sample pan.

These wires come in kits that are included in the Standard Pyris 1 TGA Spares kit or the Autosampler Spares kit.

Maintenance of the standard furnace hangdown wire consists of the following:

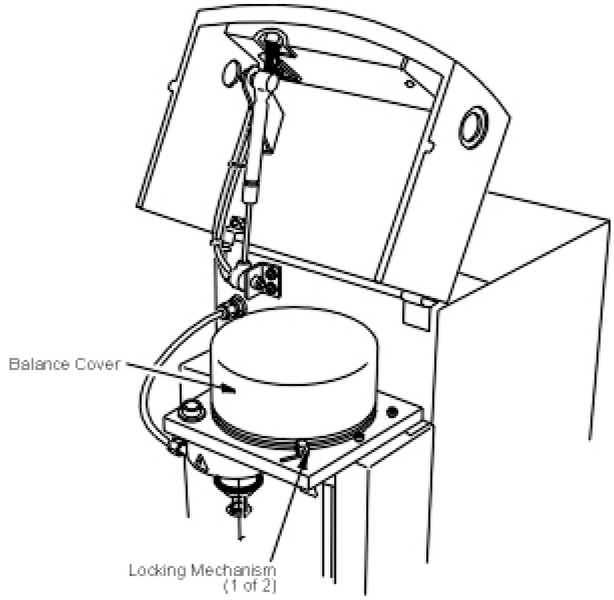
- **Removing the Standard Furnace Hangdown Wire**
- **Installing the Standard Furnace Hangdown Wire**

WARNING: Before performing the procedures below, make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.

Removing the Hangdown Wire

1. If present, lift the visor on the front of the Pyris 1 TGA all the way up. Raise the cover that allows access to the balance. The dome-shaped metal cover that protects and isolates the balance mechanism should be visible.

2. The balance dome is secured by two locking levers. Flip the levers and then pull them out so that the locking arm comes out of the groove in the dome. Lift the cover straight up and off to expose the balance mechanism.

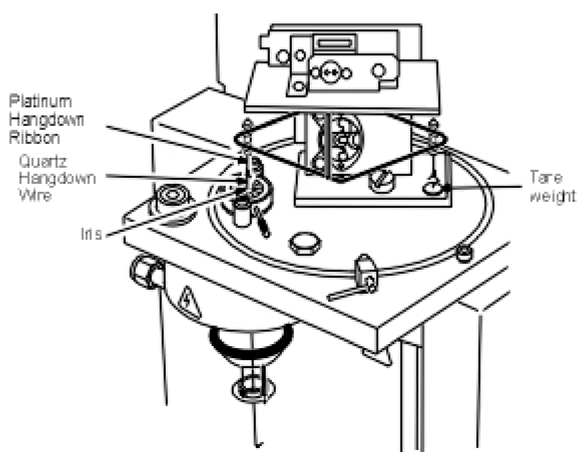


3. If there is a sample pan on the hangdown wire:

    a. If you have an autosampler, click on the **Autosampler Control** button on the control panel. In the dialog box, enter the position to which to unload the sample pan and click on Unload Sample. The autosampler will go to the Load position, the sample pan will be placed in the assigned position, and the autosampler will return to the Safe position.

    b. If there is no autosampler, move the sample loading platform under the sample pan. Raise the platform up until the pan rests on the platform and the stirrup is lifted off of the bend in the hangdown wire. Swing the platform to the right to remove the sample pan and stirrup assembly.

    A platinum balance ribbon is connected to the left and right arms of the balance. The ribbon has a loop at its end from which the hangdown wire is hung on the left side of the balance. A tare weight, specific for the particular hangdown wire used, is hung from the ribbon on the right side of the balance to offset the weight of the hangdown wire, stirrup, and sample pan or crucible.



CAUTION: The platinum ribbon is extremely fragile. Care should be taken not to exert force on the loop of the balance ribbon or on the hangdown wire after it is attached. Handle tweezers with extra care when placed in the vicinity of the platinum balance ribbon, the hangdown wire, or the stirrup. Tweezers can catch any of the components and cause damage.

4. To remove the hangdown wire from a nonautosampler Pyris 1 TGA:
   a. Make sure that the anticonvection iris in the balance area is open all the way.
   b. With the sample loading platform in the load position, carefully raise the platform until it comes in contact with the hangdown wire.
   c. While looking at the platinum ribbon loop, continue to lift the sample loading platform until the hangdown wire hook begins to rise out of the loop.
   d. Using tweezers, carefully remove the hangdown wire from the ribbon loop. Let go of the hangdown wire.
   e. Lower the sample loading platform. The hangdown wire should descend through the ball joint tube.
   f. Grasp the hangdown wire near the sample loading platform with tweezers and remove it.

   To remove the hangdown wire from an autosampler system:
   a. Place the hangdown wire guide on the end of the convection flange at the end of the ball joint.
   b. Make sure that the anticonvection iris is open all the way.
   c. Use tweezers to carefully remove the hangdown wire from the platinum ribbon loop. Let the hangdown wire drop onto the wire guide.
   d. Grasp the hangdown wire with the tweezers while removing the guide.
   e. Carefully guide the hangdown wire down and out of the ball joint tube.

Installing the Hangdown Wire

1. For a nonautosampler Pyris 1 TGA, push the sample loading platform to the load position and raise it as high as it will go; center it below the ball joint.

   For an autosampler system, place the hangdown wire guide on the convection flange below the ball joint.
2. Make sure that the convection iris in the balance chamber is open.
3. Carefully remove a new hangdown wire from its container. Handle it with one hand.
4. From the balance chamber, drop the new hangdown wire through the hangdown wire tube. The end of the wire will rest on either the guide or the loading platform.
5. For a nonautosampler system, lower the sample loading tray until the bend in the top of the hangdown wire is approximately the same level as the loop in the platinum hangdown ribbon. Using tweezers, grasp the hangdown wire with the wire tip facing the platinum ribbon loop. Insert the wire tip through the loop and release it.

   For an autosampler system, check the orientation of the hook at the end of the hangdown wire. It should face toward the autosampler. Use tweezers to grasp the hangdown wire near the top. Remove the wire gauge with your other hand. Insert the tip of the hangdown wire through the ribbon loop and release. Go to step 7.

6. For a nonautosampler system, rotate the platform to the right, place a stirrup onto the platform, then place a sample pan in the stirrup. Swing the platform underneath the hangdown wire. Use the platform to place the stirrup onto the end of the hangdown wire. Lower the sample loading platform and rotate it completely to the right.

   For an autosampler system, place an empty crucible at the end of the hangdown wire.

   NOTE: Make sure that the **proper tare weight** is in place on the other side of the balance. If you need to change the weight, carefully remove the present one using tweezers. Pick up the new tare weight with tweezers and carefully hang it from the loop of the hangdown ribbon on the right side of the balance mechanism.

   CAUTION: The platinum hangdown ribbons are extremely fragile. Be careful not to exert force on the loop of the hangdown ribbon or on the hangdown wire after it is attached. Handle tweezers carefully when placing them near the platinum hangdown ribbons, the hangdown wire, or stirrups as they may catch on one of the components.

7. Replace the balance cover over the balance and lock it into place by pushing in the lever arms and then flipping the levers.
8. Lower the access cover.

After installing a new hangdown wire on either system, you may have to **adjust the height of the furnace** with respect to the sample pan.

If you have an autosampler, you should perform the **Align Gripper** and **Align Tray** procedures which are accessed from the **Autosampler Control dialog box**.

Tare Weight Requirements

Which tare weight to use with your Pyris 1 TGA with standard furnace depends on the system, the hangdown wire, and the sample pan. Use the chart below to determine the correct tare weight to use.

Sample holder / hangdown wire combinations	**N537-0471 194 mg**	**N537-0472 272 mg**	**N537-0473 226 mg**
Manual Loading			
Platinum pan, quartz wire (standard)		✓	
Ceramic pan, quartz wire (standard)	✓		
Platinum pan, thin NiCr wire (option)			✓
Autosampler			
Ceramic pan, thick NiCr wire (autosampler standard)		✓	
Ceramic pan, quartz wire (autosampler option)	✓		

The tare weight requirement for a Pyris 1 TGA with a high temperature furnace is to use **N537-0499** (525 mg) to balance off the following:

N519-1354	Platinum stirrup
N519-1353	Platinum pan (large)
N537-6758	Platinum – rhodium hangdown wire

High Temperature Furnace Maintenance

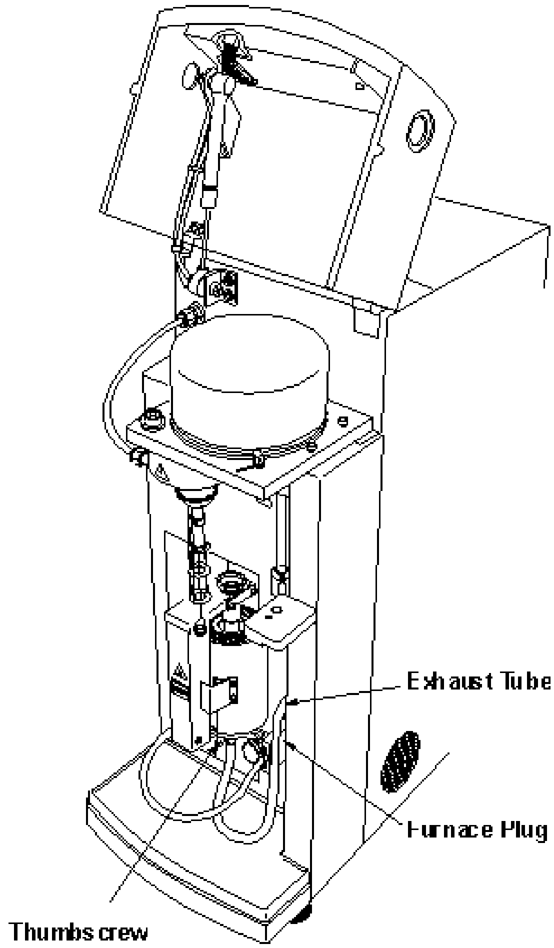
High temperature furnace maintenance consists of the following procedures:

- **Removing the High Temperature Furnace**
- **Replacing the High Temperature Furnace**
- **Adjusting the High Temperature Furnace**

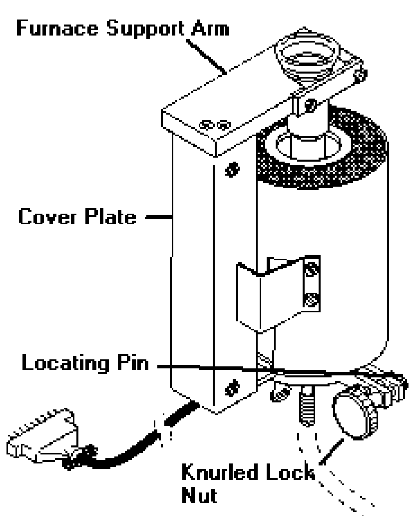
WARNING: **Before performing these procedures, shut down the system and remove the line power. Make sure that the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.**

Removing the High Temperature Furnace

1. Disconnect the furnace power cord from the connector on the front of the analyzer. To disconnect the cord, squeeze the silver locking tabs and pull forward gently on the connector.
2. Unplug the furnace exhaust line where it enters the front panel of the analyzer.



3. Support the furnace by grasping the cover plate of the furnace with one hand. Unscrew and remove the knurled lock nut located on the furnace mounting assembly with your other hand. Remove the locating pin by sliding it forward. The entire furnace assembly can now be removed.



Replacing the High Temperature Furnace

1. If replacing a high temperature furnace that already has a furnace tube installed, go to step 7.

   If replacing a removed furnace with a new one, carefully remove the high temperature furnace and the ceramic furnace tube from their protective packaging.

2. Remove the two furnace tube wing nuts from the furnace tube support arm.
3. Slide the two O-rings over the furnace tube. They should be on the upper portion of the tube just below the flange.
4. Insert the furnace tube into the high temperature furnace.
5. Secure the furnace tube in the furnace tube support arm with the furnace tube wing nuts. Be sure that the wing nuts are fingertight.
6. Attach the exhaust line to the bottom of the furnace tube.
7. Hold the furnace so that the arms on the furnace mounting assembly are positioned near the furnace lift shaft. Line up the holes in the shaft and the arms on the furnace mounting assembly and place the locating pin through the left hole.
8. Replace the knurled lock nut in the right hole, threading it until it is fingertight.
9. Plug the furnace exhaust line into its tube connector on the front panel of the Pyris 1 TGA.
10. Plug the furnace power cord into its connector on the front of the analyzer. There are male and female guides on the connector that allow the connector to go on only one way.

NOTE: The baffle assembly, thermocouple, and hangdown wire should already be in place.

After installation, you will have to **adjust the high temperature furnace** with respect to the ball joint and hangdown wire.

High Temperature Furnace Tube Maintenance

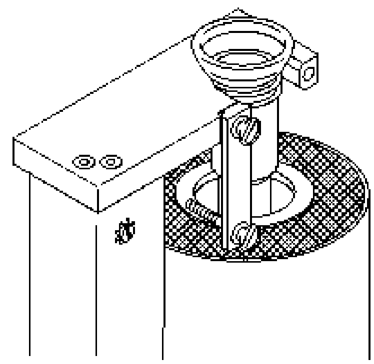
Maintenance of the high temperature furnace tube consists of the following procedures:

- **Removing the High Temperature Furnace Tube**
- **Replacing the High Temperature Furnace Tube**

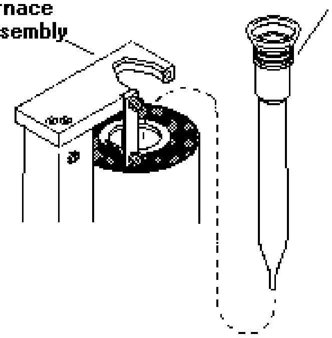
WARNING: Before performing the procedures below, shut down the system and remove the line power. Make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.

Removing the High Temperature Furnace Tube

1. Remove the high temperature furnace.
2. Remove the exhaust tube from the connector at the base of the furnace.
3. Place the furnace horizontally on a table of bench with the furnace tube wing nuts and thermocouple facing up.
4. Loosen and remove the two furnace tube wing nuts and the metal holddown bar.

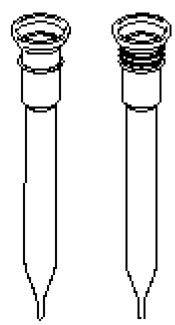


5. Gently slide the furnace tube out of the furnace. Note the location of the O-rings on the furnace tube.



Replacing the High Temperature Furnace Tube

1. Install the two O-rings (P/N 0990-2161) on the upper portion of the furnace tube. These O-rings protect the furnace tube from damage when the tube is locked in place.



2. Slide the furnace tube into the furnace from the top. The two O-rings should rest in the cutout in the furnace tube support arm.
3. Lock the furnace tube into position using the two furnace tube wing nuts and metal holddown bar.
4. Replace the furnace.

High Temperature Furnace Oven Thermocouple Maintenance

Furnace temperature control of the Pyris 1 TGA high temperature furnace is accomplished through the use of an oven thermocouple. The procedures below describe its removal and replacement.

⚠ **WARNING:** **Before performing the procedures below, shut down the system and remove the line power. Make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.**

Removing the High Temperature Furnace Oven Thermocouple

1. Remove the high temperature furnace.
2. Place the furnace horizontally on a table or bench with the furnace tube wing nuts and oven thermocouple cover facing up.
3. Loosen and remove the two screws securing the cover for the oven thermocouple. Remove the cover.
4. Loosen and remove the two screws securing the cover plate. Slide the cover plate off.

NOTE: As you slide the cover plate off, make sure that the rubber grommet is not stuck on the cover plate. The grommet should slide off as the cover plate is removed.

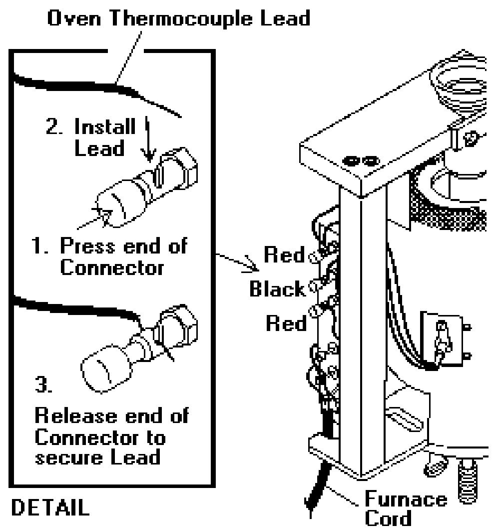
5. Loosen (but do not remove) the screw securing the oven thermocouple clip. Swing the clip to the side.
6. Pull the oven thermocouple out of the furnace.
7. If necessary, remove the ceramic liner from the oven thermocouple and replace it. (The liner rests at the base of the oven thermocouple tube. Turn the furnace over and shake gently to remove it.)

NOTE: Do not remove the other wires connected at the oven thermocouple connections.

8. Disconnect the red and black leads from the oven thermocouple connections by pressing down gently on the connectors and sliding the wires out of the groove in the connector.

Replacing the High Temperature Furnace Oven Thermocouple

1. Connect the new oven thermocouple to the thermocouple connections. Match the red thermocouple wire to the thermocouple connector that has a red wire attached to it. Repeat the procedure for the black thermocouple wire.



2. Place the black rubber grommet over the tip of the oven thermocouple. Place the thermocouple into the furnace so that its tip rests at the base of the ceramic liner.
3. Secure the thermocouple in position by swinging the oven thermocouple clip down and tightening the screw to lock the clip in place.

NOTE: As you tighten the screw to lock the oven thermocouple clip in place, make sure that the clip does not rotate or you may damage the thermocouple.

4. Replace the cover plate, making sure that the rubber grommet slides into the cutout on the plate. Secure the plate into position using the two screws.
5. Replace the furnace.

High Temperature Furnace Sample Thermocouple Maintenance

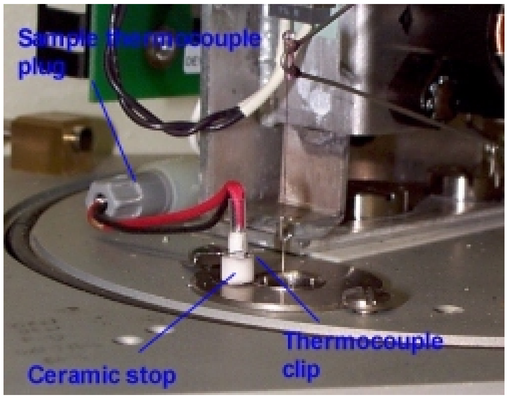
WARNING: **Before performing the procedures below, shut down the system and remove the line power. Make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.**

CAUTION: Use care when working in the area of the balance mechanism since it contains delicate parts and is highly sensitive.

Removing the High Temperature Furnace Sample Thermocouple

1. Raise the visor on the front of the Pyris 1 TGA all the way up and then raise the cover with the control panel display. The balance dome is now visible.
2. Remove the dome by flipping the two locking mechanisms and then pulling the lever arms out of the groove of the dome. Lift the cover straight up and off to expose the balance mechanism.
3. The sample thermocouple passes through a hole on the left side of the balance mechanism and down through the baffle assembly to rest near the sample pan. The thermocouple has two

wires that go into a plug on its upper end which plugs into a connector behind the balance mechanism. Unplug the thermocouple from behind the balance mechanism.



4. Loosen (but do not remove) the screw that secures the sample thermocouple clip. Swing the clip to the side.
5. Pull the thermocouple straight up from the top of the Pyris 1 TGA to remove it.

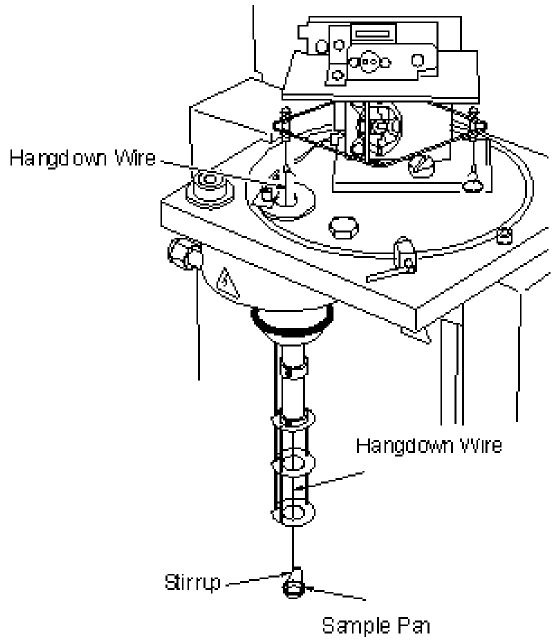
Replacing the High Temperature Furnace Sample Thermocouple

1. Feed the new thermocouple through the hole on the left side of the balance assembly and down through the thermocouple holes in the side of the baffle assembly. The ceramic stop on the end of the thermocouple should be flush with the ring on the balance plate.
2. Secure the thermocouple into position by swinging the sample thermocouple clip over and tightening the screw to lock the clip in place
3. Plug the thermocouple into the connector at the back of the balance mechanism.
4. Replace the dome cover and lock it in place by pushing the lever arms into the groove and flipping the levers.

High Temperature Furnace Hangdown Wire Maintenance

Removing the High Temperature Furnace Hangdown Wire

1. If a stirrup is at the end of the hangdown wire, move the sample loading platform directly under the stirrup. Raise the platform up until the stirrup rests on the platform and the stirrup comes off the bend in the hangdown wire.
2. Using tweezers, remove the stirrup.



3. Raise the visor all the way up and then raise the analyzer's control panel all the way back to access the balance mechanism.
4. Remove the balance dome by flipping the two locking levers, pulling the lever arms from the groove in the dome, and lifting the dome straight up and off of the balance.

A platinum balance ribbon (P/N N537-0478) is connected to the left arm of the balance. A platinum hangdown wire (P/N N519-0276) is hung from the ribbon's loop. A tare weight is hung from the right side of the balance to tare off the weight of the hangdown wire and sample pan.

5. Carefully raise the sample loading platform until it comes in contact with the hangdown wire. While looking at the loop in the platinum ribbon, continue to raise the sample loading platform until the hangdown wire hook begins to come out of the loop.
6. Using tweezers, carefully remove the hangdown wire from the platinum ribbon loop.
7. Lower the sample loading platform.
8. When you are sure that the hangdown wire is no longer attached to the ribbon loop, grasp the hangdown wire near the sample loading platform with the tweezers and remove it.

Installing the High Temperature Furnace Hangdown Wire

1. Push the sample loading platform to the load position and raise it as high as it will go. Center it below the baffle assembly.
2. Drop a new hangdown wire through the hangdown wire hole on the left side of the balance chamber. It will rest on the platform.
3. Lower the sample loading platform until the bend in the hangdown wire is at approximately the same level as the platinum ribbon loop.
4. Using tweezers, grasp the hangdown wire with the tip facing the loop. Insert it through the loop and release the wire. Withdraw the tweezers cautiously so as not to catch them on any components of the balance.
5. Replace the stirrup and sample pan.
6. Replace the dome and lock it in place.

Part Numbers

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Your instrument should be one of the following:

Pyris 1 TGA with Standard Furnace	N537-0742
Pyris 1 TGA with High Temperature Furnace	N537-0743
Pyris 1 TGA with Standard Furnace and Autosampler	N537-0744

Each system comes with its own spares kit:

Pyris 1 TGA Standard Furnace Spares Kit	(N537-0474)
Pyris 1 TGA High Temperature Furnace Spares Kit	(N537-0483)
Pyris 1 TGA Autosampler Spares Kit	(N537-0484)

Standard Furnace Spares Kit

The Standard Furnace Spares Kit (N537-0474) comprises a plastic box and bagged items, both of which are in the shipping carton's tray. The Spares Kit consists of the following items:

Part No.	**Description**	**Quantity**
0154-1498	"H" restrictor	3
0250-6483	Teflon tubing 1/8-in. o.d.	24 ft
0250-6515	Tygon tubing (black) 1/4-in. o.d.	20 ft
0250-6518	Tygon tubing	6 ft
0250-8084	Tygon FEP lined tubing (less permeable than 0250-6518)	3 ft
0990-3094	Swagelok insert for 1/8-in. i.d. tubing	6
0990-3150	Tube fitting, 1/4-in. NPT male to 1/4-in. o.d. Swagelok	1
0990-3196	Tube fitting, 1/4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3428	Tube fitting, 1/8-in. NPT male to 1/4-in. o.d. Swagelok	1
0990-3630	Air filter, 1.8-in. NPT connections	1
0990-3715	Female branch Tee, 1.4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3716	Adapter, 1.8-in. NPT female to 1/4-in. o.d. Swagelok	2
0990-3906	Tube fitting, 1/4-in. NPT female to 1/8-in. o.d. Swagelok	2

0990-8134	Teflon plastic tape	2
N537-0485	Pyris 1 TGA Standard Furnace Kit	1
N822-1178	RS232 cable	1

The plastic box shipped with the bagged items is the Pyris 1 TGA Standard Furnace Kit (N537-0485). It consists of the following items:

Part No.	Description	Quantity
0219-0071	Calibration Standards Kit	1
0319-0264	Platinum sample pan kit	1
0319-0265	Platinum stirrup kit	1
0990-2031	O-ring, packing, furnace assembly	2
0990-2131	O-ring, Pyrex ball joint seal	2
0990-2163	O-ring, dome seal	2
0990-2213	O-ring, ball assembly seal	2
0990-2223	O-ring, furnace base seal	2
0990-3011	Tube clamp	6
0990-3480	Rubber cement tube	1
0992-3481	Hex wrench 1.3 mm (for anticonvection shield setscrews)	1
0990-4905	Pinch clamp #35 (optional for dome)	1
0990-7236	Hex wrench 0.050 in.	1
0990-8397	100-mg calibration weight	1
0990-8400	Tweezers	1
0998-1611	Fuse, 1.0 A, Slow Blow (on main board)	6
0998-1614	Fuse, 2.0 A, Slow Blow (on main board)	3
0998-1617	Fuse, 5.0 A, Slow Blow (protects main board)	3
0998-1619	Fuse, 0.375 A, Slow Blow (for antistatic device)	3
0998-1753	Fuse, 10 A, Slow Blow (in power entry module, protects instrument)	3
N537-0466	Magnet assembly	1
N537-0471	Tare weight, 194 mg	1
N537-0472	Tare weight, 272 mg	1
N537-0478	Hangdown ribbon kit	1
N537-0487	Standard quartz hangdown wire kit	1
N537-6556	Quartz insert (for furnace assembly)	2
N537-6562	Teflon slip washer	2
N537-6751	Anticonvection guide, 4 mm	1

High Temperature Furnace Spares Kit

The High Temperature Furnace Spares Kit (N537-0483) comprises a plastic box and bagged items, both of which are in the shipping carton's tray. The Spares Kit consists of the following items:

Part No.	Description	Quantity
0154-1498	"H" restrictor	3
0250-6483	Teflon tubing 1/8-in. o.d.	24 ft
0250-6515	Tygon tubing (black) 1/4-in. o.d.	20 ft
0250-6518	Tygon tubing	6 ft
0250-8084	Tygon FEP lined tubing (less permeable than 0250-6518)	3 ft
0990-3094	Swagelok insert for 1/8-in. i.d. tubing	6
0990-3150	Tube fitting, 1.4-in NPT male to 1.4-in. o.d. Swagelok	1
0990-3196	Tube fitting, 1/4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3428	Tube fitting, 1.8-in. NPT male to 1/4-in. o.d. Swagelok	1
0990-3629	Pressure relief valve	1
0990-3630	Air filter, 1/8-in. NPT connections	1
0990-3715	Female branch Tee, 1/4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3716	Adapter, 1/8-in. NPT female to 1/4-in. o.d. Swagelok	2
0990-3906	Tube fitting, 1/4-in. NPT female to 1/8-in. o.d. Swagelok	2
0990-8134	Teflon plastic tape	2
N822-1178	RS232 cable	1
N537-0486	Pyris 1 TGA High Temperature Furnace Kit	1

The Pyris 1 TGA High Temperature Furnace Kit (N537-0486) is the plastic box that is shipped with the analyzer and is in the cardboard tray along with the bagged items. This kit consists of the following items:

Part No.	Description	Quantity
0990-2131	O-ring, Pyrex ball joint seal	2
0990-2161	O-ring, high temperature furnace tube	2
0990-2163	O-ring, dome seal	2
0990-2213	O-ring, ball assembly seal	2
0990-3012	Hose clamp	6
0990-3480	Rubber cement tube	1
0990-4905	Pinch clamp #35	1
0990-7222	Hex wrench 0.035 in.	1
0990-7229	Hex wrench 7/64 in.	1
0990-8397	100-mg calibration weight	1

0990-8400	Tweezers	1
0990-8689	Teflon tubing	1
0991-0555	Setscrew 2-56, 0.125 cuppt	3
0998-1611	Fuse, 1.0 A, Slow Blow (on main board)	6
0998-1614	Fuse, 2.0 A, Slow Blow (on main board)	3
0998-1617	Fuse, 5.0 A, Slow Blow (protects main board)	3
0998-1619	Fuse, 0.375 A, Slow Blow (for antistatic device)	3
0998-1753	Fuse, 10.0 A (in power entry module, protects instrument)	3
N519-0276	Platinum hangdown wire kit	1
N519-0279	Platinum stirrup kit	1
N519-0280	Platinum sample pan kit	1
N519-0284	Tare weight	1
N519-1323	Furnace thermocouple clip	1
N519-1356	Sample thermocouple clip	1
N537-0478	Platinum hangdown ribbon kit	1

Accessories

The following are accessories or optional items for use with the Pyris 1 TGA:

Tare weight 226 mg (use with platinum pan and thin NiCr hangdown wire with standard furnace)	N537-0473
Quartz hangdown wire for autosampler	N537-0488
Thin (0.005 in.) NiCr hangdown wire (use with platinum pan and tare weight N537-0473 with standard furnace)	N537-0470
Reaction gas injection port (furnace tube)	n/a
Water-cooled jacket	n/a
Purge cover	n/a
Puncture device (for autosampler system)	n/a

n/a = not available

Technical Specifications

Instrument Design	The Pyris 1 TGA is a vertical design TGA that uses a high sensitivity balance and quick response furnace. The balance is located above the furnace and is thermally isolated from the furnace. A precision hangdown wire is suspended from the balance down into the furnace. At the end of the hangdown wire is the sample pan. The sample pan's position in the furnace is extremely reproducible.
Balance	The balance is housed in a thermostatted chamber that isolates the balance from thermal effects. This design enhances sensitivity, accuracy, and weighing precision. Balance tare: Reproducible to ± 2 μg Balance sensitivity: 0.1 μg Balance accuracy: Better than 0.02% Balance weighing precision: 10 ppm (for a 100-mg sample)
Hangdown Wires	Hangdown wires that suspend the sample pan from the balance are made of high temperature quartz, nichrome, or platinum.
Sample Pans	The standard furnace sample pans are made of platinum or ceramic with a capacity of 60 μL. The high temperature furnace uses sample pans made of platinum or ceramic with a capacity of 250 μL. Additionally, disposable sample pan liners made of aluminum or stainless steel make cleanup easy.
Standard Furnace	The TGA standard furnace uses a microfurnace that has a low thermal mass for quick cooling and equilibration. The platinum heat element acts as a resistance temperature detector (PRTD) under tight feedback control. Accurate program temperature control reduces overshoot to 0.2°C at 100°C/min, which results in stepwise analysis precision. The sample temperature is sensed by a chromel–alumel thermocouple. Standard furnace temperature range: subambient to 1030°C Standard furnace scanning rates: 0.1°C/min to 200°C Temperature precision: ± 2°C
High Temperature Furnace	An optional external furnace is constructed with a platinum/30% rhodium heating element. The sample temperature is sensed by a platinum/10% rhodium–platinum thermocouple. High temperature furnace temperature range: 50°C to 1500°C High temperature furnace scanning rates: 0.1°C/min to 50°C/min Temperature precision: ± 5°C
Sample Atmosphere	The sample atmosphere may be static or dynamic. Typical gases used are nitrogen, argon, helium, carbon dioxide, air, oxygen, or other inert or reactive gases. Analyses are done at normal pressure or at reduced pressures.
Control	The Pyris 1 TGA is controlled by Pyris Software for Windows. Automation is controlled by Pyris Player.
Autosampler	The autosampler can run up to 20 samples unattended. Used most efficiently in conjunction with Pyris Player.

Gas Switching	Reduced furnace volume allows gas switching to be fast, thorough, and efficient. The time to purge the sample area of ambient gases (remove 99% of oxygen) and replace the volume with an inert gas is less than 3 minutes. It takes 10 minutes to achieve a 99.99% oxygen-free environment.
Cooling	The furnace is forced air cooled with an external fan and internal booster purge. All purge products are easily directed to the laboratory exhaust vent. Under normal operation the standard furnace cools down from 1030°C to 40°C in less than 15 minutes. The standard furnace temperature can be continuously monitored regardless of position. Under normal operation the high temperature furnace cools down from 1500°C to 100°C in less than 30 minutes.
Bench Dimensions	Height: 67 cm Width: 28 cm Depth: 60 cm
Power Requirements	100 – 240 V 50 or 60 Hz 10 A max

Updating the Pyris 1 TGA Firmware

The Pyris 1 TGA has the firmware built into the Flash EPROM, which resides on the TAC board inside the analyzer. The firmware is updated via the Pyris software. It can be updated from a floppy disk or from the Internet. Verify that the diskette contains the latest version of the software by double-checking the version printed on the label of the diskette. Then, verify the revision of the ROM software currently loaded on your Pyris 1 TGA. The revision of the firmware can be displayed in the control display on the instrument or in Pyris Configuration.

Displaying the Pyris 1 TGA Firmware Version on the Control Display

1. At the default display, press the **Menu** button.
2. Press the **Scroll Up** button twice to display Firmware Version. The version of the firmware currently installed in the analyzer is displayed on the control panel.
3. Once you verify the version currently installed, you can exit the menu by pressing the **Exit** button.

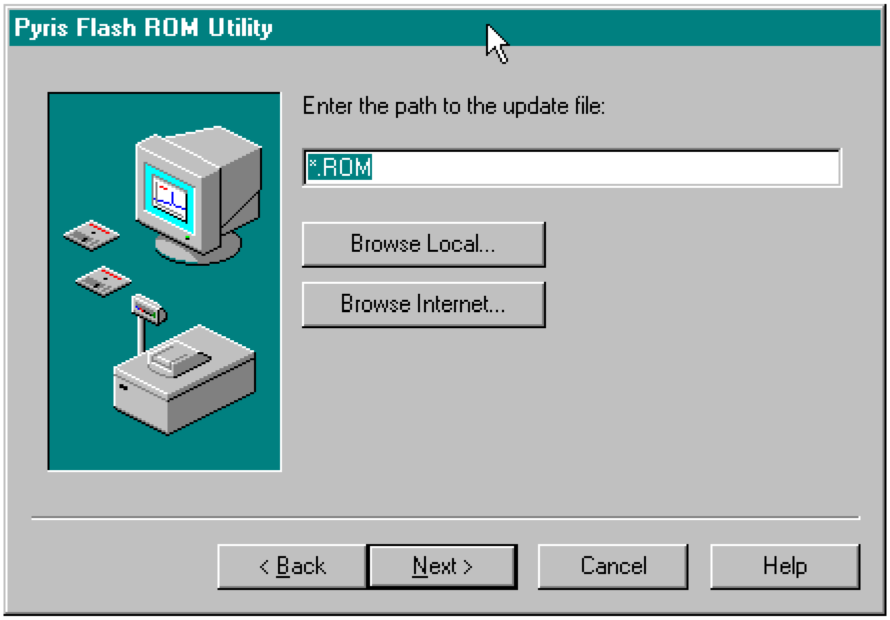
Firmware Version in Pyris Software

1. Open up the Pyris Configuration screen via the following path:
2. Start | Programs | Pyris Software for Windows | Pyris Configuration
3. Select Pyris 1 TGA and then click on the **Edit** button.
4. Click on the **Firmware Version** button to display the current firmware in the Pyris 1 TGA.

Updating the Firmware

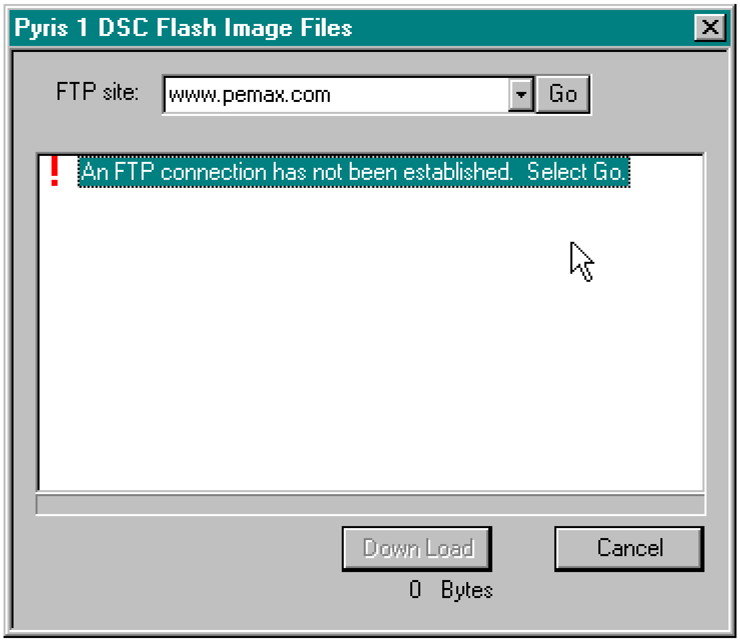
If you checked the firmware version using the instrument's menu, then you need to display the Pyris Configuration dialog box.

1. Click on the **Update Flash EPROM** button. The Pyris Flash ROM Utility is started and its dialog box is displayed.

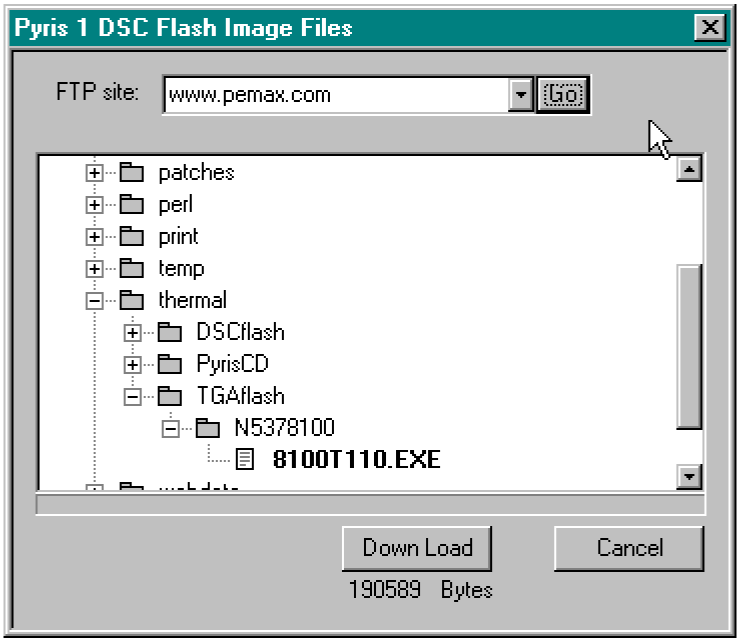


2. Select the location from where you want to download the .ROM file. If the file is on a floppy disk, place the disk in the A: drive and select **Browse Local**. The "Select Update ROM Image File" dialog box appears; the .ROM files on the floppy disk are displayed. Select the most recent .ROM file. Click on Open and the file name is displayed in the path field.

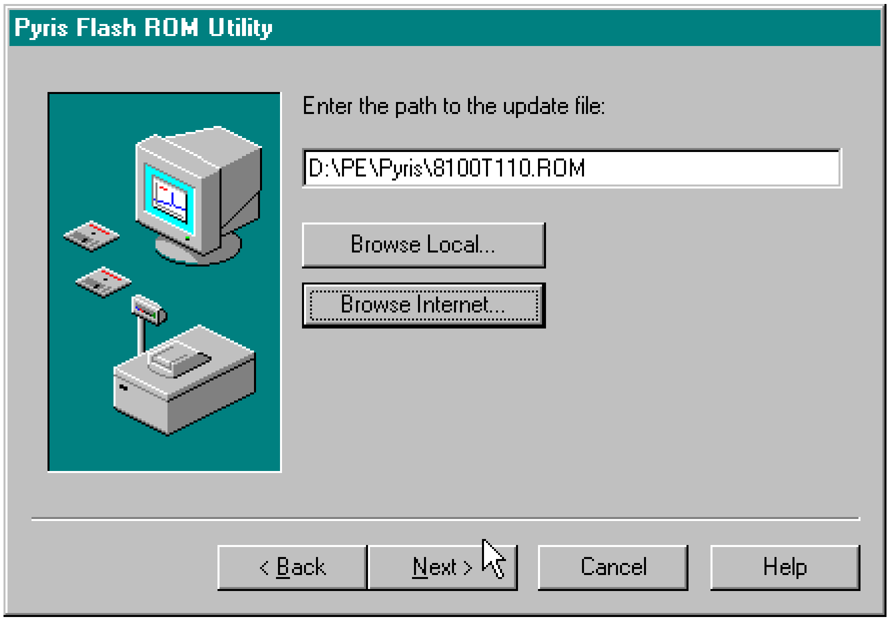
   If you want to download the file from the Internet, select **Browse Internet**. The dialog box below is seen:



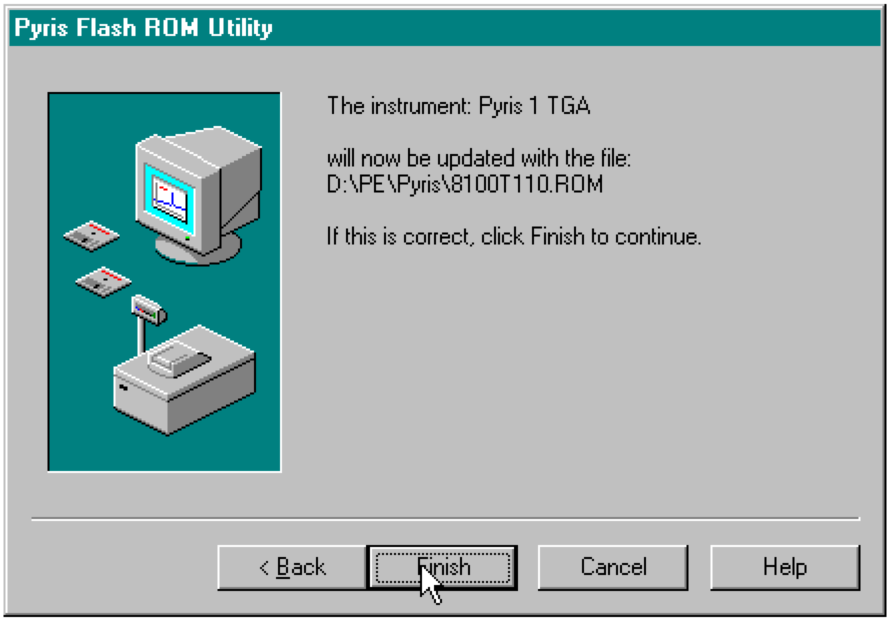
Click on **Go** to make an FTP connection to www.pemax.com. After a short time, a directory structure is displayed ending with a highlighted .exe file:



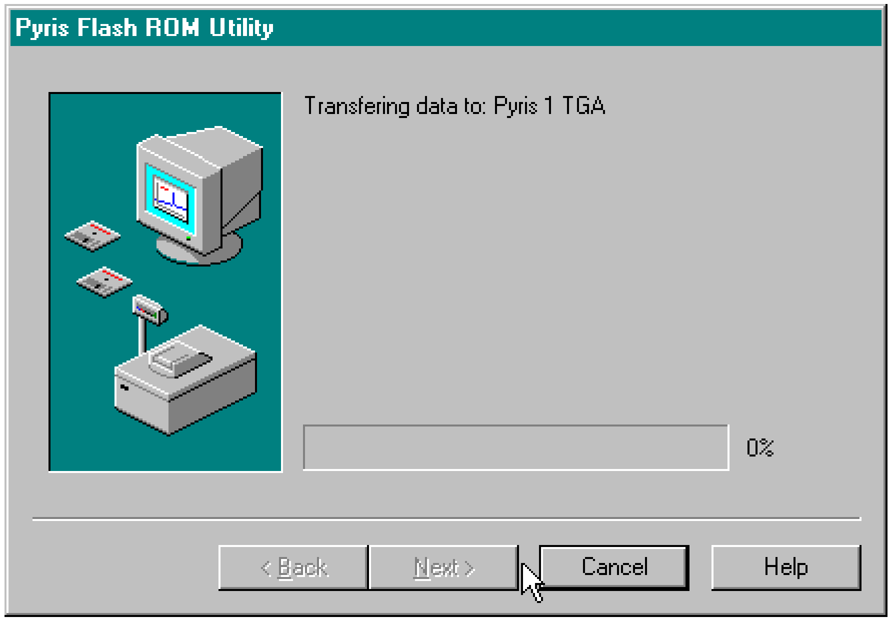
Click on **Down Load** to download this .exe file to the hard disk. This file contains the .ROM file. After downloading, the Pyris Flash ROM Utility screen displays the path and file name of the .ROM file.



3. Click on **Next** and the following is seen:



4. Click on **Finish** to begin transferring the .ROM file to the instrument to update the EPROM.



Updating takes up to 10 minutes. When complete, you will see the message:

The instrument has been successfully updated. Click Exit to end.

Pyris 1 TGA Autosampler

The Pyris 1 TGA autosampler (P/N N537-0744) brings R&D accuracy, sensitivity, and reproducibility to the real world of material inspection. Whether you need in-process product quality inspections or statistical research, the autosampler delivers fast, accurate results consistently. The autosampler's operating principle is a precision index stepper motor with a position encoder. It is directly controlled from Pyris Software for Windows on the computer to which the Pyris 1 TGA is connected. An RS 232 cable connects the autosampler PC board to the Pyris 1 TGA's main board.

The autosampler's operating range is –20°C to 1000°C.

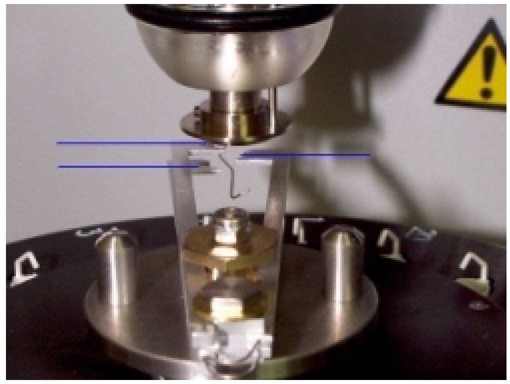
The autosampler's carousel holds up to 20 samples. The samples can be liquids, solids, gels, or powders. Carousel positions are labeled 1 through 20 for easy sample identification. New samples can be added to the carousel to replace completed samples by simply pausing the play list and manually controlling the autosampler from the PC. Increase productivity by starting the first run while preparing and loading additional samples in a second sample ring.

Once empty sample pans are loaded into the sample ring on the carousel, the Tare All feature of Pyris Player' Sample Group can be used to tare the sample pan weights. It automatically skips empty positions and tares only occupied carousel positions. The tare weights are then automatically entered into the play list.

The autosampler carousel can be covered by a transparent, plastic environmental cover. It enables you to observe the movement of the autosampler's mechanical components during operation. The enclosed carousel can be purged with dry or humidity controlled air or nitrogen and operates over the full temperature range of the Pyris 1 TGA. The cover enables the samples to be maintained in a controlled atmosphere while queued to run.

The optional Acupik device automatically pierces a sealed volatile sample pan just before loading for analysis. This unique feature ensures unmeasured weight loss is minimized before the analysis begins.

A special hangdown wire is used with the autosampler; It enables the sample pan to be positioned precisely each time in the furnace. As the autosampler loads or unloads a crucible containing a sample, the hangdown wire is stabilized by a mechanical gripper. The gripper's three arms grasp the hangdown wire using the features or "bends" of the hangdown wire.



The following topics discuss the Pyris 1 TGA Autosampler in more detail:

- **Safety Precautions**
- **How the Autosampler Works**
- **Adjusting the Standard Furnace**
- **Alignment Procedures**
- **Sample Handling**
- **Running a Play List**
- **Troubleshooting**
- **Maintenance**
- **Part Numbers**

Safety Precautions

WARNING: **Be sure that all instrument operators read and understand the following precautions. It is advisable to post a copy of these precautions on or near the Pyris 1 TGA.**

The following precautions must be observed when using the Pyris 1 TGA Autosampler. These are in addition to the precautions that apply to the Pyris 1 TGA.

WARNING: **Your Pyris 1 TGA Autosampler is either installed at the factory or is designed to be installed by a Perkin Elmer Service Engineer if purchased after the Pyris 1 TGA. Do not try to install or uninstall the autosampler without the help of a Service Engineer.**

⚠ WARNING: **Do not remove the gripper or covers. No user serviceable parts are inside the autosampler. Refer all servicing to a qualified service engineer.**

⚠ WARNING: **Never operate the Pyris 1 TGA autosampler in an explosive atmosphere.**

CAUTION: Make sure that all cables and tubes are clear of the autosampler's path when going to the Load and the Safe positions.

CAUTION: Never use the Autosampler Control dialog box to control the autosampler while it is loading or unloading a sample pan to the carousel.

How the Pyris 1 TGA Autosampler Works

The Pyris 1 TGA autosampler is computer controlled and fully automated. The Pyris 1 TGA contains a PC board at the top of the instrument that controls all the stepper motor and position control functions of the autosampler. It communicates with the Pyris TAC board via an RS232 cable that connects at the back of the instrument. If your Pyris 1 TGA has an autosampler, it should be configured into the system so the software recognizes it.

The autosampler houses three circuit boards: (1) Pyris TGA AS sensor board which has two SMT Hall effect sensors on it to determine the Safe and Load positions of the autosampler. (2) The Pyris TGA AS interface board interconnects digital signals from sensors inside the autosampler to the digital I/O cable that connects the autosampler to the autosampler control board. (3) The encoder board reads the absolute optical encoder disk which determines the position of the autosampler sample tray.

There are three identical stepper motors within the autosampler: one drives the carousel that holds the samples, one moves the autosampler from Safe to Load and back, and one moves the gripper. The carousel motor performs two functions: First, it rotates the carousel and, second, it moves the carousel up and down.

The sample tray is driven by a stepper motor with a position encoder. The sample tray can hold up to 20 crucibles. Carousel positions are labeled 1 through 20 for easy sample identification. New samples can be added to the carousel to replace samples that have been analyzed by simply pausing the play list and manually controlling the autosampler through the Autosampler Control dialog box. Once the autosampler is in the Safe position (all the way to the right), you can lift off the sample ring that holds the crucibles. Be careful to avoid the gripper. Lift the ring off of the autosampler assembly top about 1/4 inch, tilt it slightly and move it in the direction that the gripper faces until the inner edge of the ring clears the gripper, then angle the ring up and over the grippers. Remove the used samples and replace with fresh samples in the crucibles.

You can use the stainless steel sample pan liners (N537-6749) in the crucibles to keep them from getting dirty. Simply throw away the liner after use.

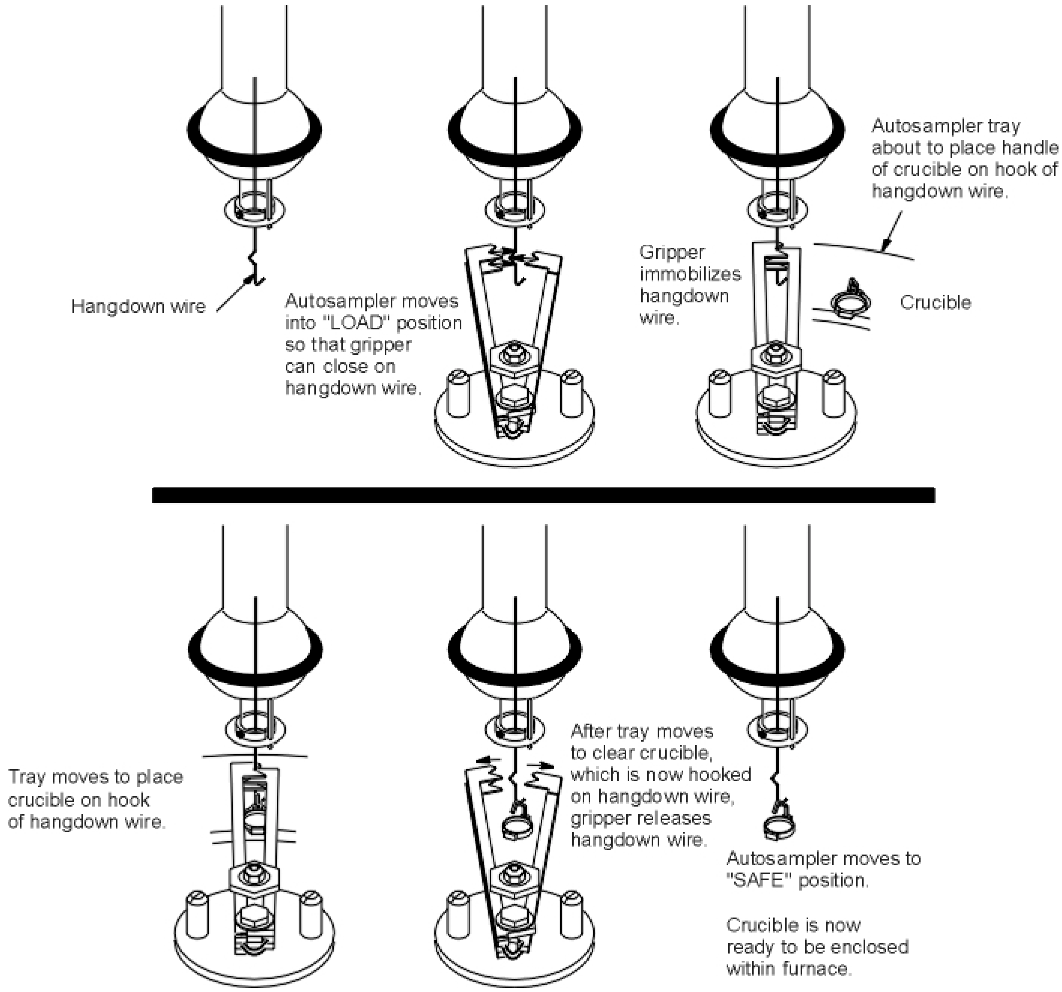
Autosampler Work Cycle

When you begin a sample run by starting a play list, the autosampler performs a typical work cycle, which includes the following steps:

NOTE: When running samples with an autosampler, the status of the Antistatic Device should be Auto Enabled and the Upper Fan should be Off.

1. Before the start of the run, the furnace is in the Cooling position or the Lowered position. The autosampler is in the Safe position.
2. Start the play list. Your play list may have an explicit **Load Sample** command or, if using a Sample Group, the entry Sample List implies **Load Sample**. Upon reaching this line in the list, the autosampler tray turns to position the selected tray location properly.
3. The autosampler swings to the Load position.
4. The gripper closes around the hangdown wire.
5. The tray rises up and turns so that the crucible handle engages the hook in the hangdown wire.
6. The tray descends (you will hear a short grinding noise) and returns to the Safe position.
7. The antistatic device turns on (indicated by the red light on the back wall of the instrument).
8. The furnace rises almost all the way to the ball joint. It stays at that position for about 15 seconds while the antistatic device deionizes the ball joint area.
9. The antistatic device turns off and the furnace finishes traveling up to engage the ball joint.
10. The method runs.
11. Your play list may contain an explicit Return Sample command. If using a Sample Group, the Sample List entry has an implied Return Sample command.
12. At the end of the run, the antistatic device comes back on as the furnace goes to the Lowered position.
13. The antistatic device turns off as the furnace goes to the Cooling position.
14. The autosampler swings to the Load position with the selected location underneath the crucible.
15. The gripper closes around the hangdown wire.
16. The tray rises so the crucible goes into the selected location.
17. The tray turns so the crucible hook comes off of the hangdown wire.
18. The tray descends (you will hear a short grinding noise) and goes to the Safe position.

The sequence of events for loading and unloading a sample are shown below:



Adjusting the Standard Furnace on an Autosampler System

When your Pyris 1 TGA with autosampler is installed by a service engineer, he performs a series of adjustments in order to optimize its performance. If you had to remove the furnace and/or furnace tube, perhaps to clean it, and then reinstalled it, you will have to perform these steps to ensure proper operation of the instrument:

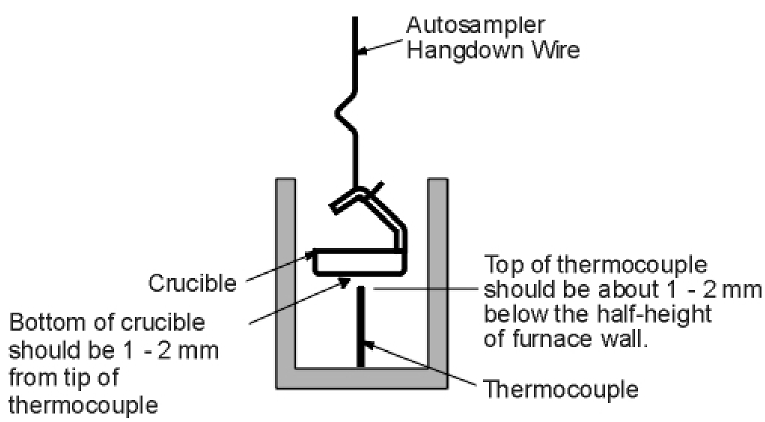
Aligning the Standard Furnace

1. Power on the Pyris 1 TGA. The furnace will go to the Cooling position and the autosampler will be in the Safe position.
2. Press the **Lower Furnace** button on the instrument's control panel. The furnace will swing out to the Lowered position.
3. Remove the lock nut from the furnace mounting assembly and swing the furnace assembly down so that it is at an angle with the instrument.
4. Look down into the furnace tube and see if the furnace is exactly centered within the tube. If it is not, loosen the clamp and move the quartz tube around until the furnace is centered. Tighten the clamp.

5. Raise the furnace assembly back up and reinsert the lock nut into the furnace mounting assembly and tighten.
6. Turn off the antistatic device by pressing the **Menu** button on the instrument's control panel three times. At the Antistatic Device display, press the **Scroll Up** button to display the status of the antistatic device. If it is ON, press the **Enter** button to respond Yes to Turn Off? If it is already OFF, press the **Exit** button to return to the default display.
7. Raise the furnace by pressing the **Raise Furnace** button. If the furnace is properly aligned, the furnace tube should move smoothly into position around the upper ball joint.
8. To adjust the horizontal position of the furnace tube, slightly loosen the furnace adjustment nut at the base of the furnace mounting assembly.
9. Press the **Lower Furnace** button.
10. Press the **Raise Furnace** button.
11. Position the furnace assembly so that the furnace tube moves smoothly onto the ball joint. Adjust the furnace assembly manually until it appears that the furnace is aligned correctly (i.e., the furnace tube is perpendicular to the table top). Lock the furnace assembly into position by tightening the furnace adjustment nut at the base of the furnace mounting assembly.

Adjusting the Height of the Furnace

1. Bring the furnace to the Lowered position by pressing the **Lower Furnace** button.
2. If not present, install a hangdown wire.
3. Place a crucible on the end of the hangdown wire; place it manually by using tweezers or load it from the autosampler via the Autosampler Control dialog box.
4. Press the **Raise Furnace** button.
5. Check the position of the crucible with respect to the top of the thermocouple. The bottom of the crucible should be approximately 1 – 2 mm above the tip of the thermocouple. The tip of the thermocouple should be 1 – 2 mm below the halfway point of the furnace wall.

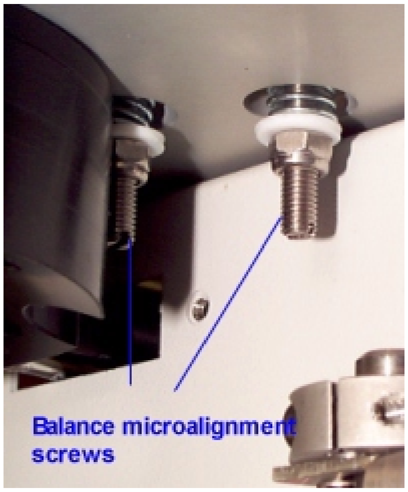


6. Adjust the height of the furnace using the furnace height adjustment knob located below the furnace adjustment ring. Turn the knob clockwise to raise the furnace and counterclockwise to lower the furnace.
7. Make sure that the crucible hangs in the center of the furnace. It is important that the crucible does not touch the side wall of the furnace or the tip of the thermocouple.

If the crucible cannot be centered in the furnace using the adjustment procedure, then you might have to adjust the balance mechanism.

Adjusting the Balance Mechanism

The balance mechanism has two cams that are used to adjust the position of the hangdown wire within the furnace. These screws are called the X-Y microbalance adjustment screws.



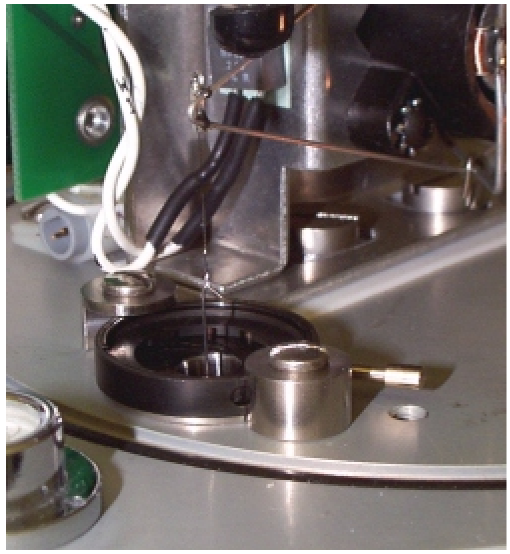
1. If not already, enable the antistatic device automode. Press the **Menu** button three times. At the Antistatic Device display, press the **Scroll Up** button; the status of the antistatic device is displayed. If it is not Auto Enabled, press the **Enter** button to enable the device. DO NOT press the **Exit** button yet.
2. Make sure that the upper fan is off; otherwise, the hangdown wire will swing. Press the **Scroll Up** button twice. With the Fan mode displayed, press the **Menu** button to see the status of the fan. If Automode is ON, turn it OFF by pressing the **Enter** button. Press the **Exit** button to return to the default display.
3. Press the **Raise Furnace** button.
4. If the hangdown wire needs to be centered, carefully use a screwdriver to adjust the position of the balance, and, therefore, the position of the hangdown wire. The left-hand screw (that closest to the antistatic device) adjusts the Y position and the right-hand screw adjusts the X position.

Once the crucible is centered within the furnace tube, it is important that the hangdown wire not touch the anticonvection iris when it is closed. The iris prevents reaction gases from rising into the balance chamber from the furnace. After adjusting the balance mechanism, the hangdown wire may no longer be centered within the iris.

Adjusting the Convection Iris

1. Lift the top cover of the analyzer up to access the balance dome.
2. Remove the balance dome by turning the locking devices and pulling the arms out of the groove in the dome. Lift the dome straight up and away from the balance. The iris is now exposed.
3. Open the iris all the way by pushing the arm clockwise.
4. Loosen the screws on the two stands that hold the iris in place.

5. Move the iris around until the hangdown wire is centered in the iris.

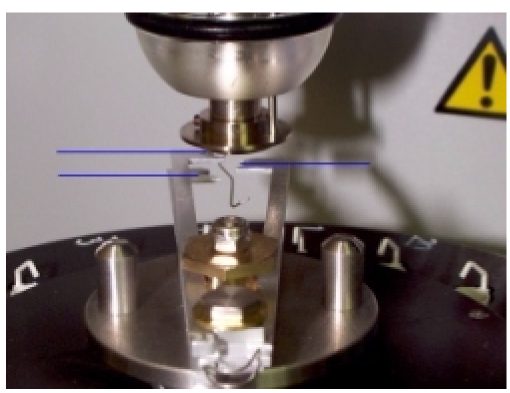


6. When the hangdown wire is centered, tighten the screws on the stands. Do not overtighten; the iris arm will not be able to move.
7. Close the iris as far as possible without it touching the hangdown wire by turning the lever counterclockwise.
8. Replace the dome back onto the balance plate and lock in place.
9. Lower the access cover.

If you installed a hangdown wire during this procedure, you will have to perform the Gripper Alignment and Tray Alignment procedures.

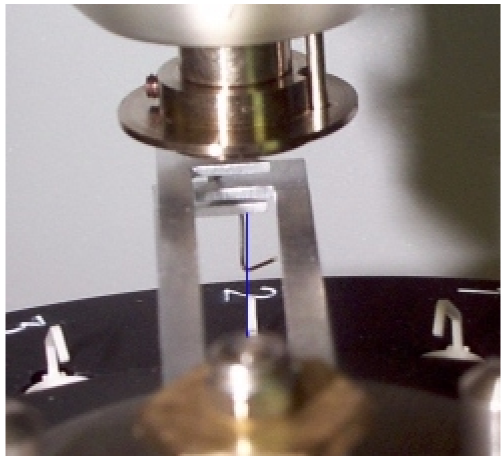
Alignment Procedures

The autosampler is aligned when the Pyris 1 TGA is installed by the service engineer. However, if you change the hangdown wire or if the hangdown wire is not picking up or returning the crucibles to the autosampler tray properly, you will have to realign the system. Alignment includes making sure that the gripper aligns correctly with the features of the special hangdown wire. The grooves of the three gripper arms should line up with the bends of the hangdown wire.



When the gripper closes around the hangdown wire, it should not pull it down or rotate it in any way. If it appears that the gripper is out of alignment with respect to the hangdown wire, you can use the Align Gripper wizard.

Alignment also includes making sure that the crucible handles align with the hook of the hangdown wire so that each one is loaded and unloaded correctly. When aligned properly, the left side of the crucible handle should line up with the right side of the hangdown wire:

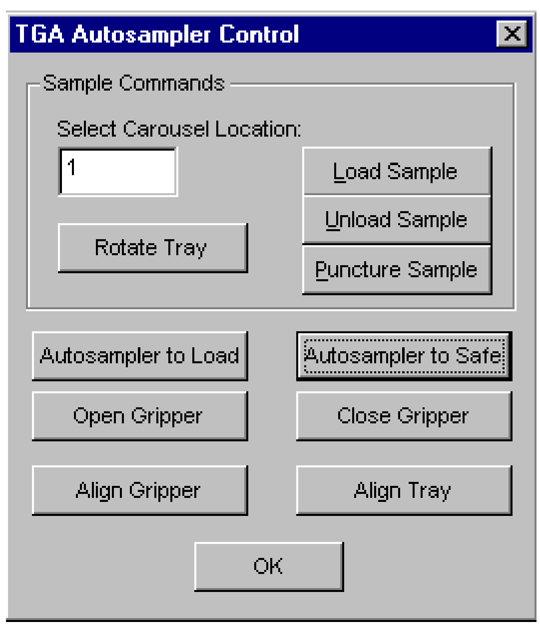


If the crucible is too far to the right or the left of the hangdown wire, it will not be picked up correctly. Run the Align Tray wizard to align each crucible with respect to the hangdown wire. The positions are "remembered" in the encoder disk.

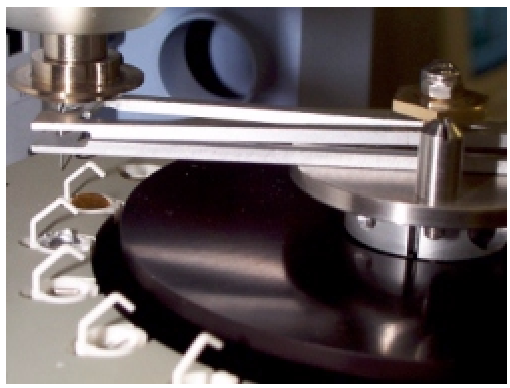
Align Gripper

To perform a gripper alignment:

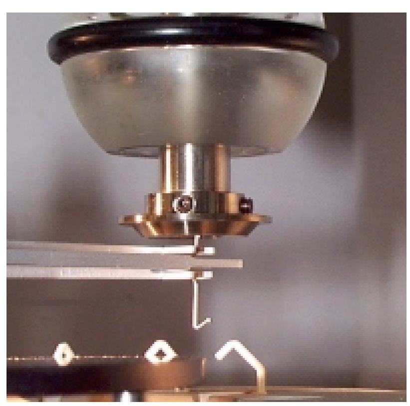
1. Open the Autosampler Control dialog box by clicking on the **Autosampler Control** button on the software's control panel:



2. Click on the **Autosampler to Load** button. Note the position of the hangdown wire's feature with respect to the "V" in each of the gripper arms.
3. Select the **Close Gripper** button.
4. Observe whether the gripper moves the hangdown wire. A properly adjusted gripper and autosampler unit will not change the rotary or the centered position of the hangdown wire significantly. It may, however, pull the wire down slightly.
5. Click on the **Open Gripper** button.
6. You can adjust the gripper location using the Align Gripper wizard. Click on the **Align Gripper** button. The first wizard screen appears.
7. Make sure that there is no crucible on the hangdown wire. Click **Next**.
8. Click **Next** in the second wizard screen to move the furnace to the Lowered position and the autosampler to the Load position.
9. Use the **Move In**, **Move Out**, and step size buttons to adjust the gripper position. **Move In** moves the gripper to the left when looking at it from the left side of the autosampler tray. **Move Out** moves the gripper to the right with respect to the hangdown wire. The hangdown wire should be positioned in the open gripper as shown below:



10. Once you are satisfied with the position of the gripper, click **Next**.
11. In the next wizard screen click on **Close Gripper**. See how the gripper encloses the hangdown wire.



12. Click on **Open Gripper**. Close and open the gripper a few times to make sure it is grasping the hangdown wire properly. Click on **Next**.

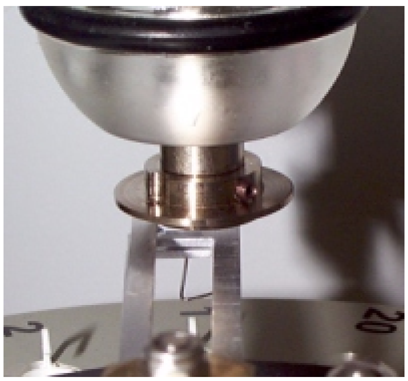
13. The final wizard screen tells you to run a Tare All from a new play list to test the alignment. However, you should perform a Tray Alignment first. Click on **All Done** to exit the Align Gripper wizard. The autosampler goes to the Safe position and the furnace goes to the Cooling position and the cooling fan goes on.

Align Tray

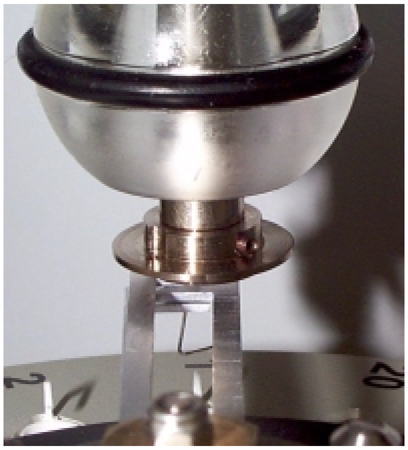
The tray alignment procedure should also be performed when you change the hangdown wire or if the crucibles are not being loaded or unloaded properly. It should also be performed after a gripper alignment.

To align the tray, follow the procedure below:

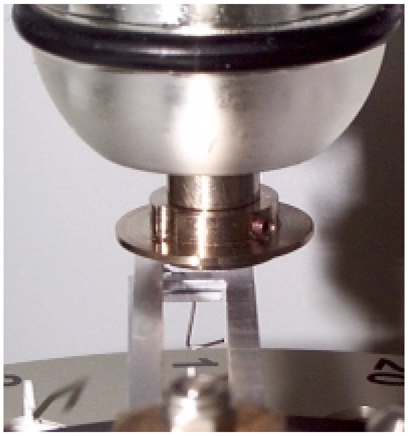
1. Fill the autosampler tray with 20 crucibles. The hooks of the crucibles should be positioned so that they face the center of the autosampler.
2. Click on the **Autosampler Control** button in the software's control panel. The Autosampler Control dialog box appears.
3. Click on the **Align Tray** button to open the Tray Alignment wizard. The first wizard screen appears.
4. Make sure that there is no crucible on the hangdown wire. Click **Next**.
5. Click **Next** in the second wizard screen to send the furnace to the Lowered position and the autosampler to the Load position. The gripper closes around the hangdown wire. The next screen appears.
6. Take a look at the position of the hangdown wire with respect to the crucible handle. The left side of the crucible handle should align with the right side of the hangdown wire. Use the **Move Left** and **Move Right** buttons along with the step size buttons to move the tray with respect to the hangdown wire. In the photo below, the crucible handle is aligned correctly. If you click on Move Left from this position, the crucible handle moves to the right; Move Right moves the handle to the left, as shown below:



Correct position of crucible handle with respect to the hangdown wire.

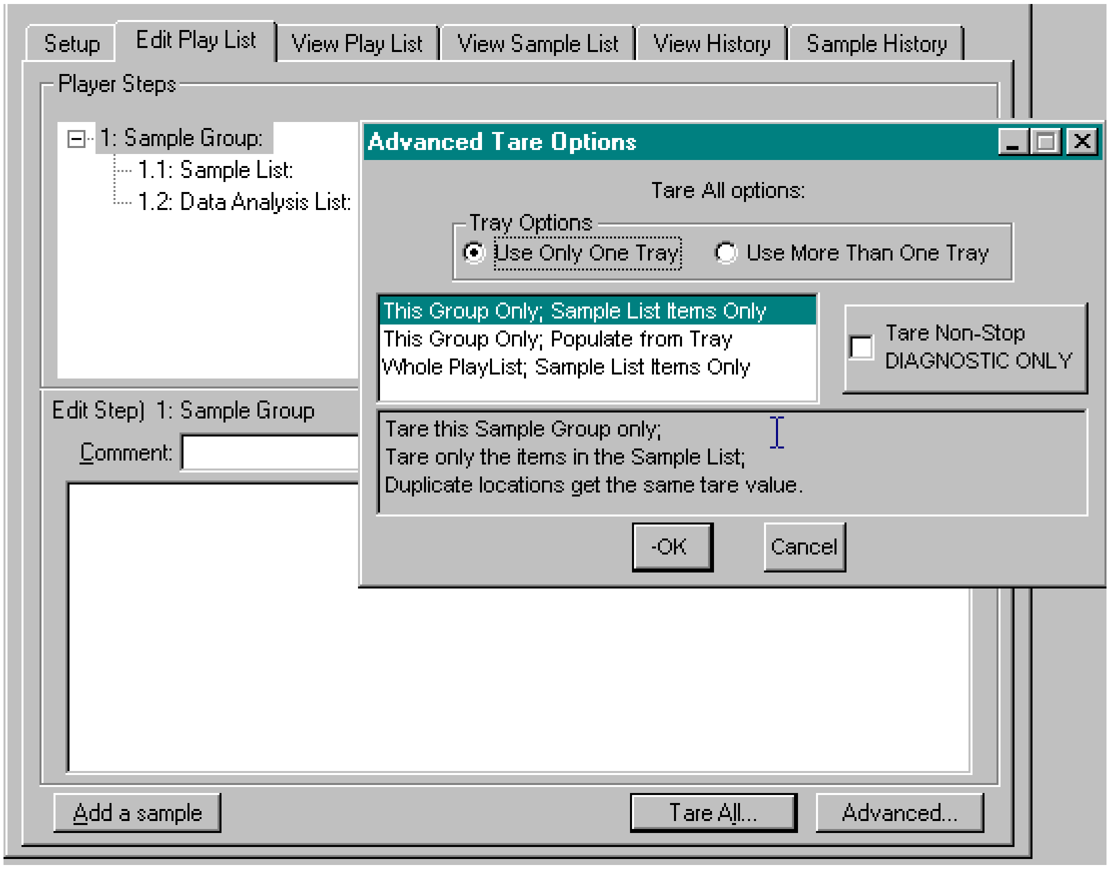


Position of the crucible after clicking on Move Left by a Small Step from the correct position. The handle moves clockwise.



Position of crucible after clicking on Move Right by a Small Step from the correct position. The handle moves counterclockwise.

7. It may take several tries to get the crucible handle aligned with the hangdown wire. After you are satisfied with the first crucible's alignment, click on **Next**.
8. The next wizard screen makes you align the crucibles in all 20 positions. Use the Next Location to move to position 2. Use the Move Left or Move Right and step sizes to position the crucible handle below the hangdown wire correctly. When you have done this for all 20 crucibles, click on **Next**.
9. The last screen instructs you to test the alignment by creating a new play list with a Sample Group as the sole entry. Click on **Tare All** to display the Advanced Tare Options dialog box.



10. Select **Use One Tray Only** and **This Group Only; Populate from Tray**. Click on **OK**. The Tare/Weigh System dialog box with a schematic of the sample tray is displayed. As each crucible is loaded, tared, and unloaded, watch how the hangdown wire picks up and unloads the sample pan. If successful, the circle representing the current crucible location will be green. If not successful, the circle is red and the tare procedure continues on to the next crucible. When finished, click on **Done** to exit the Tare/Weigh System and return to the play list. The Sample List will be populated with Sample lines of those crucibles that were tared.

Sample Handling

The Pyris 1 TGA measures the change in weight of a sample as a function of temperature or time. Proper sample preparation and handling are important for obtaining optimal results.

Sample Preparation

The thermogravimetric analyzer analyzes solid samples in powder, crystal, or granular form. Although quantitative accuracy will remain the same regardless of sample shape, the qualitative appearance of a run may be affected by the sample configuration. Proper sample preparation that maximizes the contact surface between the crucible and the sample will reduce any imbalance of the sample in the crucible and will result in maximum peak sharpness and resolution. The best sample form for optimum performance is powder or fine granules. Solid materials can be sliced into small pieces with a razor or knife.

Sample Pans

The only sample pan that you can use with the autosampler is the ceramic crucible (N537-0464). The sample tray holds up to 20. You can also place an aluminum liner (N537-0492) or a stainless steel liner (N537-0495) within the crucible to lengthen its usability. The liner can be used for runs under 600°C.

Sample Atmosphere

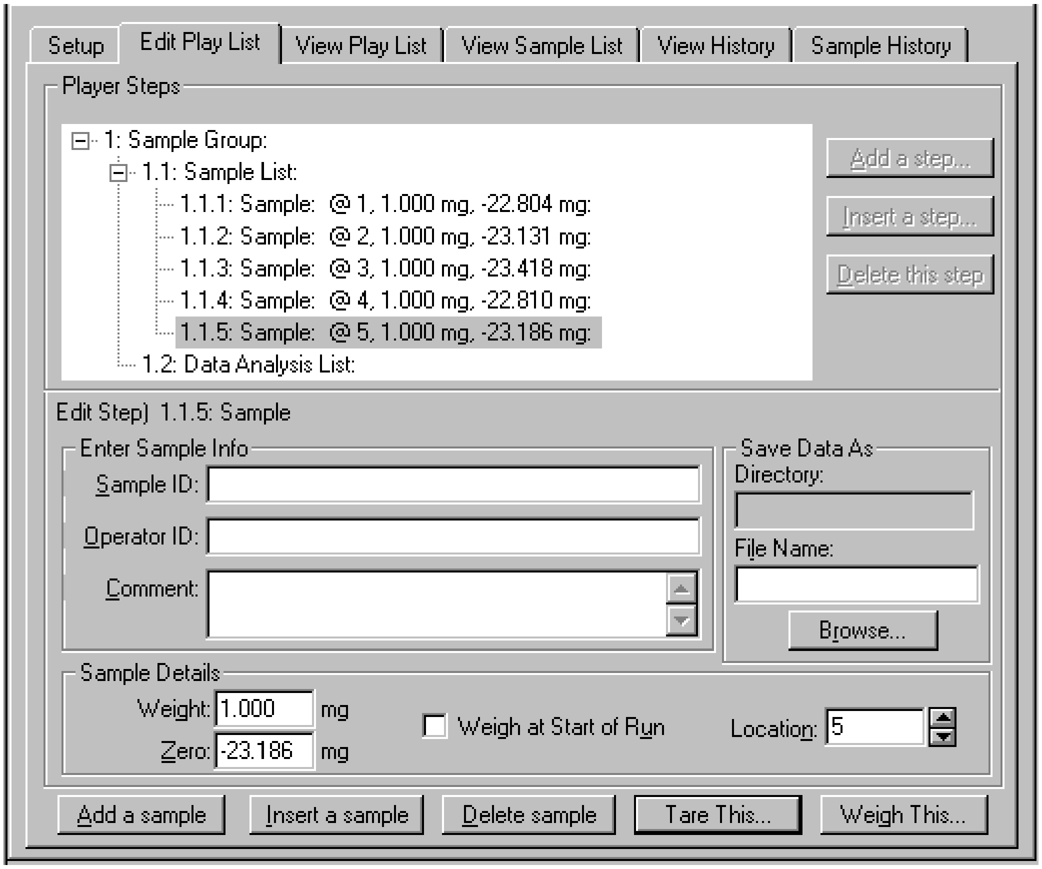
The autosampler tray can be covered with a transparent plastic cover which allows you to observe the movement of the autosampler mechanical components during operation, i.e., watch the gripper load and unload the crucible. The enclosed samples in the tray can be purged with dry or humidity controlled air or nitrogen. You can use this cover over the full temperature range of the Pyris 1 TGA.

Once the sample is loaded onto the hangdown wire and the autosampler returns to the Safe position, the sample is subject to the same atmosphere as a sample in a standard Pyris 1 TGA. The furnace rises up around the sample and the sheath gas, if used, baths the furnace liner, i.e., the gas goes between the Pyrex furnace tube and the liner, not in the furnace.

Sample Loading

Before loading the samples into the crucibles, you will have to tare the crucibles, i.e., have the system weigh them so the weight of the crucible is not included in the data. Load the empty crucibles into the sample ring. You can do this with the ring off of the autosampler assembly. After filling the locations, carefully place the ring over the gripper and settle the ring into position. There is a locating pin in the autosampler that goes into the hole in the tray. You could also keep the ring on the autosampler and use tweezers to load the crucibles into the ring. Make sure that the autosampler is in the Safe position. Be careful with the tweezers around the hangdown wire.

You can tare all of the crucibles in the tray using the Pyris Player Tare All feature. Your play list should contain a Sample Group. If you are creating a new play list, select Sample Group as the first entry. If you are using an existing play list, it should have a Sample Group in it. In a new play list, with Sample Group highlighted, click on the **Tare All** button to display the Advanced Tare Options dialog box. The tare weights are automatically put into the Sample List. If you chose Populate from Tray in the Advanced Tare Options dialog box, the Sample List is filled with a line for each position in the sample ring that contains a crucible and the tare weight of the crucible is included.

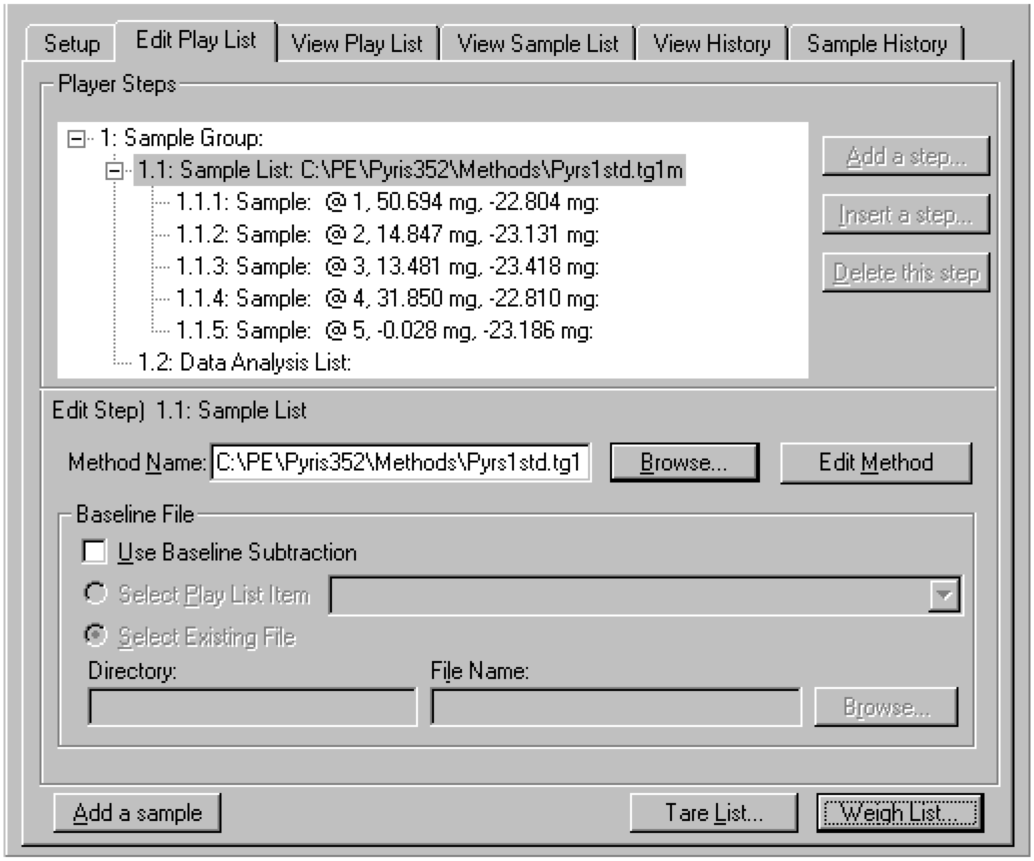


Once you have tared the empty crucibles, remove the sample ring, remove each crucible, load the sample into the crucible, and return the crucible to the same location in the sample ring. Return the sample ring to the autosampler. Now you can have the system weigh all the samples before running the play list, or you can have each sample weighed at the beginning of its run. With Sample List highlighted, select the **Weigh List** button. The TGA Tare/Weigh System dialog box appears. The system automatically starts the program to weigh each sample in the list. After the last sample, if there was a missing sample encountered, a message is displayed:

> **The following samples could not be weighed. It is possible that the crucibles are empty.**

A list of missing samples follows. Click on the dialog box's **Close** button to clear the box.

Click on the **Done** button in the Tare/Weigh System window. The Sample List will now display the weights of each sample:



For efficient use of the autosampler and to increase sample throughput, you can purchase a second sample ring (P/N N537-0489). You can prepare a second set of samples while the first set is running.

Running a Play List

Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items – Load Sample, Run Method, Return Sample – there is a Sample Group. The Sample Group simplifies grouping like samples together (as you would have in a sample tray of the autosampler). These like samples use the same test method and data analysis. A Sample Group consists of a Sample List and Data Analysis List. A specific method is selected for the sample in the Sample List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization, and calculations for automatic data analysis.

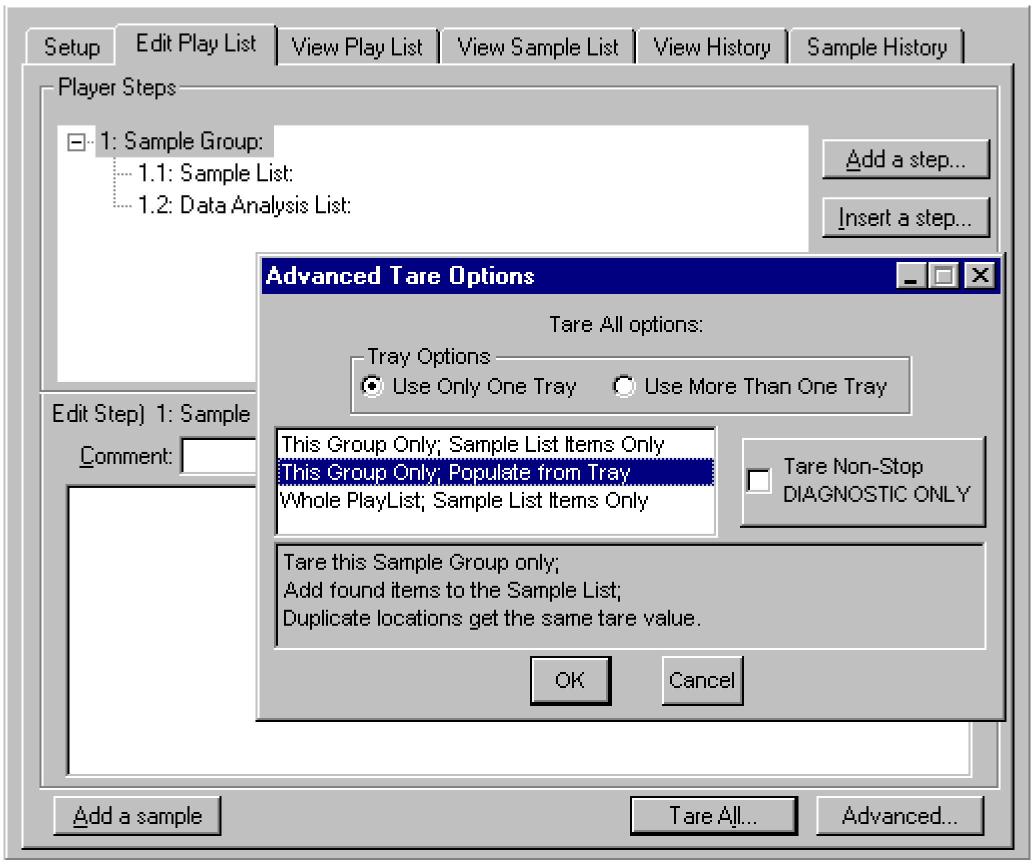
Before starting a run, perform the steps below:

1. Review the safety and warning notes for the Pyris 1 TGA and the autosampler.
2. Verify that the balance purge, sheath gas, and furnace lift mechanism gas tubing are properly connected.
3. Turn on the gases; adjust the pressures. The recommended flow rate for the balance purge is 40 – 60 mL/min. For the sheath gas, it should be 20 – 35 mL/min. For the furnace lift mechanism, use a pressure between 15 and 30 psi.
4. Verify that the electrical and cable connections between the autosampler and the Pyris 1 TGA and other components of the system are properly connected.

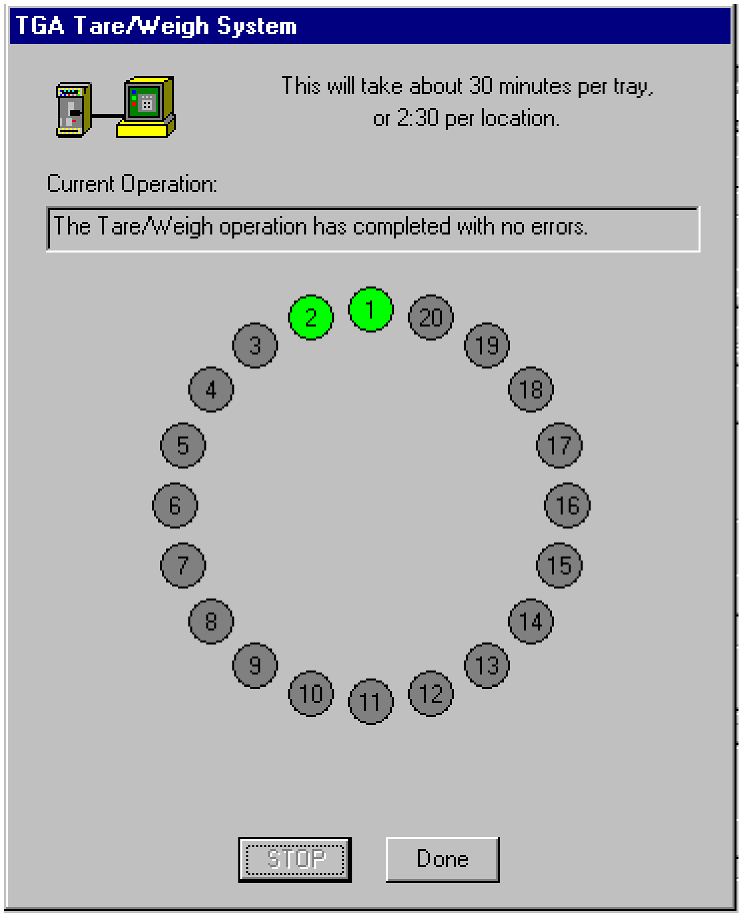
5. Turn on the power to the system components in the following order:
   - computer
   - GSA 7 or TAGS if present
   - Pyris 1 TGA
   - printer
6. Prepare your samples.
7. Start Pyris Software for Windows and click on the Pyris 1 TGA button. Click on the **Pyris Player** button on the toolbar: . Either open an existing play list or create a new one.

   There are many ways to use the play list with the autosampler. A quick way to create a play list using the Sample Group feature is given below.

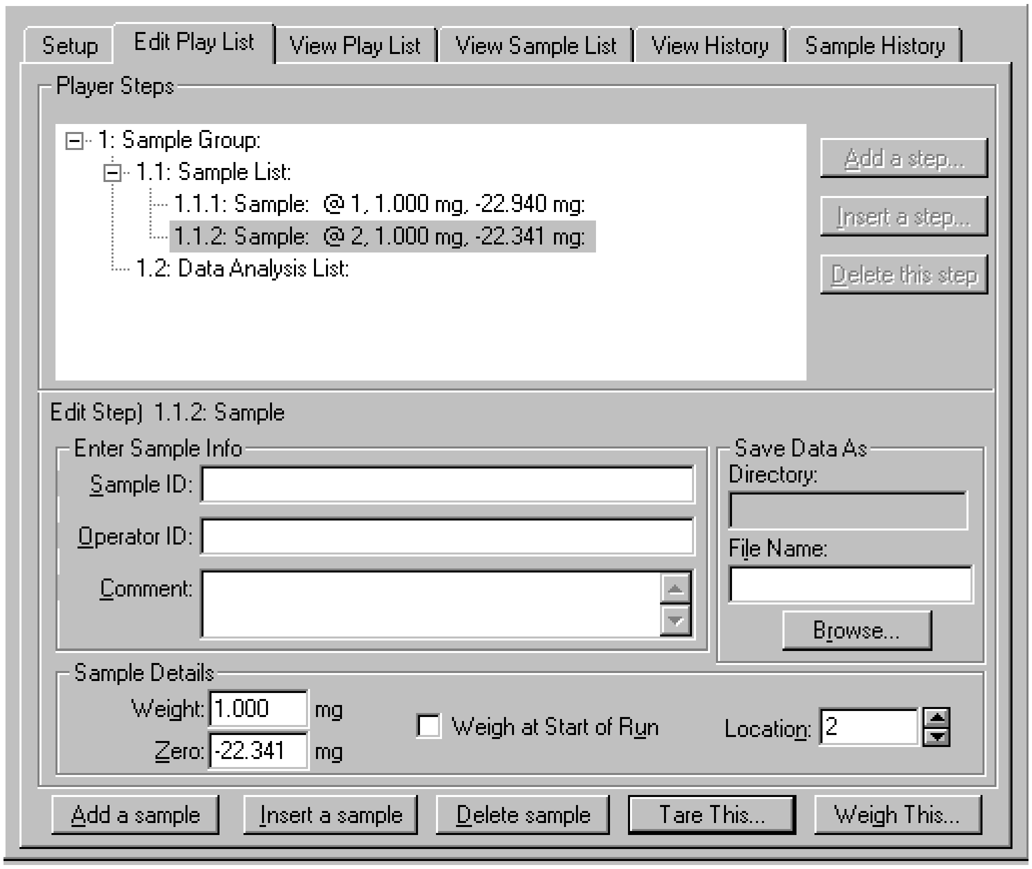
   Load empty sample pans into the locations you want to use in the sample tray. Create a new play list that contains only a Sample Group. Click on the **Tare All** button. In the Advanced Tare Options dialog box, select This Group Only; Populate from Tray.



While taring, the TGA Tare/Weigh System screen is displayed.

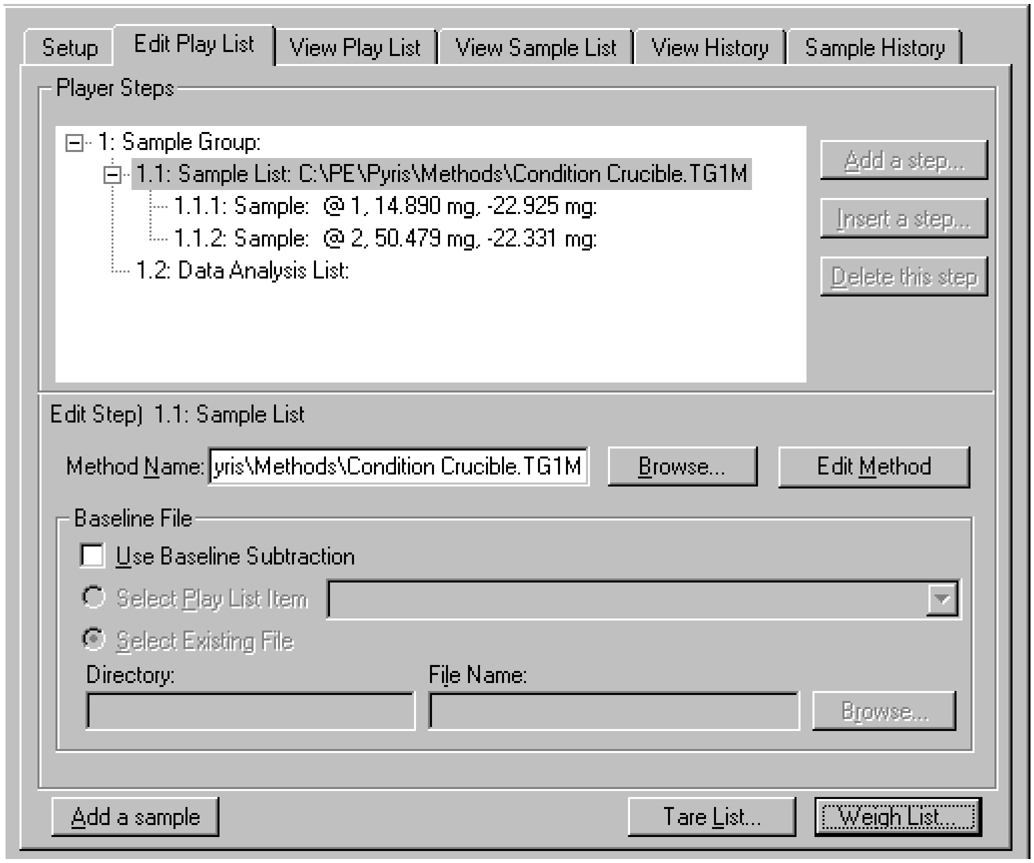


The software will tare the crucibles it finds and populate the Sample List in the Sample Group for you.



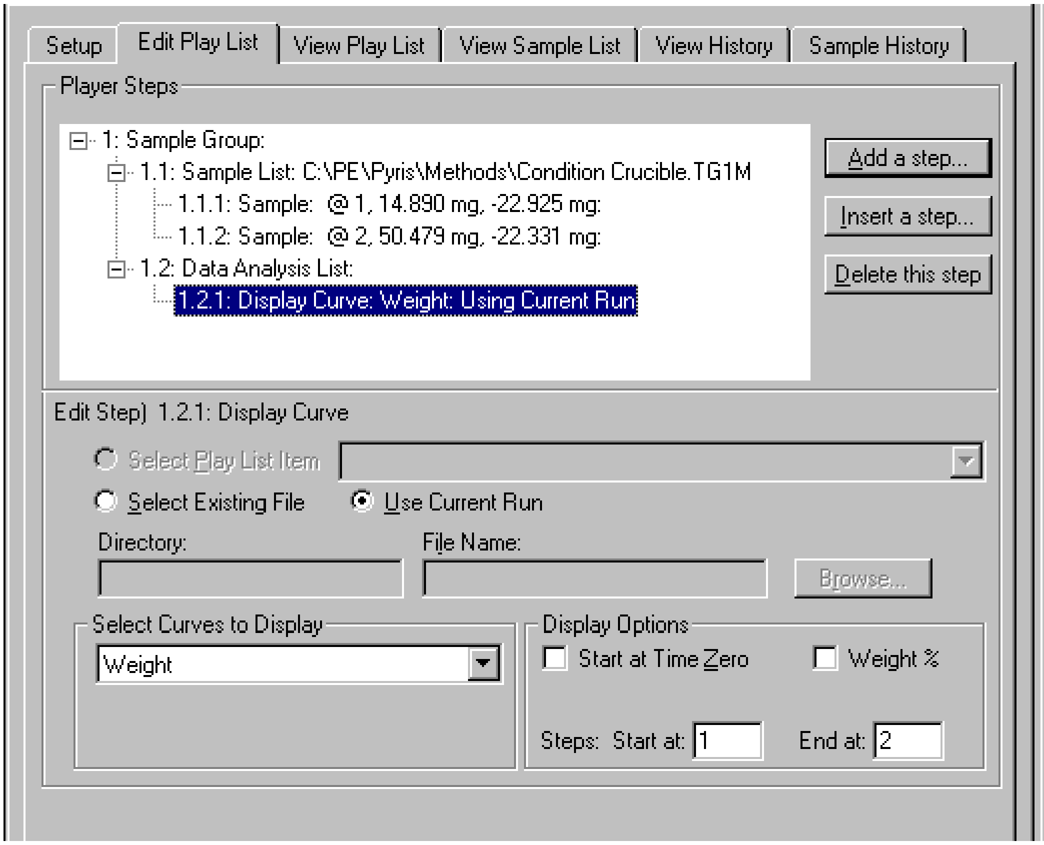
Remove the sample tray from the autosampler and place the samples in the crucibles. Carefully place the ring back on to the autosampler. Or you can remove each crucible from the tray individually and load the sample into the crucible and return it to the tray. To access position 1 of the tray while the autosampler is in the Safe position, display the Autosampler Control dialog box and enter 11 into the Location field. Click on **Rotate Tray**. The autosampler will rotate so that position 1 is facing out in order for you to access the crucible.

Now you can have the system weigh each sample in the list. You can also choose to have each sample weighed right before the run starts. To weigh all the samples before starting the play list, highlight the Sample List line. A message informing you that a method has not been selected is displayed. You can select the method after the samples have been weighed. Click on **Weigh List**. The TGA Tare/Weigh System screen appears and the system begins to weigh the samples listed in the Sample List. When complete, the weights are displayed, along with the tare weights, in each sample line.

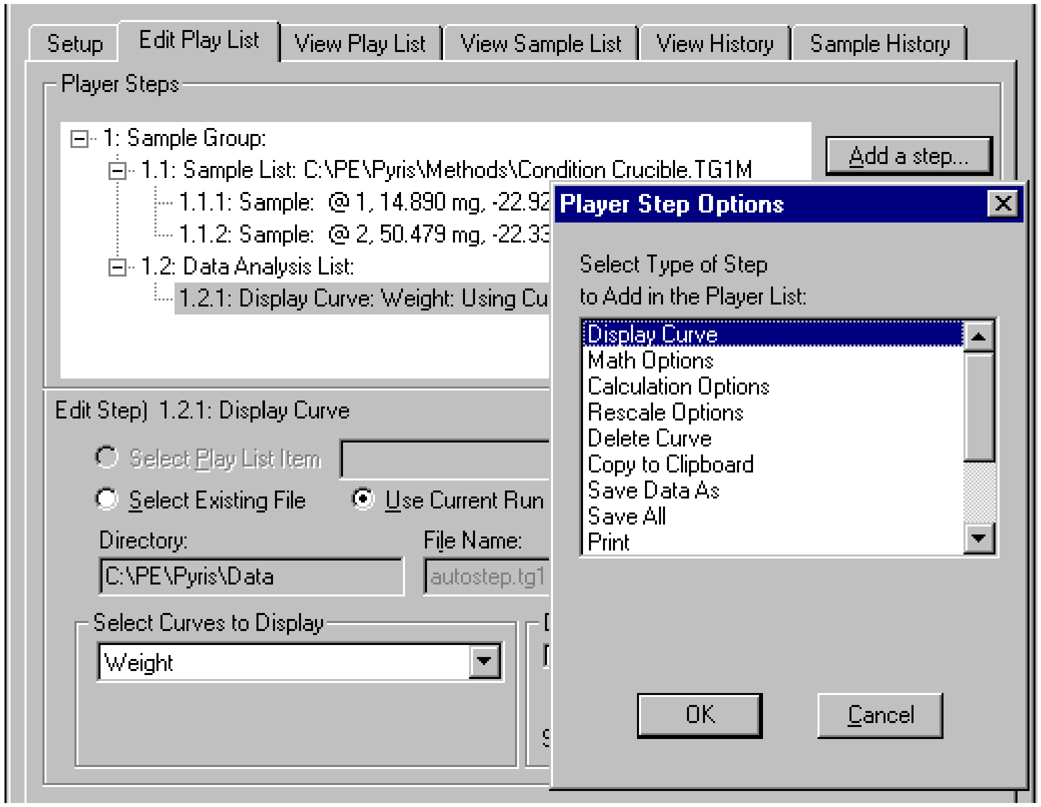


Next you need to add some items to the Data Analysis List. If you have not entered a method for the Sample List, however, you cannot fill in the Data Analysis List. To enter a method, highlight the Sample List line. Type in the name of the method in the Method Name field or click on the **Browse** button and find and select the method you want to use. Edit the method's program and initial state parameters by clicking on the **Edit Method** button. When finished editing, close the Method Editor by clicking on the window's **Close** button in the upper-right-hand corner.

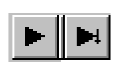
With Data Analysis List highlighted, click on **Add a step**. Select Display Curve. Use the default Use Current Run. This means that the curve from the current run will be displayed after the run is over. Add a Pause line after this line if you want to be able to view the curve.



Once you add Display Curve to the Data Analysis List, other items become available to add to the list.



8. Save the play list by selecting Save Player from the File menu.
9. Start playback of the play list by clicking on the Start at Top button or the Start at Current Item button on the Player toolbar.



NOTE: If you select the **Start at Current Step** button, the focused item must be a main-level item, i.e., Prepare Sample, Data Analysis, or Sample Group. However, if you are in a Sample Group, you can start a play list from a Sample line in the Sample List.

NOTE: If you select Go To Load, Go To Temp, or Hold at Temp from the control panel while a play list is running, the current sample run will end and the playback of the play list ends.

Troubleshooting

The Pyris 1 TGA and the autosampler were designed for routine, trouble-free operation. Although the system is relatively simple to operate, occasional problems may occur. Below are some situations that may arise that a Service Engineer will be needed to correct.

Furnace Temperature Overlimit

This error shows up as a **–34°C display for the program temperature in the Status Panel** in the Pyris software. The main power relay will click at the time of the error, thus shutting power off to the furnace. Turn the instrument off and call your service engineer.

Another situation that may cause furnace temperature overlimit is when the **furnace relay turns off when ramping to 1000°C**. This is usually a problem with the temperature and furnace calibrations. Call your service engineer.

A furnace temperature overlimit can also occur when the furnace control loop is defective. Too much power could be applied to the furnace all at once. Call your service engineer.

Balance Does Not Lift

If the balance does not lift it full load mass of 1300 mg, call your service engineer.

Keypad Does Not Work

If the Pyris 1 TGA keypad does not work upon initialization of the system, especially with the autosampler attached, wait 30 seconds and try the keypad again. If it still does not work, call your service engineer.

Autosampler Motors Run Hot

The sample tray on the autosampler sits on tiny balls so that air can circulate underneath to keep it cool. The sample tray can get as hot as 93°F (35°C). If the autosampler motors run too hot, call your service engineer.

Autosampler Does Not Respond to Commands

- Check to see of the communications cable is attached properly at the rear of the instrument. It should be connected at the labeled Autosampler at the top and at the RS232 Auxiliary connector in the center of the rear of the instrument.
- Check if the autosampler is configured for the Pyris 1 TGA. In the Pyris 1 TGA Configuration dialog box, the box next to Autosampler should be checked and the port to which it is connected should be the same as that shown for the instrument itself. Click on the Test button to make sure that the autosampler is recognized. If it is not, you should exit from the software and reset the instrument.
- Reset the analyzer by cycling the power off and on at the rear of the instrument. The autosampler will move away from the Safe position slightly and then return if it is communicating with the autosampler PCB.

If none of these work and the autosampler still does not respond to commands, call your service engineer.

Carousel Rotates to a Slightly Different Position Than Commanded

The service engineer will have to adjust the slip clutch, fix the encoder pin's engagement of the table, or adjust the brake pad that may be dragging on the top surface of the electromagnetic brake.

Carousel Rotates When an Up/Down Command Is Given

The brake is not engaging the brake disk. Call your service engineer.

Autosampler Does Not Swing to Load Position When Commanded

Check that the main power and signal cables are positioned correctly so that they do not restrict the swing; otherwise, call your service engineer.

Autosampler Grinds into Safe Position

Readjust the Safe position by loosening the collar on the sector gear and sending the unit to the Safe position again. Secure the collar on the sector gear tightly.

Tray Drives Up But Does Not Come Down

Call your service engineer.

Gripper Problems

Problem: The gripper opens when commanded to close and closes when commanded to open.

Solution: The cam is 180° out of position. Call the service engineer.

Problem: The gripper deforms the nichrome hangdown wire alignment feature.

Solution: The gripper is set too tight and must be adjusted to hold the hangdown wire alignment feature loosely while maintaining position of the sample crucible so that it slides into the sample tray in the proper orientation.

Problem: The gripper hits the hangdown wire while the autosampler unit is swinging into the Load position.

Solution: Assuming that the hangdown wire is positioned correctly, the rotary position of the gripper assembly must be adjusted. Call your service engineer.

Autosampler Maintenance

Observe the following guidelines when cleaning the autosampler:

- The black area of the autosampler is anodized aluminum. To clean it, you can use a cloth dampened with isopropyl alcohol.
- The sample tray is clear anodized aluminum which can be cleaned with isopropyl alcohol.
- To keep the ceramic crucibles clean, you can use the disposable aluminum liners (N537-0492) or stainless steel liners (N537-0495).

Part Numbers

Autosampler Spares Kit

The following items are in the Spares Kit (N537-0484) for the Pyris 1 TGA with autosampler:

Part No.	Description	Quantity
0154-1498	"H" restrictor	3
0250-6483	Teflon tubing 1/8-in. o.d.	24 ft
0250-6515	Tygon tubing (black) 1/4-in. o.d.	20 ft
0250-6518	Tygon tubing	6 ft
0250-8084	Tygon FEP lined tubing (less permeable than 0250-6518)	3 ft
0990-3094	Swagelok insert for 1/8-in. i.d. tubing	6
0990-3150	Tube fitting, 1/4-in. NPT male to 1/4-in. o.d. Swagelok	1
0990-3196	Tube fitting, 1/4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3428	Tube fitting, 1/8-in. NPT male to 1/4-in. o.d. Swagelok	1
0990-3629	Pressure relief valve	1
0990-3630	Air filter, 1/8-in. NPT connections	1
0990-3715	Female branch Tee, 1/4-in. NPT female to 1/4-in. o.d. Swagelok	1
0990-3716	Adapter, 1/8-in. NPT female to 1/4-in. o.d. Swagelok	2
0990-3906	Tube fitting, 1/4-in. NPT female to 1/8-in. o.d. Swagelok	2
0990-8134	Teflon plastic tape	2
N537-0467	**Pyris 1 TGA Autosampler Kit**	1
N537-0485	**Pyris 1 TGA Standard Furnace Kit**	1
N537-0756	Cable assembly - autosampler encoder	1
N822-1178	RS232 cable	1

Pyris 1 TGA Autosampler Kit (N537-0467) consists of the following items:

Part No.	Description	Quantity
N537-0464	Crucibles (packs of 10)	3
N537-0488	Hangdown wire kit (5 wires)	1
N537-6622	Autosampler hangdown wire alignment tool	1
N537-6627	Gripper spring	3

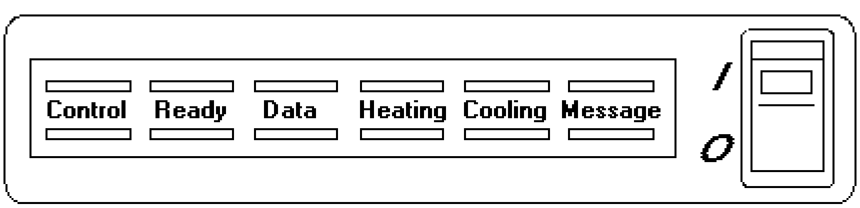
Chapter 6
TGA 7 Analyzer

Overview

The Perkin Elmer TGA 7 Thermogravimetric Analyzer is connected to your computer via a **TAC 7/DX Thermal Analysis Instrument Controller** which controls the analyzer and digitizes the analog output from the detector before sending it on to the computer. With the Pyris software installed on the computer controlling the instrument, the TGA 7 is used to measure weight changes in sample materials as a function of temperature or time. Under control of the Pyris software, the TGA 7 is programmed from an initial to a final temperature and measures weight changes resulting from chemical reactions, decomposition, solvent and water evolution, Curie point transitions, and oxidation in sample materials. Usually, the TGA 7 is programmed to scan a temperature range by changing at a linear rate over 150 temperature ramps for the study of these transitions. The analyzer can also be used to gather and analyze data at isothermal temperatures to measure weight loss or weight gain in sample materials.

Status Indicators

The TGA 7 has six status indicators on its front panel:



The current status of the TGA 7 is displayed in the Status Panel in the Pyris Software for Windows screen. You can also see the status of the instrument by looking at the front panel.

Control

The TGA 7 temperature sensors are in control of the temperature; power is supplied to the furnace to maintain the program temperature selected in the Pyris software.

The Control light should always be lit during the main section of a temperature program run in order to obtain accurate data. If the Control light goes out during an experiment, the subsequent data will not be accurate.

The temperature at which the Control indicator illuminates and, therefore, the acceptable temperature range of the TGA 7, depends on the current ambient temperature. In most cases, a TGA 7 equipped with a standard furnace can operate from temperatures as low as 25°C to 30°C. When a high temperature furnace is in use, temperatures as low as 50°C can be attained.

Ready

While blinking, Ready means that the program temperature of the TGA 7 has reached the Load Temperature defined in the Pyris software. When the Control light is also lit, you may begin the temperature program. If the Ready light is on continuously, it means that power is supplied to the TGA 7.

Upon startup of the TGA 7, the Ready indicator should be the only indicator illuminated. However, in some cases the Control light may also be lit upon startup depending on the ambient temperature.

Data

When the Data light on the TGA 7 panel is illuminated, data are being collected. The Data light remains off at all other times.

Heating

When the Heating light is illuminated, the TGA 7 is heating under the control of the Pyris software temperature program.

Cooling

When the Cooling light is illuminated, the TGA 7 is cooling under the control of the Pyris software temperature program.

Message

A blinking Message light indicates that there is a message that must be acknowledged at the computer.

Power

The power switch for the TGA 7 is on the right side of the front panel. When illuminated, line power is being supplied to the TGA 7.

Summary of the TGA 7 Status Indicators

Indicator	Blinking	On	Off
Control	Does not blink	Power is being supplied to the furnace	Analyzer is not in temperature control Power is not being supplied to the furnace
Ready	Program temperature has reached Load Temperature	Analyzer temperature has reached Load Temperature Power is being supplied to TGA 7	Analyzer temperature has not reached Load Temperature Power is not being supplied to TGA 7

Data	Does not blink	Data are being taken from the analyzer	Data are not being taken from the analyzer
Heating	Does not blink	Analyzer is heating under program control at the selected rate	Analyzer is not heating
Cooling	Does not blink	Analyzer is cooling under program control at the selected cooling rate	Analyzer is not cooling
Message	A message is waiting for you		No message is waiting

The status indicators can be used for **diagnostic troubleshooting**:

Diagnostic Troubleshooting

The status indicators can be used for diagnostic troubleshooting by observing which status indicators are illuminated:

Control	Ready	Data	Heating	Cooling	Message	Cause
On or Off	On	–	–	–	–	Computer, TAC 7, and all analyzer modules functioning properly
On or Off	Blinking	–	–	–	–	Computer, TAC 7, and all analyzer modules functioning properly. Analyzer at Load Temperature
–	On	On	On	On	On	TAC 7 for that analyzer module is not turned on or properly connected to the analyzer. Check that all cables are connected properly, TAC 7 power cord is connected to line power, and TAC is turned on
–	–	–	Blinking	Blinking	–	The cable connecting the TAC 7 to the analyzer is loose or has failed. The furnace cable is broken or not connected to the analyzer.
–	–	Blinking	Blinking or Off	Blinking or Off	Blinking or Off	A hardware component in the TAC 7 may be malfunctioning. Call your Service Representative

The TGA 7 is presented in the following topics:

- **Safety Precautions**
- **Furnaces**
- **Microbalance**
- **Operating Variables**
- **Calibration**
- **Maintenance**
- **Accessories and Parts Provided**

Safety Precautions

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

The following precautions must be observed when using the TGA 7 Thermogravimetric Analyzer:

- Never turn the computer off until the following message appears:

  It's now safe to shut off your computer.

- Never press the **Reset** button on the computer if the Pyris software appears to malfunction. Press the **Ctrl-Alt-Del** keys simultaneously and select the Task Manager. From the Task Manager, close the Pyris software.
- Never remove the outer instrument cover or side panels on the TGA 7 without shutting the instrument down and disconnecting its power cord from the power source.
- Only high-quality purge gases should be used with the TGA 7. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory (see Accessories, Replacement Parts, and Parts Provided for the TGA 7) is recommended for the removal of any moisture from the purge gases.
- Always observe the proper startup or shutdown procedures with the TGA 7 and all related instruments.
- Always maintain a positive purge flow (40 – 60 cc/min) through the balance chamber at all times. An inert gas such as nitrogen or argon is recommended as a balance purge.
- Do not raise the furnace when the clear Pyrex furnace tube has been removed. This will cause severe damage to the furnace and other components of the TGA 7 analyzer.
- Prior to performing any experiment or calibration procedure, be sure that the thermocouple currently installed on the TGA 7 is functioning properly. The thermocouple can be checked by programming the TGA 7 to 100°C and checking the temperature displayed in the status panel. The temperature should be close to 100°C if the instrument is calibrated.
- To prolong the life of the TGA 7 standard and high temperature furnaces, it is recommended that the automatic furnace cleaning procedure be performed routinely such as every 5 – 10 runs. Pyrolysis products should be burned off after every analysis that involves pyrolysis of a sample in the absence of air or oxygen.
- When the TGA 7 standard or high temperature furnace is heating, keep the protective plastic shield on the TGA 7 in the down position. This will protect you from accidentally touching the furnace when it is hot.
- Do **not** attempt to move the TGA 7 while it is on. Wait until the power is off and the furnace cooling fan has stopped spinning before moving the instrument.
- Do **not** remove cables from the TGA 7 or TAC 7/DX when the analyzer is turned on.
- Do **not** touch the opening on the furnace lift mechanism or insert objects into the opening.
- Do **not** use hydrogen gas with the TGA 7 because it is highly explosive.

Furnaces

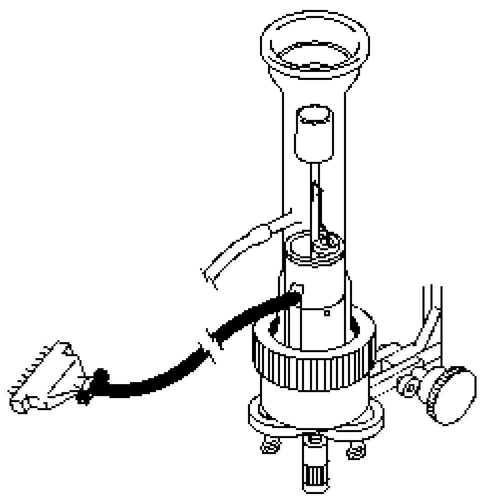
There are two furnace options that can be used with the TGA 7:

- **Standard Furnace**
- **High Temperature Furnace**

Most standard polymer applications are routinely performed with the TGA 7 standard furnace. This furnace allows routine operation to an upper temperature of 1000°C. The TGA 7 high temperature furnace is used for high temperature applications such as ceramics, metals, and geological studies. The high temperature furnace allows operation up to an upper temperature of 1500°C.

Standard Furnace

The TGA standard furnace allows operation of the TGA 7 from 30°C to 1000°C. The standard furnace is a small, platinum-wound microfurnace which allows rapid heating and cooling rates (up to 200°C/min) and rapid turnaround times between analyses. This unique furnace functions as both a heater and a resistance thermometer, detecting its own temperature and supplying power to heat the sample. This design makes it possible to obtain extremely sensitive temperature control and precision. A chromel–alumel thermocouple passes through the base of the furnace and is located close to the sample material. This provides accurate sample temperature during an analysis.



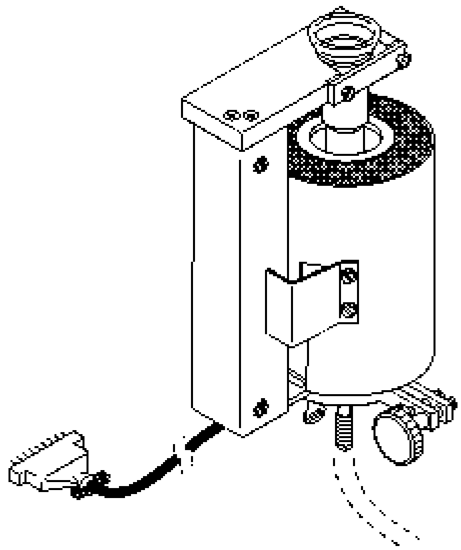
For detailed information on the TGA 7 standard furnace, see the following topics:

Furnace Positioning

Standard Furnace Maintenance

High Temperature Furnace

The TGA 7 high temperature furnace allows operation of the TGA 7 from 50°C to 1500°C. The high temperature furnace uses platinum–rhodium windings and can be operated at heating rates as fast as 100°C/min. This furnace provides excellent temperature control and precision. A sensitive platinum–rhodium thermocouple close to the sample is used to measure sample temperature during an analysis.



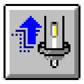
For detailed information on the TGA 7 high-temperature furnace, see the following topics:

Furnace Positioning

High Temperature Furnace Maintenance

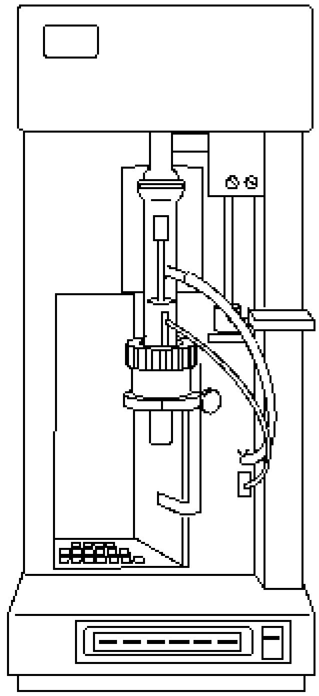
Furnace Positioning

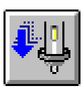
The TGA 7 has a unique automatic furnace movement and cooling feature that is controlled directly from the Pyris software by clicking on an icon. These icons are on the TGA 7 Control Panel:



Run or Raise Furnace Position

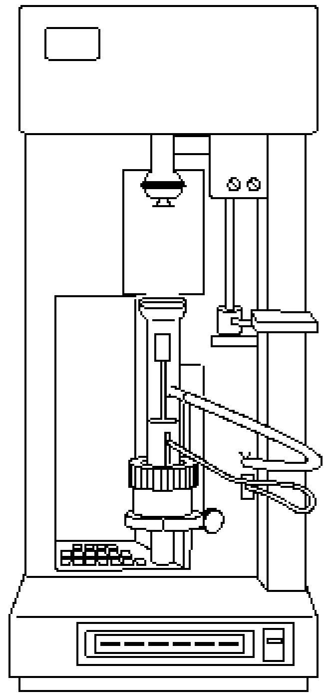
This icon automatically moves the furnace to the Run position:

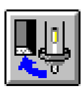




Lowered Position

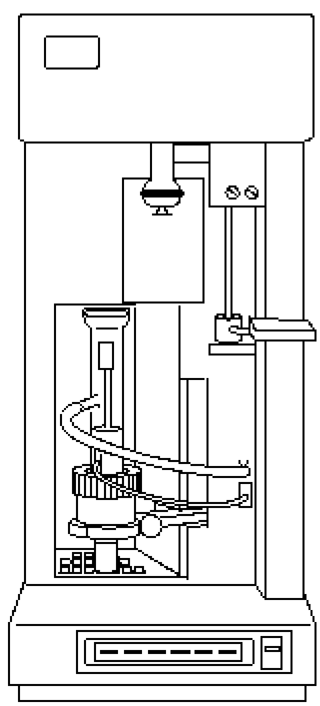
This icon automatically lowers the furnace to the Lowered position so you can gain access to the sample pan for loading or removing samples:





Cool Position

This icon automatically moves the furnace to the Cool position inside the analyzer over the large cooling fan to cool the furnace:



Microbalance

The microbalance used with the TGA 7 is extremely sensitive, capable of detecting weight changes as small as 0.1 µg, with a maximum capacity of 1300 mg. The null balance design of the microbalance uses a servo-controlled torque motor to automatically compensate for weight changes in the sample material. When the sample is placed in the sample pan, the beam that supports the sample pan deflects. A beam position detector measures the deflection with an optical sensor and uses current to return the beam to its original position. The amount of current necessary to restore the beam is a direct measure of the weight on the beam. The current is amplified and filtered. The signal is displayed in the Pyris software in milligrams or as a percentage of total sample weight.

Operating Variables

In thermogravimetric analysis, the object of an experiment is to record the change in weight of a sample as a function of temperature or time using a constant heating rate or more complex temperature program. Below are some techniques that can be used to obtain the most accurate data in a minimum amount of time.

Sample Preparation

The TGA 7 can analyze solid or liquid samples. Solid samples can be in the form of film, powder, crystal, or grains. In some cases, you may want to chop or grind the sample to create a large surface area that is exposed to the purge gas atmosphere. In other cases, larger chunks or sections of sample may be placed directly into the sample pan for analysis.

Sample Size

The size of sample that can be analyzed ranges from <1 mg to 400 – 500 mg. In most cases, the typical sample size for TGA analysis is 2 – 50 mg.

The type of transition or reaction that you expect to occur in the sample should dictate the amount of sample used. For example, in the case of polymer decomposition studies, sample sizes of 2 – 15 mg are recommended since very large weight loss values are associated with this type of experiment. If there will be a very small weight loss such as with water loss or solvent evaporation, larger sample sizes should be used. It is not uncommon to use samples as large as 40 – 50 mg.

Temperature Range

The temperature range used for your experiment will depend on the type of samples to be run as well as the specific application to be performed. The TGA 7 has two different furnaces. This permits operation over a broad temperature range. The standard furnace operates from ambient to 1000°C. This furnace has broad applications in the polymer, pharmaceutical, and organic chemical industries. For higher-temperature applications such as ceramics and metals, a high-temperature furnace is available. This furnace permits operation in the 50°C – 1500°C range.

Because of the unique design of the TGA 7, you can perform controlled heating or cooling experiments as well as isothermal temperature experiments as a function of time. You can even perform multiple-step experiments using any combination of heating, cooling, or isothermal segments, thereby allowing the performance of virtually any thermogravimetric application. In addition, there is also an **AutoStepwise Scan** available in the method program. This scan heats the sample rapidly between weight loss regions and slows down or stops heating during rapid weight loss regions.

AutoStepwise Scan

Pyris Software for Windows' AutoStepwise Scan feature uses programmable criteria to automatically determine the start and end points of a weight loss. It also switches between various heating rates or isothermal steps in order to optimize the TGA analysis.

Autostepwise thermogravimetric analysis is a technique in which thermogravimetric reactions (such as vaporization causing weight losses) may be studied more accurately by reducing the scan rate in a temperature program or holding the temperature constant when such a reaction is detected. The weight loss is detected by monitoring the rate of weight loss during a temperature scan. If the rate of weight loss is greater than a preset value, the instrument will either continue to scan the temperature at a preset reduced rate (stepwise scan) or hold the temperature constant. If during this reduced rate segment the rate of weight loss is less than a preset value, then the normal scan is resumed.

In the Method Editor Program Page, the user can enter the criteria for an autostepwise TGA temperature program. The data items collected will be the same as for a standard TGA: sample temperature, program temperature, and sample weight. Autostepwise parameters can be used in conjunction with only a temperature scan, not an isotherm.

The entrance and exit criteria during an autostepwise run is performed by the TAC7/DX interface for the TGA 7. This prevents a lengthy time delay between detection or a reaction and reduction of the scan rate. The instrument's firmware provides "alerts" that are synchronized with the data stream so that the proper data segments can be generated.

It is possible, and generally the case, that more than one extra segment is generated as a result of a change in the rate of weight loss. This occurs as a result of the following:

1. The instrument detects a weight loss rate greater than the entrance criterion and causes an autostepwise segment to be generated.
2. After a period of time the rate of weight loss drops below the exit criterion and the original temperature scan rate is restored.
3. The rate of weight loss may again go over the entrance criterion and *another* autostepwise segment is generated.

An autostepwise segment in a method consists of the original starting and ending temperatures for the segment, the original scan rate for the segment, and the complete set of entrance and exit criteria.It is this single segment that may be expanded at run time into several scans or scans and isotherms. Methods using autostepwise segments may be recalled for reuse. However, **only** the original single segment (scanning segment before runtime) is displayed in the Method Editor.

Autostepwise for TGA 7

Operation of autostepwise for a TGA 7 follows these rules:

1. The run is limited to 15 segments as recorded by the TAC.
2. If the limit is reached as segments are generated, the segments are discarded last to first.
3. If the autostepwise-generated segments have reached the point that another generated segment will go over the limit on the number of segments, the entrance criterion is ignored and the run proceeds at the original scan rate to the original final temperature of the current segment.
4. It is possible to have one scan with stepwise parameters, namely, the first.

Scanning Rate

Scanning rates for the TGA 7 range from 0.1°C/min to 200°C/min for the standard furnace and from 0.1°C/min to 100°C/min for the high-temperature furnace. The exact scanning rate used will depend on the experiment used and the end result you are trying to achieve. For example, most TGA experiments are performed at heating rates of 5°C/min to 50°C/min. However, there may be times when you want to heat or cool rapidly to a selected temperature and then hold isothermally or scan at a controlled rate. In such cases, very fast heating or cooling rates (i.e., 100°C/min or 200°C/min) are typically used to quickly increase or decrease the sample temperature.

Sample Atmosphere

WARNING: **Due to the highly explosive nature of hydrogen, it is recommended that it not be used with this instrument.**

The atmosphere to which the sample is exposed is carefully controlled by the selection of the sample **purge gas** and flow rate. Recommended sample purge gases are air, oxygen, nitrogen, and argon. Other active or reactive gases may be used provided they do not react with the furnace materials, thermocouple, or other analyzer components in which the gases come into contact.

In addition, purge gases may be switched at any point during an analysis by using a **Gas Selector Accessory GSA 7** or a **Thermal Analysis Gas Station (TAGS)**. A portion of an experiment (e.g., the pyrolysis of the polymer portion of a sample) may be performed in an inert gas such as nitrogen. The purge gas may then be switched to an active gas such as air or oxygen to selectively oxidize the other components in the sample such as carbon black.

Purge Gases

The TGA 7 requires the use of two purge gas lines that individually purge the balance chamber and the sample/furnace area. In addition, a gas supply is required for automatic furnace positioning. If you want to use only the balance purge, clamp off the Tygon sample purge line where it enters the upper glassware of the TGA 7, just above the furnace. Only one purge gas line is required in this case. That gas line should be connected to the Balance Purge connection at the rear of the TGA 7. The balance purge gas supply flows through the balance chamber, down through the sample/furnace area, and then out of the system through an exhaust line.

The sample purge is bypassed around the balance chamber and enters directly above the sample/furnace area. This purge gas flows directly over the sample area and then out through the same exhaust line as the balance purge. This design permits you to rapidly switch the sample atmosphere without having to purge the entire balance chamber. In addition, you will always have controlled purge through the balance chamber, protecting the balance components at all times.

The recommended balance purge gas is an inert gas such as nitrogen or argon with a minimum purity of 99.9%. **The gas must be dry**. The recommended flow rate for the balance purge gas is 40 – 60 cc/min. These rates will be realized by an output pressure of 27,000 – 41,400 Pa (4 – 6 psi) on the output side of the regulator when using the type “A” flow restrictor.

The sample purge gas can be nitrogen, argon, oxygen, helium, or other inert or active gases. (The gas used should not be reactive with the materials used in the construction of the TGA 7, including Pyrex, stainless steel, and platinum.) A size 1A cylinder equipped with a suitable regulator is recommended.

Complete instructions on installing the purge gases and how to connect them to the TGA 7 are given in the online Installation Help.

The flow rate of the sample purge **must be less than** the balance purge flow rate at all times. If the sample purge flow rate is greater than the balance flow rate, decomposition products may be deposited in the balance chamber and may eventually cause damage to the microbalance. Therefore, the recommended flow rate for the sample purge line are 20 – 35 cc/min. These rates will be realized by an output pressure of 13,800 – 24,150 Pa (2 – 3.5 psi) on the output side of the regulator when using the type "A" flow restrictor.

If you wish to use more than one sample purge gas, you can use either a **GSA 7 Gas Selector Accessory** for two gases or a **Thermal Analysis Gas Station (TAGS)** for four gases.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the plug attached to the N519-0310 analog cable at the back of the TAC 7/DX Thermal Analysis Instrument Controller. The TAC 7 then connects to the analyzer.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Instructions for installing the TAGS are in the online Installation Help.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

WARNING: FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 1/4-in. pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

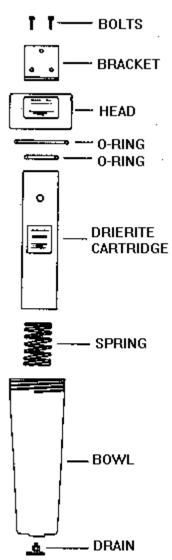
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4-in. pipe fitting using Teflon tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4-in. pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2-1/2" o.d. O-ring on top of cartridge.
8. Place the 4" o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD SOAP AND WATER. DO NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

Calibration

There are three calibration routines for the TGA 7. All calibrations should be performed upon installation of the TGA 7. Periodic calibration checks will be necessary to verify accurate calibration.

The TGA 7's calibration should remain unchanged for quite some time, provided that there are no changes in the instrument's operating conditions. Even when the system is shut off, the calibration values are stored so that the next time the instrument is turned on, it will still be calibrated. Some of the conditions that could change either the temperature or weight calibration of the TGA 7 and require recalibration are as follows:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
- If the purge gas type or purge gas flow rate is changed, the calibration should be checked for highest accuracy.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new thermocouple is installed or if the position of the thermocouple changes, you should perform the temperature calibration again.
- If the analyzer is moved or releveled, you should perform the weight calibration again.

The calibration routines for the TGA 7 are

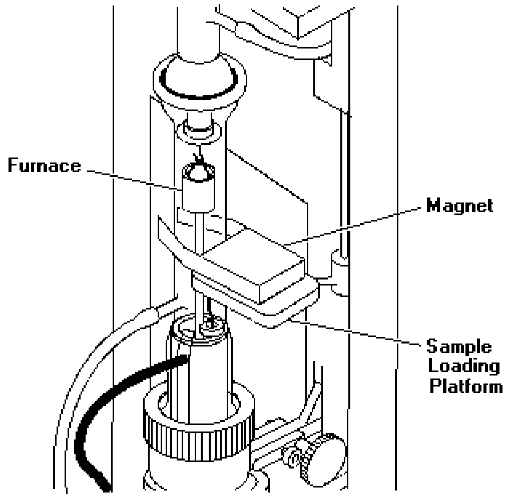
- **Calibrate Temperature**
- **Calibrate Weight**
- **Calibrate Furnace**

Calibrate Temperature

The temperature calibration for a TGA 7 is performed when you are using a standard furnace. You do not need to perform a temperature calibration for a high temperature furnace. However, if you want to calibrate a high temperature furnace for temperature, you may do so by following the procedure below, but you will need to supply a magnet that will work with the high temperature furnace and you will have to determine where you can place the magnet.

1. From the Method Editor, select Open Method from the File menu. Double click on the Nickcal.tgm method file which is in the Method directory.

   The nickel calibration method is displayed on the screen.
2. Select the Sample Info tab and fill in the Sample ID, Operator ID, Comment, and File Name if you wish to do so.
3. Cut a small piece of nickel approximately 1 – 2 mm long and place it in the sample pan.
4. Click on the **Raise Furnace** button in the control panel.
5. Click on the **Zero Weight** button in the control panel. The analyzer reads the weight of the sample.
6. After the weight reading has stabilized, click on the **OK** button. The weight is entered into the Zero field of the Enter Sample Weight area on the Sample Info page.
7. Place the magnet onto the sample loading platform and move the platform next to the furnace tube.



8. Adjust the height of the sample loading platform so that you get a weight reading between 3 and 6 mg. The apparent weight reading is a result of the magnetism of the sample. As the sample loses magnetism, the apparent weight reading decreases.
9. After the weight reading has stabilized, click on the **Sample Weight** button on the control panel. The weight is entered into the Weight field of the Enter Sample Weight area of the Sample Info page.
10. Click on the **Start Method** button on the control panel.
11. Repeat steps 1 – 10 to obtain a run for iron, opening the Ironcal.tgm method file to use fo the calibration run.

You are now ready to calculate the Onset value for both iron and nickel.

1. In Data Analysis, open the data file from either the nickel or the iron run. Be sure that the curve is displayed with the x axis as Temperature (change by selecting Rescale X from the Display menu and selecting Temperature for the Set Axis Units value) and the y axis as Weight (change by selecting Weight from the Curves menu; you may have to use Swap Y Axes from the Rescale Tools bar to place Weight on the left-hand axis.)
2. Select Derivative from the Math menu. The first derivative of the curve is displayed. Use the inflection point to accurately determine what the left limit should be for the onset calculation.
3. Click on the sample curve to make it the focused or active curve.
4. Select Onset from the Calc menu.
5. Move the two red **X** marks on the curve. Position the left limit marker at the inflection point of the first derivative curve and place the right limit marker past the inflection point on a level portion of the curve.
6. Click on the **Calculate** button in the Onset dialog box. The Adjust Tangents dialog box is seen.
7. Adjust the tangents of each point until they cross.
8. Click on the **Calculate** button in the Adjust Tangents dialog box. The onset temperature is calculated for the sample and displayed on the screen (Onset X).
9. Repeat steps 1 – 9 to calculate the onset temperature for the second reference material.
10. In the Method Editor, select Calibrate from the View menu.

11. Click on the Temperature tab in the Calibration window. Enter the Measured Onset values for nickel and iron and click in the check box in the Use column for each reference material.
12. Click on the **Save and Apply** button to send the calibration values to the analyzer and save the current calibration file.
13. Select the **Close** button to exit the Calibration window.

Calibrate Weight

1. While in the Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Enter the weight of your reference material in the Reference Weight edit box.
4. Select the **Begin Calibration** button.
5. If there is a sample pan already on the hangdown wire, remove any sample from the pan. If there is no sample pan already in place, then place an empty sample pan on the sample loading tray and load the pan onto the hangdown wire. Move the sample loading tray away from the sample pan. Click on **OK** in the dialog box.
6. When the zero weight reading is stable in the Read Zero field, click on **OK** in the dialog box.
7. Place the reference weight in the sample pan. Move the sample loading tray away from the sample pan. Click on **OK**.
8. When the weight reading is stable in the Read Value field, click on **OK**. The calibration is complete.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.
10. Go to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

Calibrate Furnace

Before performing the furnace calibration of the TGA 7, make sure that the thermocouple is functioning properly and that the sample pan is empty. To verify thermocouple operation, raise the TGA 7 furnace and program the temperature to 100°C. Enter 100 in the Go To Temp entry field of the Control Panel and click on the **Go To Temp** button.

Check that the current sample temperature displayed in the Status Panel is at or below the intended minimum calibration temperature. The easiest way to accomplish this is to click on the **Go To Load** button on the Control Panel.

When selecting minimum and maximum temperatures for the furnace calibration, select the limits so that they encompass the temperature range in which you plan to operate.

The furnace must be in the raised position to perform a furnace calibration. Lifting and clamping by hand is not sufficient. You must select the **Raise Furnace** button on the Control Panel.

NOTE: Furnace calibration must be performed **after** Temperature calibration.

1. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
2. If applicable, complete the Temperature calibration.
3. Remove any sample from the sample pan in the stirrup.

4. Select the Furnace tab in the Calibration window.
5. In the Minimum field, enter a minimum temperature where the calibration will begin. This should be below your normal operating region.
6. In the Maximum field, enter a maximum temperature where the calibration will end. This should be above your normal operating region.

NOTE: Maximum temperature minus minimum temperature must be greater than 100°C or the furnace calibration will not be performed.

7. Select the **Begin Calibration** button.
8. The approximate time remaining in the furnace calibration is displayed.
9. Select the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.
10. Select the **Close** button to close the Calibration window and begin using the new calibration values.

Calibration Reference Materials

Your company may have to comply with ISO 9000. Perkin Elmer Thermal Analysis calibration materials are ISO 9000 compatible. They are called Reference Materials. ISO has two classifications for calibration materials: Reference Materials and Certified Reference Materials.

Reference Materials (RMs) comply with ISO for instrumentation calibration. They are available from Perkin Elmer and Perkin Elmer provides documentation with each RM that cross references the material's lot code. This declaration should be filed with all other material certificates and documents in your ISO file.

ISO Guide 30 defines a RM as a

> "Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials."

Certified Reference Materials (CRMs) are traceable to national or international standards through an unbroken chain of comparisons. Not every calibration material is a CRM. CRMs can be very expensive and are not available for all thermal analysis techniques.

If you are not sure what material to use, then check with your Quality Assurance Department and discuss the differences between RMs and CRMs. If you find that you need CRMs, you may order them from the National Institute of Standards and Technology (301 975-6776).

Magnetic Calibration Reference Materials are used to perform the Curie point temperature calibration on the standard furnace. They are available from PE XPRESS in kit form and separately. We recommend that you use fresh reference materials each time you calibrate your instrument. This way you can avoid the effects of hysteresis and oxidation on the Curie point of the reference materials. The transition temperature for each of the magnetic standards is indicated in the documentation included with the reference material.

Part No.	Reference Material	Magnetic Transition Temperature
0219-0071	Kit of 5 reference materials including alumel, nickel, Perkalloy, iron, and Hisat-50	
0998-8015	Three 2-in. pieces of 99.99% alumel wire	163°C
N519-0869	Three 2-in. pieces of 99.99% nickel wire	354°C
N519-0616	Three 2-in. pieces of Perkalloy wire	596°C
0998-8017	Three 2-in. pieces of 99.99% iron wire	780°C

Maintenance

The TGA 7 needs little routine maintenance other than proper treatment as a sensitive electronic device. Occasionally, the furnace or sample pans may become coated with sample residue and require cleaning, or furnace components and other accessories, such as hangdown wires and thermocouples, may need to be replaced.

Maintenance of the TGA 7 includes the following procedures:

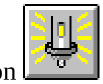
- **Cleaning the Furnace**
- **Cleaning the Sample Pan**
- **Standard Furnace Maintenance**
- **Standard Furnace Tube Maintenance**
- **Standard Furnace Hangdown Wire Maintenance**
- **High Temperature Furnace Maintenance**
- **High Temperature Furnace Tube Maintenance**
- **High Temperature Furnace Oven Thermocouple Maintenance**
- **High Temperature Furnace Sample Thermocouple Maintenance**
- **High Temperature Furnace Hangdown Wire Maintenance**

Cleaning the Furnace

An automatic procedure for cleaning the TGA 7 standard and high temperature furnaces is included in the Pyris software. The procedure involves lowering the furnace to expose it to the air and then heating it to approximately 900°C to burn off any materials coated onto the furnace surfaces.

WARNING: **Since the furnace will be programmed to 900°C, make certain that the protective plastic visor on the TGA 7 is in the down position. DO NOT touch the furnace during this procedure.**

1. Start up the Pyris software and make sure the Control Panel is displayed.

2. Select the **Clean Furnace** icon  from the Control Panel.
3. The furnace will move to the lowered position and programmed to 900°C. When the cleaning procedure is complete, the furnace will move to the cool position and cool down to 30°C.

NOTE: To abort the furnace cleaning procedure at any time, select the **Cool Furnace** icon.

Cleaning the Sample Pan

The platinum sample pans used with both the standard and high temperature furnaces in the TGA 7 can be cleaned with one of the following techniques:

1. Place the sample pan on the hangdown wire, raise the furnace, and, using air or oxygen as a sheath gas, heat the instrument to 950°C to burn off any material coated on the pans. After the pans cool, scrape off any residue or tap out any ash residue.
2. Carefully flame the pan over a Bunsen burner.

Standard Furnace Maintenance

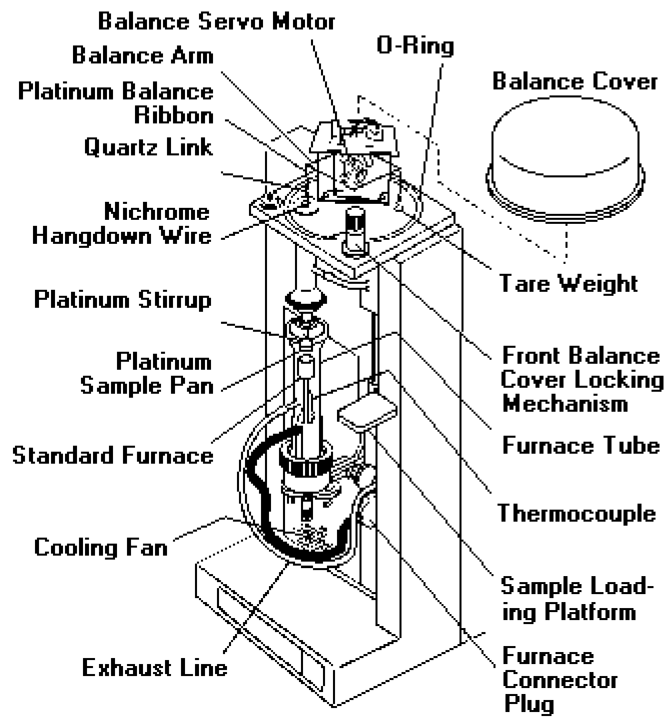
Standard furnace maintenance consists of the following procedures:

- **Removing the Standard Furnace**
- **Replacing the Standard Furnace**
- **Adjusting the Standard Furnace**

Refer to the diagram below of the parts of the standard furnace while performing these procedures.

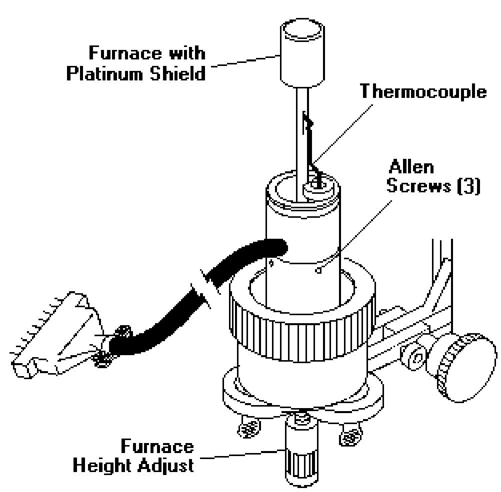
WARNING: **Before performing these procedures, shut down the system and remove the line power. Make sure the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.**

TGA 7 with a Standard Furnace

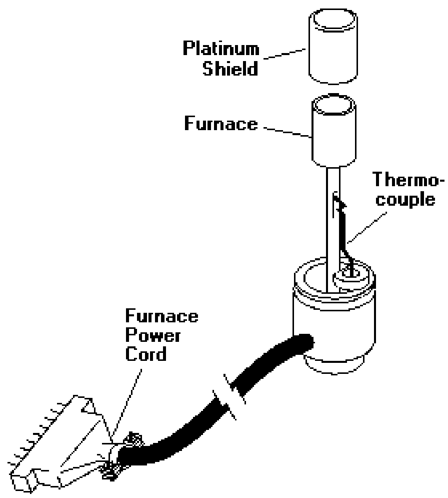


Removing the Standard Furnace

1. Remove the standard (Pyrex) furnace tube.
2. Disconnect the furnace power cord from its connector on the front of the TGA 7. To disconnect the cord, squeeze the silver locking tabs and gently pull forward on the connector.
3. Using the hex wrench (P/N 0990-7236) supplied with the TGA 7 Spares and Accessories Kit, loosen the three Allen screws on the furnace mounting assembly. Remove the furnace by lifting it straight up and off the mounting assembly.



4. Remove the platinum furnace shield by sliding it off. Do not deform the shield.



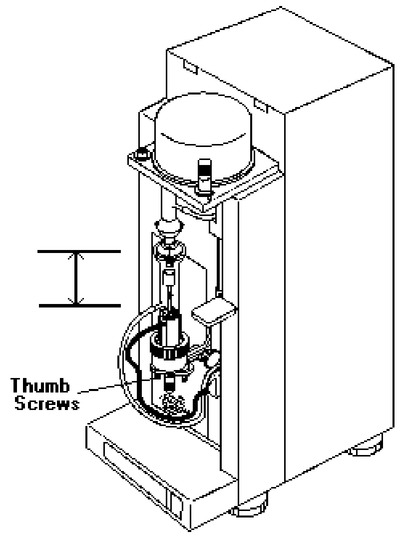
If you plan to use the thermocouple from the removed furnace on the new furnace, remove the thermocouple as described in the subtopic below.

Replacing the Standard Furnace

1. Slide the platinum furnace shield over the furnace.
2. Place the furnace on the furnace mounting assembly. Note that there is a locating pin on the mounting assembly that fits into a hole on the furnace base to assist alignment. Using the hex wrench (P/N 0990-7236), tighten the three set screws on the furnace mounting assembly to secure the furnace in place.
3. Replace the standard (Pyrex) furnace tube.
4. Plug in the furnace power cord to its connector on the front of the TGA 7. There are male and female guides on the connector that allow the connector to go on in only one way.
5. Adjust the furnace position.

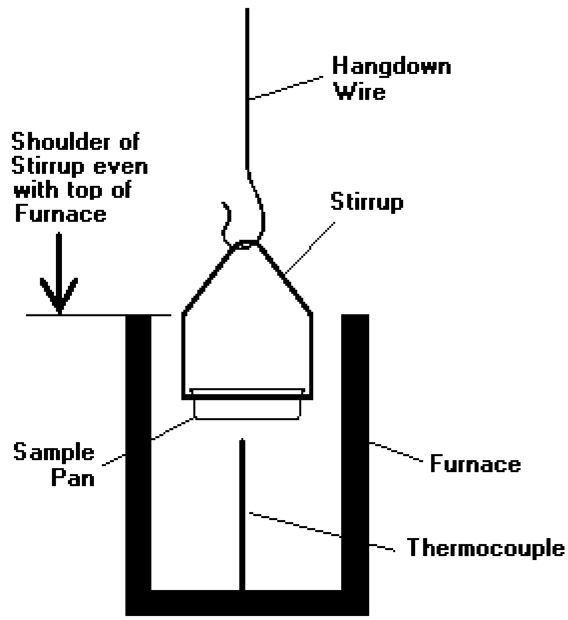
Adjusting the Standard Furnace

1. Make sure that the TGA 7 Control Panel is displayed on the screen.
2. From the TGA 7 Control Panel select the **Lower Furnace** icon. The TGA 7 furnace lowers automatically (if it was not already in the lowered position).
3. To adjust the horizontal position of the furnace, loosen the three thumbscrews that lock the furnace mounting assembly in place.



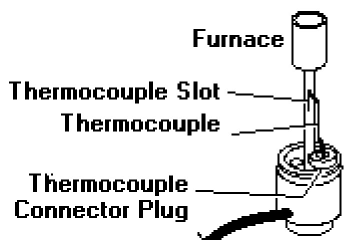
4. Select the **Cool Furnace** icon.
5. Select the **Raise Furnace** icon. If it is properly aligned, the furnace tube should move smoothly into position around the upper ball joint. Use the sample loading platform as a furnace movement control by pushing the platform to the safe position and then moving it back out again. Notice that each time you put the platform in the safe position, the furnace moves up a little further.
6. As the furnace tube gets close to the upper ball joint, position the furnace assembly so that the furnace tube moves smoothly onto the ball joint. When the tube and ball joint appear to be aligned, allow the furnace to go all the way up by placing the sample loading platform to the safe position and leaving it there.
7. Adjust the furnace assembly with your hand until it appears that the furnace is aligned correctly (i.e., the tube is at a right angle to the table top).
8. Lock the furnace assembly into position using the three thumbscrews at the base of the furnace mounting assembly.
9. Adjust the vertical position of the furnace using the furnace adjusting knob located below the furnace mount. Clockwise adjustment of this knob raises the furnace; counterclockwise adjustment lowers the furnace.

   The top of the furnace should be approximately 10 mm below the end of the hangdown wire and even with the shoulder of the stirrup. The sample should be just below the halfway point on the furnace side wall.



Removing and Installing the Thermocouple in a Standard Furnace

1. Disconnect the plastic thermocouple connector plug from the base of the furnace by gently pulling up on the connector plug.



CAUTION: Do not bend the thermocouple severely as this may damage it. If the thermocouple appears old, worn, or broken, replace it (P/N 0319-1253).

2. Remove the tip of the thermocouple from inside the furnace by pulling the thermocouple straight down toward the bottom of the furnace.
3. Take the new thermocouple and, while looking inside the furnace, feed the tip of the new thermocouple through the thermocouple slot in the furnace stem until it is approximately 1/3 of the way up inside the furnace.
4. Center the thermocouple using tweezers.
5. Plug the plastic thermocouple connector plug on the base of the thermocouple into the base of the furnace. There are two small pins in the furnace base where the thermocouple connects. Be careful not to bend or break the thermocouple pins in the furnace base.

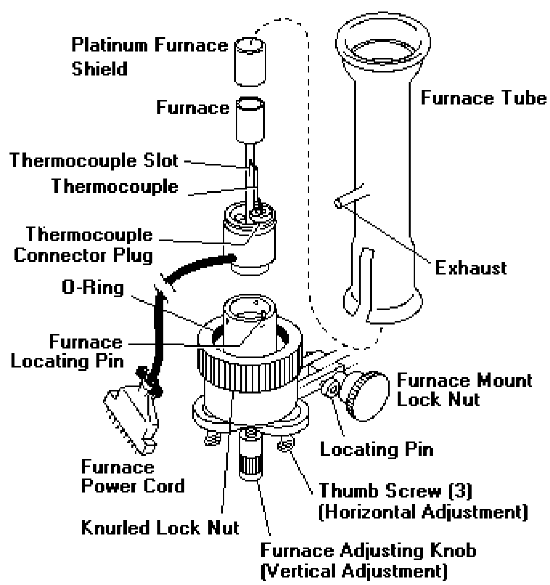
Standard Furnace Tube Maintenance

Maintenance of the standard furnace tube consists of the following procedures:

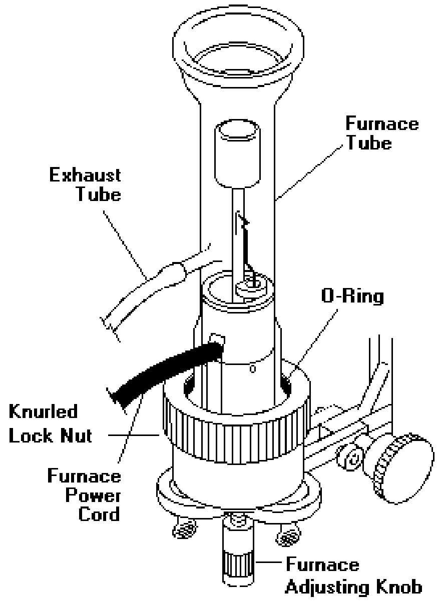
- **Removing the Standard Furnace Tube**
- **Cleaning the Standard Furnace Tube**
- **Replacing the Standard Furnace Tube**

Refer to the schematic below while performing the procedures:

TGA 7 Standard Furnace and Furnace Tube



Removing the Standard Furnace Tube



1. Make sure that the furnace is in the lowered position.
2. Remove the exhaust line from the furnace tube side arm.
3. Loosen the black plastic knurled lock nut at the base of the furnace tube.
4. Remove the O-ring located at the base of the furnace tube platform. The O-ring is split for easy removal and replacement.
5. Grasp the furnace adjusting knob at the base of the furnace with one hand and the furnace tube with the other hand. Gently pull up on the furnace tube and remove the tube.

CAUTION: To avoid breaking the furnace's thin stem, do not pull the furnace too fast or at an angle.

Cleaning the Standard Furnace Tube

In order to clean the furnace tube, you must first remove the tube from the furnace, as described above. Clean the furnace tube using one of these methods:

1. Soak and scrub the furnace tube in a strong detergent (e.g., alconox).
2. Soak the furnace tube in a strong solvent such as cleaning solution (concentrated H_2SO_4 and K_2CrO_7).

WARNING: **Concentrated H_2SO_4 and K_2CrO_7 is a very strong, caustic acid solution. Wear protective gloves and safety glasses and perform this procedure in a fume hood.**

3. Dip the furnace tube in a dilute hydrogen fluoride (HF) solution (10% solution, 5 minutes maximum).

WARNING: **Hydrogen fluoride is a dangerous skin irritant. Wear protective gloves and safety glasses and perform this procedure in a fume hood.**

4. Rotate the furnace tube over a Bunsen burner (heat up slowly to avoid cracking).

After cleaning, reinstall the furnace tube.

Replacing the Standard Furnace Tube

1. Carefully place the furnace tube over the furnace and press the tube down so that it rests on the base of the furnace tube platform. Position the furnace tube so that the cutout at the base of the tube allows the furnace electrical connections to exit without being bent or pinched.

NOTE: If you are replacing the O-ring, split the new O-ring at a right angle with a razor blade before placement.

2. Place the O-ring around the outside of the furnace tube base. (The tube flares out at the base.)
3. Tighten down on the furnace tube and O-ring by threading the black plastic knurled lock nut at the base of the furnace tube.
4. Replace the exhaust line to the furnace side arm.

Standard Furnace Hangdown Wire Maintenance

A nichrome hangdown wire has a platinum stirrup on the end of it. This stirrup supports the sample pan in the furnace. These wires come in preformed kits (P/N N519-0516), kits containing straight wires (P/N N519-0515) that you can form into hangdown wires, or in kits containing both straight and formed (P/N N519-0285). A kit of six preformed and six straight nichrome wires is included with the TGA 7 standard furnace option.

The procedures below describe how to remove and replace the hangdown wire. For multimedia help on how to remove and replace the hangdown wire, see **Removing the Hangdown Wire** and **Installing the Hangdown Wire** in Pyris Multimedia Presentations Help.

NOTE: The multimedia presentations are also applicable to hangdown wire removal and replacement for a TGA 7 with a high temperature furnace.

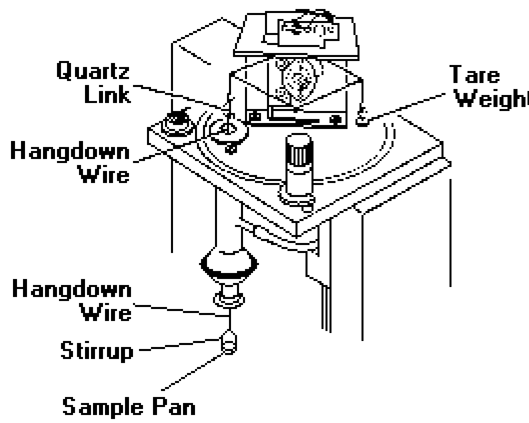
WARNING: **Before performing these procedures, make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.**

Removing the Hangdown Wire

1. Before removing the hangdown wire, familiarize yourself with the parts of the TGA 7 involved in replacing the hangdown wire.
2. Lift the clear plastic visor on the front of the TGA 7 all of the way up. Then raise the cover that allows access to the balance. The dome-shaped metal cover that protects and isolates the balance mechanism should now be visible.

3. The dome-shaped cover is secured in place by two locking mechanisms: one in the front of the balance chamber and one at the rear. Turn both locks counterclockwise simultaneously to unlock the cover.
4. Lift the cover straight up and off to expose the balance mechanism.
5. Move the sample loading platform directly under the sample pan.
6. Raise the platform up until the pan rests on the platform and the stirrup is lifted off of the bend in the hangdown wire.
7. Swing the platform to the right to remove the sample pan and stirrup assembly.

   A platinum balance ribbon is connected to the left arm of the balance. This ribbon has a loop at its base. A quartz link is connected to the loop on the balance ribbon. The nichrome hangdown wire hangs from the quartz link. This arrangement must be maintained for proper operation. A tare weight is hung from the right side of the balance beam to tare off the weight of the hangdown wire and sample pan.



CAUTION: The platinum balance ribbon is extremely fragile. Care should be taken not to exert force on the loop of the balance ribbon or on the hangdown wire after it is attached. Handle tweezers with extra care when placed in the vicinity of the platinum balance ribbon, the hangdown wire, or the stirrup. Tweezers can catch any of these components and cause damage.

8. Carefully raise the sample loading platform until it comes in contact with the hangdown wire. While looking at the quartz link attached to the platinum balance ribbon on the balance arm, continue to lift the sample loading platform until the hangdown wire begins to rise in the quartz link.
9. Using tweezers, carefully remove the hangdown wire from the quartz link.
10. Lower the sample loading platform. The hangdown wire should descend through the tube.
11. When the hangdown wire is no longer attached to the link on the platinum balance ribbon, grasp the hangdown wire near the sample loading platform with the tweezers and remove it.

Installing the Hangdown Wire

The steps below describe how to select and install a new hangdown wire for the standard or high temperature furnace. The balance cover should be off, exposing the balance mechanism.

1. Raise the sample loading platform as high as possible and center it below the hangdown tube.

2. Carefully remove a new hangdown wire from its container. Handle it with one hand.
3. From the top of the balance chamber, drop the new hangdown wire through the hangdown tube.
4. Lower the sample loading platform until the bend in the hangdown wire is at approximately the same level as the quartz link on the platinum balance ribbon.
5. Using tweezers, grasp the hangdown wire with the wire tip facing the link. Insert the wire tip through the link and release the hangdown wire from the tweezers. Withdraw the tweezers carefully so they do not get caught on any components of the balance.
6. Rotate the platform underneath the hangdown tube and carefully replace the sample pan and stirrup on the end of the new hangdown wire.
7. Lower the sample loading platform and rotate it completely to the right.
8. Replace the dome-shaped metal balance cover and lock it into place by turning the two locking mechanisms clockwise simultaneously.
9. Lower the access cover.

NOTE: Calibrate the TGA 7 after replacing the hangdown wire.

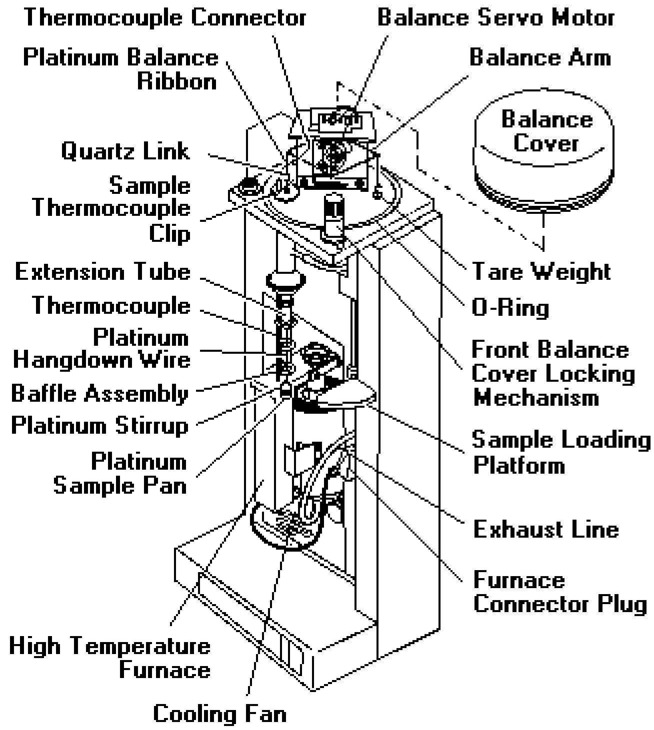
High Temperature Furnace Maintenance

High temperature furnace maintenance consists of the following procedures:

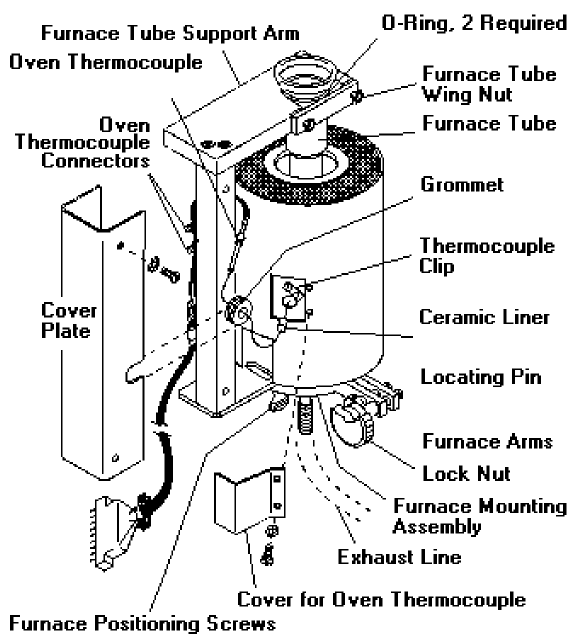
- **Removing the High Temperature Furnace**
- **Replacing the High Temperature Furnace**
- **Adjusting the High Temperature Furnace**

Refer to the schematics below while performing the procedures below.

TGA 7 with a High Temperature Furnace



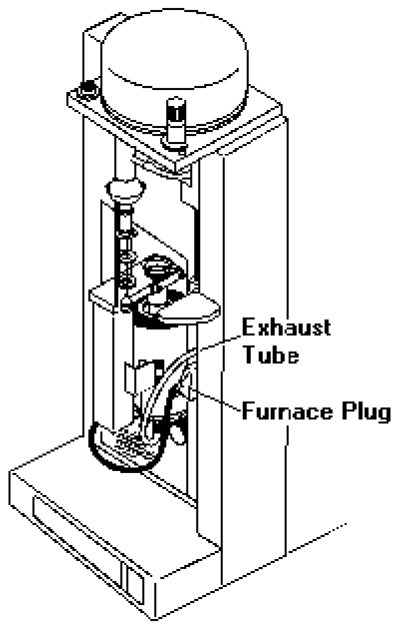
TGA 7 High Temperature Furnace



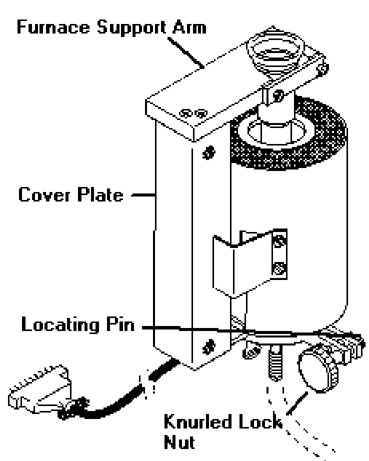
WARNING: Before performing these procedures, shut down the system and remove the line power. Make sure the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.

Removing the High Temperature Furnace

1. Disconnect the furnace power cord from its connector on the front of the TGA 7 by squeezing the silver locking tabs and gently pulling forward on the connector.
2. Unplug the furnace exhaust line where it enters the front panel of the analyzer.



3. Support the furnace by grasping the cover plate of the furnace with one hand. Unscrew and remove the knurled lock nut located on the furnace mounting assembly with the other hand. Remove the locating pin by sliding it forward. The entire furnace assembly can now be removed.

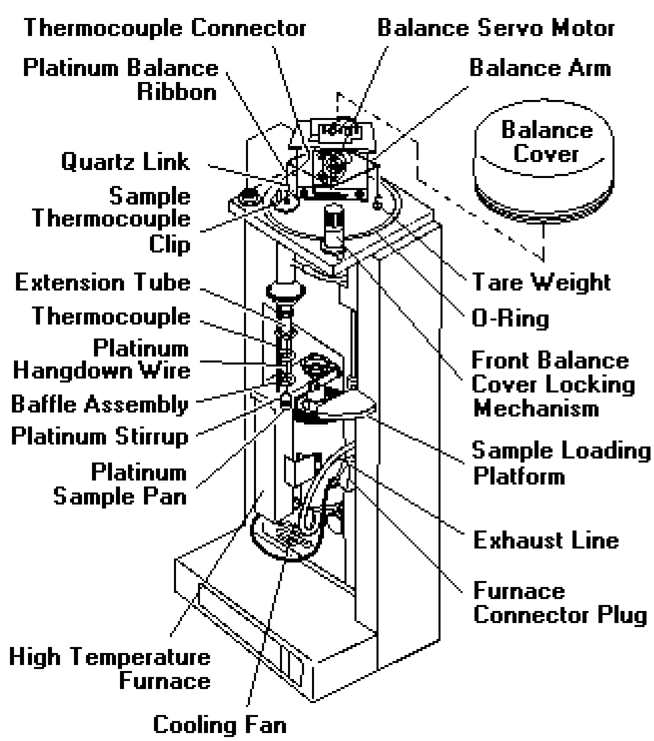


Replacing the High Temperature Furnace

1. Hold the furnace so that the arms on the furnace mounting assembly are positioned near the furnace lift shaft. Line up the holes in the shaft and the arms on the furnace mounting assembly and place the locating pin through the left-hand hole.
2. Replace the knurled lock nut in the right-hand hole, threading it until it is fingertight.
3. Plug the furnace exhaust line into its tube connector on the front panel of the analyzer.
4. Plug the furnace power cord into its connector on the front of the analyzer. There are male and female guides on the connector that allow it to go on in only one way.
5. Adjust the furnace position.

Adjusting the High Temperature Furnace

1. Make sure that the TGA 7 Control Panel is displayed on the screen.
2. Select the **Lower Furnace** icon. The TGA 7 furnace is lowered automatically (if it was not in the lowered position already).
3. Loosen the three furnace positioning thumbscrews that lock the furnace mounting assembly in place.



4. Select the **Cool Furnace** icon.
5. Select the **Raise Furnace** icon. As soon as the furnace begins to move, move the sample loading platform out slightly from the safe position. (You need to move it only about 0.5 – 1 in.)
6. Use the sample loading platform as a furnace movement control by pushing the platform to the safe position and then moving it back out again. Notice that each time you put the platform in the safe position for furnace moves up a little further.

7. As the furnace tube gets close to the upper ball joint, position the furnace assembly so that the furnace tube moves smoothly onto the ball joint. When the tube and ball joint appear to be aligned, allow the furnace to go all the way up by placing the sample loading platform to the safe position and leaving it there.
8. Adjust the furnace assembly with your hand until it appears that the furnace is aligned correctly (i.e., the tube is at a right angle with the table top).
9. Lock the furnace assembly into position using the three thumbscrews at the base of the furnace mounting assembly.

High Temperature Furnace Tube Maintenance

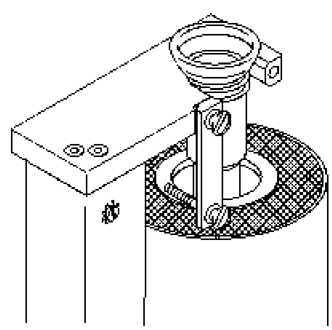
Maintenance of the high temperature furnace tube consists of the following procedures:

- **Removing the High Temperature Furnace Tube**
- **Replacing the High Temperature Furnace Tube**

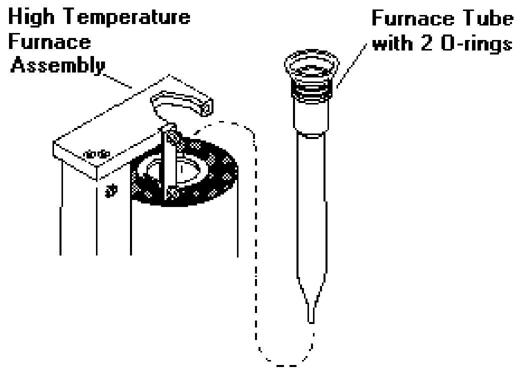
WARNING: **Before performing these procedures, make sure the furnace has cooled sufficiently so that you do not accidentally burn yourself when removing the furnace components.**

Removing the High Temperature Furnace Tube

1. Remove the TGA 7 high temperature furnace.
2. Put the furnace on the table or bench horizontally with the furnace tube wing nuts and thermocouple cover facing up.
3. Loosen and remove one of the two furnace tube wing nuts and let the metal holddown bar hang down.

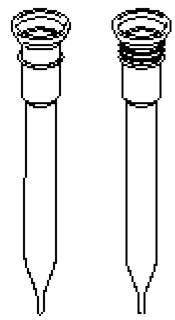


4. Gently slide the furnace tube out of the furnace. Note the location of the O-rings on the furnace tube.



Replacing the High Temperature Furnace Tube

1. Install the two O-rings (P/N 0990-2161) on the upper portion of the furnace tube. These O-rings protect the furnace tube from damage when the tube is locked in place.



2. Slide the furnace tube into the furnace from the top. The two O-rings should rest in the cutout in the furnace tube support arm.
3. Lock the furnace tube into position by swinging up the metal holddown bar and tighten the wing nut.
4. Replace the furnace.

High Temperature Furnace Oven Thermocouple Maintenance

Furnace temperature control of the TGA 7 high temperature furnace is accomplished through the use of an oven thermocouple. The procedures below describe its removal and replacement.

WARNING: **Before performing these procedures, shut down the system and remove line power. Make sure the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.**

Removing the High Temperature Furnace Oven Thermocouple

1. Remove the high temperature furnace from the TGA 7.
2. Place the furnace on a table or bench horizontally with the furnace wing nuts and oven thermocouple cover facing up.

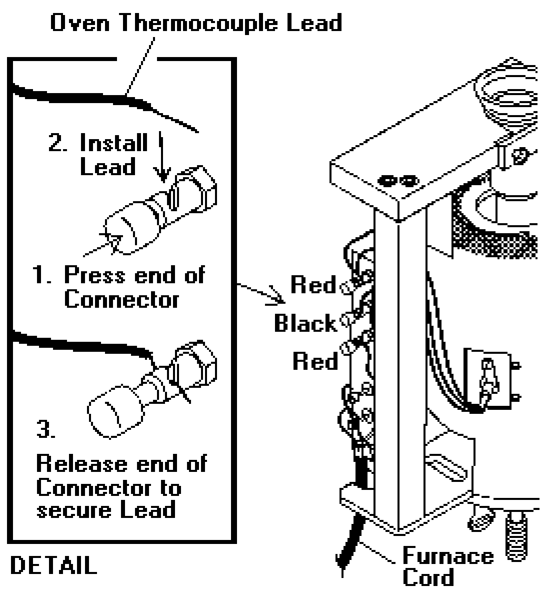
3. Loosen and remove the two screws securing the cover for the oven thermocouple. Remove the cover and the cover plate.

   NOTE: As you slide the cover plate off, make sure that the rubber grommet is not stuck on the cover plate. The grommet should slide off as the cover plate is removed.

4. Loosen (but do not remove) the screw securing the oven thermocouple clip. Swing the clip to the side.
5. Pull the oven thermocouple out from the furnace.
6. If necessary, remove the ceramic liner from the oven thermocouple and replace it. (The liner rests at the base of the oven thermocouple tube. To remove it turn the furnace over and shake gently.)
7. Disconnect the red and black leads from the oven thermocouple connections by pressing down gently on the plastic caps on the ends of the three connectors and sliding the wires out of the groove in each connector.

Replacing the High Temperature Furnace Oven Thermocouple

1. Connect the new oven thermocouple's three wires to the thermocouple connections. The black wire goes on the middle connector. Push the plastic cap in as you wrap the wire around connector. Releasing the cap will secure the wire.



2. Place the black rubber grommet over the tip of the oven thermocouple. Place the thermocouple into the furnace so that its tip rests at the base of the ceramic liner.
3. Secure the thermocouple in position by swinging the oven thermocouple clip down and tightening the screw to lock the clip in place.

   NOTE: As you tighten the screw to lock the oven thermocouple clip in place, make sure that the clip does not rotate or you may damage the thermocouple.

4. Replace the cover plate, making sure that the rubber grommet slides into the cutout on the plate. Secure the plate into position using the two screws.
5. Replace the furnace.

High Temperature Furnace Sample Thermocouple Maintenance

The procedures necessary to remove and replace the TGA 7 high temperature furnace sample thermocouple are discussed below.

WARNING: **Before performing these procedures, shut down the system and remove the line power. Make sure the furnace has cooled sufficiently so that you do not accidentally burn yourself.**

CAUTION: Use care when working in the area of the balance mechanism so as not to change the delicate components in this area.

Removing the High Temperature Furnace Sample Thermocouple

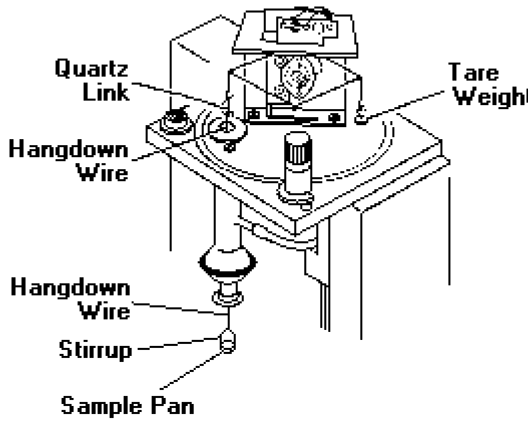
1. Lift the clear plastic visor on the front of the TGA 7 as far as possible and then lift the metal cover that allows access to the balance. The dome-shaped metal cover that protects and isolates the balance mechanism should now be visible.
2. The dome-shaped cover is secured in place by two locking mechanisms, one in front of the balance chamber and one at the rear. Turn both of these locks counterclockwise simultaneously to unlock the cover. Lift the cover straight up and off to expose the balance mechanism.
3. The TGA 7 high temperature sample thermocouple passes through a hole on the left side of the balance mechanism and down through the baffle assembly to rest near the sample pan. The thermocouple has two wires and a plug on its upper end. These are plugged into a connector behind the balance mechanism. Unplug the sample thermocouple from the point where it is connected behind the balance mechanism.
4. Loosen (but do not remove) the screw that secures the sample thermocouple clip. Swing the clip to the side.
5. Pull the thermocouple straight up from the top of the TGA 7 to remove it.

Replacing the High Temperature Furnace Sample Thermocouple

1. Feed the new thermocouple through the hole on the left side of the balance and down through the thermocouple holes in the side of the baffle assembly. The ceramic stop on the end of the thermocouple should be flush on the base of the balance support.
2. Secure the thermocouple into position by swinging the sample thermocouple clip over and tightening the screw to lock the clip in place.
3. Plug the thermocouple into the connector at the back of the balance mechanism.
4. Replace the dome-shaped metal balance cover and lock it into place by turning the two locking mechanisms clockwise simultaneously.

High Temperature Furnace Hangdown Wire Maintenance

The TGA 7 with a high temperature furnace uses a platinum hangdown wire with a platinum stirrup on the end of it. (The standard furnace TGA 7 uses a nichrome hangdown wire.) This stirrup supports the sample pan in the furnace. These wires can be purchased in preformed kits (P/N N519-0276). A kit is included with the TGA 7 high temperature furnace option.



The procedures for removing and replacing the hangdown wire are the same as those for the standard furnace hangdown wire.

NOTE: The multimedia presentations are also applicable to hangdown wire removal and replacement for a TGA 7 with a high temperature furnace.

Parts Provided

Your instrument should be one of the following:

	115 V, 60 Hz	230 V, 50 Hz	100 V, 50 Hz
TGA 7 Standard Furnace	N519-0151	N519-0150	N519-0500
TGA 7 High Temperature Furnace	N519-0321	N519-0320	N519-0501

TGA 7 Standard Furnace Spares and Accessories

The following Spares and Accessories Kit is provided with the TGA 7 Standard Furnace:

Spares and Accessories Kit (N519-0349)

Part No.	Description	Quantity
N519-0207	Standard Furnace Spares Kit (packaged in plastic box)	1
Includes:		
N519-0275	Magnet	1
N519-0282	Balance Tare Weight	1
N519-0285	Hangdown Wire Kit (nichrome; 6 preformed, 6	1

	straight)	
N519-0330	Quartz Hangdown Wire Links (10 each)	1
0219-0071	Curie Point Calibration Standards (6)	1
0319-0264	Platinum Pan Kit (4 each)	1
0319-0265	Platinum Stirrup Kit (4 each)	1
0990-8400	Tweezers (nonmagnetic)	1
0990-8397	Calibration Weight, 100 mg, Class M	1
0990-4905	Pinch Clamp, No. 35	1
0990-7222	Hex Wrench, 0.035 in.	1
0990-7236	Hex Wrench, 0.050 in.	1
0990-2031	O-Ring 1 7/8-in. o.d. x 3/16-in. wide (furnace mount, split)	2
0990-2131	O-Ring 1 7/16-in. o.d. x 1/8-in. wide (ball joint)	2
0990-2163	O-Ring 5 7/8-in. o.d. x 1/8-in. wide (under balance cover)	2
0990-2213	O-Ring 1 1/4-in. o.d. x 1/16-in. wide (under ball joint tube holddown plate)	2
0990-2223	O-Ring 1-in. o.d. x 3/22-in. wide (around furnace base)	2
0998-1785	Fuse, 12 A, 250 V (line power)	2
0990-3011	Tube Clamp, 3/8-in. diameter	6

TGA 7 Sample and Balance Purge Kit (N519-0277)

Part No.	Description	Quantity
0154-1498	Type “H” Purge Gas Restrictor	3
0990-3906	Female Connector	2
0250-6483	Teflon Tubing 1/8-in. o.d. x 1/16-in. i.d.	24 ft
0990-8134	Thread Sealant Tape	1

TGA 7 Pneumatic Kit (N519-0278)

Part No.	Description	Quantity
0990-3630	Air Filter	1
0990-3629	Pressure Relief Valve	1
0990-3428	Male Connector	1
0990-3094	Tube Insert	6
0990-3716	Adapter Tube to Pipe	2
0990-3150	Tube Fitting	1
0990-3196	Tube Fitting	1

0990-3715	Branch Tee, Female	1
0990-6515	Tygon Tubing, 1/4-in. o.d. x 1/8-in. i.d.	20 ft
0990-8134	Thread Sealant Tape	1

Miscellaneous

Part No.	Description	Quantity
N519-1352	TGA 7 Dust Cover	1
N519-0203	TGA 7 Standard Furnace Tube	1
0319-0253	Thermocouple for Standard Furnace	1
0319-1628	Standard Platinum Furnace Shield	1
N519-0264	TGA 7 Standard Furnace	1
N519-0171	TGA 7 Standard Furnace Assembly Mount	1

TGA 7 High Temperature Furnace Spares and Accessories

The following Spares and Accessories Kit is provided with the TGA 7 High Temperature Furnace.

Spares and Accessories Kit (N519-0319)

Part No.	Description	Quantity
N519-0323	High Temperature Furnace Spares Kit (packaged in plastic box)	1
Includes:		
N519-0284	Tare Balance Weight (high temperature furnace)	1
N519-0276	Hangdown Wire Kit (platinum, 6 preformed)	1
N519-0330	Quartz Hangdown Wire Links (10 each)	1
N519-0280	Platinum Sample Pan Kit (4 each)	1
N519-0279	Platinum Stirrup Kit (4 each)	1
0990-8400	Tweezers (nonmagnetic)	1
0990-8397	Calibration Weight (100 mg, Class M)	1
0990-4905	Pinch Clamp (No. 35)	1
N519-1356	Sample Thermocouple Clip	1
0990-3012	Hose Clamp	3
0990-8689	Flexible Teflon Tubing	1
0990-7222	Hex Wrench, 0.035 in.	1
0990-7229	Hex Wrench, 7/64 in.	1
0991-0555	Set Screws	3

0990-2131	O-Ring 1 7/16-in. o.d. x 1/8-in. wide (ball joint)	2
0990-2161	O-Ring 1 5/16-in. o.d. x 1/8-in. wide (high temperature furnace tube)	2
0990-2163	O-Ring 5 7/8-in. o.d. x 1/16-in. wide (under ball joint tube hold down plate)	2
0998-1785	Fuse 12 A, 250 V (line power)	2

TGA 7 Sample and Balance Purge Gas Kit (N519-0277)

Part No.	Description	Quantity
0154-1498	Type “H” Purge Gas Restrictor	3
0990-3906	Female Connector	2
0250-6483	Teflon Tubing 1/8-in. o.d. x 1/16-in. i.d.	24 ft
0990-8134	Thread Sealant Tape	1

TGA 7 Pneumatics Kit (N519-0278

Part No.	Description	Quantity
0990-3630	Air Filter	1
0990-3629	Pressure Relief Valve	1
0990-3428	Male Connector	1
0990-3094	Tube Insert	6
0990-3150	Tube Fitting	1
0990-3196	Tube Fitting	1
0990-3716	Adapter Tube to Pipe	2
0990-3715	Branch Tee, Female	1
0250-6515	Tygon Tubing, 1/4-in. o.d. x 1/8-in. i.d.	20 ft
0990-8134	Thread Sealant Tape	1

Miscellaneous

Part No.	Description	Quantity
N519-1352	TGA 7 Dust Cover	1
N519-0172	TGA 7 High Temperature Furnace Assembly	1
N519-9084	TGA 7 High Temperature Furnace Printed Circuit Board (already installed in TGA 7)	1
N519-0267	TGA 7 High Temperature Furnace Baffle Assembly	1
N519-1324	TGA 7 High Temperature Furnace Extension Tube (for anticonvection tube)	1
N519-0197	TGA 7 High Temperature Furnace Sample Thermocouple	1

N519-1356	TGA 7 High Temperature Furnace Sample Thermocouple Clip	1
0993-8676	TGA 7 Users Manual	1

Accessories and Replacement Parts

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Hangdown Wires, Links, Stirrups, and Sample Pans

Part No.	Description	Quantity
N519-0285	Nichrome Hangdown Wire Kit for TGA 7 Standard Furnace	6 preformed straight
N519-0516	Nichrome Hangdown Wire Kit for TGA 7 Standard Furnace	6 preformed
N519-0515	Nichrome Hangdown Wire Kit for TGA 7 Standard Furnace	6 straight
N519-0276	Platinum Hangdown Wire Kit for TGA 7 High Temperature Furnace	6 preformed
N519-0330	Quartz Hangdown Wire Link Kit	10
0319-0265	Platinum Stirrup Kit for TGA 7 Standard Furnace	4
N519-0279	Platinum Stirrup Kit for TGA 7 High Temperature Furnace	4
0319-0264	Platinum Sample Pan Kit for TGA 7 Standard Furnace	4
N519-0280	Platinum Sample Pan Kit for TGA 7 High Temperature Furnace	4
N519-0283	Ceramic Liner Kit for TGA 7 High Temperature Furnace Sample Pans	6 covers 6 liners

Calibration Materials

Part No.	Description
0219-0071	Curie Point Reference Materials (5) for Temperature Calibration

0990-8397	Calibration Weight, 100 mg, Class M
N519-0275	Magnet Assembly for Temperature Calibration
N519-0282	Balance Tare Weight for TGA 7 Standard Furnace
N519-0284	Balance Tare Weight for TGA 7 High Temperature Furnace

Thermocouples, Furnaces, and Furnace Accessories

Part No.	**Description**
0319-0253	Thermocouple for TGA 7 Standard Furnace
N519-0197	Sample Thermocouple for TGA 7 High Temperature Furnace
N519-1356	Sample Thermocouple Clip for High Temperature Furnace
N519-0196	Oven Thermocouple for High Temperature Furnace
N519-1323	Oven Thermocouple Clip for High Temperature Furnace
0419-0166	Ceramic Liner for Oven Thermocouple (6 each)
N519-0264	TGA 7 Standard Furnace
N519-0203	Pyrex™ Furnace Tube for TGA 7 Standard Furnace
0319-1628	Platinum Furnace Shield for TGA 7 Standard Furnace
N519-0171	Furnace Assembly Mount for TGA 7 Standard Furnace
N519-0172	TGA 7 High Temperature Furnace (complete assembly including N519-0202)
N519-0202	Furnace Tube for TGA 7 High Temperature Furnace
N519-0267	Baffle Assembly for TGA 7 High Temperature Furnace
N519-1324	Extension Tube for TGA 7 High Temperature Furnace Anticonvection Tube
0991-0555	Collar Set Screw for Extension Tube (2 required)

Purge Gas Accessories, Pressure Regulators, Flow Meters

Part No.	**Description**
N519-0277	TGA 7 Sample and Balance Purge Kit
Includes:	
0154-1496	Type “A” Purge Gas Restrictor (2)
0990-3906	Female Connector (2)
0250-6483	Teflon Tubing 1/8-in. o.d. x 1/16-in. i.d. (24 ft)
0990-8134	Thread Sealant Tape
0212-1127	Pressure Regulator for Gas Line Use (secondary regulator 0 – 100 psi)
0240-0084	Pressure Regulator for Oxygen (CGA #540)

0240-0085	Pressure Regulator for Helium and Nitrogen (CGA #580)
0290-1624	Float Displacement Flow Meter, 0 – 100 cc/min
0023-0522	Two-Stage, Soap Bubble Gas Flow Meter
0990-8134	Thread Sealant Tape
0154-1496	Type "A" Purge Gas Restrictor (provides a flow of roughly 10 cc/min purge/lb pressure)
0154-1495	Type "H" Purge Gas Restrictor (provides a flow of roughly 1 cc/min purge/lb pressure)

Pneumatic Accessories for Furnace Lift Mechanism

Part No.	Description
N519-0278	TGA 7 Pneumatic Kit
Includes:	
0990-3630	Air Filter
0990-3629	Pressure Relief Valve
0990-3428	Male Connector
0990-3094	Tube Insert (3)
0990-3716	Adapter Tube to Pipe (2)
0990-3715	Branch Tee, Female
0250-6515	Tygon Tubing, 1/4-in. o.d. x 1.8-in. i.d. (20 ft)
0990-8134	Thread Sealant Tape

Purge Lines and Tubing

Part No.	Description
0250-6483	1/8-in. o.d. x 1/16-in. i.d. Teflon Tubing for TGA 7 Sample and Balance Purge Gas Supply (24 ft)
0250-6515	1/4-in. o.d. x 1/8-in. i.d. Tygon Tubing for TGA 7 Pneumatic Furnace Lift Mechanism (20 ft)
0250-6518	Tygon Tubing for Standard Furnace Exhaust Line (6 ft)
0990-8689	Flexible Teflon Tubing for High Temperature Furnace Exhaust Line

O-Rings

Part No.	Description
0990-2031	O-Ring 1 7/8-in. o.d. x 3/16-in. wide (TGA 7 standard furnace mount, split)
0990-2131	O-Ring 1 7/16-in. o.d. x 1/8-in. wide (ball joint)
0990-2163	O-Ring 5 7/8-in. o.d. x 1/8-in. wide (under balance cover)

0990-2213	O-Ring 1 1/4-in. o.d. x 1/16-in. wide (under ball joint tube holddown plate)
0990-2223	O-Ring 1-in. o.d. x 3/32-in. wide (around base of TGA 7 standard furnace)
0990-2161	O-Ring 1 5/16-in. o.d. x 1/8-in. wide (around top of TGA 7 high temperature furnace tube, 2 required)
N519-1204	Silicone Gasket (under TGA 7 standard furnace tube)

Fuses

Part No.	**Description**
0998-1611	Fuse, 1.0 A, 250 V (power supply board, Service Engineer only)
0998-1614	Fuse, 2.0 A, 250 V (power supply board, Service Engineer only)
0998-1785	Fuse, 12 A, 250 V (line power)
0998-1610	Fuse, 0.5 A, 250 V Slow Blow (TAC 7 for 230 V)
0998-1611	Fuse, 1.0 A, 250 V Slow Blow (TAC 7 for 100 and 115 V)
0999-1633	Fuse, 3/16 A, 250 V, Slow Blow (GSA 7 for 220 V)
0998-1619	Fuse, 3/8 A, 250 V Slow Blow (GSA 7 for 100 and 115 V)

Sample Handling Accessories

Part No.	**Description**
0240-1286	Hayman Micro Spatula
0228-5005	Syringe (10 µL)

Miscellaneous

Part No.	**Description**
N519-1352	TGA 7 Dust Cover
0990-4905	Pinch Clamp, No. 35
0990-7222	Hex Wrench, 0.035 in.
0990-7236	Hex Wrench, 0.050 in.
0990-7229	Hex Wrench, 7/64 in.
0990-8400	Tweezers (nonmagnetic)
N519-0282	Balance Tare Weight for TGA 7 Standard Furnace
N519-0284	Balance Tare Weight for TGA 7 High Temperature Furnace
N519-0191	Anticonvection Tube
0319-0274	Anticonvection Tube, Bottom Disk, and Collar
0319-1585	Bottom Disk (Anticonvection Tube)

0991-1908	Collar (Anticonvection Tube)
0991-0555	Set Screw for Disk and Collar (Anticonvection Tube) (2)
N519-1324	Extension Tube for TGA 7 High Temperature Furnace Anticonvection Tube
0991-0555	Set Screw for Extension Tube for High Temperature Furnace (2)
N519-0267	High Temperature Furnace Baffle Assembly
0990-5932	Antistatic Compound, Liquid

Gas Selector Accessory

Part No.	Description
N519-0270	GSA 7 Gas Selector Accessory (115 V, 50/60 Hz)
N519-0271	GSA 7 Gas Selector Accessory (230 V, 50/60 Hz)
N519-0269	GSA 7 Gas Selector Accessory (100 V, 50/60 Hz)

High Temperature Furnace Install Kit (P/N N519-0510)

The High Temperature Furnace Install Kit has been designed to add high temperature capability to a TGA 7 with a standard furnace. This kit includes the following items:

Part No.	Description
N519-0172	TGA 7 High Temperature Furnace
N519-0323	High Temperature Furnace Spares Kit (packaged in plastic box)
N519-9084	High Temperature Furnace Printed Circuit Board (requires service installation)
N519-0341	High Temperature Furnace Baffle Install Kit

Standard Furnace Install Kit (P/N N519-0511)

The Standard Furnace Install Kit has been designed to add standard furnace capability to a TGA 7 with a high temperature furnace. This kit includes the following items:

Part No.	Description
N519-0264	TGA 7 Standard Furnace
0319-1628	Platinum Shield for TGA 7 Standard Furnace
0319-0253	Thermocouple for TGA 7 Standard Furnace
N519-0203	Furnace Tube for TGA 7 Standard Furnace
N519-0171	TGA 7 Standard Furnace Assembly Mount
N519-0309	Anticonvection Tube, Bottom Disk, and Collar
0991-0555	Set Screw for Anticonvection Disk (3)
N519-0207	Standard Furnace Spares Kit (packaged in plastic box)

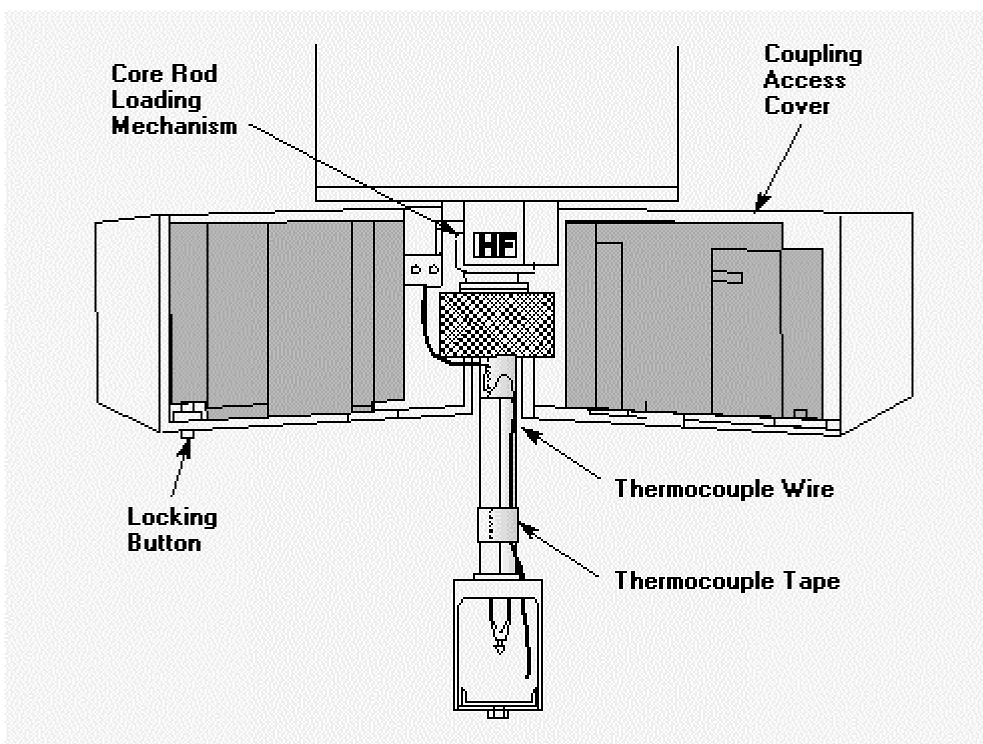
Chapter 7
DMA 7/7e Analyzer

Overview

NOTE: Any reference made to the DMA 7e applies equally to the DMA 7 unless specifically stated.

NOTE: Analyzers shipped prior to July 1, 1993, are considered DMA 7. If your instrument is a DMA 7e, the casting of the core rod locking mechanism is labeled “HF” (see below).

View of DMA 7e Showing HF Label



The DMA 7e Dynamic Mechanical Analyzer is used for mechanical analysis of solid and near-solid samples. It measures mechanical properties such as modulus (elasticity) and viscosity (damping) as a function of time, temperature, frequency, stress, or combinations of these parameters. The DMA 7e provides the performance and flexibility necessary for the characterization of a broad range of materials, from soft samples such as elastomers, thin films, and single filament fibers to hard samples like composites, ceramics, and some metals. The DMA 7e is also used in the food industry. Its patented temperature and stress control and enhanced sensitivity provide new capabilities for accurate material characterization. The DMA 7e can also apply a constant force to perform standard thermomechanical analysis (TMA).

The DMA 7e is controlled by Pyris Software for Windows which is on the computer to which the DMA 7e is attached via a TAC 7/DX Thermal Analysis Instrument Controller which controls the analyzer and digitizes the analog output from the detector before sending it on to the computer. Comprehensive calibration, verification, and validation assure the highest confidence in results reported by the DMA 7e. It has been certified to perform standard test methods defined by international organizations including ASTM and ISO.

Typical applications for which the DMA 7e is used are

Polymers

- Stiffness/Modulus
- Quality factor/tan delta
- Glass transition
- Impact performance
- Effects of filler, modifiers, blending, grafting, and copolymerization

Fiber/Film

- Shrinkage
- Heat set
- Orientation
- Strength

Food/Pharmaceutical

- Effect of drying
- Effect of additives
- Effect of heating

Biological/Environmental

- Effect of chemicals, solvents, or humidity
- Effect of aging, drying, or sunlight
- Effect of stress or pressure

The DMA 7e is composed of four functional components:

motor

detector

measuring system

environmental system

Motor

The motor is a linear force motor that can produce 8,500 mN downward force and 6,500 mN upward force. Dynamic force can be applied at frequencies from 0.01 to 51 Hz. The force is applied to the sample through the central core rod which is suspended in a magnetic field. The force motor supports the weight of the probe and core rod and applies the force to the sample. The vertical design of the DMA 7e permits accurate control of the moving components and a very low compliance frame for applying force. This design also uses gravity to help in mounting samples.

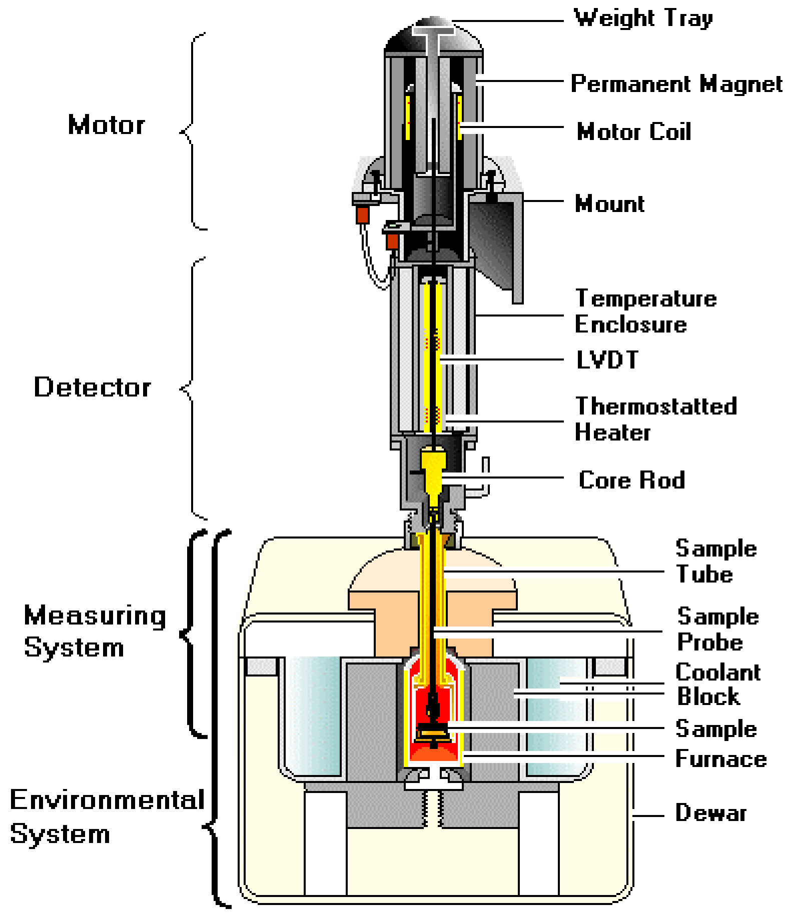
Detector

The detector, a linear variable differential transducer (LVDT), measures the vertical deflection of the sample. The magnetic core of the LVDT maintains a constant horizontal distance from the coils through the 25-mm range. The LVDT provides high sensitivity and repeatability for accurate measurements as well as a broad dynamic range to accommodate a variety of sample sizes and geometries.

Environmental System

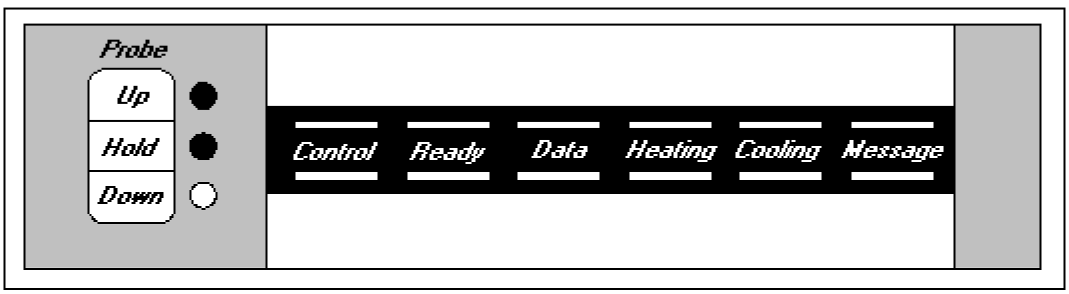
The environmental system includes the purge gas, cooling device, and furnace. The purge gas is supplied through the top of the measuring system and provides uniform atmosphere for the sample. A number of cooling devices can be connected to the heat sink that surrounds the furnace. The heat sink is maintained at a relatively constant temperature and provides a uniform thermal environment for the furnace to heat or cool the sample. Two types of low-mass furnace are available for use with the DMA 7e and each can be programmed for high and low temperatures. The low-mass furnace design permits a rapid cool-down at the end of the experiment, often requiring only a few minutes to cool to the starting temperature. The DMA 7e allows you to analyze materials during cooling as well as heating experiments. The furnace is closely coupled to the sample to enable precise temperature control.

Cross-Sectional View of DMA 7e



Operating Controls

Another important part of the DMA 7e is the front panel. It contains three probe control keys and six status indicators.



A light next to each probe control key is illuminated when that key is selected.

Control

The DMA 7e temperature sensors are in control of the temperature; power is being supplied to the furnace to maintain the program temperature selected in Pyris. The Control light should always be lit in conjunction with the Heating or Cooling and/or Data control lights during the main section of a temperature program run.

Ready

A blinking Ready light means that the program temperature has reached the user-defined load temperature. When the Ready and Control lights are both lit (nonblinking), the system is ready to begin a temperature program. Upon system startup, the Ready indicator should be the only indicator that is lit. In some cases the Control indicator may also be illuminated on startup depending on the heat sink and coolant temperature.

Data

If the Data indicator is illuminated, data are being collected. The Data indicator is off at all other times.

Heating

If the Heating indicator is illuminated, the DMA 7e is heating under program control.

Cooling

If the Cooling indicator is illuminated, the DMA 7e is cooling under program control.

Message

If the Message light is blinking, there is a message to be read at the computer. A blinking Message light may also mean that the measuring system and sample may be stuck if the sample has melted and fused to the probe tip. If this should occur, detach the probe tip from the probe holder (for a stainless steel measuring system) and remove the sample from the probe tip outside of the analyzer.

Probe Up

This button causes the probe assembly to rise and lock into place. Selecting Probe Up with a sample in place engages the extension analysis accessory and applies the desired stress to the sample while using the Extension Analysis measuring system.

Probe Hold

Pressing this button holds the probe assembly at its current location between the up and down positions.

Probe Down

Selecting this button causes the probe assembly to lower to the base of the sample platform (or on the sample currently resting on the sample platform). The weight of the core rod plus the measuring system will be measured by the DMA 7e each time the probe is lowered (except when using the Extension Analysis measuring system). This allows accurate measurement of the system inertia.

Status Indicators

The current status of the DMA 7e is displayed in the Status Panel in the Pyris Software for Windows screen. You can also see the status of the instrument by looking at the front panel.

Summary of the DMA 7e Status Indicators

Indicator	Blinking	On	Off
Control	Does not blink	Power is being supplied to the furnace	Analyzer is not in temperature control Power is not being supplied to the furnace
Ready	Program temperature has reached Load Temperature	Analyzer temperature has reached Load Temperature Power is being supplied to DMA 7e	Analyzer temperature has not reached Load Temperature Power is not being supplied to DMA 7e
Data	Does not blink	Data are being taken from the DMA 7e	Data are not being taken from the DMA 7e
Heating	Does not blink	Analyzer is heating under program control at the selected rate	Analyzer is not heating
Cooling	Does not blink	Analyzer is cooling under program control at the selected cooling rate	Analyzer is not cooling
Message	A message has been sent to the computer		No message is waiting

NOTES: If the indicators are blinking in sequence, the DMA 7e is calibrating. **Do not touch the front panel.**

The Control indicator may go out momentarily at the beginning of a run.

The temperature at which the Control indicator lights and, therefore, the acceptable temperature range of the DMA 7e, is dependent on the type of coolant or cooling accessory that you use. For example, with an ice water bath in the DMA 7e dewar, temperatures as low as 20°C can be attained. With liquid nitrogen as the coolant, temperatures as low as –170°C can be attained.

The status indicators change during **start up** of the DMA 7e system.

The status indicators can be used for **diagnostic troubleshooting**.

Start Up a DMA 7e System

Your DMA 7e Thermal Analysis System should be started up as follows:

1. Turn on the computer and access Pyris Software for Windows program group.
2. Turn on all of the analyzers.

   The **Ready, Heating, Cooling,** and **Message** status indicators on the front of the analyzers should be illuminated.
3. Turn on and adjust all of the purge gases and pneumatic pressure gases (or check them if they are already on).

4. Turn on the TAC 7/DX Instrument Controller for each analyzer.

   NOTE: If the TAC 7/DX is already on, turn it off, wait 5 – 10 seconds, and then turn it back on.

   All of the status indicators on the front of the analyzer should go out for a few seconds. Then the **Ready** indicator should illuminate. Depending on the cooling accessory or coolant in use, the **Control** indicator may also illuminate or flash.

   NOTE: If any other status indicators are illuminated at this point, turn off the TAC 7/DX and then the analyzer module. Wait 5 to 10 seconds. Turn on the analyzer and then the TAC 7/DX.

5. Turn on the power to the printer.
6. Turn on the power to the GSA 7 or the TAGS, if these units will be used with your system.
7. Select the Pyris Manager or the Pyris Data Analysis icon to start the Pyris Software for Windows software.

Diagnostic Troubleshooting

When a problem with the DMA 7e is encountered by the system, it is indicated by the status indicators on the front panel. The problem can be diagnosed and corrected quickly by observing which status indicators are illuminated. The table below is a diagnostic troubleshooting guide for the DMA 7e.

Control	Ready	Data	Heating	Cooling	Message	Status
On	On					Analyzer is ready and in temperature control.
On	On	On	On/Off	On/Off		Analyzer is storing data in the TAC 7/DX.
On	On		On			Analyzer is heating at a constant rate.
On	On			On		Analyzer is cooling at a constant rate.
On/Off	On				Blinking	Movement of the probe is restricted or the probe is stuck. Remove obstruction or free up probe.
					Blinking	Analyzer is in its initialization sequence. Do not disturb the analyzer.
		Blinking			On	Error Number U32. Contact Service Rep.
		Blinking		On		Error Number U33. Contact Service Rep.
		Blinking		On	On	Error Number U34. Contact Service Rep.
			Blinking	On		Error Number U35. Contact Service Rep.
				On	Blinking	Error Number U36. Contact Service Rep.
			Blinking		On	Error Number U6. Contact Service Rep.
			Blinking	On		Error Number U7. Contact Service Rep.
			Blinking	On	On	Error Number U8. Contact Service Rep.
		On	Blinking			Error Number U9. Contact Service Rep.
		On	Blinking		On	Error Number U10. Contact Service Rep.

			Blinking	Blinking		TAC 7/DX did not recognize DMA 7e. Cable N519-0310 connecting TAC to DMA is loose or has failed.
		Blinking			Blinking	TAC failure. Contact Service Rep.
	Blinking				On	Error Number U69. Contact Service Rep.
	Blinking			On		Error Number U70. Contact Service Rep.

The DMA 7e is presented in the following topics:

- **Features**
- **Safety Precautions**
- **Optimum Performance**
- **Calibration**
- **Measuring Systems**
- **Types of Test Methods**
- **Operational Guidelines**
- **Subambient Operation**
- **Maintenance**
- **Part Numbers**
- **Glossary**

Features of the DMA 7/7e

To get an idea of the versatility and range of features of the DMA 7e, check each of the features below:

- **Force Control**
- **Temperature Control**
- **Displacement Sensitivity**
- **First Principle Sample Mounting**
- **Multiple Sample Types and Geometries**
- **Broad Modulus Range**
- **Broad Frequency Range**
- **Furnace Systems**
- **Worldwide Standard Tests**

Force Control

Force motors are used to cause the sample to deflect. Precise force control is critical to obtaining accurate modulus results. The DMA 7e force motor can accurately control from very low forces for the analysis of samples in the molten or flowing state to high forces for the analysis of very high modulus solids. Forces can be calibrated and verified using traceable mass standards providing traceable modulus results.

Temperature Control

Mechanical properties of polymers are thermally dependent. Precise temperature control is critical to obtaining reproducible mechanical properties. The DMA 7e sample and furnace size has been optimized for precise temperature control. The low mass furnace gives low thermal lags for quicker control. Close coupling of the furnace to the sample gives faster heating rates. Furnace and cooling device options provide a wide range of thermal performances and ranges to suit your application or laboratory requirements.

Displacement Sensitivity

Displacement sensors are used to measure sample deflections. High displacement sensitivity is important for characterizing a wide variety of sample types from polymer pellets to single filament fibers and thin films. High dynamic displacement sensitivity allows measurement of subtle mechanical transitions. Long static displacement range allows the sample to expand or contract many times its original size, up to 300%.

First Principle Sample Mounting

Measuring systems hold the sample in place for analysis. Sample mounting must apply simple, first principle deformations to produce fundamental, easy-to-understand results. Measuring systems are available for tensile, flexural, and compressive modulus determinations for a wide range of sample types, shapes, and sizes including squares, rods, cylinders, rectangles, disks, cubes, fluids, films, and fibers.

Sample Types and Geometries

The practical advantages of the DMA technique have expanded to almost every material and sample type. It is not uncommon for a single laboratory to test raw materials, intermediate products, and finished products to verify materials and end-use performance. To accommodate this broad range of sample types and test geometries, numerous new measuring systems have been developed for the DMA 7e. These systems are designed to handle materials in a variety of geometries, from semisolids in the form of flat bars, pellets, cylinders, disks, films, and even fibers. Included are Extension Analysis systems for the analysis of thin films and fibers; 3-Point Bending, Dual Cantilever, and Single Cantilever systems for the analysis of a variety of thermoplastics and thermosets; and nine different Parallel Plate systems.

Modulus Range

The broad modulus range of the DMA 7e provides full characterization capabilities in one instrument. For example, rubber and other elastomers can be completely characterized from subambient temperatures to above the glass transition in a single experiment with a single instrument. Neat resins may be analyzed directly along with intermediate and final products without instrument modifications or changes.

Frequency Range

With a frequency range of 0.01 to 50 Hz, the DMA 7e provides a valuable tool for the characterization or “fingerprinting” of materials. Frequency scanning provides a convenient way to observe differences between materials as a result of long and short chain branching, chain entanglements, and molecular weight distribution differences. Frequency scanning also provides a means for calculating the “zero shear viscosity” or for identifying the Newtonian region in polymers, which may then be applied to molecular weight calculations. Lower frequency data, such as one cycle per day or one cycle per week, is generated through creep recovery tests.

Furnace Systems

Two furnace systems are available for use with the DMA 7e. These systems allow DMA measurements over the range of –170°C to 1000°C. Based on a resistance-heating design, these furnace systems allow continuous monitoring and temperature control, resulting in improved accuracy and reproducibility. Low-mass design permits rapid cool-down at the completion of an experiment, often requiring only a few minutes to cool back to the starting temperature. Fast-cooling experiments are best performed with the DMA 7e’s unique quartz measuring systems. The result is that you can run more samples and characterize more materials. The precise temperature control features of this furnace allow heating and cooling rates from 0.1°C/min to 100°C/min.

Standard Tests

Worldwide standardization is increasing. The DMA 7e can be programmed to perform standard test procedures defined by ASTM, ANSI, ISO, BSI, JIS, and others. The DMA 7e can determine the flexural modulus, compressive modulus, tensile modulus, DTUL, CLTE, Melt characterization, Cure characterization, and many other standard test methods.

Some of the tests are

ASTM D4065, ISO, JIS	Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics
ASTM D790, D4476, D5023, ISO 178, JIS	Test Method for Flexural Properties of Plastics
DTUL (see ASTM D648)	Test Method for Deflection Temperature of Plastics under Flexural Load
See ASTM D1525	Test Method for Vicat Softening Temperature
See ASTM D638, D5026, D1708, D882, and D2923	Test Method for Tensile Properties of Plastics
See ASTM D695 and D5024	Test Method for Compressive Properties of Rigid Plastics
See ASTM D746	Test Method for Brittleness Temperature of Plastics and Elastomers by Impact
See ASTM D4440	Practice for Rheological Measurement of Polymer Melts Using Dynamic Mechanical Procedures
See ASTM D1790	Impact
See ASTM D4473, D2765, D2471	Practice for Measuring the Cure Behavior of Thermosetting Resins Using Dynamic Mechanical Properties
See ASTM D696, D1042, D1204, D2732, D2838	Test Method for Coefficient of Linear Thermal Expansion of Plastics
See ASTM D747, D4812, D5418	Test Method for Apparent Bending Modulus of Plastics by Means of a Cantilever Beam
See ASTM D2990	Test Method for Tensile, Compressive, and Flexural Creep and Creep Rupture of Plastics

Safety Precautions

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

The following precautions must be observed when using the DMA 7e Dynamic Mechanical Analyzer:

- Never turn off the power to the computer until the appropriate shutdown message appears.
- Never remove the outer instrument cover or side panels on the DMA 7e without turning the instrument off and disconnecting its power cord from the line power.
- Always observe the precautions indicated for the use of the various cooling devices.
- Only high-quality purge gases should be used with the DMA 7e. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory, such as the **Drierite Compressed Air Filter Dryer**, is recommended for the removal of any moisture from the purge gases.
- Always observe the startup and shutdown procedures with the DMA 7e and all related instruments.
- The DMA 7e will operate best when using helium as the purge gas.
- Prior to performing any experiment or calibration, be sure that the thermocouple currently installed in the DMA 7e is functioning properly. The thermocouple can easily be checked by programming the DMA 7e to a temperature of 100°C and checking the temperature displayed in the status window. (The temperature should read a value close to 100°C if the instrument is calibrated.)
- Make sure the furnace is programmed to between 20°C and 40°C between runs and if you leave the DMA 7e unattended for a period of time when using a low-temperature cooling accessory (below –20°C).
- If you are using an Intracooler or liquid nitrogen as a cooling device, raise the furnace temperature to 150°C before shutting down the cooling device when finished with normal operation. Leave the DMA 7e on and the furnace at 150°C for at least 2 hours or overnight. This will expel any moisture and prevent it from refreezing and damaging the furnace.

In addition to these precautions, follow the points in **Optimum Thermal Performance** of the DMA 7e to get the most out of your instrument:

Optimum Thermal Performance

The following are hints to follow to obtain the optimum in performance from your DMA 7e:

1. Be sure the control light is lit before beginning a run.
2. Check your purge gas regularly. Variations in the purge rate can cause nonuniformities in the furnace temperature.
3. Use helium for the purge gas whenever possible. Helium provides optimum thermal conductivity at low, moderate, and high temperatures. Nitrogen should be used when operating to very high temperatures (i.e., >500°C).
4. Heat at reasonable heating rates. Heating rates are typically 10°C/min for TMA (i.e., when the DMA 7e is using a quartz measuring system and a small furnace) and 5°C/min for the DMA 7e (i.e., when using the stainless steel measuring system and large furnace).
5. Avoid bending or kinking the thermocouple during installation or use.

6. Do not move the thermocouple after you have calibrated the instrument.
7. When installing a new furnace or changing the furnace, use heat sink compound. Apply to the furnace mounting area and the mounting block and around the furnace leads at the heat sink and the base of the heat sink.
8. Avoid overfilling the dewar because liquid could come in contact with the furnace. This situation could also occur if liquid seeps between the walls of the dewar liner and the dewar insulation. If liquid enters the furnace, it can cause premature failure or damage.

Calibration

All calibrations of the DMA 7e are performed by a Perkin Elmer Service Engineer upon installation so it will not be necessary for you to perform a complete calibration. A periodic check of the calibration is all that is needed. The DMA 7e's calibration should remain unchanged for an extended period of time provided there are no changes in operating conditions. Even when the instrument is turned off, the calibration values are stored so that the next time the instrument is turned on, it will still be calibrated. Some of the changing operating conditions that could affect either the temperature or the eigendeformation calibration of the DMA 7e are as follows:

- If the operating range of your experiments changes, temperature calibration may be necessary. Check the temperature calibration in the range of interest (to determine if the current calibration is still valid) by measuring the melting point of a reference material.
- If the purge gas or purge gas flow rate is changed, the temperature calibration should be checked.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new thermocouple is installed or if the position of the thermocouple is changed, the temperature calibration should be checked.
- The eigendeformation calibration should be performed if the sample tube or probe is changed and if the samples being analyzed are very stiff (i.e., they have a modulus of greater than 10 GPa).
- If a different measuring system is installed, the temperature and other calibrations should be checked.

There are six calibration routines used to calibrate the DMA 7e:

DMA Calibration	This initial calibration must be completed before any other calibration is performed.
Height Calibration	This calibrates the displacement transducer that is used to measure the position and amplitude of samples.
Force Calibration	This calibrates the force motor that is used to apply the static and dynamic forces to the sample.
Eigendeformation Calibration	This calibrates the very small movement of the analyzer itself when large forces are applied. After performing the calibration, the compliance of the system is subtracted from the probe position signal. Therefore, increasing force with no sample present will yield zero displacement. Perform the Eigendeformation calibration after Height and Force calibrations.

Temperature Calibration	This calibration allows you to run one or more reference materials to match thermocouple temperature and sample temperature.
Furnace Calibration	This is a nine-point temperature calibration between operator-selected upper and lower temperature limits. The thermocouple temperature will be matched to the programmed furnace temperature when this calibration is complete.

Perkin Elmer ships reference materials and standards in the Spares Kit and the Accessories Kit for the DMA 7e. Additional standards and reference materials are available through PE XPRESS.

For a summary of the steps involved in calibrating the DMA 7e, see **Calibrating the DMA 7e.**

DMA Calibration

DMA calibration performs necessary operations in the firmware and generates look-up tables that are stored in memory. Perform a DMA calibration as follows:

1. Install the desired measuring system as described in the instructions provided with the system.

   See the topic **Installing the 3-Point Bending Measuring System** in Pyris Multimedia Presentations Help.

   NOTE: For other measuring systems such as extension, you need to change only the probe tip and platform. In other cases, you need to change the sample tube as well.

   See the topic **Removing the Sample Tube and Probe** under Maintenance in Pyris Multimedia Presentations Help.

   See the topic **Removing the Probe Tip** under Maintenance in Pyris Multimedia Presentations Help.

   CAUTION: Do not attempt to perform any calibration before the DMA 7e has been on for at least 40 minutes to allow the LVDT temperature to stabilize.

2. Restore DMA Calibration values by first selecting Calibrate from the View menu and then **DMA Calibration** from the Restore menu.
3. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
4. Select the **DMA** tab in the Calibration window.
5. Enter your name in the Operator field and click on **Begin Calibration** to initiate the DMA calibration.
6. Remove any samples from the analyzer.
7. Lower the probe by pressing the **Probe Down** button on the front panel of the DMA 7e.
8. Press the furnace locking mechanism and raise the furnace assembly. Guide the furnace with your hand to make sure that it does not bump the probe assembly. Make sure that the furnace assembly locks in place.
9. Click on **OK** in the dialog box. When calibration begins, a dialog box that indicates the progress of the calibration by showing the elapsed time is seen. The status indicators on the instrument will flash in sequence during the calibration.
10. When the calibration is finished, click on **OK** to clear the dialog box.

11. Select **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.

You can now proceed to the next calibration procedure: Height Calibration.

If the initial DMA calibration fails, an error message and error code indicating the failure appears. Report the code letter or number to your Perkin Elmer Service Representative.

Height Calibration

To perform a Height Calibration, follow the procedure below:

NOTE: A sapphire height displacement standard (P/N N539-1051) is provided in the DMA 7e Accessories Kit. It is suitable for height calibration over the full range of the system. However, you can use other materials of known height if you wish. These materials should be very hard (i.e., have a high modulus) and should have flat, parallel surfaces. Use a micrometer (P/N 0990-7281) to measure the height of the displacement standard. The measured height is used in the procedure that follows.

1. Restore the Height calibration values by first selecting **Calibrate** from the View menu and then **Height** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Height** tab in the Calibration window.
4. Enter your name in the Operator field and the height of the displacement standard that you are using in the Ref. Height field. (The current calibration reference material height value is displayed in the Measured field.) Click on **Begin Calibration**.
5. The Height Calibration dialog box displayed instructs you to prepare the analyzer for the zero reading. Carefully lower the furnace assembly by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
6. Press the **Probe Up** button on the front panel of the instrument to raise the probe.
7. Make sure there are no samples on the sample platform.
8. Press the **Probe Down** button on the front panel of the instrument to lower the probe so that it rests on the empty sample platform. Click on **OK** to clear the dialog box. It will take from 20 to 30 seconds for the Y signal value to be displayed in the Read Zero dialog box once the probe tip rests on the platform.
9. When the Y signal stabilizes, select **OK** if you want the current Y value entered as the new zero value. The Y signal will automatically set to zero. The calibration sets the location of the empty sample platform as 0 mm.
10. The next dialog box gives you further instructions. Press the **Probe Up** button on the front panel of the analyzer. Place the sapphire height displacement standard on the sample platform. Press the **Probe Down** button to lower the probe so that it rests on the displacement standard. Click on **OK** to clear the dialog box.
11. It will take 20 – 30 seconds for the Y signal value to be displayed in the Read Value field in the next dialog box. When the Y signal stabilizes, click on **OK** to enter this value as the height calibration value and clear the dialog box.
12. Click on **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.

The Height Calibration is now complete.

13. Press the **Probe Up** button on the front panel and remove the displacement standard.

You can now perform the next calibration, **Force Calibration.**

Force Calibration

NOTE: Locate the 50-g-force calibration weight (P/N 0990-8825) and weight platform (P/N N519-0392) provided in the DMA 7e Accessories Kit.

To perform a Force Calibration:

1. Restore the Force default calibration values by first selecting Calibrate from the View menu and then **Force** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Force** tab in the Calibration window.
4. Enter your name in the Operator field.
5. Click on **Begin Calibration**.
6. Remove the dust cover, remove any sample from the sample platform, install the weight tray in the analyzer, and lower the probe. Click on **OK**. The next dialog box tells you to place the standard 50-g weight onto the tray. After a short time, the calibration will be finished.
7. Remove the 50-g weight and the weight tray from the analyzer at the same time. Replace the dust cover.

The force calibration value is automatically saved to the instrument.

You can now proceed to perform the **Eigendeformation Calibration**.

Eigendeformation Calibration

NOTES: A 9.5-mm (0.375-in.) steel eigendeformation calibration cylinder (P/N N519-1469) is provided in the DMA 7e Accessories Kit and must be used for the second part of the Eigendeformation Calibration.

You **must** perform a Height Calibration and a Force Calibration before proceeding to an Eigendeformation Calibration.

Perform an Eigendeformation Calibration as follows:

1. Restore the Eigendeformation default calibration values by first selecting Calibrate from the View menu and then **Eigendeformation** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Eigendeformation** tab in the Calibration window.
4. Enter your name in the Operator field and click on Begin Calibration.
5. Remove any sample from the platform, lower the probe by pressing the **Probe Down** button on the front panel of the DMA 7e, wait 1 minute, and then proceed.

6. Raise the probe by pressing the **Probe Up** button on the instrument, insert the steel reference material, and then lower the probe by pressing the **Probe Down** button.
7. After a short time, the calibration is completed. Raise the probe by pressing the **Probe Up** button on the analyzer and remove the steel reference material.
8. Click on **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.

You can now proceed to perform the **Temperature Calibration.**

Temperature Calibration

To perform a Temperature Calibration using indium as the standard, perform the following steps:

1. **Set up a run and collect data for indium.**
2. **Perform an onset calculation.**
3. **Perform the calibration.**

In this calibration procedure, the onset of melting temperature for indium is determined using the 3-Point Bending measuring system.

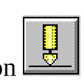
Indium Run and Data Collection

For a temperature calibration for the DMA 7e, you must perform an indium run as follows:

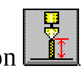
1. Install the large bending platform and the 3-mm sphere probe tip onto the analyzer as described in the Three-Point Bending Measuring System User’s Manual, P/N 0993-8545.

   See the topic **Installing the 3-Point Bending Measuring System** in Pyris Multimedia Presentation Help.
2. While in the Instrument Viewer or the Method Editor, select Calibrate from the View menu.
3. Restore the default temperature calibration values by selecting **Temperature** from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the **All** command.
4. Click on the **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
5. Click on **Close** to close the Calibration window.

NOTE: If the furnace assembly is not already locked in place at the base of the analyzer, carefully lower it by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base.

6. If there are any samples on the sample tube platform, press the **Probe Up** button on the front panel of the analyzer and remove them using the tweezers (P/N 0990-8400) provided with the instrument.
7. Select the calibration method IndCal.dmm provided with Pyris by selecting **Open Method** from the File menu and choosing the method from the dialog box.
8. Press the **Probe Down** button to lower the probe so that it rests on the sample tube platform. The Y signal value will be displayed in the Status Panel; it will stabilize several seconds later.
9. Click on the **Zero Height** button  in the Control Panel.

This zeros the probe for height. The Y value reading displayed in the Status Panel should now be set to 0.000 mm.

10. Press the **Probe Up** button to raise the probe.
11. Using tweezers, prepare a small amount of the indium reference material (P/N 0319-0033) (3 mm square x 0.5 mm thick). Use the tweezers' handle to flatten the indium.
12. Place the indium in the center of a sample pan so that it lies flat. [A kit of sample pans (P/N N539-0129) is in the Spares Kit (P/N N539-0139).] Place a sample lid over the indium reference material. Place the sample pan in the center of the 3-point bending platform. This is for temperature calibration only. This is not the way in which the 3-point bending platform is normally used.
13. Press the **Probe Down** button. Just before the probe tip comes in contact with the sample pan lid, press the **Probe Hold** button.
14. Check that the sample pan and probe are positioned properly. The pan should be in the middle of the platform, and the center of the probe tip should be over the center of the sample pan lid. Reposition the sample pan as needed.
15. Press the **Probe Down** button so that the probe rests on the sample pan. In approximately 30 seconds, the Y signal value will appear in the Status Panel and stabilize several seconds later.
16. Click on the **Read Height** button  on the Control Panel:

    The DMA 7e will read the height of the sample automatically and it will appear in Sample Height entry field in the Method Editor. The reading will be stored in memory.
17. Carefully raise the furnace assembly all the way up, making sure that it locks in place. Place the diffusion cap around the sample tube at the top of the furnace.
18. Select the Instrument Viewer icon from the toolbar to display the graphics screen for your indium run.
19. Enter 100°C in the **Go To Temp** entry field in the Control Panel and click on the **Go To Temp** button.

    NOTE: Do not begin the run until the temperature signal stabilizes in the Status Panel.
20. Click on the **Start** button in the Control Panel to start the run.

    The system begins to collect data immediately. The following status indicators on the front panel of the DMA 7e should be illuminated: Control, Ready, Data, and Heating. These indicators show that the instrument is in temperature control, data is being collected, and the furnace is heating. The Status Panel should indicate that the DMA 7e is heating and Y values should be updating.

Once the data are collected, you should perform an onset calculation of the results.

Onset Calculation for Indium

The data file you just collected should be displayed in the Data Analysis window. Perform an onset calculation on the data you just collected as follows:

1. Select **Onset** from the Calc menu. The Onset Calculation dialog box appears.
2. Enter the Left Limit and the Right Limit. You can also click on the two red **X**'s that appear on the curve and drag them to the desired left and right limits. The Left Limit and the Right Limit fields will be filled in on the dialog box.
3. Click on the **Calculate** button.

The Onset result is used in the temperature calibration. You can now perform the temperature calibration.

Performing a Temperature Calibration

The measured onset value for indium is used to calibrate the temperature of the analyzer. A temperature calibration consists of the following steps:

NOTE: You should have restored the default Temperature calibration value before you performed the indium run.

1. Select **Calibrate** from the View menu.
2. Select the Temperature tab in the Calibration window. Enter your name in the Operator field.
3. Enter the name of the reference material used. (Indium should be displayed as the Ref. Material for Run 1.)
4. Enter the expected onset temperature for the reference material in the Expected Temperature field.
5. In the Measured Temperature field, enter the onset temperature you obtained when you performed the onset calculation.
6. Click on the check box in the Use column.
7. Select **Save and Apply**; select the file to which to save the new calibration values or enter a new file name. This sends the new calibration value to the analyzer and saves the calibration file specified.
8. Select **Close** to exit from the Temperature Calibration box.

Furnace Calibration

NOTE: Perform a Furnace Calibration **after** a Temperature Calibration.

NOTES: Prior to performing the nine-point Furnace Calibration, verify that the sample temperature is at or near the program temperature and that the sample platform is empty. Also, make sure that the current sample temperature is at or below the intended minimum calibration temperature.

You **must** use some form of coolant that will maintain a constant block temperature below 45°C throughout the entire calibration. This can be achieved by using ice water or other coolant in the dewar, or by using a circulator or an Intracooler 1 or 2 as a cooling device.

1. Select **Calibrate** from the View menu.
2. Select the **Furnace** tab in the Calibration window.
3. On the DMA 7e, press the furnace locking mechanism and raise the furnace until it locks in place.
4. Enter your name in the Operator field.
5. In the Minimum field, enter the temperature where the calibration will begin (e.g., 50).
6. In the Maximum field, enter the temperature where the calibration will end (e.g., 250).

NOTES: The difference between the minimum and the maximum temperatures must be greater than 100°C.

Excellent calibration can be achieved for both very low temperatures (down to –170°C) and very high temperatures (up to 500°C) when a circulator or ice in the dewar is used as a constant temperature heat sink and the calibration is performed between these limits.

7. Click on the **Begin Calibration** button.

   The Furnace Calibration is performed automatically. The program and sample temperature values are matched at nine different points over the temperature range selected. When the calibration is complete, the furnace automatically cools to 30°C.

8. To accept the Furnace Calibration values, click on **Save and Apply**. Select an appropriate filename in which to save the calibration.

9. Click on **Close** to exit from the Furnace Calibration window.

Upon completion of the Furnace Calibration, the analyzer is fully calibrated.

Calibration Steps for the DMA 7e

A typical DMA 7e calibration consists of the following steps:

1. **Restore the default calibration values.**
2. **Perform a DMA Calibration.**
3. **Perform a Height Calibration.**
4. **Perform a Force Calibration.**
5. **Perform an Eigendeformation Calibration.**
6. **Perform a Temperature Calibration using indium as the reference material.**
7. **Perform a Furnace Calibration.**

NOTE: Be sure to perform the calibration procedures in this order.

Restore Default Calibration Values

You can restore the default calibration values for the analyzer by performing the following steps:

1. Select **Calibrate** from the View menu.
2. Click on the **Restore** option on the menu bar to display the Restore menu.

   Select **Height** to restore the default height calibration values.

   Select **Temperature** to restore the default temperature **and** furnace calibration values.

   Select **Force** to restore the default force calibration values.

   Select **Eigendef** to restore the default eigendeformation calibration values.

   Select **All** to restore all of the default calibration values.

In a typical DMA 7e calibration, the next step is **DMA Calibration.**

DMA 7/7e Measuring Systems

The measuring system comprises a central core rod and probe. The central core rod runs the length of the analyzer and is the device through which all stresses are applied to the sample. Measuring systems and probes are attached at the lower end of the core rod through the use of a "quick connect" fixture. This fixture allows the rapid installation or removal of the measuring systems. The measuring system holds the sample in place for testing and can accommodate a number of sample geometries and types.

A wide variety of stainless steel (gold-colored coating) and quartz measuring systems are available. Each measuring system is easily installed and removed to facilitate changing and cleaning the system outside of the analyzer.

The following measuring systems are available for the DMA 7e:

- **3-Point Bending Measuring System**
- **Extension Analysis Measuring System**
- **Parallel Plate Measuring System**
- **Dual Cantilever Measuring System**
- **Single Cantilever Measuring System**

Three-Point Bending Measuring System

The 3-point bending measuring system is used to perform many of the classical mechanical testing applications. It is typically used with high-modulus materials such as engineering thermoplastics, resins, and composites. Sample geometries include solid materials such as sheet stock material, as well as wire, rods, and tubes.

Three different 3-point bending measuring system kits are available:

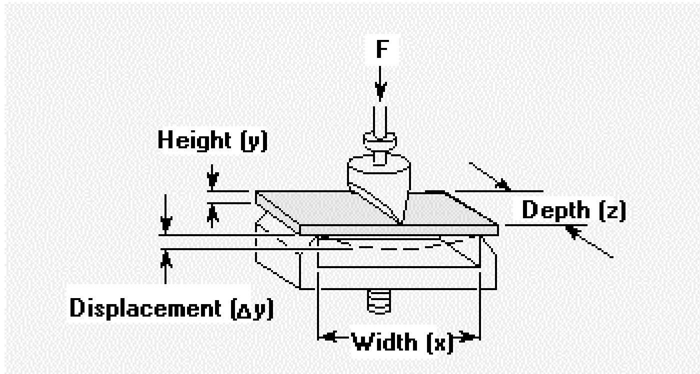
The Stainless Steel 3-Point Bending Kit (P/N N539-0130) is provided with the DMA 7e analyzer. The kit contains a 3-mm-sphere probe tip, 5- and 10-mm knife probe tips, and 5-, 15-, and 20-mm bending platforms. An optional 10-mm bending platform is also available.

The Quartz Small Bending Kit (P/N N539-0137) and the Quartz Large Bending Kit (P/N N539-0136) have a 1-mm round probe and 3- and 5-mm knife probes. The Small Quartz Bending Kit is used with analyzers that have a high-temperature DMA 7e furnace. The Large Quartz Bending Kit is used with analyzers that have a standard DMA 7e furnace.

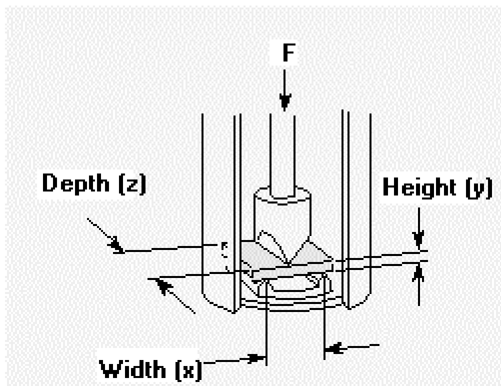
See the three systems below.

See the topic **Installing the 3-Point Bending Measuring System** in Pyris Multimedia Presentations Help to view the procedure for installing the 3-Point Bending Kit. (The same procedure can be used for the quartz kit as well as the stainless steel kit.)

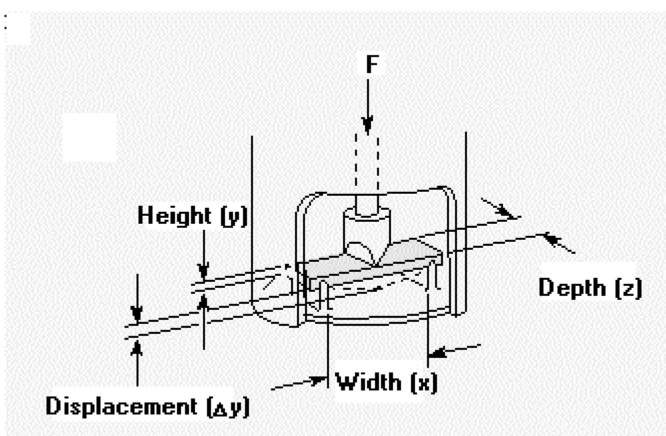
Three-Point Bending Measuring Systems



Stainless Steel



Quartz Small Bending



Quartz Large Bending

Extension Analysis Measuring System

The extension analysis measuring systems are used to study low-modulus materials such as thin films, textile, and other fibers and hair. There are two types of extension measuring systems. The Parallel Fixture provides flat parallel clamping surfaces for analyzing films and thin materials. The Tangential Fixture provides tangential clamping surfaces for handling fibers and wire.

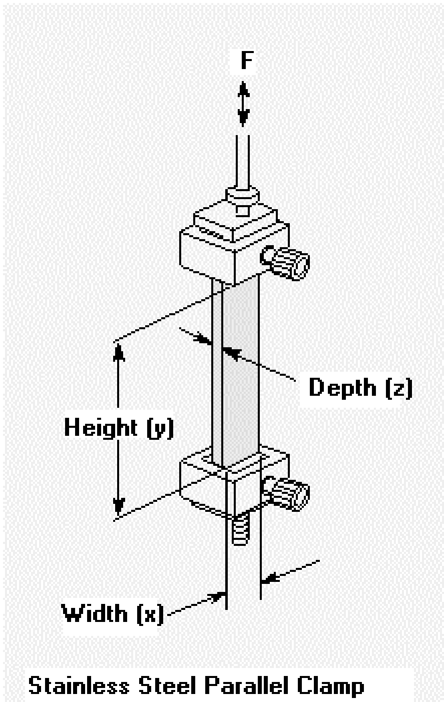
The Stainless Steel Extension Kit (P/N N539-0132) contains two clamping fixtures: a Parallel and a Tangential.

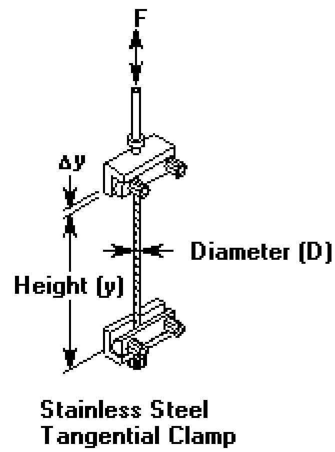
The Quartz Extension Kit (P/N N539-0134) has a clamp assembly with top and bottom clamps for samples such as films that require flat, parallel clamping surfaces. Fiber pins are also provided and are recommended for very thin samples.

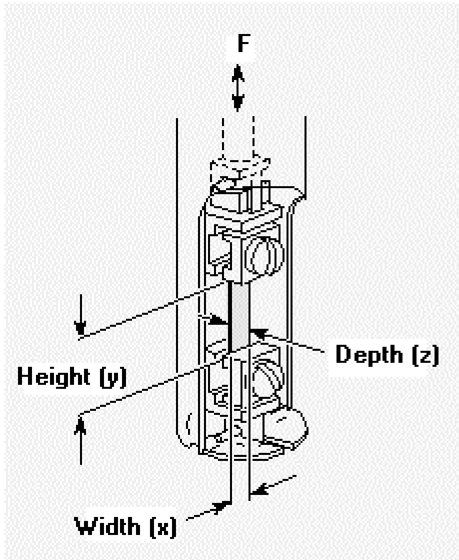
See the Extension Analysis measuring systems below.

See the topic **Mounting Samples on an Extension Measuring System** in Pyris Multimedia Presentations Help.

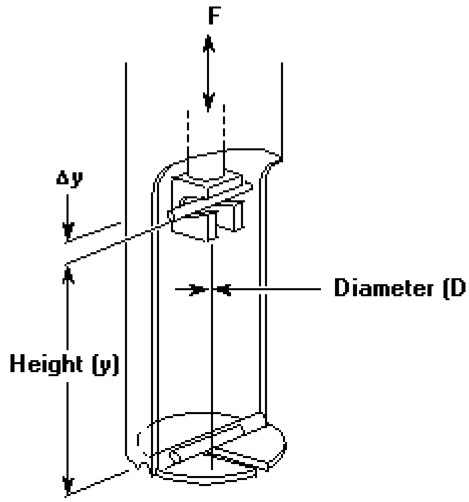
Four Extension Analysis Measuring Systems







Quartz Film Clamp



Quartz Fiber Pin Clamp

Parallel Plate Measuring System

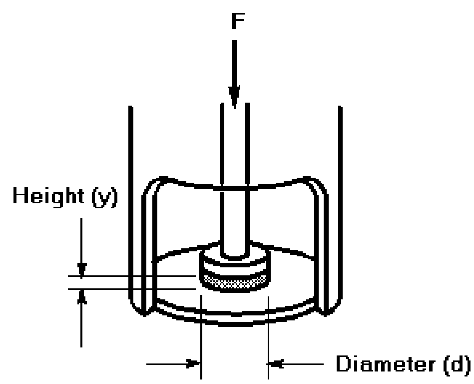
The parallel plate measuring systems are used to study materials ranging from those with honeylike consistency to thermoplastics and rubbers above their glass transition temperature. Three different Parallel Plate Measuring System Kits are available:

The Stainless Steel Parallel Plate Kit (P/N N519-0133) has stainless steel parallel plates with diameters of 1, 3, 5, 10, 15, and 20 mm. With unique mounting fixtures, the parallel plates can be easily aligned to provide exact positional location for the most accurate measurements.

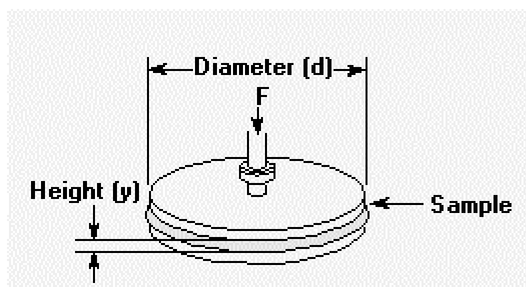
The Quartz Large Parallel Plate Kit (P/N N519-0135) and the Quartz Small Parallel Plate Kit (P/N N519-0138) have quartz probe plates with diameters of 1, 3, and 6 mm. The Quartz Small Parallel Plate Kit is used with analyzers that have a high-temperature DMA 7e furnace. The Quartz Large Parallel Plate Kit is used with analyzers that have a standard DMA 7e furnace. Optional accessories available for the Parallel Plate measuring system include cup and plate accessories, parallel plate with tray accessories, and sintered parallel plates.

See the various parallel plate measuring systems below.

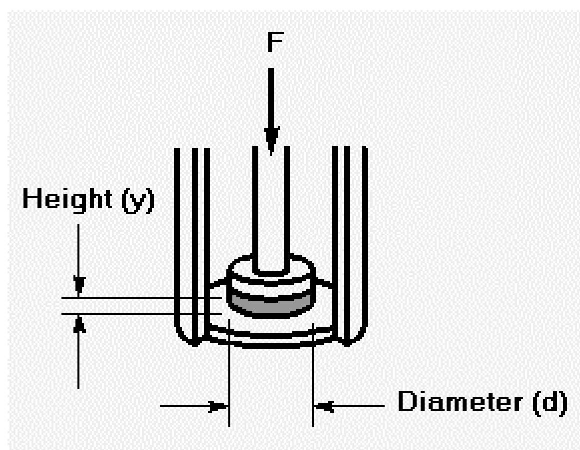
Parallel Plate Measuring Systems



Large Quartz



Stainless Steel



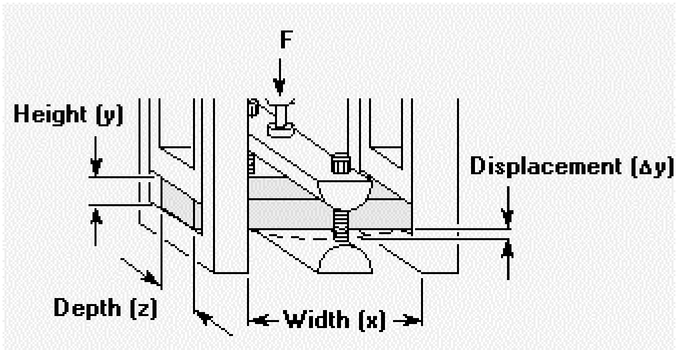
Small Quartz

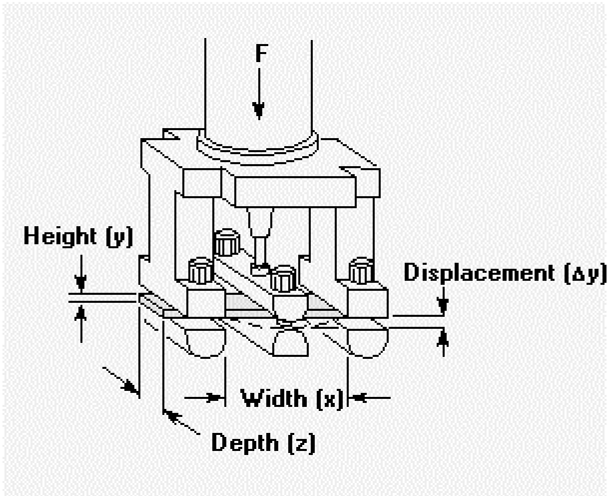
Dual Cantilever Measuring System

The dual cantilever measuring system is used to study materials with midrange modulus such as elastomers and materials above their glass transition temperature. This measuring system uses a clamping assembly with a hemispherical center clamp that increases effective sample size and reduces localized clamping stress.

The Stainless Steel Dual Cantilever Kit (P/N N539-0131) comes with two sample tube assemblies: One is recommended for high-modulus samples and the other for low-modulus samples.

Dual Cantilever Measuring Systems

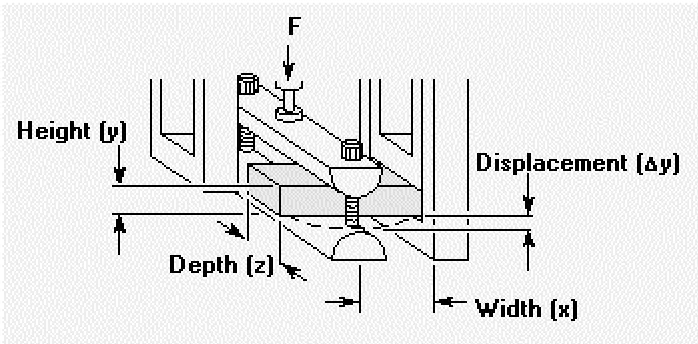


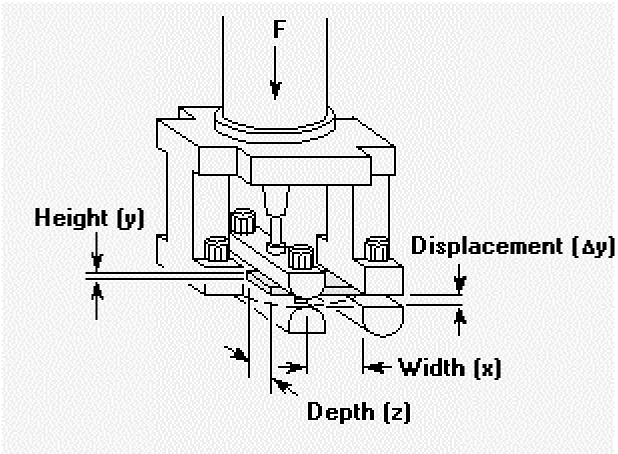


Single Cantilever Measuring System

The single cantilever measuring system is used for the same classical mechanical testing applications as the dual cantilever measuring system. When only one side clamp is attached, the dual cantilever can be used as a single cantilever measuring system.

Single Cantilever Measuring Systems





DMA 7/7e Test Methods

The DMA can be used to run many different types of tests. These different types of tests provide the user with the ability to program a selected variable (or variables) while all other variables are held constant. The DMA 7e can run the following types of test methods:

- **Temperature Scan**
- **Time Scan**
- **Frequency Scan**
- **Frequency MultiPlex Scan**
- **Dynamic Stress Scan**
- **Creep-Recovery**
- **Static Stress Scan**
- **Constant Position**
- **Constant Force**

Temperature Scan

In a Temperature Scan test, the temperature is programmed and the frequency and stress are held constant. Temperature-dependent behavior is characterized by monitoring changes in strain and phase. Alpha, Beta, and Gamma transitions may be identified as a function of temperature or time. Modulus and viscosity, as well as other standard reporting variables, may be readily quantitated for these materials as a function of temperature or time.

See the chart in **Zeroing the Probe** that shows the relationship among the DMA test (temperature scan), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Time Scan

In a Time Scan test, the frequency, temperature, and stress are held constant over time. Time-dependent behavior is characterized by monitoring changes in strain and phase. This test is useful for examining time-dependent curing behavior in materials such as coatings, adhesives, rubbers, and epoxies. Modulus and viscosity, as well as other standard reporting variables, may be readily quantitated for these materials as a function of temperature or time.

See the chart in **Zeroing the Probe** showing the relationship among the DMA test (time scan), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Frequency Scan

In a Frequency Scan test, the frequency of oscillation is programmed over a specific range with time. Temperature and stress are held constant. Rate-dependent behavior is characterized by monitoring the change in strain and phase over time. A Frequency Scan test is useful in the detection of subtle differences in the molecular structure of materials. This information can be used to predict material behavior or emulate processes.

See the chart in **Zeroing the Probe** that shows the relationship among the DMA test (frequency scan), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Frequency MultiPlex Scan

In a Frequency MultiPlex test, a series of frequency scans can be programmed at different temperatures. The data can be analyzed as independent frequency scans to detect subtle differences in molecular structure. Or the scans can be analyzed as a group, using the Time–Temperature Superposition calculation, to produce data to predict material behavior over long periods of time.

Dynamic Stress Scan

In a Dynamic Stress Scan test, the dynamic and static stresses are linearly programmed up or down over time and the frequency and temperature are held constant. The stress-dependent behavior is characterized by monitoring changes in strain and phase. This test is useful for conducting DMA analysis in the stress-dependent, linear, viscoelastic region. Dynamic Stress Scan is also useful for performing standard test methods and identifying stress-dependent differences in materials.

See the chart in **Zeroing the Probe** that shows the relationship among the DMA test (dynamic stress scan), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Creep Recovery

In a Creep Recovery test, a Creep or a Recovery analysis can be performed or both analyses can be performed during one experiment. In the Creep part of the test, a static stress is instantaneously applied and held at a constant value for a set time. In the Recovery part of the test, a static stress is instantaneously removed. The resultant sample displacement is measured. The Creep Recovery test is used to generate modulus and viscosity values at long times (very low frequencies) that are not within practical limits of instrument testing.

The temperature in these tests can be held constant or can be changed during a run; there is no dynamic stress applied. Time and stress-dependent behavior are characterized by monitoring the strain. Compliance, modulus, and stress are calculated and displayed on the screen as a retardation spectrum in the Creep test and as a relaxation spectrum in the Recovery test.

See the chart in **Zeroing the Probe** that shows the relationship among the DMA test (creep recovery), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Static Stress Scan

The Static Stress Scan applies a linearly increasing or decreasing static stress to the sample. The starting and final stress values, as well as the scanning rate, can be programmed. This test is useful for characterizing stress-dependent properties of the sample.

See the chart in **Zeroing the Probe** that shows the relationship among the DMA test (static stress scan), DMA measuring system geometry (e.g., 3-point bending), and zeroing the probe.

Constant Position Test

The Constant Position test is a variation of the Creep Recovery test. The sample is programmed over a selected temperature range or held at a constant temperature over time. Temperature- or time-dependent behavior is characterized by monitoring changes in the sample dimensions. Constant length experiments are performed using this test.

Constant Force Test

Only static forces are applied during a Constant Force test. The sample run is programmed over a selected temperature range or held at a constant temperature versus time. Temperature- or time-dependent behavior is characterized by monitoring changes in the sample dimensions. Constant force experiments, such as softening points and expansion coefficients, are performed using this test.

Operational Guidelines

The operational guidelines for using your DMA 7e are given for the following topics:

- **Purge Gas**
- **Temperature Scanning Rate**
- **Force/Stress Display Choice**
- **Static Stress**
- **Dynamic Stress**
- **Frequency**
- **Amplitude**

- Phase Angle
- Resonance
- Sample Preparation and Mounting
- Theoretical Constraints
- Reference Materials
- Zeroing the Probe
- Instrument Controls

Purge Gas

Helium of 99.9% minimum purity is required for purging the sample environment. Helium is used instead of nitrogen because it has better thermal conductivity and provides superior purge performance. **The gas must be dry**. This can be achieved with the use of a **filter dryer**. A size 1A cylinder equipped with a suitable regulator is recommended for the gas supply. The regulator should have a shutoff valve at the outlet. The shutoff valve should have a 0.25-in. NPT male thread on the outlet side for connection to the DMA 7e purge gas line. Constant pressure from the purge gas will also prevent contaminants from getting up into the core rod. The cleanliness of the core rod must be maintained or the analyzer's performance will be affected.

When using helium as a purge, you may want to install a helium flow meter and a control valve in line in place of the H restrictor that is provided with the DMA 7e. In general, the temperature performance may be greatly affected by the purge gas flow rate. By controlling the rate, it is possible to control the temperature performance more precisely. You may want to try different flow rates to optimize temperature performance.

You can use a **gas selector accessory** with the sample purge gas in order to allow for the automatic switching of purge gases.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the plug attached to the N519-0310 analog cable at the back of the TAC 7/DX Thermal Analysis Instrument Controller. The TAC 7 then connects to the analyzer.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Instructions for installing the TAGS are in the online Installation Help.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 1/4-in. pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

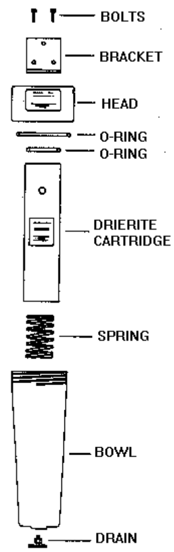
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4-in. pipe fitting using Teflon tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4-in. pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.

5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2-1/2-in. o.d. O-ring on top of cartridge.
8. Place the 4-in. o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD SOAP AND WATER. DO NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

Temperature Scanning Rate

Scanning rates should be sufficiently low to prevent excessive lag between the program and the sensor temperatures. “Excessive” is determined by the desired accuracy and extent of calibration. Stainless steel measuring systems have a much higher thermal mass and higher thermal inertia than quartz measuring systems. Depending on the desired accuracy, scanning rates are typically between 1°C/min and 5°C/min for stainless steel measuring systems and between 2°C/min and 10°C/min for quartz measuring systems.

Force/Stress Display Choice

There are two ways to enter parameter values: Forces and Stresses. You select which you would like to use in the Display page in Preferences. The default is Forces. Your choice affects inputs and displays. Force is measured in millinewtons (mN); stress in pascals (Pa).

Static Stress

- The static stress (including static force, static stress, position control, and tension control) should be sufficient (relative to dynamic stress) to prevent loss of contact between the sample and the measuring system.
- Static stress should be low enough to prevent excessive bending or drift of the sample. This is measuring system dependent.
- Static stress that is too high can cause errors in the damping (phase angle) signal.
- Static stress should be maintained within analyzer limits throughout the entire analysis. Motor controls can be used to regulate this value.

Dynamic Stress

- The dynamic stress (including dynamic force, dynamic stress, force control, amplitude control, strain control, and stress control) should be sufficient to produce a dynamic amplitude between 5 and 500 µm throughout the entire analysis (see **Amplitude**).
- The dynamic stress provides the dynamic oscillatory response in the sample that is used to generate dynamic mechanical analysis data.
- The dynamic stress should be sufficient to produce average dynamic amplitude above 1 µm (the minimum detection limit).
- It should be low enough to produce an average amplitude below 650 µm (the maximum detection limit).

Frequency

- The frequency should be selected to produce a reasonable phase angle while avoiding resonant (harmonic) frequencies. Typical industry standards are between 1 and 11 Hz for practical applications.
- Resonant frequencies are affected by the sample spring constant and sample dimensions. The resonant frequency can be altered by changing the sample size.
- Extremely low frequencies are not practical because of the long times between cycles and data points:

Frequency (Hz)	0.1	0.1	0.01
Seconds/cycle	10	10	100
Seconds/point	5	50	500

Amplitude

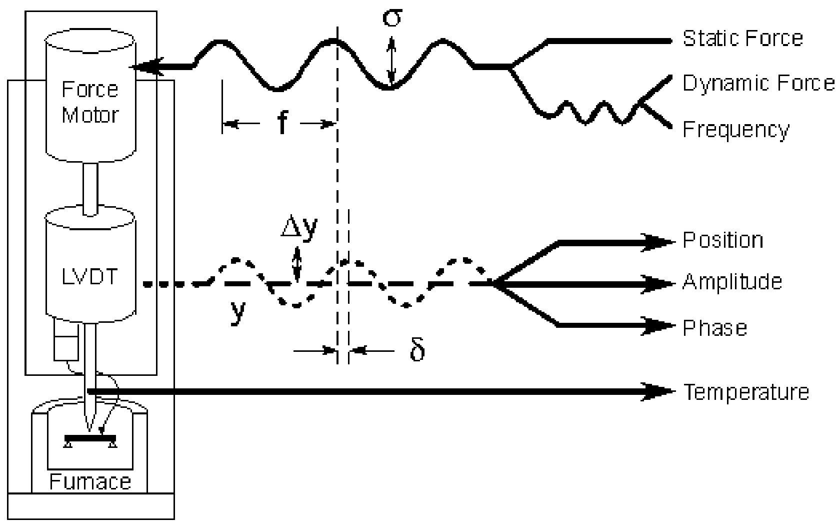
The amplitude is the most important output signal. It is generated by the LVDT (see figure below). Some instruments specify peak-to-peak amplitudes. Amplitude (1/2 peak-to-peak amplitude) and phase angle are closely related; they are derived from the same measured vector.

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

The data quality of primary results depends on both phase angle and amplitude:

		Amplitude	
		<1 µm	>1 µm
Phase lag (delta)	<0.5	limited	good
	>0.5	good	best

DMA 7e Motor Control Variables



Phase Angle

Phase angle values between 1° and 45° can produce very-high-quality data, even at much lower amplitudes. A phase angle can be changed, but only for a sample that has been mounted properly and is viscoelastic in nature. In that case, changing the frequency of dynamic oscillation changes the phase angle. Reducing the phase angle generally increases data quality.

You cannot alter the equilibrium phase angle of a material. The reported phase angle can be altered only by introducing or eliminating error.

Sources of error in the phase results include sample mounting, sample rocking, loose probes, probe misalignment (damping), and resonance.

Resonance

The resonant frequency for the analyzer system with no sample is approximately 75 Hz. The resonant frequency of the sample and system can be found on the status line of the frequency scan when using force entry. The symbol for resonance is f0 and is reported in Hz.

Avoid using a frequency that is near the resonant frequency in the method.

Sample Preparation and Mounting

Calibration, sample preparation, and mounting have the greatest impact on data quality.

- Samples should not be subjected to extreme stresses (mechanical, thermal, etc.) during preparation and mounting.
- Samples should be mounted to prevent any undesired motion of the sample, such as twisting, with respect to the measuring system. Such motion can cause noise in both the amplitude and phase values obtained.
- Sufficient time should be allowed for sample-to-measuring-system stress relaxation. The sample should not be left mounted for an extended period of time before the analysis begins.

See the topic **Mounting Samples onto the 3-Point Bending Measuring System** in the Pyris Multimedia Presentations Help.

See the topic **Mounting Samples onto the Extension Measuring System** in the Pyris Multimedia Presentations Help.

Theoretical Constraints

The DMA 7e is qualitative for all samples, modes, measuring systems, and sample geometries. It is quantitative for all samples, modes, measuring systems, and sample geometries under the following conventions:

The sample should be isotropic (independent of direction), homogeneous (single component), amorphous (random orientation), and linearly viscoelastic (stress proportional to strain and particulate matter less than 10% of sample critical dimension). It should be tested at small strains (<1%) using primary deformation, such as simple extension/compression, simple bending, or simple shear.

Reference Materials

Reference materials are provided in the Spares Kit (P/N N539-0139). They are used for performance verification and training.

Reference Materials (RMs) comply with ISO for instrumentation calibration. They are available from Perkin Elmer and Perkin Elmer provides documentation with each RM that cross references the lot code. This declaration should be filed with all other material certificates and documents in your ISO file.

ISO Guide 30 defines a RM as a

> Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Certified Reference Materials (CRMs) are traceable to national or international standards through an unbroken chain of comparisons. Not every calibration material is a CRM. CRMs can be very expensive and are not available for all thermal analysis techniques.

If you are not sure which material to use, check with your Quality Assurance Department and discuss the differences between RMs and CRMs. If you find that you need CRMs, you may order them from the National Institute of Standards and Technology (301 975-6776).

Reference Material E is a standard epoxy–glass composite known as FR4. It is bisphenol A-type epoxy supported by four layers of woven E-type glass fibers. This type of material can be assayed by DMA or TMA thermal analysis. The supporting glass permits analysis well above the glass transition temperature. The epoxy resin exhibits mechanical relaxations down to liquid nitrogen temperatures. It is highly crosslinked and changes little under normal storage and handling conditions.

This type of sample has minimum mounting, edge, end, or clamping effects when using the DMA 7e 3-Point Bending 15-mm stainless steel measuring system or the 18-mm quartz measuring system.

Reference material E has been analyzed by various techniques and has been found to agree with the manufacturer’s specifications. Assay numbers are not indicated on the samples. Recent lots indicate a storage modulus of 1.1e10 to 1.8e10 Pa.

The performance of the DMA 7e can be measured by data quality, which can be affected significantly by several factors. For best results from your reference material, observe the following guidelines:

- Measure each sample carefully. The DMA 7e allows the entry of sample dimensions in 0.001 increments.
- Do not use a sample that has been heated and remounted.
- Store the sample in a cool dry place, away from liquid or gas contaminants.
- For the highest repeatability, rerun the sample without removing it from the measuring system. This reduces any error caused by sample-to-measuring system stress relief, internal stress in the sample, of additional curing.

Zeroing the Probe

To zero the probe means to set the LVDT to zero height. To zero the probe height:

1. Set the current static force to 100 mN in the Initial Values page.
2. Make sure that there is no sample on the sample platform.
3. Press the **Probe Down** button on the analyzer.
4. Click on the **Read Zero** button on the Control Panel. The Zero Height value is displayed in the Zero field in the Method.

The analyzer is now zeroed for height. The sample zero reflects the difference between calibration zero and the current probe position. When a sample is mounted in the analyzer, the reported height should be close to the sample height.

When & Where to Read Height and Zero to Get Meaningful Results

Type of Analysis	Oscillating DMA	Static DMA	TMA	Sample Height
Type of DMA Mode / Type of DMA Geometry	Temperature Scan Time Scan Frequency Scan Dynamic Stress Scan	Creep-Recovery Static Stress Scan	TMA	For each measuring system, all modes.
Flexure 3-Point Bending 4-Point Bending Dual Cantilever Single Cantilever	ZERO NOT USED The sample height is typed in.	ZERO on the Top of the Sample so the sample height and deflection are in the same direction. BASE	BASE ZERO on the Bottom of the Sample	Always type in the sample height. The automatic height is not used
Tensile Extension Tension	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	Sample height is automatically read. The typed value is not used.
Compressive Parallel Plate Cup and Plate Plate and Tray Sintered Plates	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	Automatically read. Typed-in values are not used.
How Displacement is Calculated	Amplitude (micrometers) Start, Y, ΔY, Time	Displacement (millimeters) Start Run, End Run, Y, ΔY, Time	ΔY is calculated using "SELECT CALC"	

Instrument Controls

Operational guidelines for the instrument controls are given in the following topics:

- Temperature Control
- Force Control
- Total Force
- Tension Control
- Position Control
- Amplitude Control
- Stress Control
- Strain Control

The table below summarizes the function of each of these controls:

Static Control	**Description**
Force Control	Force is controlled, independent of any other parameter
Position Control	Force is changed to hold the position constant
Tension Control	Static force is changed to maintain a constant ratio of static and dynamic force
Dynamic Control	
Force Control	Dynamic force is controlled, independent of any other parameter
Amplitude Control	Dynamic force is changed to keep the amplitude at setpoint
Stress Control	Dynamic force is changed to keep stress at setpoint as sample dimensions change
Strain Control	Dynamic force is changed to keep strain at setpoint

Temperature Control

If the temperature in the Status Panel and Control Panel is not at approximately the temperature in step 1 of your temperature program, in the Control Panel enter the temperature to which you want to heat or cool the analyzer and click on the **Go To Temp** button.

When the Control indicator on the front panel is lit, the Go To Temp value should agree with the initial temperature in the temperature program. When the program temperature reaches the selected temperature, the Heating and Cooling indicator lights will turn off. When the analyzer reaches the selected temperature, Control will be lit, indicating that the analyzer is equilibrated. If this does not occur, the furnace is cooling to the Go To Temp value. In this case, wait until the furnace has cooled sufficiently to allow power to be supplied to the furnace (i.e., Control light is on). For best results, the Control light should be on at least two minutes prior to starting a run.

Force Control

CAUTION: The DMA 7e motor operates in force control by default. You may select force control or stress control in Preferences. Force control maintains a constant control of the static force (and dynamic force where applied) at the values entered throughout the run.

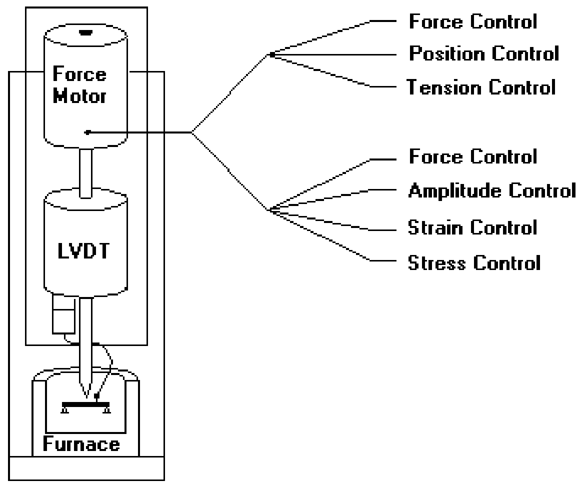
We recommend that you become familiar with the operation of the DMA 7e and the applicability of your sample to the control systems before you use the instrument control systems.

The DMA 7/DMA 7e Motor Control System is in force control by default. Both static and dynamic forces are maintained at fixed values throughout the experiment. The calculated stresses may vary. The strain, phase, position, and amplitude are not controlled and are allowed to vary.

- You can enter a static stress (Pa) or a static force (mN) and the analyzer will program a static force (mN).
- You can enter a dynamic stress (Pa) or a dynamic force (mN) and the analyzer will program a dynamic force (mN).

Motor Control System

Motor control systems can be used with any measuring system and almost any type of method.



Total Force

DMA 7

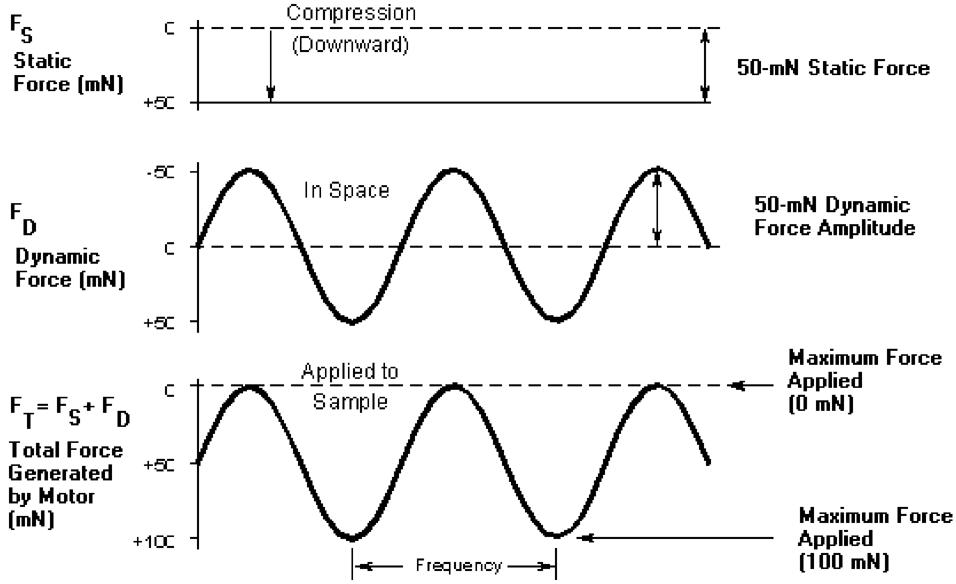
The total force is the sum of the dynamic and the static forces and must be less than the force of the motor. When using the DMA 7, the total force should be between 10 and 2500 mN throughout the entire run. This total force range is more than sufficient for almost all materials when used in conjunction with the numerous modes, measuring systems, and geometries.

- The total force should not exceed the limits of the analyzer.
- The total compressive force that the analyzer can exert is between 1 and 2500 mN. (The total tensile force that can be exerted is between 5 and 1300 mN.)

DMA 7e

The total force for the DMA 7e is also the sum of the dynamic and the static forces. It should be between 10 and 8500 mN throughout the entire run. This total force range is more than sufficient for almost all materials when used in conjunction with the numerous modes, measuring systems, and geometries.

- The total force should not exceed the limits of the analyzer.
- The total compressive force that the DMA 7e can exert is between 1 and 8500 mN. (The total tensile force that can be exerted is between 5 and 5600 mN.)



Tension Control

Tension Control is recommended when using Strain Control or Stress Control. The Tension Control applies a static force as a percentage of the dynamic force. For example, if Tension Control is 110%, then the analyzer will apply 10% more static force than dynamic force.

When Tension Control is off, the static force necessary to maintain sample-to-probe contact is applied automatically. When Tension Control is activated, the analyzer does not use the user-entered value for the static force but rather a percentage of the dynamic force. That percentage can be entered by the user.

Tension Control setpoint values depend on the measuring system used and the state of the sample, including its tackiness and compliance. Typical Tension Control setpoint values are given below:

Setpoint Minimum	**Setpoint Maximum (%)**	**Measuring System**
105	110	3-Point Bending
0	5	Dual Cantilever
0	1	Single Cantilever
120	150	Extension
0	110	Parallel Plate
0	10	Parallel Plate (tacky sample)

Position Control

The Position Control varies the static force as the sample changes position as detected by the LVDT. For example, if a 5.000-mm fiber sample shrinks during a run and Position Control is on, the force will be increased automatically to maintain a 5.000-mm length.

When Position Control is activated, the analyzer no longer applies the user-entered value for Static Force. It applies a static force needed to maintain a constant position.

Amplitude Control

The DMA 7e can operate as a constant amplitude analyzer. In order to extend the application range of the analyzer, the Dynamic Force can be programmed to maintain a constant dynamic displacement. The amplitude will be controlled at a fixed setpoint throughout the experiment.

Stress Control

The DMA 7e can operate as a constant stress analyzer. In order to extend the application range of the analyzer, the Dynamic Force can be programmed to maintain a uniform force per sample contact area (stress). The stress will be controlled at a fixed setpoint throughout the experiment.

Strain Control

The DMA 7e can operate as a constant strain analyzer. In order to extend the application range of the analyzer, the Dynamic Force can be programmed to maintain a displacement amplitude. The strain will be controlled at a fixed setpoint throughout the experiment. If a sample passes through more than two orders of magnitude of Modulus, Strain Control can be used to extend the analysis.

Subambient Operation

Different coolants and cooling accessories are available for use with the DMA 7e. The type of experiments you will perform determine your choice of coolant and accessory. If you are using a DMA 7e with a furnace dewar assembly, cooling the system is performed either ambiently or subambiently.

NOTE: There must always be some form of coolant in the dewar assembly when operating the DMA 7e. The heatsink temperature must not rise abouve 45°C.

For standard operation of the DMA 7e from ambient to 500°C, use an ice water bath in the dewar assembly. Access the dewar space by removing the top dewar cover. Fill the dewar space with ice water. You could also use tap water circulation with the turbulent chamber. Attach the turbulent chamber to a standard faucet so that tap water can continuously circulate through the turbulent chamber. This arrangement allows operation from 50°C to 500°C. An optional high temperature furnace is available for operation to 1000°C.

For subambient operation, use liquid nitrogen in the dewar assembly or use an Intracooler 1 or 2. Liquid nitrogen allows operation from –170°C to 500°C. When the dewar assembly is filled with liquid nitrogen, the system reaches the boiling point of liquid nitrogen (–195°C) at atmospheric conditions.

NOTE: When using liquid nitrogen, do not heat faster than 20°C/minute.

Intracooler 1 is a mechanical refrigeration unit that allows operation of the DMA 7e from –30°C to 500°C. Intracooler 1 automatically maintains the DMA 7e heatsink at a constant temperature.

Intracooler 2 is also a mechanical refrigeration unit that allows operation of the DMA 7e from –65°C to 500°C. It automatically maintains the DMA 7e heatsink at a constant temperature.

Follow the procedure below when using **liquid nitrogen** for subambient operation of the DMA 7e:

1. Program the furnace to –180°C.
2. Fill the dewar with LN2 using the funnel.
3. Wait a few minutes for the heat sink to cool and equilibrate.
4. Continue to top off the dewar as necessary as the analyzer cools.
5. Program the furnace to your starting (minimum) temperature. Wait a few minutes for the analyzer to reach the programmed temperature. The programmed temperature is reported in the Status Panel.
6. Set up the method in the Method Editor.
7. **Mount the sample**.
8. Begin the run.

Mounting the Sample

For most standard DMA 7e experiments, mount the sample following the steps below. Standard experiments include penetration, expansion, and compression tests. For performing experiments using the dilatometer cell, the extension analysis kit, or the flexure kit, refer to the specific instructions that come with the accessory.

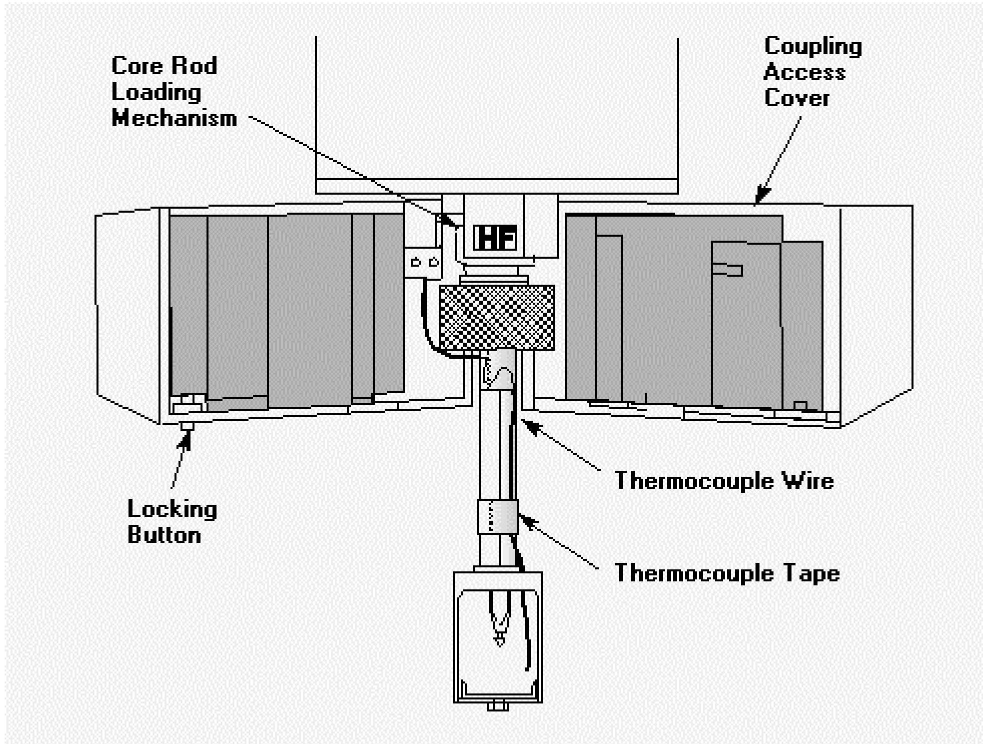
1. Carefully lower the furnace assembly to the base of the analyzer by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
2. Press the **Probe Up** button on the front panel to raise the probe assembly and lock it into place.
3. Make sure that all traces of previous samples have been removed.
4. Cover the furnace opening with a business card or some other suitable material.
5. Using the tweezers provided with the instrument, place the sample to be analyzed on the sample tube platform.

NOTE: If you are running very soft samples in the subambient range (i.e., < 20°C), you should perform step 6 after steps 7 and 8.

6. Press the **Probe Down** button to lower the probe assembly so that it rests on the sample.
7. Carefully raise the furnace assembly all the way up, making sure that it locks in place.
8. Enter the desired temperature in the Go To Temp field in the Control Panel and click on the **Go To Temp** button.
9. When the height signal (mm) displayed in the Status Panel stabilizes, select the **Read Height** button from the Control Panel to automatically read the height of the sample and store it in memory.
10. Place the diffusion cap around the sample tube at the top of the furnace.

Maintenance

Maintenance topics discuss maintaining the DMA 7 and the DMA 7e. The DMA 7 analyzers that were shipped prior to July 1, 1993, are the older analyzers. The new analyzers — DMA 7e — have a core rod labeled “HF.”



The standard shipping version of the DMA 7e includes a turbulent chamber, a large (28 mm) furnace, a 3-point bending measuring system, and a dewar. In order to install the Dewar Kit onto the DMA 7e, the turbulent chamber must be removed. If you have only one furnace, you will have to remove the furnace from the turbulent chamber and install it into the dewar.

Maintenance also includes changing from the large (28 mm) furnace to the small (15 mm) furnace on the DMA 7e.

A Dewar Retrofit Kit is available from Perkin Elmer for those users who wish to upgrade their DMA 7 to use the new dewar. The new dewar provides extended LN2 residence time, allowing you to perform longer experiments. The kit must be installed by a Perkin Elmer Service Representative.

See the following topics for maintenance of the DMA 7e:

- **Dewar Installation**
- **Furnace Replacement in the New Dewar**
- **Furnace Replacement in Turbulent Chamber**
- **Replacing Large Furnace with Small Furnace**

See the following topics for maintenance of the DMA 7:

- **Dewar Installation**
- **Furnace Replacement in Old Dewar**
- **Furnace Replacement in the New Dewar**
- **Furnace Replacement in Turbulent Chamber**
- **Replacing Large Furnace with Small Furnace in DMA 7**
- **Upgrade DMA 7 with New Dewar**

In addition, maintenance also includes:

- **Replacing the Thermocouple**
- **Maintenance of Sample Tubes and Probes of DMA 7e**

Dewar Installation

NOTE: The instructions for installing a new dewar into a DMA 7e should be used to install a new dewar into a DMA 7 that has been retrofitted for a new dewar.

The dewar in the DMA 7e analyzers is used to obtain data in the ambient to subambient temperature range. Liquid nitrogen may be used in the dewar for operation from –170°C. Ice water may be used for operation from 25°C. The standard furnace size for the DMA 7e is the large (28 mm) furnace which gives the analyzer a temperature of –170°C to 500°C, while the TMA 7 analyzer uses a small (15 mm) furnace for a temperature range –170°C to 1000°C. It is possible to use the small furnace in the DMA 7 and DMA 7e and to use the large furnace in the TMA 7. The new large dewar can also be used on the DMA 7 and on older TMA 7 analyzers. Contact your local Perkin Elmer Service Representative for details.

The dewar is easily installed and removed from the analyzer, allowing the turbulent chamber and other environmental systems to be used. It is recommended that you purchase a second furnace if you will be switching between the dewar and the turbulent chamber often. This way you can keep a furnace in each assembly, making the switch faster.

Installation of the new dewar into the DMA 7e consists of the following steps:

- **Turbulent Chamber Removal**
- **Furnace Removal from the Turbulent Chamber**
- **Furnace Installation into the Dewar**
- **Connecting the Dewar**
- **Preparing the Dewar for Use**

Turbulent Chamber Removal

NOTE: If you have only one furnace, you will have to remove it from the turbulent chamber and install it into the dewar.

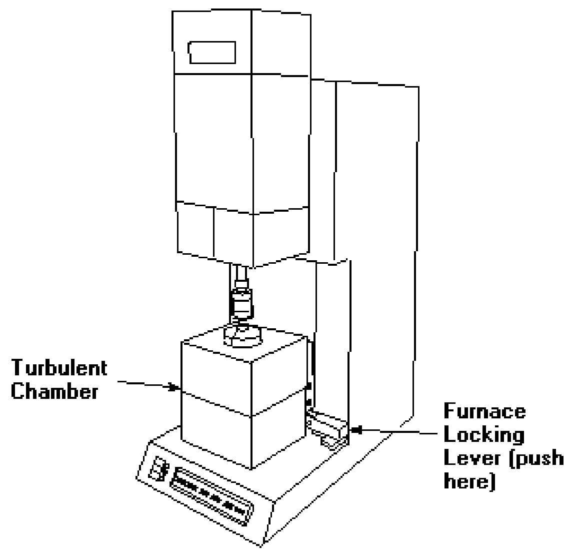
We recommend that you purchase a second furnace if you intend to use both devices. You may want to create and store calibration sets in conjunction with the furnace change.

CAUTION: If you are using a Quartz measuring system and no sample is mounted, you should place the septum (P/N 0009-0652) on the base of the sample tube directly under the probe to prevent damage to the probe or the sample tube.

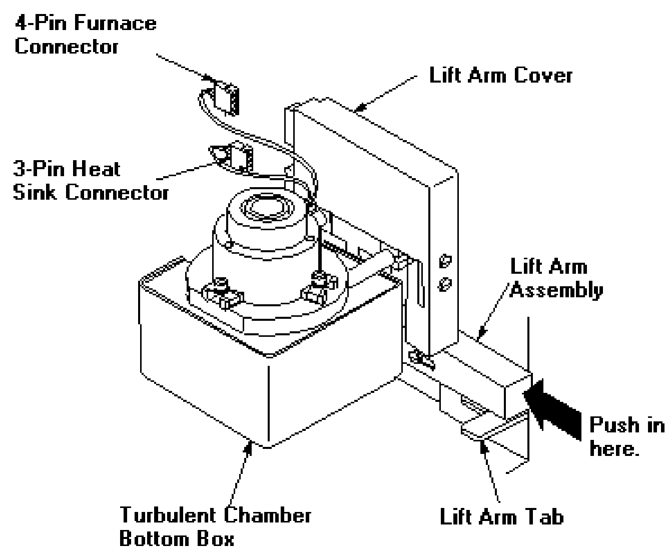
1. Turn off the power to the DMA 7e and the TAC 7/DX.

CAUTION: Be sure to turn the TAC 7/DX off, especially if a new calibration set is going to be used.

2. Remove the diffusion cap.
3. Completely lower the furnace. Make sure that the latch locks the furnace in the fully lowered position.

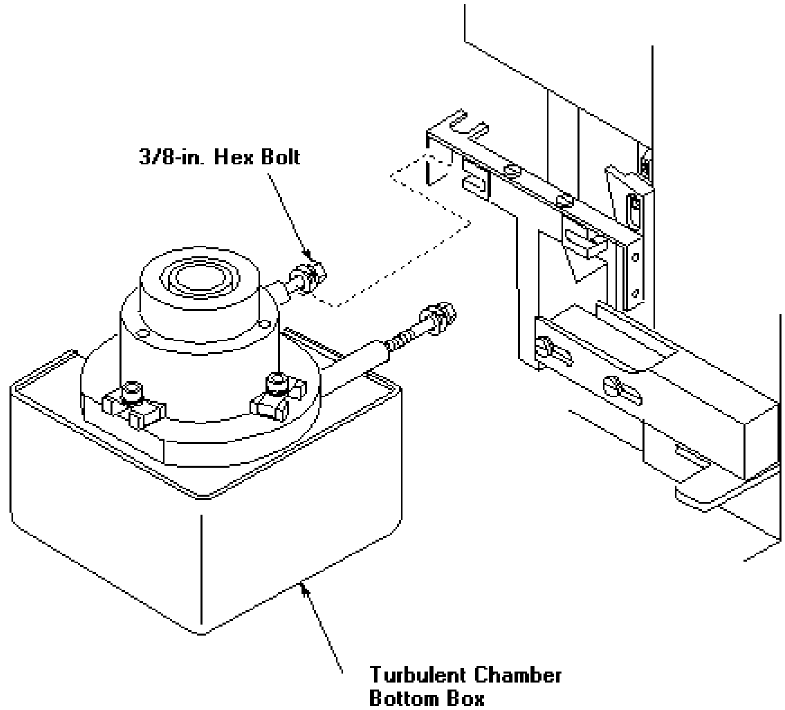


4. Remove the top furnace cover carefully to avoid damaging the measuring system. (If necessary, tilt the turbulent chamber cover toward you.)
5. Unplug the top white (4-pin) furnace connector which is on the left side of the furnace lift arm housing cover.
6. Unplug the bottom white (3-pin) heat sink connector which is also on the left side of the furnace lift arm housing cover.



7. Remove the three flat head screws on the furnace lift arm cover (see figure above). Make sure that the screws do not drop inside the analyzer.
8. Remove the furnace lift arm cover by lifting it straight up and to one side. Make sure that all wires have been cleared out of the way.

For the next five steps refer to the figure below.

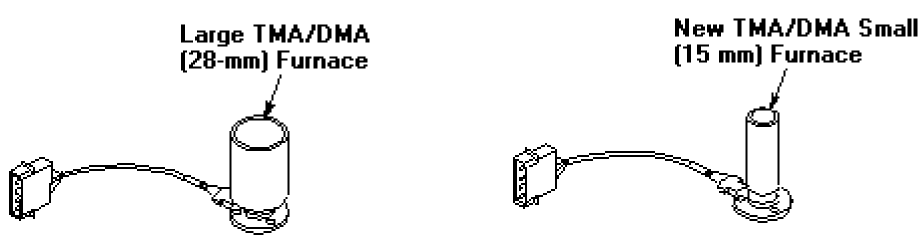


9. Locate the two 3/8-in. mounting box hex bolts that are attached to the mounting ring and furnace assembly lift arm.
10. Completely loosen and remove the leftmost bolt by turning the wrench clockwise.
11. Loosen the remaining bolt to the right by turning the wrench clockwise.
12. Gently move the turbulent chamber bottom box, which contains the circulator and furnace, to the right and remove it from the analyzer.
13. Carefully remove the rightmost bolt from the analyzer and place it into the mounting ring for storage.

Now that the turbulent chamber has been removed from the analyzer, you can remove the furnace from the turbulent chamber.

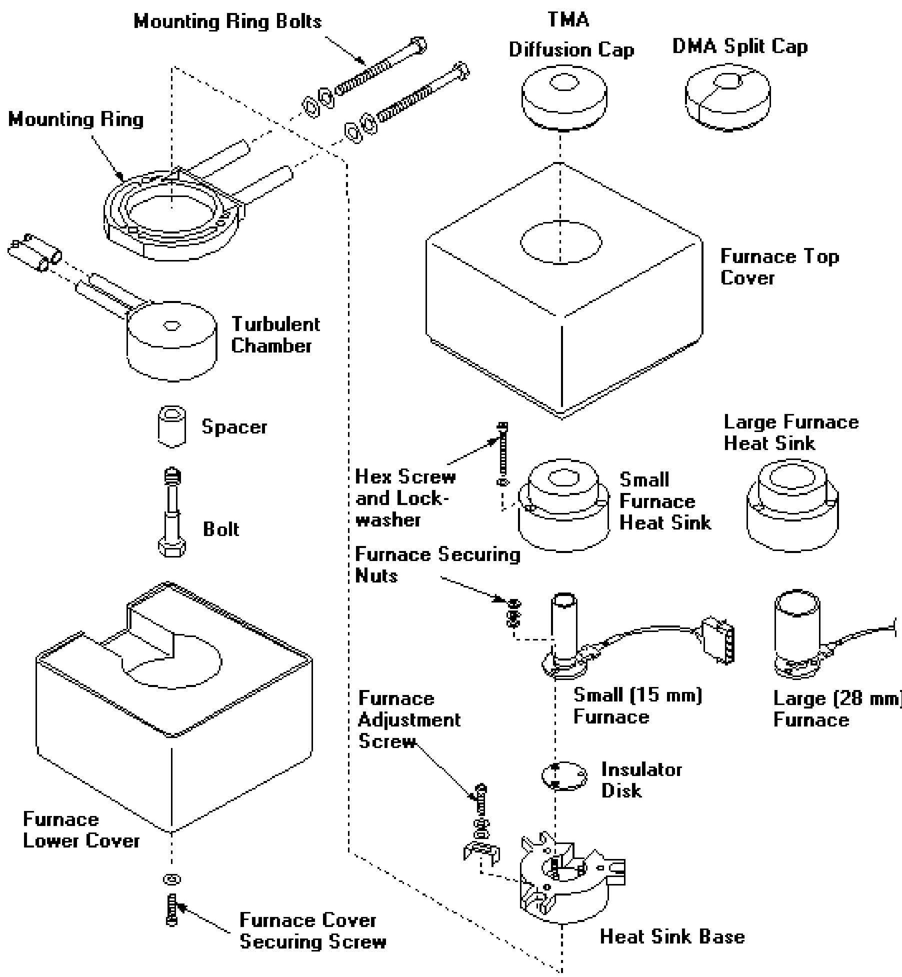
Furnace Removal from the Turbulent Chamber

If you do not have a second furnace to install into the dewar, the furnace in the turbulent chamber will have to be removed and installed in the dewar. Follow the steps here to remove either a **large (28 mm) furnace** or a **small (15 mm) furnace**.



Refer to the schematic below and use it to follow the instructions below.

Turbulent Chamber Disassembly Schematic



1. Remove the heat sink by loosening the three 9/64-in. hex head screws using the Allen wrench provided.
2. Carefully lift off the heatsink from the heat sink base; do not bump the sides of the furnace. **The furnace is fragile; it can be broken easily.** Keep the heat sink upright to prevent losing the screws and lockwashers.

   The furnace is now exposed.
3. Use a 1/4-in. open-end wrench to loosen the three nuts that hold the furnace in place.

   NOTE: The size of the furnace determines the type of mounting screw used. To determine the furnace size, measure the inner diameter of the furnace element.

4. Gently lift the furnace straight up and off of the three setscrews.
5. Replace the three furnace securing nuts onto the setscrews for storage.

Now that the furnace has been removed, it can be installed into the dewar.

Furnace Installation into the Dewar

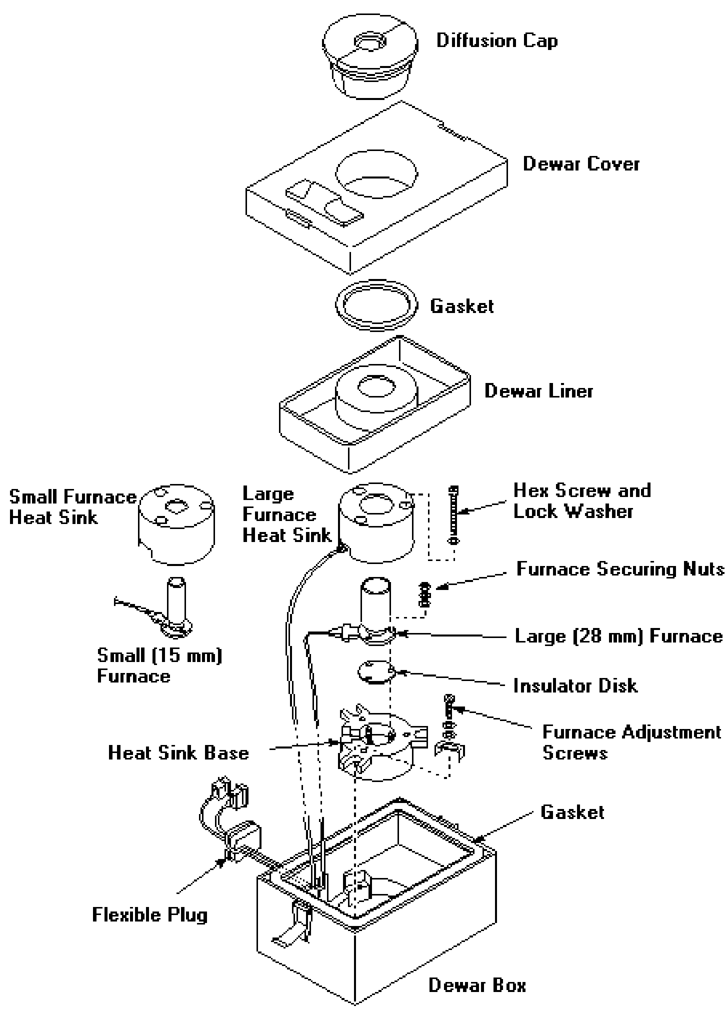
In installing a new dewar into the DMA 7e, you first had to remove the turbulent chamber and then remove the furnace from the turbulent chamber. You now can install that furnace into the new dewar. The same instructions can be used for installing a large (28 mm) furnace or a small (15 mm) furnace. Furnace installation consists of the following three steps:

- **Disassembling the Dewar**
- **Installing the Furnace**
- **Assembling the New Dewar**

Disassembling the Dewar

Use the schematic below while following the instructions below.

Dewar Disassembly Schematic



1. Remove the dewar cover by lifting the two latches on the sides of the dewar box.
2. Remove the two gaskets. One is on top of the dewar and the other is on top of the dewar box.

3. Lift the dewar liner out of the dewar box, exposing the heat sink.
4. Loosen the three 9/64-in. hex screws from the heat sink.
5. Lift the heat sink out of the dewar box, taking care to keep the heat sink upright to prevent losing the washers.

   The heat sink base is exposed. If a furnace is installed in the dewar box, it will also be exposed.
6. Remove the three flathead screws from the heat sink base and lift the base out of the dewar box.
7. Place the insulator disk over the three setscrews of the heat sink base.

Now you can install the furnace.

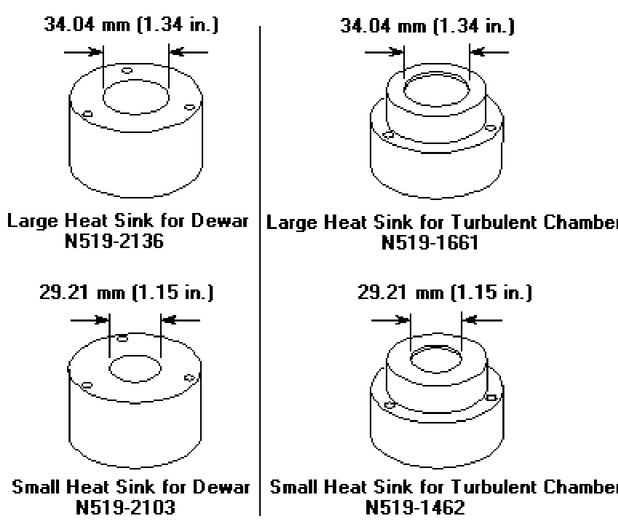
Installing the Furnace into the New Dewar

1. Place the furnace onto the heat sink base by lining up the three holes on the furnace with the three setscrews on the heat sink base.
2. Place a flat washer, a lock washer, and 1/4-in. hex nut onto each setscrew. Tighten the hex nut carefully until it is snug.

CAUTION: DO NOT overtighten the hex nuts; it could cause the furnace to break.

NOTE: You may want to have a second furnace so that the dewar and the turbulent chamber can each have a dedicated furnace.

3. Place the heat sink over the furnace and onto the heat sink base. Be sure to select the appropriate heat sink for the furnace you are using.



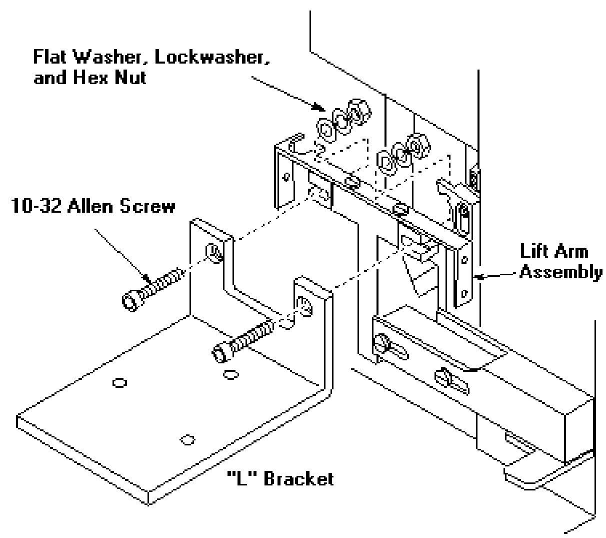
4. Place the three lock washers and 9/64-in. hex screws into the heat sink and tighten them.

Finally, you can assemble the new dewar.

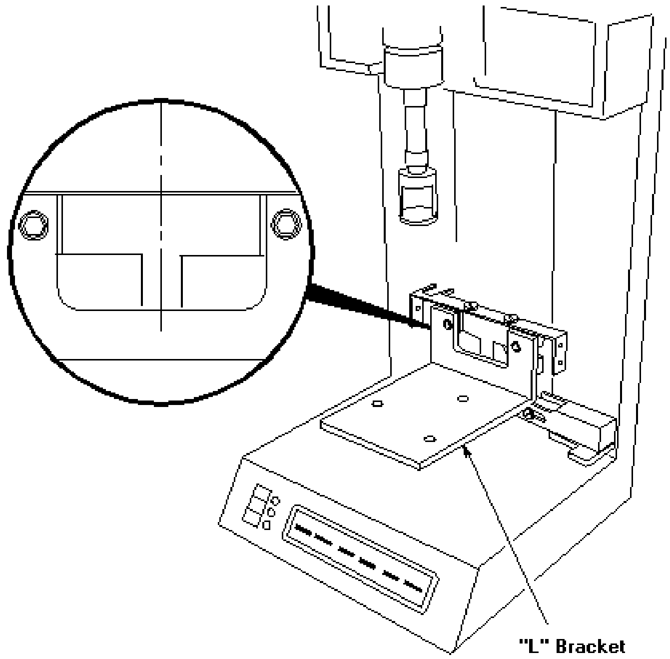
Assembling the New Dewar

At this point a furnace has been installed into the heat sink assembly of the dewar. In the instructions here, when heat sink is mentioned, the furnace is included.

1. Thread the electrical connectors for the furnace and the heat sink through the hole in the back of the dewar box.
2. Place the heat sink base (with furnace and heat sink on top) into the bottom of the dewar box.
3. Install the three brackets, washers, and flathead alignment screws. Make sure to secure them in place. **DO NOT OVERTIGHTEN**. The assembly will be moved for alignment later.
4. Place the flexible plug over the wires in back of the dewar box, pressing the plug into the hole at the back of the box. When the plug is properly seated, it will snap into place.
5. Attach the “L” bracket to the lift arm assembly by placing a 10-32 Allen screw into the left-hand hold of the bracket. Secure the screw by placing a lockwasher, a washer, and a hex nut on the back of the screw and tighten the hex nut.



6. Place a 10-32 Allen screw in the right-hand hole of the “L” bracket. Secure the screw by placing a lockwasher, a washer, and a hex nut on the back of the screw and tighten the hex nut.
7. Adjust the bracket so that it is centered. Once the bracket is centered, tighten the two Allen screws.

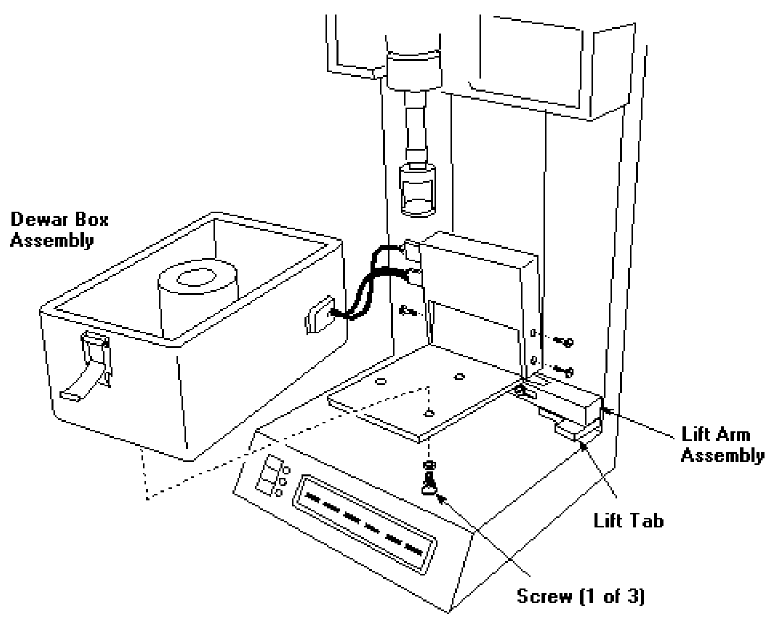


8. Replace the furnace lifting arm cover with the three flathead screws.

Now you can connect the dewar.

Connecting the Dewar

1. Plug the heat sink's 3-pin connector into the left side of the analyzer.



2. Plug the furnace's 4-pin connector (P/N N519-0781) into the left side of the analyzer above the 3-pin connector.

3. Slide the dewar into place on the furnace “L” bracket.
4. Holding the dewar in place with your left hand, unlock the furnace latch with your right hand and raise the dewar and furnace into position. Make sure that it locks into position.

CAUTION: DO NOT let the furnace assembly rise too quickly and impact the measuring system since the furnace or the measuring system may be damaged.

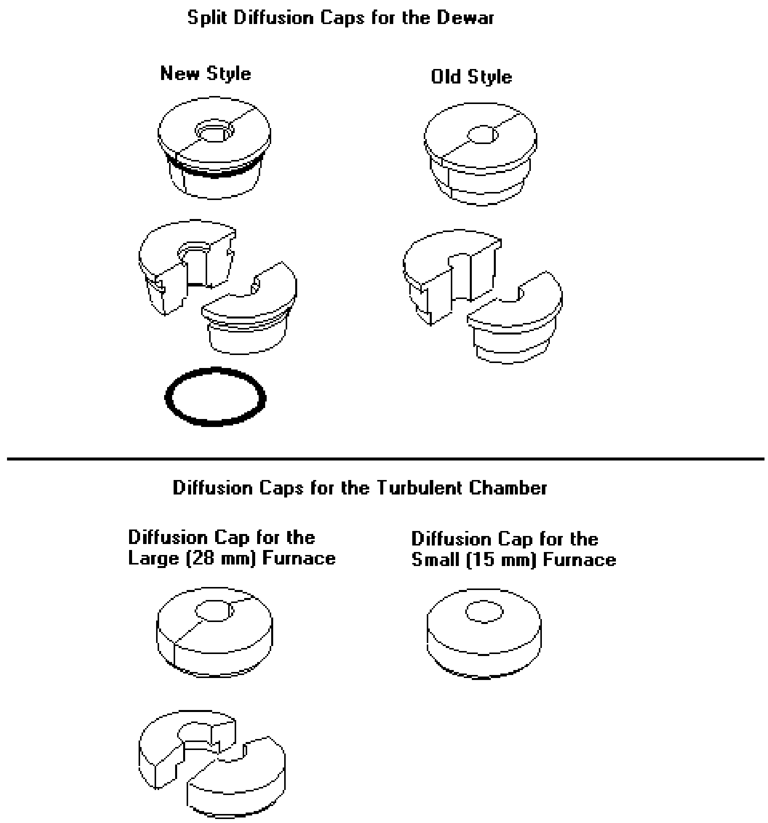
5. Insert the knurled head screws into the base of the dewar, going through the bottom of the “L” bracket. Tighten the screws; however, do not overtighten.
6. Align the furnace by moving either the heat sink assembly or the dewar box so that the measuring system is centered inside of the furnace.
7. Tighten the knurled nut on the base of the dewar box.
8. Lower the base.
9. Tighten the three flathead alignment screws that are on the heat sink base.
10. Insert the dewar liner.
11. Place the rectangular gasket inside the dewar box and around the ridge between the dewar liner and the dewar box.
12. Place the circular flat gasket on top of the heat sink cover in the dewar liner.
13. Replace the dewar lid.
14. Visually inspect the circular gasket so that it does not extend toward the furnace. A good seal is necessary to sustain the low temperature for an extended period.
15. Lock the dewar lid into place using the latches on the side of the dewar box.
16. Insert the funnel into the dewar opening if you are going to fill it with liquid nitrogen, or put the filler cover in place if you do not intend to fill the dewar immediately.

Finally, you can prepare the dewar for use.

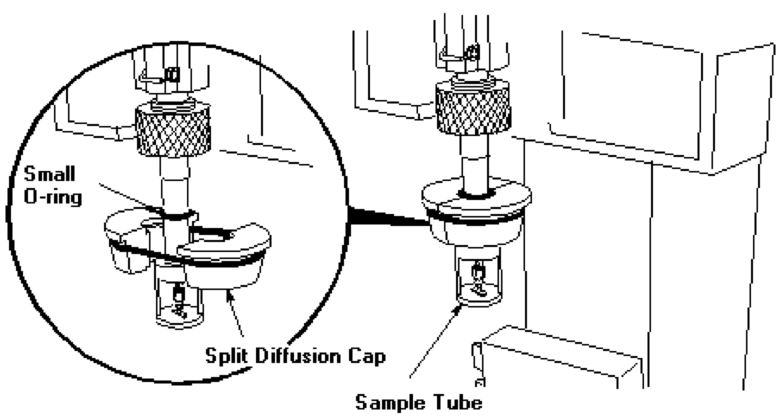
Preparing the Dewar for Use

The final step in installing the new dewar into a DMA 7e is preparing the dewar for experiments.

1. Slide an O-ring over the two halves of the split diffusion cap. The O-ring should slip into the groove under the lip of the cap.



2. Place a small O-ring over the measuring system and slide it up the shaft of the universal sample tube.
3. Pull the two halves of the split diffusion cap open so that they go around the measuring system. Slide the split diffusion cap up the universal sample tube so that the cap rests on top of the tube.



4. Roll the small O-ring down into the diffusion cap.
5. If the coupling access cover is open, close it.
6. Raise the furnace and, if necessary, press the O-rings into place.

The system is now ready to use. Power up the system. The Ready and Down lights on the front panel should light up. If any other lights come on, there is an improper connection. Check the cables and connections. If necessary, disassemble the dewar and reassemble it, making sure to follow all of the steps in the procedure.

Furnace Replacement in the New Dewar

These instructions are for replacing the furnace in the dewar if you have a DMA 7e or an older DMA 7 that has been retrofitted with a new dewar.

NOTE: The older DMA 7 analyzers can be retrofitted with the new style dewar by having a Perkin Elmer Service Representative install the Dewar Retrofit Kit onto the analyzer. The new style dewars provide extended LN2 residence time, allowing longer experiments to be performed. Contact your local Perkin Elmer Representative for details.

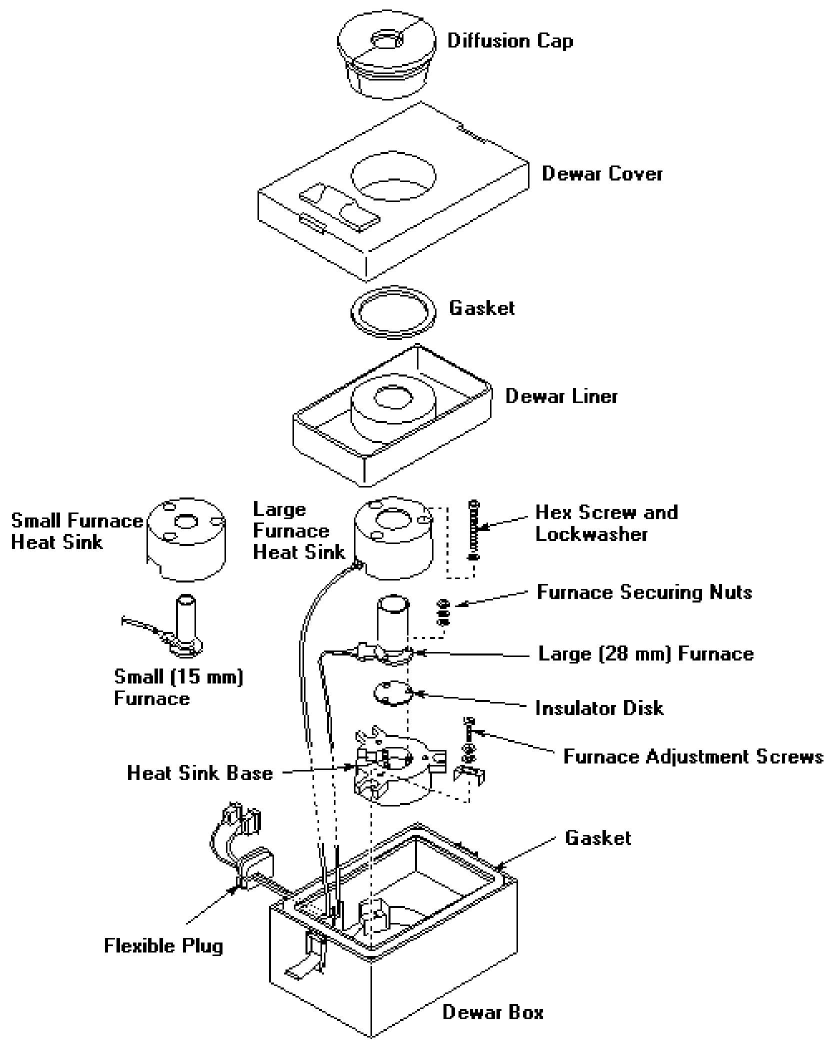
Replacing the furnace in the new dewar consists of three major steps:

- **Remove the Furnace from the New Dewar**
- **Install the Furnace in the New Dewar**
- **Align the Furnace in the New Dewar**

Furnace Removal from the New Dewar

See the schematic of the new dewar with furnace below and use it to follow the instructions below.

New Dewar Assembly with Furnace



1. Lower the dewar assembly and lock it into place at the base of the analyzer.
2. Remove the dewar cover by lifting the two latches on the sides of the dewar box and disconnecting them from the top cover.
3. Remove the two gaskets. One is on top of the dewar liner and the one is on top of the dewar box.
4. Lift the dewar liner out of the dewar box, exposing the heat sink.
5. Remove the three hex screws from the heat sink.
6. Lift the heat sink out of the dewar box, taking care not to lose the washers.
7. Unplug the furnace's 4-pin connector and the heat sink's 3-pin connector from the analyzer.
8. Remove the flexible plug from the rear of the dewar and guide the connectors through the hole.
9. Remove the three hex screws from the heat sink base and lift the base out of the dewar box.

10. Remove the three furnace securing nuts, lockwashers, and flat washers from the setscrews using a 1/4-in. open-end wrench.
11. Lift the furnace straight up and off of the setscrews.

NOTE: DO NOT remove the insulator disk. However, if it is broken, replace it.

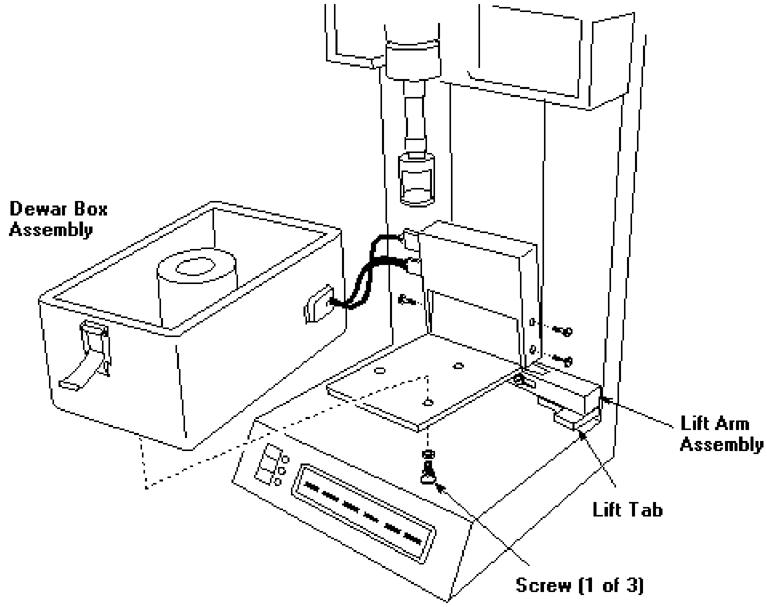
The next step is installing the furnace into the new dewar.

Furnace Installation into the New Dewar

1. Examine the insulator disk that is on top of the heat sink base. If the disk is broken, replace it with a new one.
2. Place the furnace onto the heat sink base by lining up the three holes on the furnace base with the three setscrews on the heat sink base.
3. Place a flat washer, a lockwasher, and a 1/4-in. hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since you could damage the furnace.

4. Thread the connectors for the furnace and the heat sink base through the hole in the back of the dewar box.
5. Place the heat sink base (with furnace attached) into the bottom of the dewar box.
6. Insert the three hex head alignment screws and secure them in place. Do not overtighten the screws since later on you will be moving the assembly to align the furnace.
7. Carefully place the heat sink onto the heat sink base. Take care not to bump the furnace; it is extremely fragile.
8. Place the three hex screws into the heat sink and tighten them.
9. Place the flexible plug over the wires in back of the dewar box. Press the plug into the hole at the back of the dewar box. When the plug is properly seated, it will snap into place.
10. Plug the heat sink’s 3-pin connector into the left side of the analyzer above the lift arm cover.
11. Attach the furnace’s 4-pin connector (P/N N519-0781) into the left side of the analyzer above the 3-pin connector.



12. While holding the dewar in place with your left hand, unlock the furnace latch with your right hand and raise the dewar and furnace into position. Make sure that it locks into position.

The sample tube should be in the center of the furnace. Proceed with aligning the furnace.

Furnace Alignment in the New Dewar

The final step in replacing the furnace in a new dewar in the DMA 7e or a DMA 7 retrofitted with the new dewar is aligning the furnace.

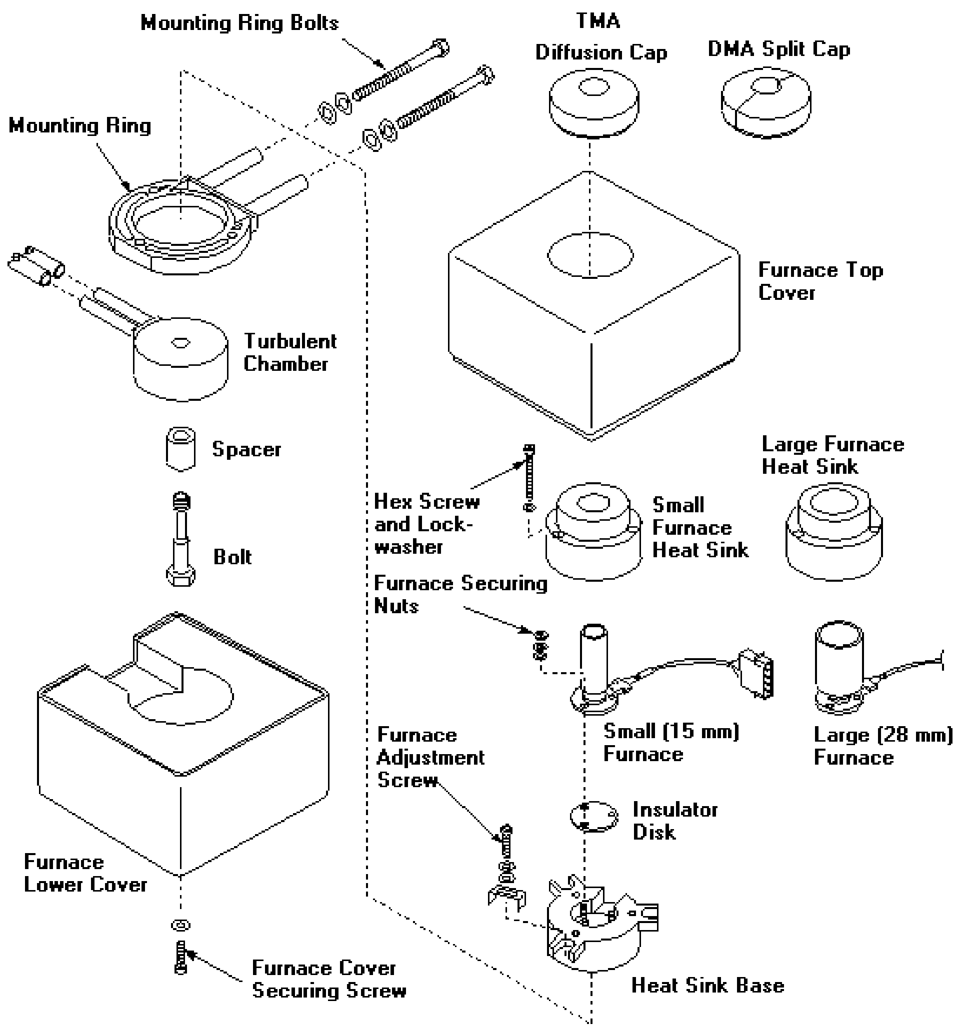
1. Loosen the three furnace adjustment screws that secure the heat sink base to the dewar box assembly.
2. Slowly elevate the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the tip of the sample tube but does not come into contact with it. Make sure that the furnace assembly is locked into the elevated position.
3. Continue making final adjustments to the furnace/heat sink assembly. Make sure that the sample tube is centered inside the furnace. When centering is complete, lock the furnace/heat sink assembly into place by tightening the three furnace adjustment screws.
4. Insert the dewar liner.
5. Place the rectangular gasket inside the dewar box and around the ridge between the dewar liner and the dewar box.
6. Place the circular flat gasket on top of the heat sink cover (which is part of the dewar liner).
7. Replace the dewar cover.
8. Visually inspect the circular gasket so that it does not extend outward toward the furnace. A good seal is necessary to sustain the low temperatures for an extended period.
9. Lock the dewar cover into place with the latches on the side of the dewar box.
10. Insert the funnel into the dewar opening if you are going to fill it with liquid nitrogen, or place the filler cover in place if you do not intend to fill the dewar immediately.

Furnace Replacement in Turbulent Chamber

The instructions below are for replacing the furnace in the turbulent chamber of a DMA 7e.

- Remove the Furnace from the Turbulent Chamber
- Install the Furnace in the Turbulent Chamber
- Align the Furnace in the Turbulent Chamber

Furnace Assembly with Turbulent Chamber in DMA 7e



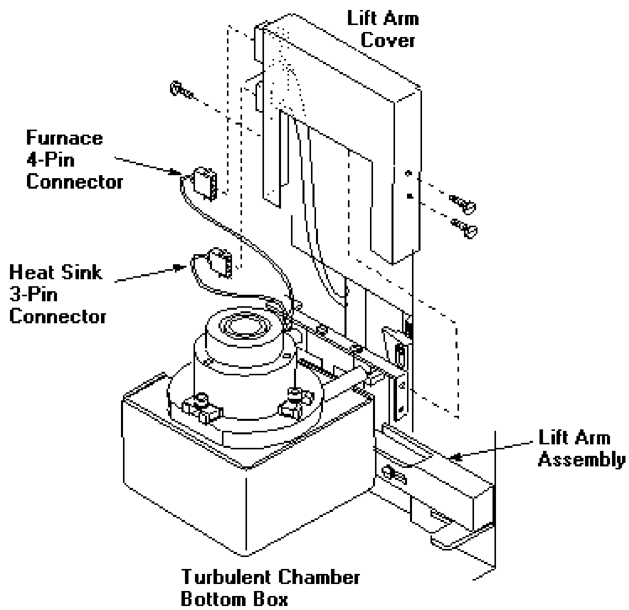
Furnace Removal from the Turbulent Chamber

See the display above of the furnace assembly with the turbulent chamber to refer to as needed as you perform the steps below.

The first step in replacing the furnace in the turbulent chamber in a DMA 7e is to remove the existing furnace:

1. Lower the furnace assembly and lock it in place at the base of the analyzer.

2. Remove the top furnace cover from the furnace assembly.
3. Unplug the 4-pin furnace connector from the analyzer.



4. Remove the three hex screws and the three lockwashers from the heat sink and lift off the heat sink.
5. Remove the three furnace securing nuts on the base of the furnace.
6. Lift the furnace straight up and off of the setscrews.

NOTE: Do not remove the insulator disk. If the disk is broken, replace it.

The next step in replacing the furnace in the turbulent chamber is installing the furnace.

Furnace Installation in the Turbulent Chamber

Install the new furnace into the turbulent chamber of the DMA 7e as follows:

1. Examine the insulator disk that is on top of the heat sink base. If it is broken, replace it.
2. Place the furnace over the three setscrews, with the furnace connector facing the rear of the analyzer.
3. Place a flat washer, a lockwasher, and hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since it can cause the furnace to break.

4. Attach the 4-pin furnace connector to the left side of the analyzer.
5. Install the heat sink. Use the three hex screws and lock washers that you removed from the heat sink earlier to secure the heat sink in place.

The final step in replacing the furnace in the turbulent chamber is alignment.

Furnace Alignment in the Turbulent Chamber

1. Loosen the three furnace adjustment screws which secure the heat sink base to the furnace lift arm.
2. Slowly raise the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the tip of the sample tube but does not come into contact with it. Make sure that the furnace assembly is locked into place in the highest position on the analyzer.
3. Continue to make final adjustments to the furnace/heat sink assembly. Make sure that the sample tube is centered inside the furnace. When centering is complete, lock the furnace/heat sink assembly into place by tightening the three furnace adjustment screws.
4. Place the cover over the turbulent chamber.

Replacing Large Furnace with Small Furnace

To replace a large furnace with a small furnace in a DMA 7e, see the appropriate subtopic for your system setup:

- **Furnace Replacement in the Turbulent Chamber**
- **Furnace Replacement in New Dewar in DMA 7 and DMA 7e**

Dewar Installation in the DMA 7

Before beginning the installation of the old dewar into the DMA 7, make sure that you have the tools required.

The parts included in the old DMA 7 Dewar Kit (P/N N519-0514) are

N519-0446	Cover Assembly
N519-1589	Dewar Insert
N519-1590	Dewar Mounting Screw
N519-0385	Dewar Box

Installation of the old dewar into the DMA 7 is a two-step process:

- **Turbulent Chamber Removal from DMA 7**
- **Install Old Dewar Assembly in DMA 7**

Tools Required for Maintenance Procedures

9/64-in. hex head driver (heat sink)

1/4-in. open end wrench (furnace)

3/8-in. open end wrench (heat sink mount)

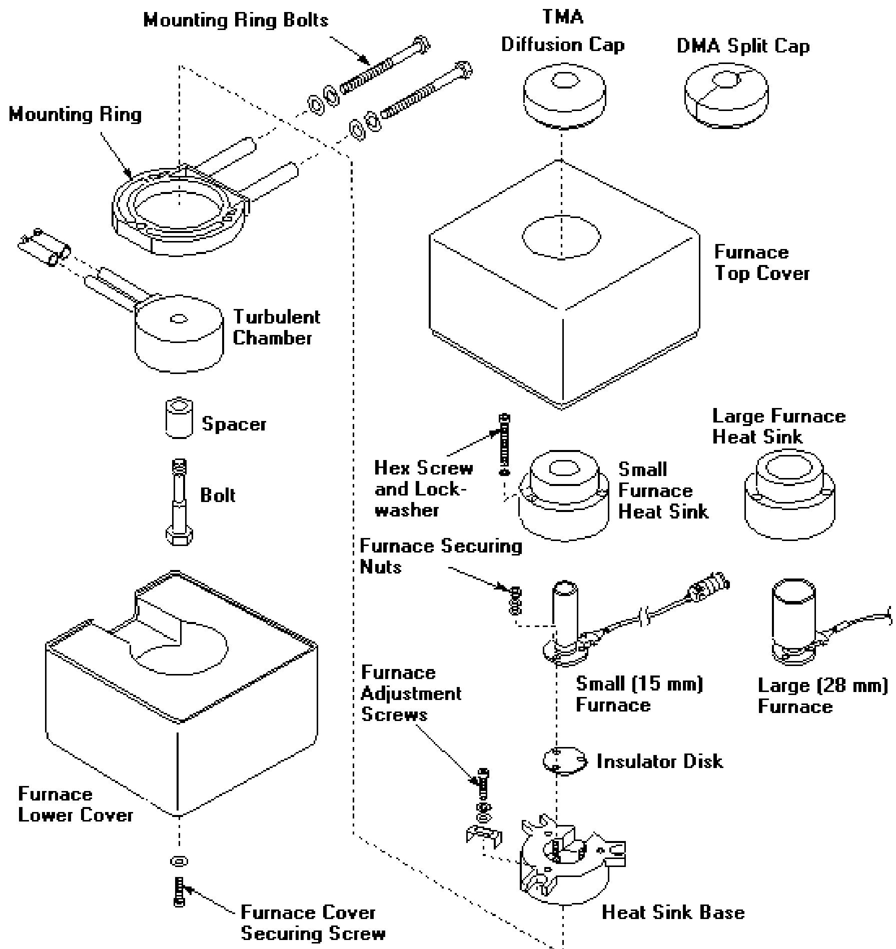
flat head screwdriver

Turbulent Chamber Removal from DMA 7

If you are using a turbulent chamber with a DMA 7, you must remove it before installing the old dewar assembly. Follow the steps below to perform this procedure.

See the schematic of the turbulent chamber below and use it to follow the instructions below.

Detail of DMA 7 with Turbulent Chamber

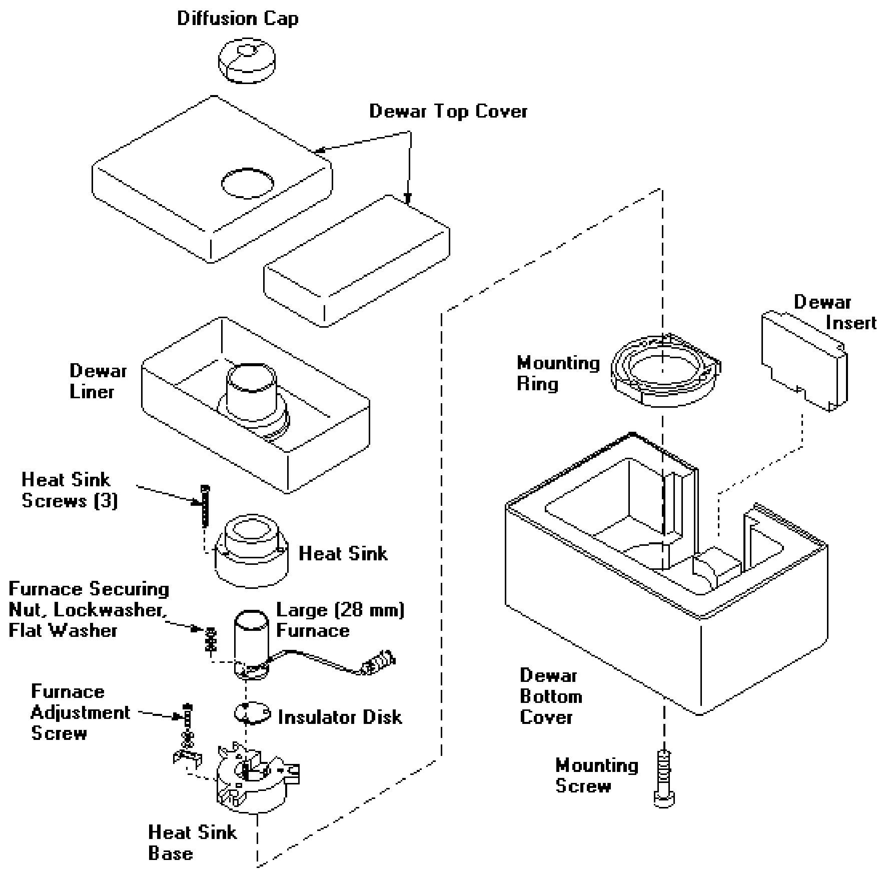


1. Remove the top furnace cover.
2. Using the Allen key hex wrench (P/N 0990-7278) provided in the Accessories Kit, remove the bottom furnace cover securing screw. Remove the bottom furnace cover.
3. Press the furnace locking mechanism and slowly lower the furnace.
4. Using an adjustable wrench, remove the turbulent chamber mounting bolt and spacer.

Install Old Dewar Assembly in DMA 7

See the diagram below showing alignment of furnace when using a dewar and use it to follow the instructions below.

Furnace Alignment with Dewar in DMA 7



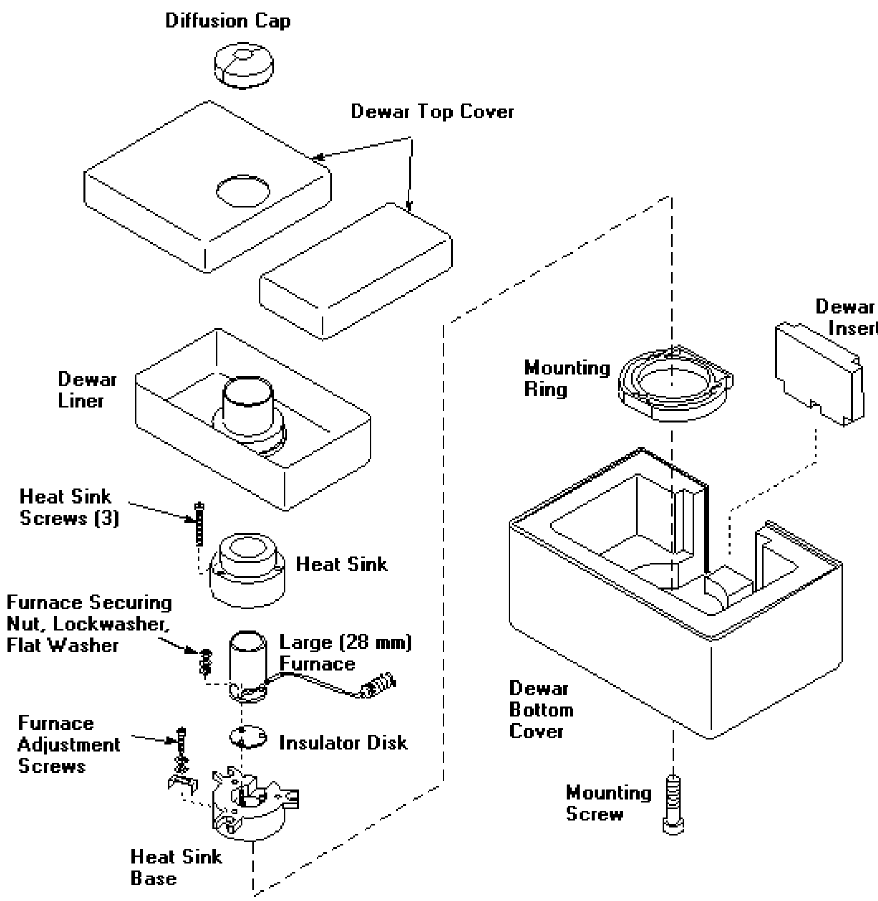
1. Press the furnace locking mechanism and slowly raise the furnace.
2. Hold the bottom dewar cover under the mounting ring and insert the mounting screw into the bottom of the dewar cover. Thread the screw into the heat sink base. Leave the screw loose enough to allow alignment of the dewar assembly.
3. Press the furnace locking mechanism and slowly lower the furnace.
4. Place the dewar insert inside the bottom dewar cover. Be sure to position the insert so that the small opening in the bottom allows room for the wires. The bottom dewar cover should now be around the mounting furnace ring.
5. Place the dewar box inside the dewar; place back the covers.
6. Raise the furnace and check the alignment of the assembly.
7. Align the furnace so that the sample tube fits into the center of the furnace. If necessary, loosen the furnace adjustment screws and gently move the furnace to center it.
8. Place the diffusion cap into the cover so that it rests on the heat sink.

Furnace Replacement in Old Dewar

These instructions apply to both the DMA 7 and the TMA 7. Replacing the furnace in the old style dewar involves three main steps:

- **Remove the Furnace from Old Dewar**
- **Install a New Furnace in Old Dewar**
- **Align the New Furnace in Old Dewar**

Furnace Assembly with Old Style Dewar



Remove the Furnace from Old Dewar

See the schematic above of the furnace assembly with the old style dewar and use it to follow the instructions below.

The first step in replacing the furnace in the old style dewar is to remove the furnace, following the procedure below:

1. Lower the dewar assembly and lock it in place at the base of the analyzer.
2. Remove the top dewar cover (two pieces) from the furnace assembly.
3. Loosen the three flathead screws from the lift arm cover (behind the furnace); remove the lift arm cover by lifting it straight up.

4. Remove the dewar liner inside the bottom dewar cover.
5. Remove the three hex screws and the three lockwashers from the heat sink and lift off the heat sink.
6. Remove the dewar insert from the back of the bottom dewar cover.
7. Rotate the bottom of the furnace connector plug to unlock it. Unplug the furnace connector from the analyzer.
8. While holding the dewar in place with your left hand, use your right hand to unlock the furnace latch and raise the dewar and furnace into position. Make sure that the dewar locks into position.
9. Remove the bottom dewar cover from the furnace assembly by unscrewing the nylon mounting screw.
10. Lower the furnace assembly.
11. Remove the three furnace securing nuts on the base of the furnace.
12. Lift the furnace straight up and off of the setscrews.

NOTE: Do not remove the insulator disk. If the disk is broken, however, replace it.

Now you can install a new furnace into the old dewar.

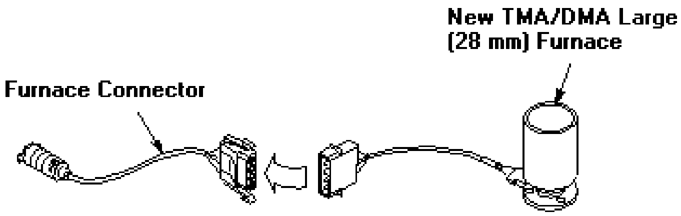
Install a New Furnace in Old Dewar

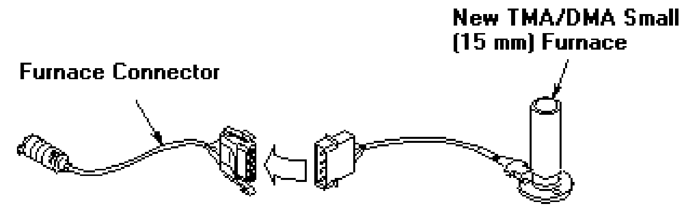
Install a new furnace into an old style dewar as follows:

1. Examine the insulator disk that is on top of the heat sink base. If it is broken, replace it.
2. Place the new furnace over the three setscrews, with the furnace connector facing the rear of the analyzer.
3. Place a flat washer, a lock washer, and a hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since that could break the furnace.

4. Attach the 4-pin connector (P/N N519-0781 in the furnace kit) to the furnace connector, if necessary.





NOTE: If you are inserting an old style furnace into an old style dewar, the 4-pin connector is not used.

5. Plug the round furnace connector into the analyzer. One of the pins is larger than the rest, so the connector can be plugged in in only one way.
6. Install the heat sink using the three hex screws and lockwashers that you removed earlier.
7. Install the lift arm cover and secure it with the three flathead screws.

Now you can align the furnace.

Align the New Furnace in Old Dewar

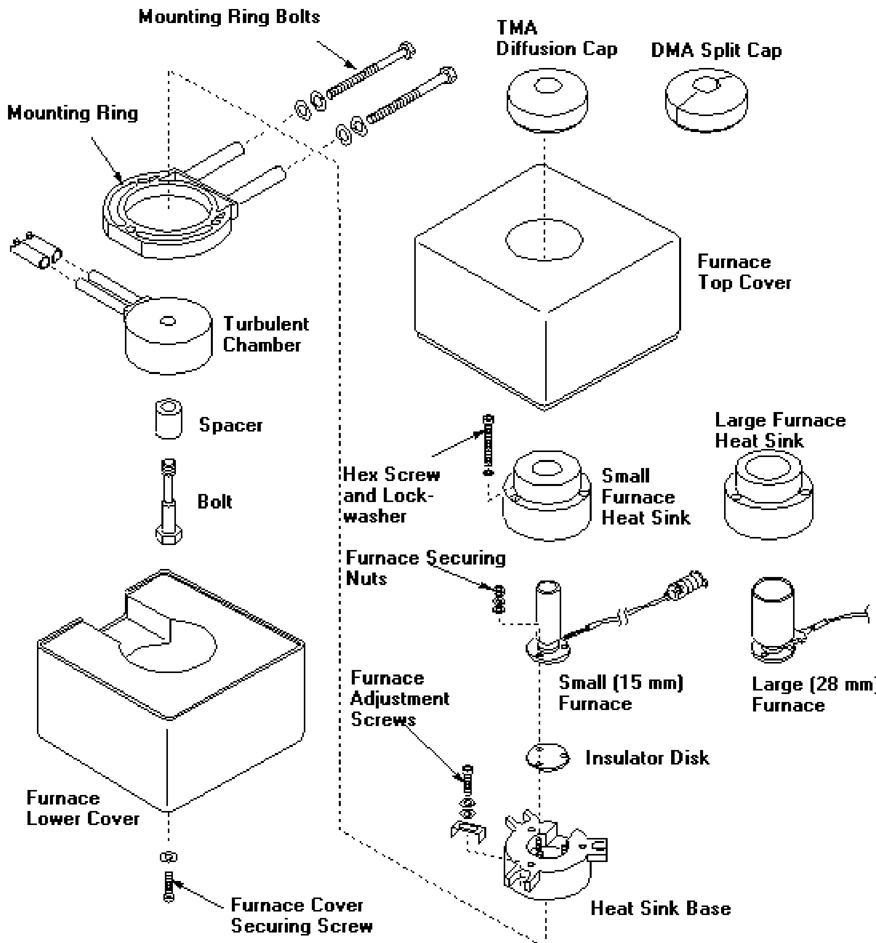
1. Loosen the three furnace adjustment screws that secure the heat sink base to the furnace lift arm.
2. Slowly raise the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the sample tube but does not come in contact with it. Make sure that the furnace assembly is locked into place.
3. Continue to make final adjustments to the furnace/heat sink assembly making sure that the sample tube is centered inside the furnace. When completely centered, lock the furnace/heat sink assembly into place by tightening the three furnace adjustment screws.
4. Replace the bottom dewar cover and secure it in place by securing the nylon mounting screw to the underside of the bottom dewar cover. Make sure that the mounting screw is tight.
5. Lower the dewar assembly and lock it in place.
6. Replace the dewar insert, dewar liner, and covers.

Furnace Replacement in Turbulent Chamber of DMA 7/TMA 7

The instructions below are for replacing the furnace in the turbulent chamber of a DMA 7 or older TMA 7.

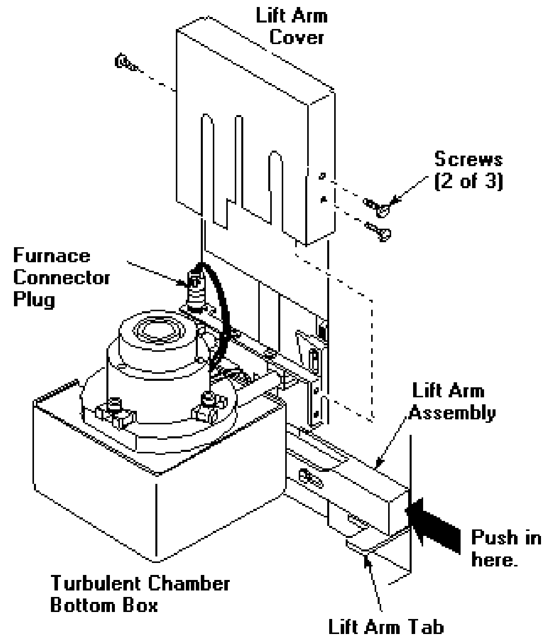
- **Remove the Furnace from Turbulent Chamber**
- **Install the Furnace in Turbulent Chamber**
- **Align the Furnace in Turbulent Chamber**

Furnace Assembly with Turbulent Chamber

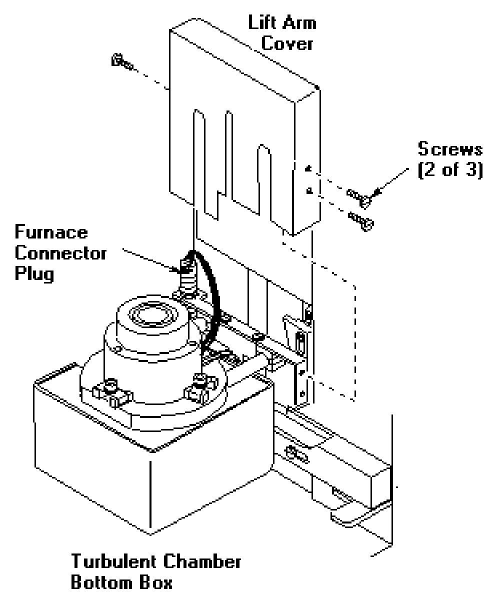


Furnace Removal from the Turbulent Chamber

1. Lower the furnace assembly and lock it in place at the base of the analyzer.



2. Remove the top furnace cover from the furnace assembly.
3. Loosen the three screws from the lift arm cover (behind the furnace) and then remove the lift arm cover by lifting it straight up.



4. Remove the three hex screws and the three lock washers from the heat sink and lift and remove the heat sink.
5. Rotate the bottom of the furnace connector plug to unlock it. Unplug the furnace connector from the analyzer.

6. Remove the three furnace securing nuts on the base of the furnace.
7. Lift the furnace straight up and off of the setscrews.

NOTE: Do not remove the insulator disk. If it is broken, however, replace it.

Now you can install the furnace into the turbulent chamber.

Furnace Installation into the Turbulent Chamber

Install the new furnace into the turbulent chamber as follows:

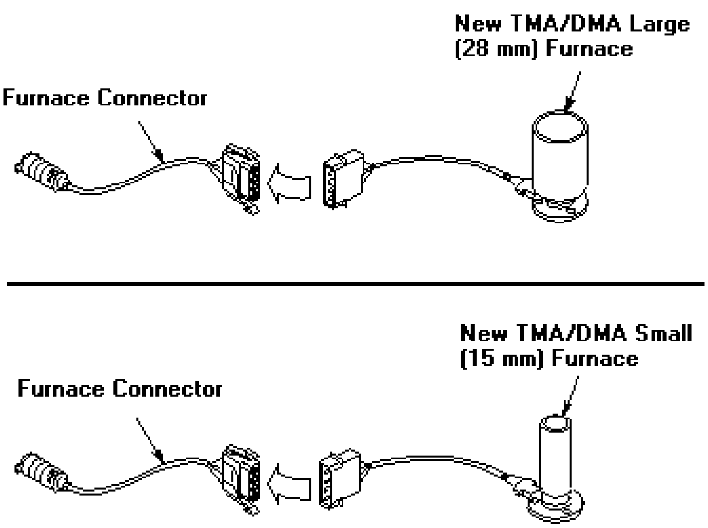
NOTE: If you are replacing the furnace with a new style furnace, the connectors will be different. Remember to use the 4-pin adapter cable that is included with the furnace kit.

1. Examine the insulator disk that is on top of the heat sink base. If it is broken, replace it.
2. Place the furnace over the three setscrews, with the furnace connector facing the rear of the analyzer.
3. Place a flat washer, a lockwasher, and a hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since it cause the furnace to break.

4. Attach the 4-pin connector (P/N N519-0781) that was supplied with the furnace kit to the furnace connector if necessary.

NOTE: If you are installing an old style furnace into the turbulent chamber, the 4-pin connector is not used.



5. Plug the round furnace connector into the analyzer. One of the pins is larger than the others so the connector can be plugged in only one way.

6. Install the appropriately sized heat sink [the large heat sink is used with the large (28 mm) furnace and the small heat sink is used with the small (15 mm) furnace]. Use the three hex screws and lockwashers that you removed from the heat sink earlier.
7. Install the lift arm cover and secure it with the three flathead screws.

The furnace now needs to be aligned.

Furnace Alignment in the Turbulent Chamber

After installing the furnace into the turbulent chamber, it must be aligned as follows:

1. Loosen the three furnace adjustment screws that secure the heat sink base to the furnace lift arm.
2. Slowly raises the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the sample tube but does not come into contact with it. Make sure that the furnace assembly is locked into place at the highest position on the analyzer.
3. Continue making final adjustments to the furnace/heat sink assembly. Make sure that the sample tube is centered inside the furnace. When centering is complete, lock the furnace/heat sink assembly in place by tightening the three furnace adjustment screws.
4. Place the cover over the turbulent chamber.

Replacing Large Furnace with Small Furnace in DMA 7

To change from the large furnace to the small furnace in the DMA 7, see the appropriate topic depending on your system setup:

- **Furnace Replacement in Old Dewar**
- **Furnace Replacement in Turbulent Chamber of DMA 7/TMA 7**
- **Furnace Replacement in the New Dewar in the DMA 7 and the DMA 7e**

Upgrade DMA 7 with New Dewar

The DMA 7 analyzer shipped prior to July 1, 1993, is considered the older of the DMAs. A Dewar Retrofit Kit is available from Perkin Elmer for those users who want to upgrade their DMA 7 for use with the new dewar. The new style dewar provides extended LN2 residence time, allowing you to perform longer experiments. The kit must be installed by a Perkin Elmer Service Representative.

Replacing the Thermocouple

A thermocouple is a device that generates a differential voltage as temperature is increased or decreased. The voltage is generated by fusing wires made from two different metals at the tip. When this device is repeatedly flexed, the point at which the two wires are fused is stressed and can occasionally fail. Restricting the movement of the thermocouple can greatly improve its service life. The high-temperature tape, provided in the Spares Kit, restricts the movement of the thermocouple when placed at the top and bottom of the sample tube. This tape also serves as an insulator.

Although the calibration software can compensate for thermocouple placement, careful routing and placement of the thermocouple can improve thermal performance of the analyzer, especially when stainless steel measuring systems are used.

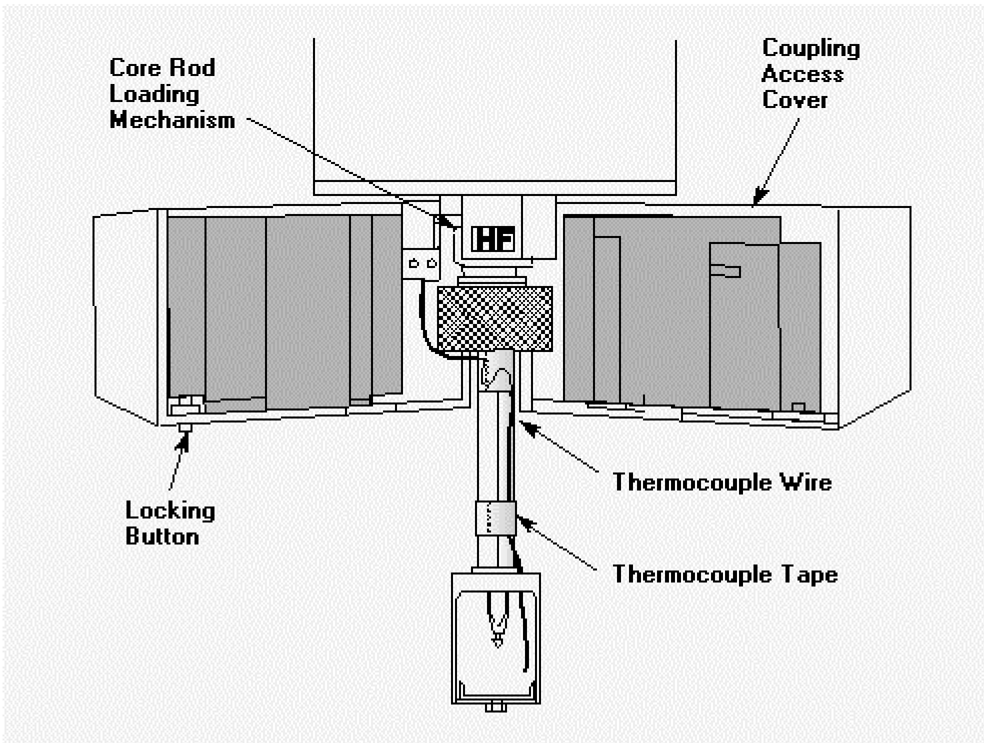
CAUTION: The thermocouple should not touch any metal parts.

To replace the sample thermocouple, you must first remove the old thermocouple and then install the new one.

Removing the Thermocouple

NOTE: Before performing this procedure, shut the system down and turn off the analyzer. Remove the line power from the analyzer. Make sure that the furnace, sample tube, and probe are sufficiently cooled so that you do not burn yourself.

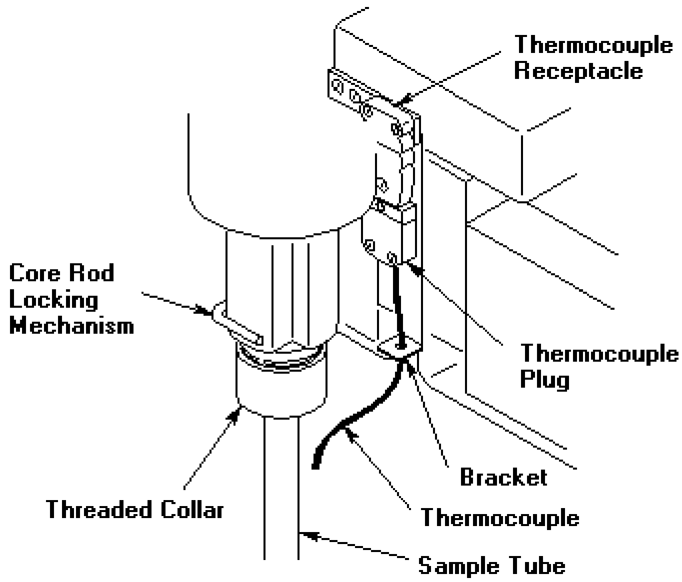
1. Remove the furnace diffusion cap and carefully lower the furnace assembly to the base of the analyzer by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
2. Press the locking button that secures the coupling access covers and separate the doors.



3. Remove the thermocouple from the sample tube by grasping the thermocouple just above the point where it enters the sample tube and then pulling it straight out.

NOTE: The thermocouple may be taped to the sample tube. This is usually done to provide consistent thermocouple positioning in the measuring system.

4. Unplug the thermocouple from the receptacle at the back of the probe assembly by gently grasping the sides of the thermocouple connector and pulling the connector straight down.
5. Pull the thermocouple up through the bracket.



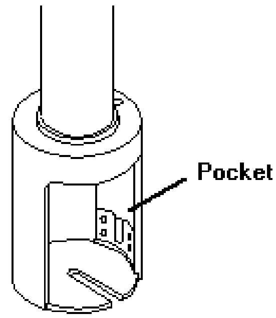
Installing the Thermocouple

Follow the instructions below when installing for the first time or replacing the DMA 7e sample thermocouple.

1. Take the new thermocouple and plug it into the receptacle at the back of the probe assembly. Observe the polarity (+ or –) as marked on the plugs.
2. Apply a piece of tape along the length of the sample tube. This insulates the thermocouple from the sample tube.
3. Route the thermocouple along side of the sample tube on top of the tape, making sure to include the loop at the top to prevent tip movement.

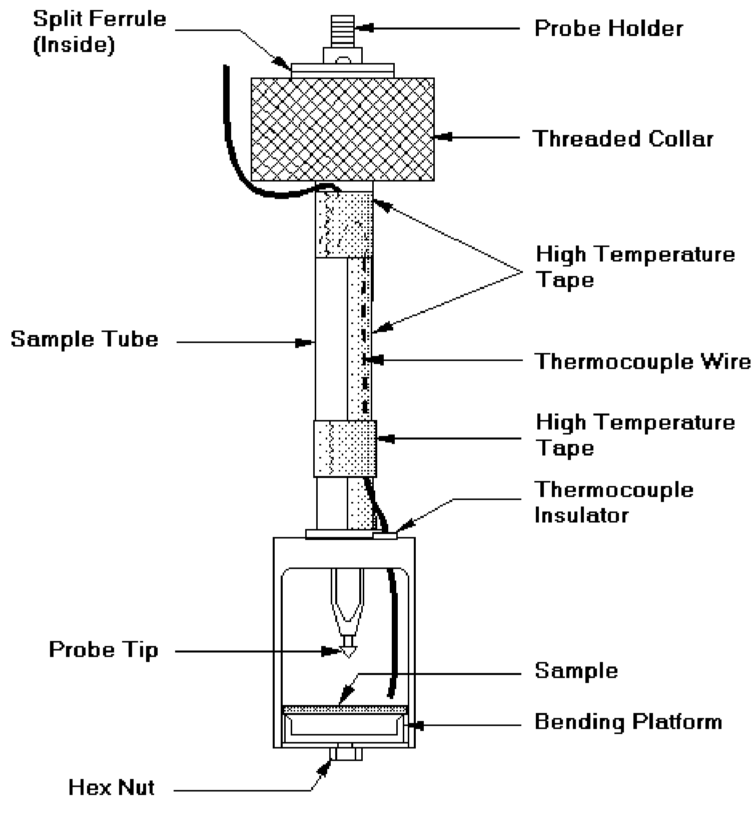
CAUTION: Do not route the thermocouple through the mounting bracket. This allows you to remove the sample tube from the analyzer without disturbing the thermocouple route or position. Remember, do not make any sharp bends in the thermocouple. This may damage the thermocouple.

4. Place the thermocouple insulator (a white ceramic sleeve about 2 mm long and 1 mm wide) in the hole in the top of the base of the sample tube.
5. Route the tip of the thermocouple through the insulator.
6. If you are using the stainless steel sample tube (P/N N539-0107), place the tip of the thermocouple all the way down into the bottom of the thermocouple pocket on the inside side wall of the sample tube.



If you are using the standard quartz sample tube, the thermocouple should extend straight down the tube’s sidewall and rest near the platform surface without touching the sample, the probe, or the sample holder.

7. Apply another piece of tape over the thermocouple and over the first piece of tape. This will hold the thermocouple in place.
8. Apply a piece of tape around the thermocouple at the top and bottom of the sample tube. This will secure the thermocouple.



NOTE: The top of the sample tube does not reach extreme temperatures during normal use. However, the adhesive on the high-temperature tape may age over time or if very high temperatures over long periods of time are used. Positioning the lower tape wrap approximately 1 cm from the thermocouple insulator should reduce the aging of the tape adhesive. Replace the tape if discoloration or other signs of wear appear.

9. Slowly close the coupling access covers, making sure that you do not pinch the thermocouple in the doors as they are closed.
10. The thermocouple should exit through the rear of the access covers. Notice that a space is present at the back of the access covers where the thermocouple can pass without being bent or pinched.

The thermocouple installation is now complete.

Maintenance of Sample Tubes and Probes

Maintenance of the sample tube and probe of the DMA 7e entails:

- **Removing the Sample Tube and Probe**
- **Cleaning the Sample Tube and Probe**
- **Replacing the Sample Tube and Probe**

These are topics in Pyris Multimedia Presentations Help.

Cleaning Sample Tubes and Probes

To clean the quartz sample tube and probe, follow the instructions below:

WARNING: **Wear protective gloves and safety glasses when cleaning the sample tube and probe. Use a fume hood when working with solvents or cleaning solutions.**

1. For organic contaminants, insert the tip of the probe or the base of the sample tube over a Bunsen burner for several seconds.

CAUTION: Heat the quartz glassware for only a few seconds. Prolonged heating could damage the glassware.

2. In some cases, it may be necessary to dissolve contaminants from the glassware using suitable solvents. Dip the glassware in the solvent to dissolve the contaminant.

Accessories, Replacement Parts, and Parts Provided

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

See the following topics for detailed lists of parts and accessories for the DMA 7e:

Measuring Systems Parts Lists

TAC 7/DX Interface

Analyzer Accessories

Measuring Systems Parts Lists

The parts list for each of the measuring systems available for the DMA 7e and its associated illustration showing the parts are seen in the following:

- **Stainless Steel 3-Point Bending Kit (P/N N539-0130)**
- **Stainless Steel Dual Cantilever Kit (P/N N539-0131)**
- **Stainless Steel Extension Kit (P/N N539-0132)**
- **Stainless Steel Parallel Plate Kit (P/N N519-0133)**
- **Quartz Extension Kit (P/N N539-0134)**
- **Quartz Large Parallel Plate Kit (P/N N519-0135)**
- **Quartz Large Bending Kit (P/N N539-0136)**
- **Quartz Small Bending Kit (P/N N539-0137)**
- **Quartz Small Parallel Plate Kit (P/N N519-0138)**

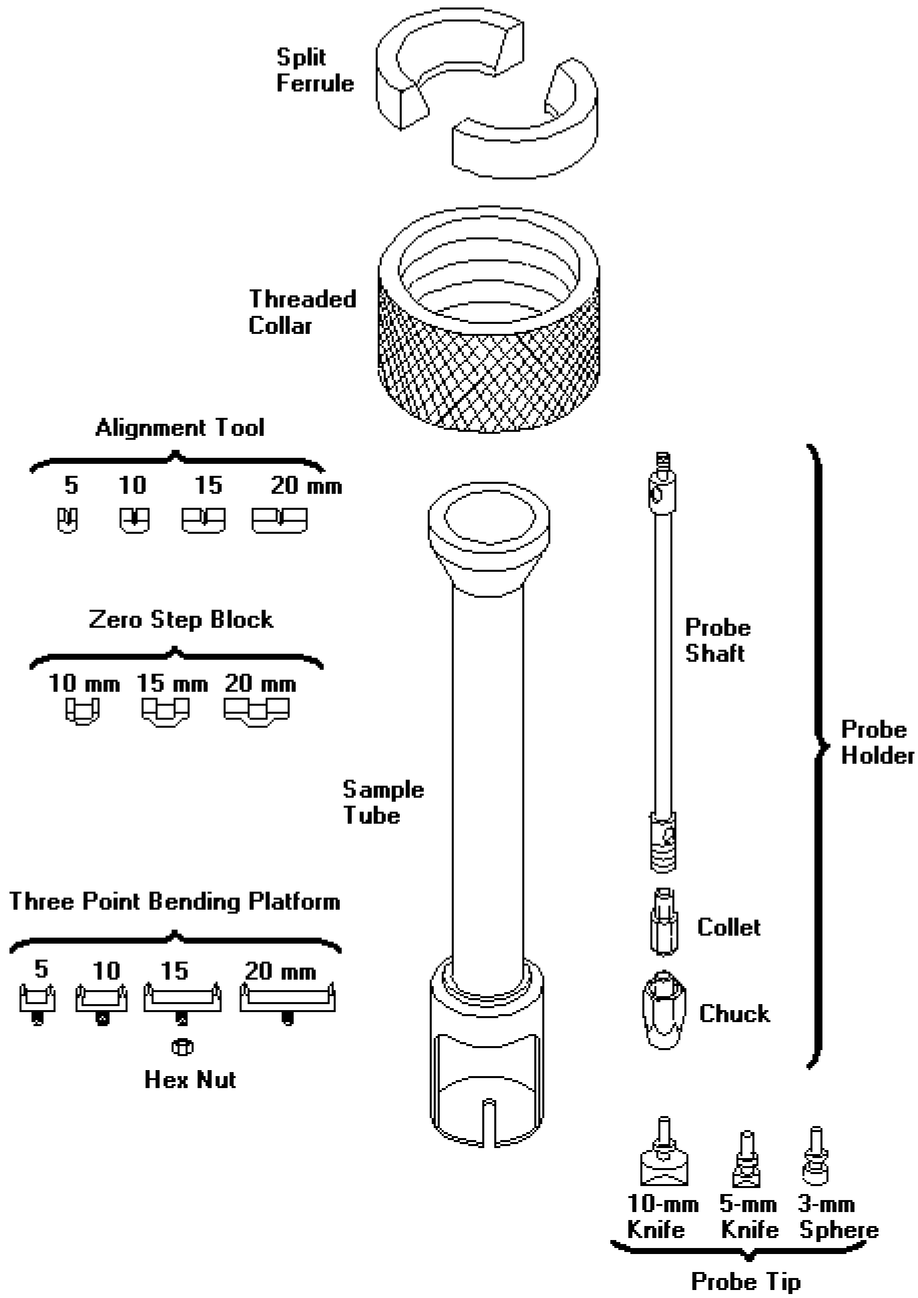
Stainless Steel 3-Point Bending Kit (P/N N539-0130)

Item	Part No.	Description	Quantity
1	0991-1173	Nut 4-40 S/S	7
2*	0991-1243	#4 Flat Washer	3
3*	0991-1285	Split Lockwasher	3
4	0993-8545	Instructions	1
5	N519-1662	Sample Tube Ferrule	1
6	N539-0101	15-mm Bending Platform	1
7	N539-0102	5-mm Bending Platform	1
8	N539-0105	Probe Holder	1
9	N539-0107	Sample Tube	1
10	N539-0122	20-mm Bending Platform	1
11*	N529-0142	Case Assembly	1
12	N539-0197	10-mm Bending Platform	1
13	N539-1016	10-mm Knife-Edge Probe Tip	1
14	N539-1017	3-mm Round Probe Tip	1
15	N539-1054	Sample Tube Collar	1

16	N539-1063	3-mm Round Probe Tip	1
17*	N539-1075	Nameplate	1
18	N539-1090	20-mm Zero Step Block	1
19	N539-1093	20-mm Alignment Tool	12
20	N539-1094	5-mm Alignment Tool	1
21	N539-1095	15-mm Alignment Tool	1
22	N539-1104	15-mm Zero Step Block	1
23	N539-1183	10-mm Alignment Tool	1
24	N539-1184	10-mm Zero Step Block	1

* = Item not shown in illustrated parts list.

Stainless Steel 3-Point Bending Kit (N539-0130) Illustrated Parts

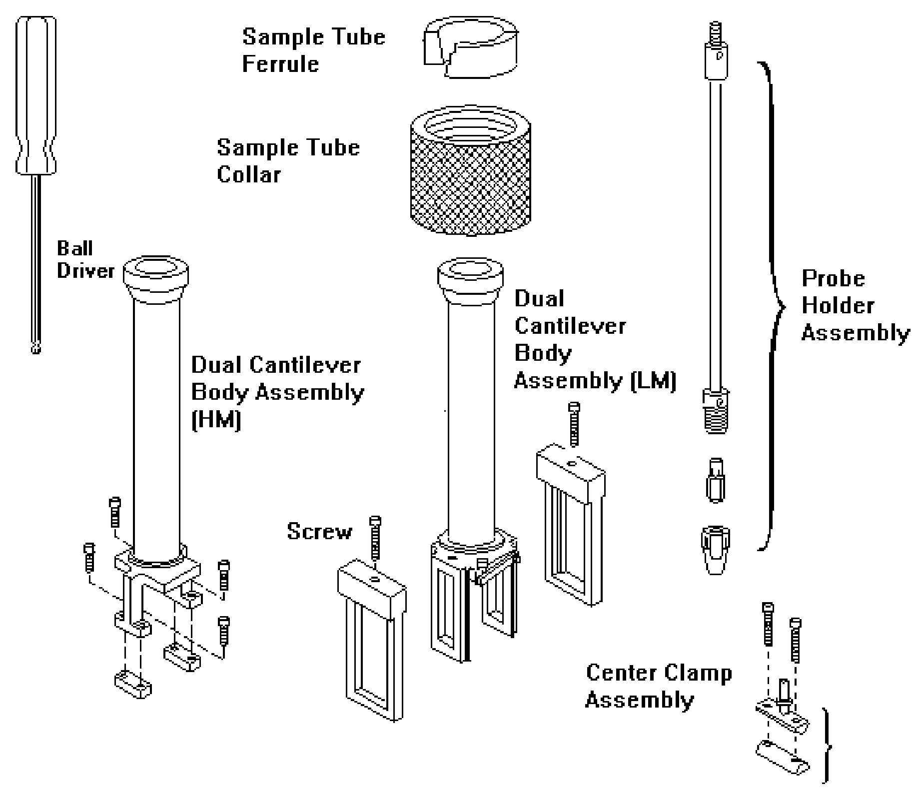


Stainless Steel Dual Cantilever Kit (P/N N539-0131)

Item	Part No.	Description	Quantity
1	0991-0469	Screw, Socket Head 2-56 x 1/2 in.	10
2*	0993-8548	Instructions	1
3	N519-1662	Sample Tube Ferrule	2
4	N539-0105	Probe Holder Assembly	1
5*	N539-0142	Case Assembly	1
6	N539-0174	Dual Cantilever Body Assy (LM)	1
7	N539-0175	Center Clamp Assembly	1
8	N539-0176	Dual Cantilever Body Assy (HM)	1
9	N539-1054	Sample Tube Collar	2
10*	N539-1073	Spring Clip	1
11*	N539-1076	Nameplate	1
12	N539-1083	Ball Driver, 5/64 Hex Tip	1

* = Item not shown in illustrated parts list.

Stainless Steel Dual Cantilever Kit (N539-0131) Illustrated Parts

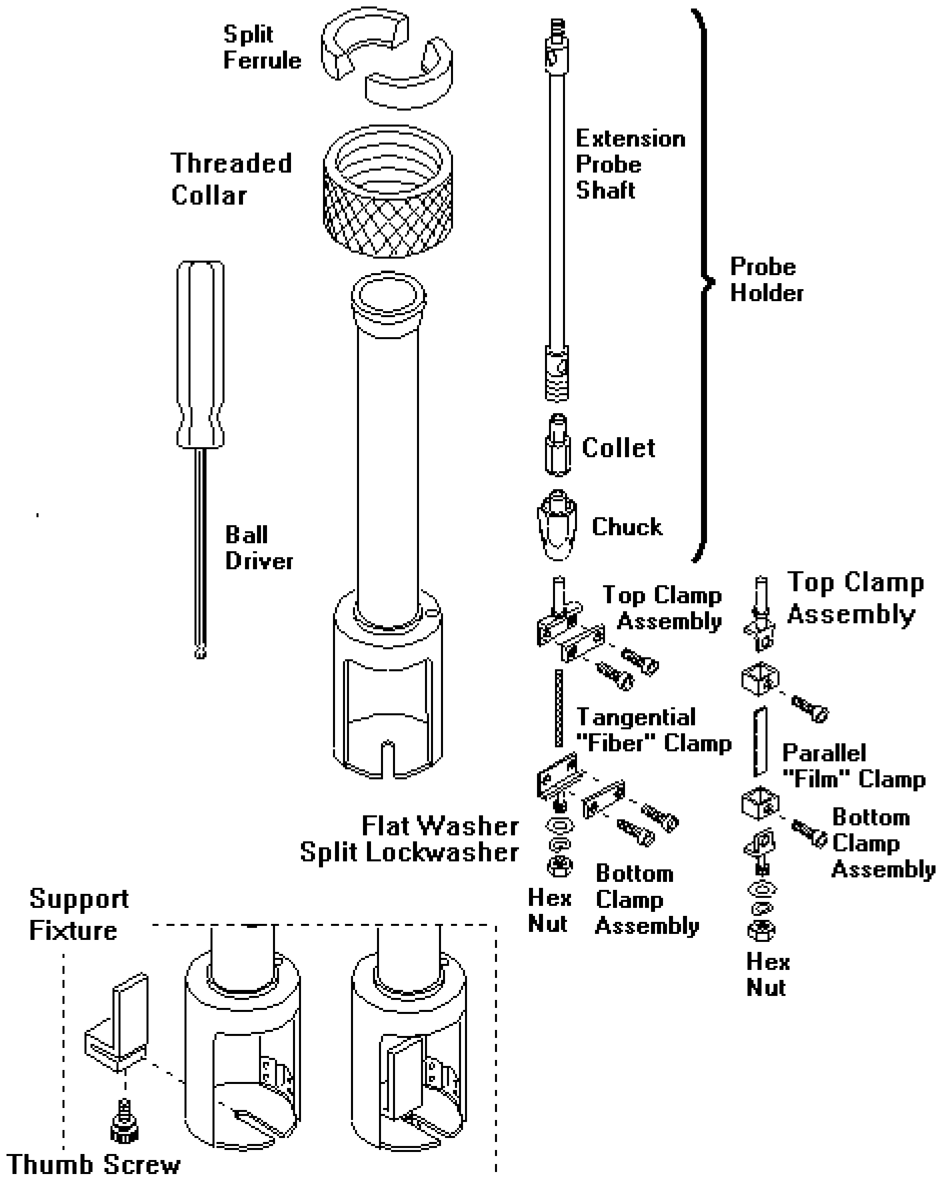


Stainless Steel Extension Kit (P/N N539-0132)

Item	Part No.	Description	Quantity
1	0991-1173	Nut 4-40 S/S	4
2	0991-1243	Flat Washer	2
3	0991-1285	Split Lockwasher	2
4	0991-1902	Socket Hd. Cap Screw	6
5	0993-8546	Instructions	1
6	N519-1662	Sample Tube Ferrule	1
7	N519-2096	Support Fixture	1
8	N519-2097	Thumb Screw	1
9	N539-0107	Sample Tube	1
10*	N539-0142	Case Assembly	1
11	N539-0154	Film Extension Top Clamp Assy	1
12	N539-0156	Film Extension Bottom Clamp Assy	1
13	N539-0177	Fiber Extension Bottom Clamp Assy	1
14	N539-0178	Fiber Extension Top Clamp Assy	1
15	N539-0193	Probe Holder Assembly	1
16	N539-1054	Sample Tube Collar	1
17*	N539-1078	Nameplate	1
18	N539-1083	Ball Driver, Hex Tip 5/64	1

* = Item not shown in illustrated parts list.

Stainless Steel Extension Kit (N539-0132) Illustrated Parts

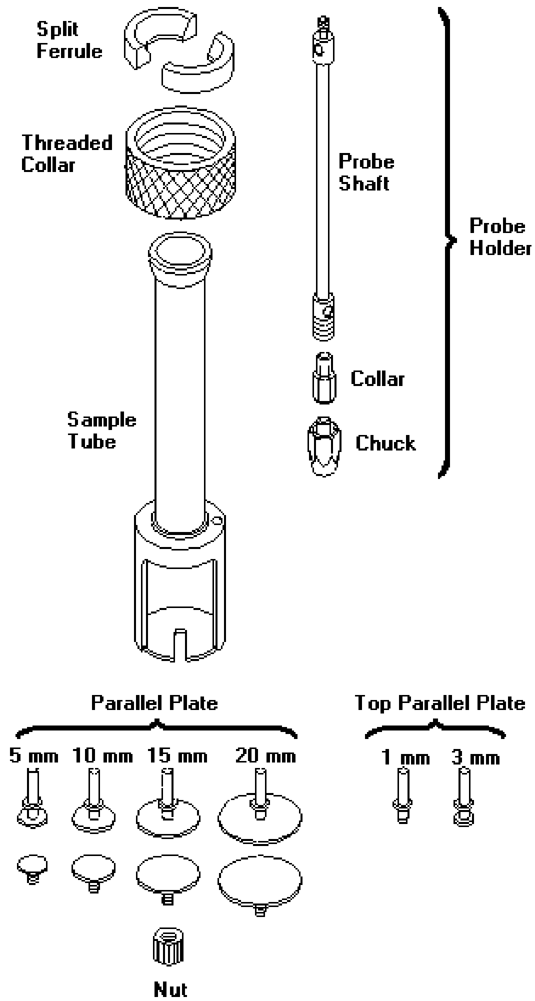


Stainless Steel Parallel Plate Kit (P/N N539-0133)

Item	Part No.	Description	Quantity
1	0993-8547	Instructions	1
2*	N519-1173	Nut 4-40 S/S	4
3	N519-1662	Sample Tube Ferrule	1
4	N539-0105	Probe Holder	1
5	N539-0107	Sample Tube	1
6*	N539-0142	Case Assembly	1
7	N539-0143	5-mm Parallel Plate Assy	1
8	N539-0144	10-mm Parallel Plate Assy	1
9	N539-0145	15-mm Parallel Plate Assy	1
10	N539-0146	20-mm Parallel Plate Assy	1
11	N539-1014	1-mm Top Parallel Plate	1
12	N539-1015	3-mm Top Parallel Plate	1
13	N539-1054	Sample Tube Collar	1
14*	N539-1077	Nameplate	1

* = Item not shown in illustrated parts list.

Stainless Steel Parallel Plate Kit (N539-0133) Illustrated Parts

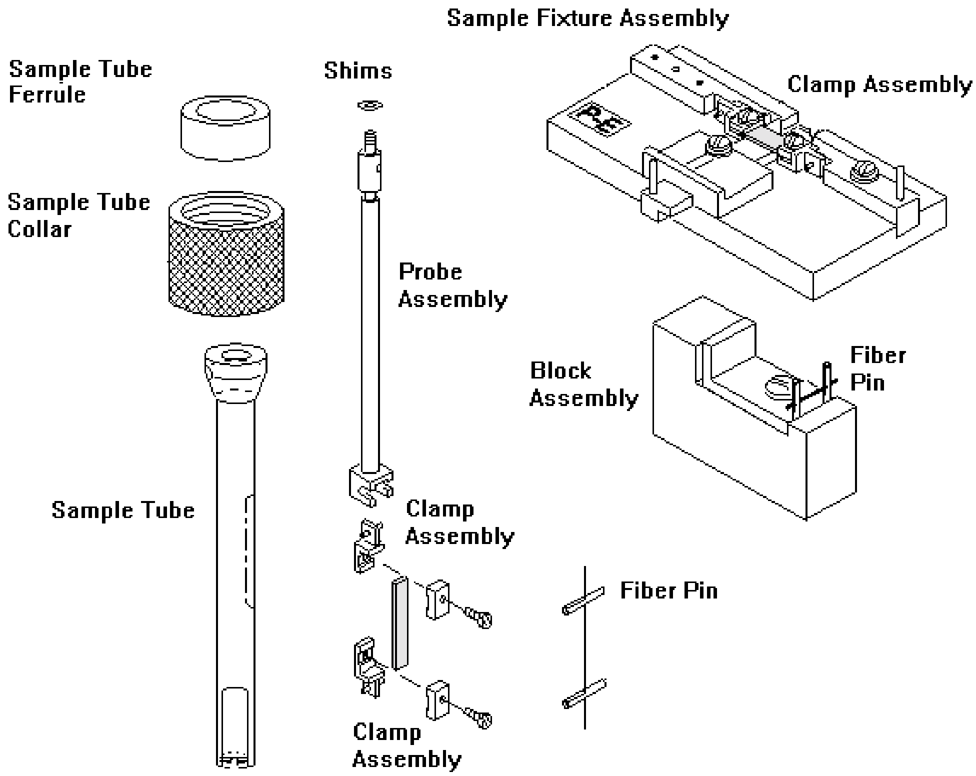


Quartz Extension Kit (P/N N539-0134)

Item	Part No.	Description	Quantity
1	0990-7245	Sample Pliers	1
2	0993-8554	Instructions	1
3	N519-0609	Probe Assembly	1
4	N519-0611	Clamp Assembly	1
5	N519-0615	Sample Fixture Assembly	1
6	N519-1532	Sample Tube Ferrule	1
7	N519-1700	Sample Tube	1
8	N519-1781	45° Shim	12
9	N519-1782	90° Shim	12
10*	N539-0142	Case Assembly	1
11	N539-0482	Block Assembly	1
12	N539-1054	Stainless Steel Collar	1
13*	N539-1078	Nameplate	1
14	N539-1089	Fiber Pin	100
15*	N539-1205	Retainer	1
16*	N539-1206	Setup Spacer	1

* = Item not shown in illustrated parts list.

Quartz Extension Kit (N539-0134) Illustrated Parts

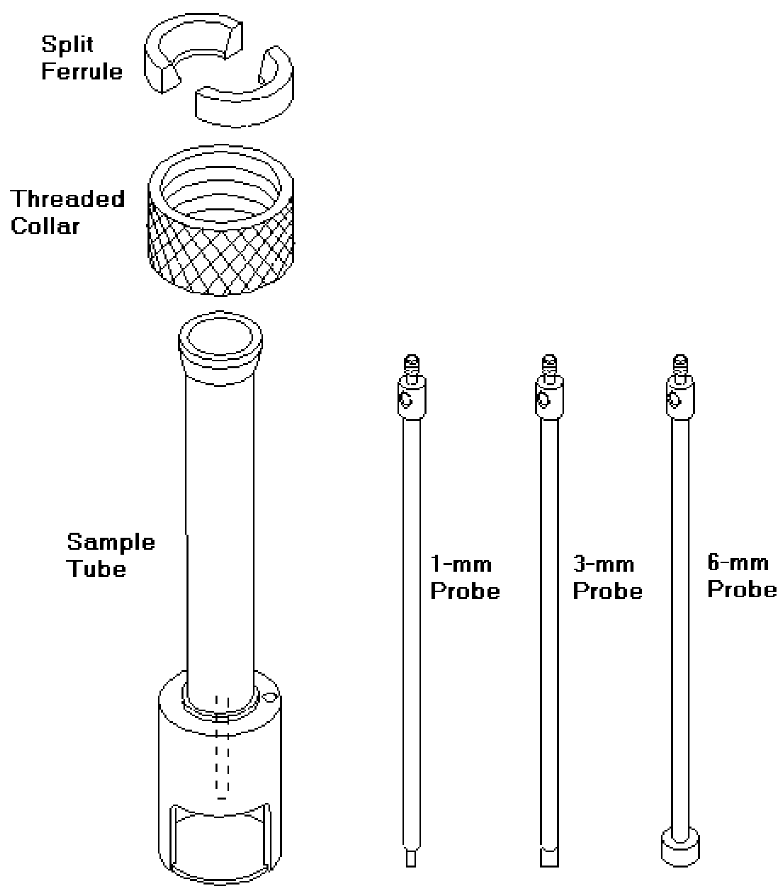


Quartz Large Parallel Plate Kit (P/N N519-0135)

Item	Part No.	Description	Quantity
1*	0009-0652	Septum (pkg. of 50)	1
2*	0993-8550	Instructions	1
3	N519-0376	1-mm Parallel Plate Probe	1
4	N519-0378	3-mm Parallel Plate Probe	1
5	N519-0606	6-mm Parallel Plate Probe	1
6	N519-1662	Sample Tube Ferrule	1
7	N519-1837	Sample Tube	1
8*	N539-0142	Case Assembly	1
9*	N539-1077	Nameplate	1
10	N539-1054	Sample Tube Collar	1

* = Item not shown in illustrated parts list.

Quartz Large Parallel Plate Kit (N539-0135) Illustrated Parts

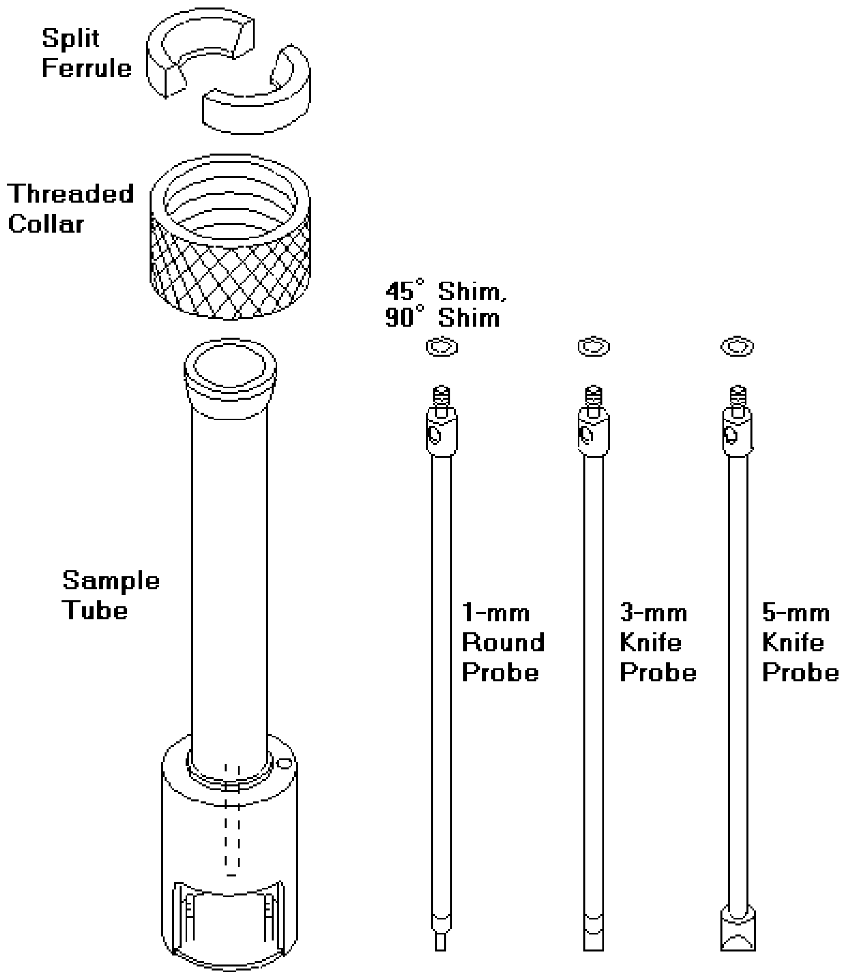


Quartz Large Bending Kit (P/N N539-0136)

Item	Part No.	Description	Quantity
1*	0009-0652	Septum (pkg. of 50)	12
2*	0993-8551	Instructions	1
3	N519-0377	1-mm Round Probe	1
4	N519-0393	3-mm Knife Probe	1
5	N519-0614	5-mm Knife Probe	1
6	N519-1472	Sample Tube	1
7	N519-1662	Sample Tube Ferrule	1
8	N519-1781	45° Shim	12
9	N519-1782	90° Shim	12
10*	N529-0142	Case Assembly	1
11	N539-1054	Sample Tube Collar	1
12*	N539-1075	Nameplate	1

* = Item not shown in illustrated parts list.

Quartz Large Bending Kit (N539-0136) Illustrated Parts

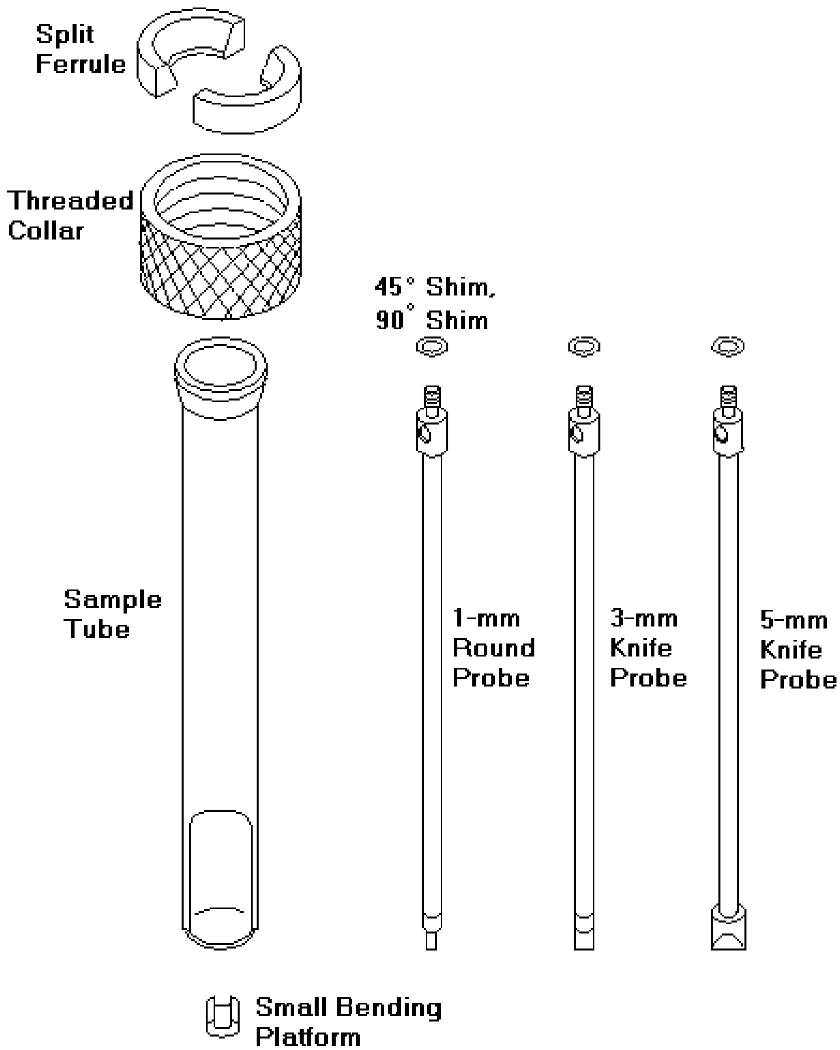


Quartz Small Bending Kit (P/N N539-0137)

Item	Part No.	Description	Quantity
1*	0009-0652	Septum (pkg. of 50)	1
2	0219-0215	Small Bending Platform	1
3*	0993-8553	Instructions	1
4	N519-0377	1-mm Round Probe	1
5	N519-0393	3-mm Knife Probe	1
6	N519-0614	5-mm Knife Probe	1
7	N519-1532	Sample Tube Ferrule	1
8	N519-1549	Sample Tube	1
9	N519-1781	45° Shim	12
10	N519-1782	90° Shim	12
11*	N539-0142	Case Assembly	1
12	N539-1054	Sample Tube Collar	1
13*	N539-1075	Nameplate	1

* = Item not shown in illustrated parts list.

Quartz Small Bending Kit (N539-0137) Illustrated Parts

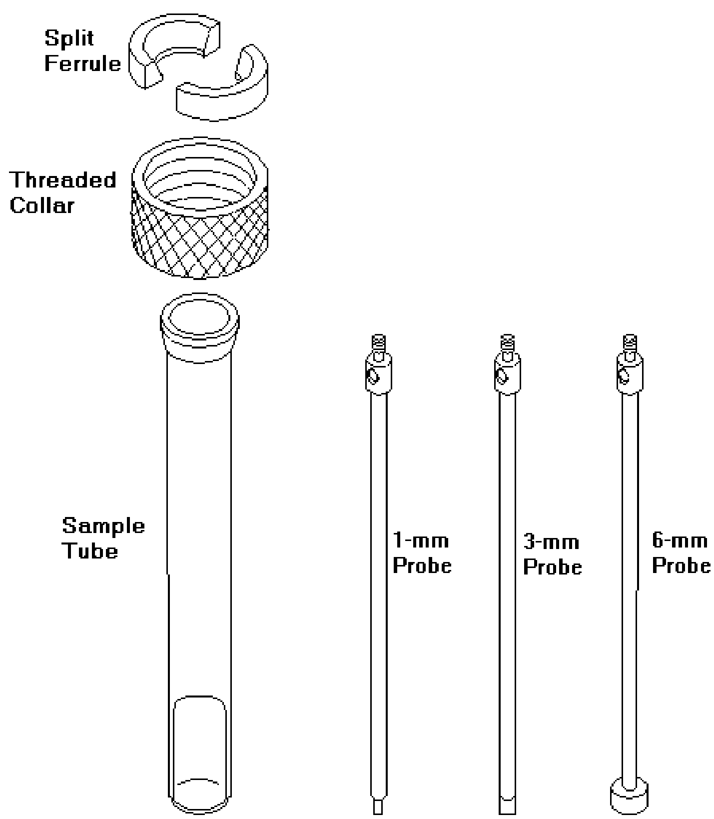


Quartz Small Parallel Plate Kit (P/N N519-0138)

Item	Part No.	Description	Quantity
1*	0009-0652	Septum (pkg. of 50)	1
2*	0993-8552	Instructions	1
3	N519-0376	1-mm Parallel Plate Probe	1
4	N519-0378	3-mm Parallel Plate Probe	1
5	N519-0606	6-mm Parallel Plate Probe	1
6	N519-1532	Sample Tube Ferrule	1
7	N519-1549	Sample Tube	1
8*	N539-0142	Case Assembly	1
9	N539-1054	Sample Tube Collar	1
10*	N539-1077	Nameplate	1

* = Item not shown in illustrated parts list.

Quartz Small Parallel Plate (N539-0138) Illustrated Parts



TAC 7/DX Interface

N519-0070 100 V

N519-0071 120 V, 60 Hz

N519-0072 230 V, 50 Hz

All three TAC interfaces use the same analyzer cable (P/N N519-0310).

DMA 7e Analyzer Accessories

The DMA 7e accessories consist of the following:

- **DMA 7e Spares Kit (P/N N539-0139)**
- **DMA 7e Accessories Kit (P/N N539-0140)**
- **High Temperature Small (15 mm) Furnace Kit (P/N N539-0141)**
- **Large (28 mm) Furnace Kit (P/N N519-0617)**
- **DMA 7e Cooling Accessories**
- **Purge Gas Accessories**
- **Gas Selector Accessories**
- **Miscellaneous DMA 7e Accessories**

DMA 7e Spares Kit (P/N N539-0139)

Part No.	Description	Quantity
0319-0033	Indium Calibration Standard	1
0319-0036	Zinc Calibration Standard	1
0401-0203	Plastic Box	1
0992-3184	Kapton Tape	1
0998-1614	2A Fuse	3
0998-1616	A4 Fuse	3
N519-0417	Thermocouple	2
N539-0128	Sample Kit with Reference E	1
N539-0129	Sample Pan Kit	1
N539-1048	Thermocouple Insulator	1

DMA 7e Accessories Kit (P/N N539-0140)

Part No.	Description	Quantity
0990-7233	Open end wrench	1
0990-7236	Hex wrench	1
0990-7278	5/32 Allen wrench	1
0990-8400	Tweezers	1

0990-8825	Force Calibration Standard (50 g)	1
N519-0392	Weight Tray	1
N519-1469	Eigendeformation Calibration Standard	1
N539-0142	Case Assembly	1
N539-1006	Dual Cantilever Block	1
N539-1007	Auto Zero Bending Block	1
N539-1051	Height Calibration Standard (10 mm)	1
N539-1066	Nameplate	1
N539-1083	Ball-Tip Hexhead Screwdriver	1
N539-1085	Dual Cantilever Height Standard	1

High Temperature Small (15 mm) Furnace Kit (P/N N539-0141)

Part No.	Description	Quantity
0990-7233	Open End Wrench	1
0990-7236	Hex Wrench	1
0990-7983	Screw	4
0991-1173	Hex Nut	4
0991-1243	Flat Washer	4
0991-1285	Split Lock Washer	4
N519-0370	Furnace (15 mm) with Instructions	1
N519-1431	Platinum Shield	1
N519-1462	Small Heat Sink	1
N519-1523	Furnace Base Insulator	2
N519-1580	Diffusion Cap	1

Large (28 mm) Furnace Kit (P/N N519-0617)

Part No.	Description	Quantity
0990-7233	Open End Wrench	1
0990-7236	Hex Wrench	1
0990-7983	Screw	4
0991-1173	Hex Nut	4
0991-1243	Flat Washer	4
0991-1285	Split Lock Washer	4
0998-1616	4A Fuse	2
N519-0613	Large Furnace Assembly with Instructions	1

N519-1523	Furnace Base Insulator	2
N519-1661	Heat Sink	1
N519-1715	Flexure Tube Cap	1

DMA 7e Cooling Accessories

Part No.	Description
0319-0205	Intracooler 1 (115 V)
0319-0206	Intracooler 1 (230 V)
0319-0207	Intracooler 2 (115 V)
0319-0208	Intracooler 2 (230 V)
N519-0514	DMA 7e Dewar Kit
0319-0101	DMA 7e Turbulent Chamber

All Intracoolers include P/N 0319-0327 which contains the following parts:

Part No.	Description	Quantity
0319-0209	Probe Insulated Bolt	1
0319-0247	Insulated Bolt Kit	1
0990-9960	Instructions	1
0991-1827	3/8 FW 25/64-in. i.d. 1-in. o.d. 1/16 TK SS	
N519-1845	Air Seal Ring	4

DMA 7e Dewar Kit (P/N N519-0514)

Part No.	Description	Quantity
0990-2222	O-Ring Silicone 2/113	2
0990-7617	Hext Nut 10/32 SS	2
0990-7983	Screw 4/40 x 3/4-in. SS	3
0991-0488	Screw 8/32 x 1/2 in.	3
0991-0493	Cap 8/32 x 1 1/2-in. SS	3
0991-0499	Cap 10/32 x 7/8-in. SS	2
0991-1173	Nut 4/40 SS	3
0991-1243	Flat Washer #4 SS	3
0991-1245	Flat Washer #8 SS	6
0991-1246	Flat Washer #10 SS	6
0991-1285	Split Lock Washer #4 SS	3
0991-1287	Split Lock Washer #8 SS	3
0991-1288	Lock Washer	2

0991-1319	Lock Washer	1
0992-1022	O-Ring Silicone 2/228	1
0993-8879	Users Manual	1
N519-0768	Box Assembly	1
N519-0771	Dewar Assembly	1
N519-0775	Funnel	1
N519-0789	Bracket Assembly – Adapter	1
N519-2079	Thumbscrew	3
N519-2102	Gasket – Dewar	1
N519-2105	Plug – Filler	1
N519-2115	Antidiffusion Cap	1
N519-2130	Grommet	1
N519-2132	Gasket	1
N519-2136	Heat Sink – Large Furnace	1

Purge Gas Accessories

Part No.	Description
0023-0522	Two-Stage, Soap Bubble Gas Flow Meter
0154-1496	Type “A” Purge Gas Restrictor (provides a flow of roughly 10 cc/min purge per pound of pressure
0212-1127	Pressure Regulator for Gas Line Use (secondary regulator 0–100 psi)
0240-0085	Pressure Regulator, Helium/Nitrogen (CGA 580)
0290-1624	Float Displacement Flow Meter, 0–100 cc/min
0319-0039	Gas Filter Dryer Assembly
N519-0603	Purge Gas Kit

A newer filter dryer is available from Perkin Elmer: the Drierite Compressed Air Filter Drier (P/N N537-0103). Instructions for its installation are given below.

The Purge Gas Kit contains the following items:

Part No.	Description	Quantity
0154-1498	Type “H” Purge Gas Restrictor (provides a flow of roughly 1 cc/min purge per pound of pressure	1
0250-6483	Teflon Tubing, 1/16-in. i.d. x 1/8-in. o.d.	24 ft
0250-6519	Tygon Tubing, 3/8-in. i.d. x 1/2-in. o.d.	24 ft
0990-3906	Female Connector 1/8-in to 1/4-in.	1
0990-8134	Thread Sealant Tape	1

0992-0008	Hose Clamp	10

Gas Selector Accessories

See below for instructions on installation of the GSA 7. Installation instructions for the TAGS are in the online Installation Help.

Part No.	Description
N519-0270	GSA 7 Gas Selector Accessory (115 V, 60 Hz)
N519-0271	GSA 7 Gas Selector Accessory (230 V, 50 Hz)
N519-0269	GSA 7 Gas Selector Accessory (100 V, 50/60 Hz)
N520-2019	Thermal Analysis Gas Station (TAGS) (110/120 and 220/240 V)

Miscellaneous DMA 7e Accessories

Part No.	Description
0419-0197	Al2O3 Dilatometer Packing Media (28 g)
0990-8353	Teflon Spray Lubricant
0990-7281	Micrometer Caliper
N519-1643	Dust Cover
0990-8400	Tweezers, Nonmagnetic Flat Forceps

Glossary

This glossary presents the terminology used in performing mechanical analysis and interpreting mechanical analysis results. The definitions are designed to promote understanding of the DMA 7e and may not apply to rheology in general. Different terms may be used by other instrument manufacturers. Consult standard reference texts for broader or more detailed definitions.

3-Point Bending	Measuring system that allows flexing at a central point of a sample supported between two knife edges.
Alloy	Mechanically blended combination of polymers. An alloy does not depend on chemical bonds but often requires special compatibilizers to join different constituent polymers together to improve mechanical performance (impact properties and chemical resistance), to lower cost, or to improve processability. Alloys produce tailored variations in polymer performance.
Amorphous	Chemical structure that allows the polymer chains to fold on themselves and intertwine in a random manner. Thermoplastics are an example of amorphous materials and are often described as independent, intertwined strings resembling spaghetti. Typical examples are polystyrene, polycarbonate, PVC, ABS, SAN.
Amplitude (p–p)	Magnitude of total cyclical displacement. Also referred to as the extent of the vibratory movement measured from one extreme to another (peak-to-peak amplitude).

Amplitude (true)	Magnitude of cyclical displacement. Also referred to as the extent of the vibratory movement measured from the mean position to an extreme. This is programmed and measured by DMA 7e.
Amplitude Control	System that controls the dynamic force or stress applied to a sample to maintain the sample at a constant amplitude setpoint.
Analytical train	The motor, LVDT, and furnace.
Anisotropy	Inconsistency of material properties with respect to direction. Physical properties of an anisotropic material depend on the direction in which they are measured. Varying degrees of anisotropy depend on the amount of symmetry in a material. Natural wood is a good example of anisotropy.
Behavior	Observed mechanical qualities of a material. Mechanical-dependent behavior includes temperature, time, rate, stress, and strain.
Bending	Deforming a sample by pushing or pulling in the direction normal to the sample.
Brittleness	Lack of toughness. Brittle materials often have lower impact and higher stiffness properties. Many materials in the glassy state are very brittle.
Bulk Modulus	Change in volumetric stress (hydrostatic) divided by the resultant change in volumetric strain; compressibility.
Calibrate DMA	Automatic calibration system that initiates the DMA electronics.
Calibrate Furnace	Calibration procedure that linearizes the heating and cooling of the furnace and matches the program temperature to the thermocouple temperature at 9 different points between the minimum and maximum temperatures selected by the user.
Cold forming	Polymer’s shape changes permanently while it is not heated. Also known as cold set. Stress a polymer beyond its yield point so that the material tends to stay in the shape or size to which it has been deformed. See Plasticity.
Complex Modulus	Ratio of maximum stress to maximum strain. Describes the combined elastic and viscous (bulk viscoelastic) behavior. It can often be used to correlate the results from older mechanical analysis tests since the older tests do not separate elasticity from viscosity. The notation used to report results depends on the type of deformation imparted to the sample. Modulus is reported as pressure (Pa).
Compliance	Ratio of strain to corresponding stress. Material’s ability to dissipate mechanical energy.
Compression	Uniaxial stress or strain applied to a sample. Deformation of a sample by pushing in the axial direction to the sample.
Creep Recovery	Combined technique in which the end point of the first analysis is the starting point of the second analysis. See Creep Mode and Recovery Mode.
Creep Mode	Measurement of strain growth versus time after a sudden stress is applied. Creep compliance is calculated; referred to as Retardation Spectrum.
Crossover	Point in a frequency scan where the Storage Modulus and the

	complex viscosity cross over when plotted on the same log-log-log scales. This point is related to the molecular weight and molecular weight distribution of a polymer.
Copolymers	A polymer with two or more different repeating units.
Crystalline	Chemical structure that allows the polymer chains to fold on themselves and pack together in an organized manner producing the lowest possible energy state. Typical examples are acetal, nylon, polyethylene, polypropylene, polyester (PBT, PET).
Current Height	Instantaneous position of the analyzer probe tip. Average position output of the LVDT.
Damping (D)	Absorption of energy. Decrease of mechanical input. Force needed to deform a liquid (water) divided by velocity (rate) yields a constant value (D) (Newton's law). Often reported in units of mN s/mm.
Deborah number (De)	Dimensionless number used in rheology. Equal to the relaxation time for some process divided by the time it is observed. In DMA, the measured events are molecular relaxation times.
Deformation	Change in sample's shape.
Density	Measure of the mass per unit volume expressed in g/cm^3. Used to calculate the relationship between the weight and volume of a material.
Depth	Dimension of a sample. Measured back to front and represents the z direction.
Diameter (d)	Dimension of a sample.
Dilatometer	Measuring system designed to translate a three-dimensional expansion into a uniaxial deflection.
Displacement	Measure of dimensional change in a material.
DMA	Dynamic Mechanical Analysis, analytical tests performed by DMA 7e.
DMS	Dynamic Mechanical Spectrometry, analytical tests performed by DMA 7e.
DMTA	Dynamic Mechanical Thermal Analysis, analytical tests performed by DMA 7e.
Ductility	Ability of a material to be stretched, pulled, or rolled into a shape without damaging its integrity.
Dynamic Control	System that controls the dynamic force or stress applied to a sample to maintain the sample at a constant setpoint.
Dual Cantilever	Measuring system that allows the flexing of a sample by a clamp in its center and which is statically clamped at both ends.
Dynamic Force	Magnitude of total cyclical force. Referred to as vibratory force as measured from the midpoint to the extreme deviation.
Dynamic Oscillation	Application of a stress or strain in a cyclic fashion (up and down) over time. Measuring elasticity or viscosity at a single temperature is of limited interest. To facilitate characterization of materials over temperature ranges, dynamic oscillations produce a continuous test.

Dynamic Stress	Oscillatory pressure applied to a sample. Magnitude of cyclical force normalized for the sample's dimensions. Also referred to as vibratory stress as measured from the midpoint to the extreme deviation.
Elasticity	Ability of a material to return to its original size and shape after being deformed. Material's ability to store mechanical energy. Elastic materials that follow Hooke's law can be described using Young's Modulus.
Elastomers	Low modulus flexible materials that can be stretched repeatedly and are able to return to their approximate original shape.
Extension	Uniaxial stress or strain applied to a sample. Measuring system that allows the uniaxial tension of a sample that is clamped at both ends.
Extension ratio	Variable expressing how a given dimension is changing in a sample undergoing deformation.
Force	Mass times acceleration (Newton's 2nd law). An applied force is stress.
Force amplitude	Magnitude of cyclical force normalized for sample's dimensions. Also referred to as vibratory force as measured from the mean position to an extreme.
Force Control	System that controls the static or dynamic force applied to a sample to maintain the sample at a constant force setpoint.
Force Motor	Device for generating force. A coil mounted inside a permanent magnet. When current is supplied to the coil, a magnetic field is generated that pushes against the magnetic field of the permanent magnet.
Frequency (f)	Amount of time required to repeat a periodic processes. Measured in Hz (cycles per second).
Friction	Force required to overcome surface molecular attraction between two materials.
Galling	Wearing away by friction; caused by poor lubricity.
Geometry	Physical shape of a sample, e.g., rod, bar, disk, film, fiber.
Glass Transition (Tg)	Temperature range at which segmental motion occurs for amorphous polymers within the time frame of the experiment. Temperature at which melting begins.
Height	Dimension of a sample or position of the analyzer probe tip. Measured bottom to top and represents the y direction.
Heterogeneity	Variability; inconsistency of material content. Different properties from a small sample than those of the whole body.
Homogeneity	Uniformity; consistency of material content. Same properties from a small sample as those of the whole body.
Hooke's Law	Displacement times the spring constant is equal to the applied force. This law is valid for all elastic materials. It is not valid for materials that exhibit viscous flow, such as water.
Hysteresis	Amount of energy stored after a cyclic deformation.
Isotropy	Independence of direction; consistency of material properties. The

	physical properties of an isotropic material at some point are the same, independent of the direction in which they are measured. Metals are typically isotropic.
Loss Modulus	Indication of a material's ability to dissipate mechanical energy.
Lubricity	Load-bearing characteristics of a material under load and relative motion. Measured with coefficient of friction.
LVDT	Linear Variable Differential Transducer. Analog sensing device that measures linear displacement by passing a magnetic core through the center of a coil.
MA	Mechanical Analysis, analytical tests performed by DMA 7e.
Measuring System	Combination of devices such as probes, clamps, sample tubes, etc., that hold the sample in place for testing within the analyzer.
Mechanical analysis	Testing of samples to elicit mechanically dependent behavior. Mechanical properties of a material affect the use, life, and manufacturing of many products.
Mechanical System	In a mechanical system the properties of force and damping are used to characterize materials. A mechanical system also has mass and friction which are present in models and real systems but are eliminated from most of DMA analysis using calibrations.
Mode	Programming of a single variable while other variables are held constant or are measured.
Modulus	Ratio of a stress to a corresponding strain.
Mold shrinkage	Ratio of expected reduction in the dimension of the plastic part as it solidifies in the mold and cools to room temperature versus the original mold dimensions.
Motor	Device for generating displacement. A coil mounted inside a permanent magnet. When a current is supplied to the coil, a magnetic field is generated that pushes against the magnetic field of the permanent field.
MS	Mechanical Spectrometry, analytical tests performed by DMA 7e.
Notch sensitivity	Measure of the ease with which a crack propagates through a material from a pre-existing notch, crack, or sharp corner. Not to be confused with brittleness.
Opacity	Measure of haze or luminous transmittance. Haze is a measure of the percentage of transmitted light through a test specimen that is scattered more than 2.5° from the incident beam. Luminous transmittance is a measure of the ratio of transmitted light to incident light.
Original Height	Measured dimension of a sample. Measured from bottom to top and represents the y direction.
Parallel Plate	Measuring system that allows the uniaxial compression of a sample supported between two parallel disks of equal diameter.
Pascal	The SI unit for pressure (1 Pa = 1 N/m^2).
Peak-to-peak force	Magnitude of total cyclical force. Also referred to as vibratory force measured from one extreme to another.

Permanent set	Degree of irrecoverable flow that exists after a material has been deformed.
Phase	Time lag between a disturbance and the result. Time difference between input and output. Measured in radians or degrees. Also called phase lag or phase angle. For materials that exhibit viscoelastic behavior (between a perfect steel spring and water), stress is function of strain and strain rate. These materials are between 90° out of phase and completely in phase with the applied stress. Almost all polymers fall within this range.
Phase Lag	Angular difference between the directed sine wave and the sample's response.
Plasticity	Inverse of elasticity. Material that tends to stay in the shape or size to which it has been deformed has high plasticity. See Viscosity. Polymers exhibit plasticity when they are stressed beyond the yield point.
Poisson's ratio	Volumetric changes exhibited by a material as it is stressed or strained.
Position Control	System that controls the static force or stress applied to a sample to maintain the sample at a constant position setpoint.
Probe Position	Average position of the probe tip at any time before, during, or after a run. Probe position relates directly to sample dimensions or height.
Proportional Limit	Greatest applied stress that a material can sustain without deviating from the proportionality of stress and strain.
Recovery Mode	Measurement of strain recovery versus time after a stress is removed. The Recovery Modulus is calculated and the result is referred to as the Recovery Spectrum.
Resonant frequency	Amplification of natural harmonics.
Rheology	Study of flow and deformation of materials. Analyzer is considered a DMA or rheometer if it reports complex, storage, and loss moduli.
Setpoint	Value a control system controls around. Setpoint can be set using "current" or typed in values.
Shear Modulus (G)	Shear stress divided by shear strain. Measured in Pa.
Shear Strain (γ)	Angle of deformation measured in radians as one layer of a material slides over another to produce a shear deformation. Shear strain is reported as dimensional percentage.
Specific Gravity	Ratio of mass of a given volume of a material to the mass of the same volume of water measured at 23°C. Dimensional. Used in determining part cost, weight, and quality control.
Spring Constant	Describes the force exerted by a material as it is deformed.
Static Control	System that controls the static force or stress applied to a sample. Includes Position Control, Tension Control, and Force Control.
Static Force	Steady force applied to a sample.
Static Friction	Force required to overcome surface molecular attraction between two materials at rest.

Static Stress	Steady pressure applied to a sample.
Storage Modulus	Indication of a material’s ability to store mechanical energy.
Strain (ε)	Ratio of the sample size under load to the sample size under no load. Reported as dimensionless %.
Strain amplitude	Magnitude of cyclical displacement normalized for the sample dimensions. Referred to as vibratory movement measured from the mean position to an extreme.
Strain Control	Automatic adjustment of the applied dynamic force to achieve a constant strain value defined by setpoint.
Strain rate	Time-dependent dimensional change of a material.
Stress (σ)	Applied force per unit area; pressure. Force is normalized for the contact area. Reported in dyn/cm^2, bar, psi, or Pa.
Stress amplitude	Magnitude of cyclical pressure normalized for the sample dimensions. Referred to as vibratory pressure measured from mean position to an extreme.
Stress Control	Automatic adjustment of the applied dynamic force to achieve a constant stress value defined by the setpoint.
Tangent delta	Indication of state (or phase) of a material. Most fundamental measurement in DMA. Rate-dependent relaxation time of a material. Derived from relationship between Loss and Storage Moduli. It indicates relative importance of both viscous and elastic behaviors. Materials with a tangent delta of <1exhibit more elastic behavior. They may have a relatively high Storage Modulus and may behave like a solid. Materials with a tangent delta of >1 exhibit more viscous behavior. They may have relatively high Loss Modulus and behave more like a liquid.
Tension	Uniaxial stress or strain applied to a sample.
Tension Control	System that controls the static force or stress applied to a sample to maintain a constant relationship between the dynamic and static controls. Automatic adjustment of static force to maintain constant sample contact. Tension Control of 110% applies 10% more static force or stress than dynamic force of stress.
Terpolymer	Copolymer with three different kinds of repeating units.
Thermoplastic	Polymers that repeatedly soften when heated and harden when cooled, e.g., water and ice. Thermoplastics are often described as independent, intertwined strings resembling spaghetti. When heated, individual chains slip, causing flow. When cooled, contraction eliminates the freedom of movement and prevents the chains from slipping or rotating. Thermoplastics exhibit the reversible effect of heating.
Thermoset	Polymers that undergo chemical change during processing to become permanently insoluble and infusible. Boiled egg is a thermoset: reheating cannot reverse the process. Thermosets are often described as independent, intertwined strings resembling spaghetti. When crosslinked, individual chains are prevented from slipping. Reheating degrades the polymer rather than melts it. Thermosets exhibit the irreversible effect of heating.

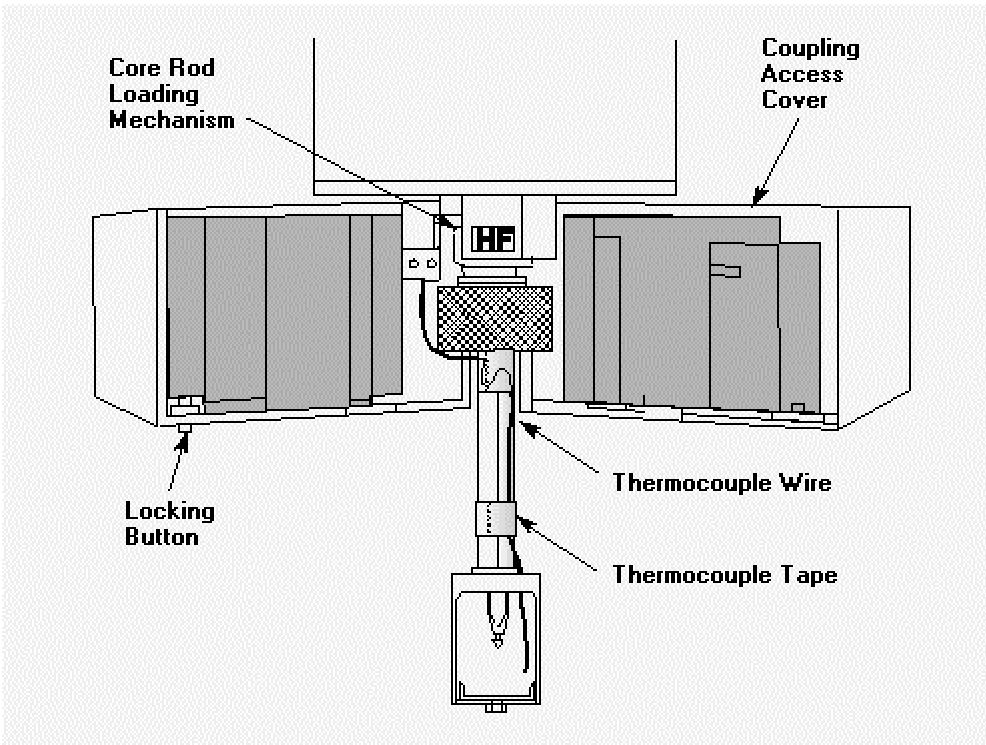
Toughness	Material’s ability to absorb mechanical energy without fracturing or excessively changing shape. Tough material can absorb mechanical energy with either plastic or elastic deformation. Toughness can be measured as the area under the stress – strain curve before failure.
Transparency	See Opacity.
Ultimate strength	Maximum applied stress that a material withstands before failure.
Viscoelasticity	Characteristic exhibited by most polymeric materials. Some materials will behave like springs, some like water. Most materials will exhibit both properties in varying degrees. These materials are considered to exhibit viscoelastic behavior.
Viscosity	Indicates material’s ability to dissipate mechanical energy. Equal to stress divided by strain rate.
Water absorption	Percent increase in weight of a material due to absorption of water.
Width	Dimension of a sample. Measured left to right and represents the x direction.
Yield Point	Increase in strain without increase in applied stress.
Yield Strength	Greatest applied stress that a material can sustain without deviating more than a given strain above the proportional limit.
Young’s Modulus (Y)	Indicates material’s ability to store mechanical energy. Measure of resistance of a material to deformation when external forces are applied. Reported in Pa.

Chapter 8
TMA 7 Analyzer

Overview

NOTE: Analyzers shipped before July 1, 1993, are considered older TMA 7 analyzers. In order to see if your analyzer is an older model, check the casting of the core rod locking mechanism. The newer models have "HF" on the mechanism.

View of the TMA 7 Showing HF Label



The TMA 7 Thermomechanical Analyzer measures the dimensional and viscoelastic changes in nearly all types of sample materials as a function of temperature or time. Typical applications of the TMA 7 include coefficient of expansion studies, softening point determinations, fiber and thin film studies, and glass transition determinations. The TMA 7 can be programmed at a linear heating or cooling rate or held at a constant temperature. It is completely automated including electromagnetic probe control, probe positioning, and calibration. Closed loop electromagnetic circuitry provides constant load force throughout the experiment. Forces are selected in Pyris Software for Windows which is on the computer to which the instrument is connected. The TMA 7 is controlled by Pyris Software for Windows which is on the computer to which the TMA 7 is attached via a TAC 7/DX Thermal Analysis Instrument Controller which controls the analyzer and digitizes the analog output from the detector before sending it on to the computer.

The TMA 7 comprises three major components: a highly sensitive linear variable differential transformer (LVDT), a low-mass furnace, and a precise linear motor for probe loading.

The LVDT is extremely sensitive, capable of measuring dimensional changes with a displacement sensitivity of 50 nm and a resolution of 3 nm. The dynamic range of the LVDT allows the study of samples as large as 1.91 cm (0.75 in.) high. The combination of this sensitive temperature-controlled LVDT with the precise linear probe control motor permits accurate, reproducible analyses of virtually any sample type.

The low-mass furnace, when combined with the appropriate cooling accessories, allows operation from –170°C to 1000°C. Heating and cooling rates ranging from 0.1°C/min to 100°C/min can be used with this furnace. This broad temperature range and wide variety of heating and cooling rates encompasses virtually the complete range of sample materials for thermomechanical analyses.

Some features of the TMA 7 are as follows:

- High-resolution (3 nm) detector allows the measurement of the smallest dimensional changes.
- Electromagnetic probe control, not springs, provides constant load force regardless of sample dimensional changes.
- Computer-controlled loading (force application) eliminates the use of weights, improving experimental reproducibility.
- Temperature-controlled detector (LVDT) eliminates baseline variation as a function of temperature, improving measurement accuracy.
- One-touch automatic probe control simplifies probe positioning and removes experimental variability.
- Multiple probe types, including **expansion, compression, flexure, extension, and dilatometer**, provide the flexibility needed to analyze a broad range of materials. The probes are constructed of durable quartz for long life and minimal background interference.
- Single furnace allows operation from –170°C to 1000°C, eliminating the need for time-consuming furnace changes when temperature ranges are changed.

Expansion Probe

The expansion probe (P/N N519-0378) permits the direct determination of the coefficient of linear expansion (α), a characteristic and important property of any material.

Compression Probe

Narrow tip compression probes are used for penetration measurements. A broad tip compression probe permits the determination of the compressive modulus and additional properties of soft rubbery materials.

0.5-mm radius spherical tip	P/N N519-0377
1.5-mm radius spherical tip	P/N N519-0416

Flexure Probe

The deflection temperature of materials under load and other types of three-point bending studies can be performed with the flexure analysis probe (P/N N519-0393).

Extension Probe

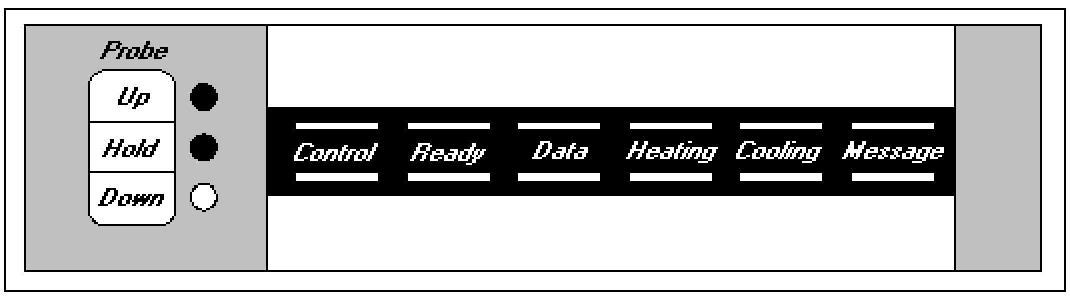
For the extension and contraction characterization of fibers and thin films, an extension analysis probe (P/N N519-0609) and extension analysis kit (P/N N519-0619) are available for the TMA 7. With the extension probe you can perform distortion studies of drawn fibers and films. The system measures the length of an extended sample as a function of the force and temperature applied to the sample.

Dilatometer Accessory

A dilatometer accessory permits the determination of volumetric changes of materials as a function of temperature. The accessory is useful for materials whose linear coefficient of expansion cannot be easily determined by the expansion probe. The dilatometer is useful for determining the volume change of materials upon melting, or upon cooling from a melt. A typical application would be determining shrinkage from a mold.

Operating Controls

An important part of the TMA 7 is the front panel which contains three probe control buttons and six status indicators.



A light next to each probe control button is illuminated when that button is selected.

Control

The TMA 7 temperature sensors are in control of the temperature; power is being supplied to the furnace to maintain the program temperature selected in Pyris. The Control light should always be lit in conjunction with the Heating or Cooling and/or Data control lights during the main section of a temperature program run.

Ready

A blinking Ready light means that the program temperature has reached the user-defined load temperature. When the Ready and Control lights are both lit (nonblinking), the system is ready to begin a temperature program. Upon system startup, the Ready indicator should be the only indicator that is lit. In some cases the Control indicator may also be illuminated on startup depending on the heat sink and coolant temperature.

Data

If the Data indicator is illuminated, data are being collected. The Data indicator is off at all other times.

Heating

If the Heating indicator is illuminated, the TMA 7 is heating under program control.

Cooling

If the Cooling indicator is illuminated, the TMA 7 is cooling under program control.

Message

If the Message light is blinking, there is a message to be read at the computer. A blinking Message light may also mean that the measuring system and sample may be stuck if the sample has melted and fused to the probe tip. If this should occur, detach the probe tip from the probe holder (for a stainless steel measuring system) and remove the sample from the probe tip outside of the analyzer.

Probe Up

This button causes the probe assembly to rise and lock into place. Selecting Probe Up with a sample in place engages the extension analysis accessory and applies the desired stress to the sample while using the Extension Analysis measuring system.

Probe Hold

Pressing this button holds the probe assembly at its current location between the up and down positions.

Probe Down

Selecting this button causes the probe assembly to descend to the base of the sample platform (or on the sample currently resting on the sample platform). The weight of the core rod plus the measuring system will be measured by the TMA 7 each time the probe is lowered (except when using the Extension Analysis measuring system). This allows accurate measurement of the system inertia.

Status Indicators

The current status of the TMA 7 is displayed in the Status Panel in the Pyris Software for Windows screen. You can also see the status of the instrument by looking at the front panel.

Summary of the TMA 7 Status Indicators

Indicator	Blinking	On	Off
Control	Does not blink	Power is being supplied to the furnace	Analyzer is not in temperature control Power is not being supplied to the furnace
Ready	Program temperature has reached Load Temperature	Analyzer temperature has reached Load Temperature Power is being supplied to TMA 7	Analyzer temperature has not reached Load Temperature Power is not being supplied to TMA 7
Data	Does not blink	Data is being taken from the TMA 7	Data are not being taken from the TMA 7
Heating	Does not blink	Analyzer is heating under program control at the selected rate	Analyzer is not heating
Cooling	Does not blink	Analyzer is cooling under program control at the selected cooling rate	Analyzer is not cooling
Message	A message has been sent to the computer		No message is waiting

NOTES: If the indicators are blinking in sequence, the TMA 7 is calibrating. **Do not touch the front panel.**

The Control indicator may go out momentarily at the beginning of a run.

The temperature at which the Control indicator lights and, therefore, the acceptable temperature range of the TMA 7, is dependent on the type of coolant or cooling accessory that you use. For example, with an ice water bath in the TMA 7 dewar, temperatures as low as 20°C can be attained. With liquid nitrogen as the coolant, temperatures as low as –170°C can be attained.

The status indicators change during **startup** of the TMA 7 system.

The status indicators can be used for **diagnostic troubleshooting**.

Start Up a TMA 7 System

Your TMA 7 Thermal Analysis System should be started up as follows:

1. Turn on the computer and access Pyris Software for Windows program group.
2. Turn on all of the analyzers.

   The **Ready, Heating, Cooling,** and **Message** status indicators on the front of the analyzers should be illuminated.
3. Turn on and adjust all of the purge gases and pneumatic pressure gases (or check them if they are already on).
4. Turn on the TAC 7/DX Instrument Controller for each analyzer.

NOTE: If the TAC 7/DX is already on, turn it off, wait 5 – 10 seconds, and then turn it back on.

All of the status indicators on the front of the analyzer should go out for a few seconds. Then the **Ready** indicator should illuminate. Depending on the cooling accessory or coolant in use, the **Control** indicator may also illuminate or flash.

NOTE: If any other status indicators are illuminated at this point, turn off the TAC 7/DX and then the analyzer module. Wait 5 to 10 seconds. Turn on the analyzer and then the TAC 7/DX.

5. Turn on the power to the printer.
6. Turn on the power to the GSA 7 or the TAGS, if these units will be used with your system.
7. Select the Pyris Manager or the Pyris Data Analysis icon to start the Pyris Software for Windows software.

Diagnostic Troubleshooting

Control	Ready	Data	Heating	Cooling	Message	Status
On or Off	On					Computer, TAC, and TMA 7 functioning properly.
On or Off	Blinking					Computer, TAC, and TMA 7 functioning properly. Analyzer at load temperature.
	On	On	On	On	On	TAC is not turned on or properly connected to TMA 7. Check that all cables are connected properly, TAC power cord is connected to line power, and TAC is turned on.
			Blinking	Blinking		Cable connecting TAC to TMA 7 is loose or has failed. Furnace cable is broken or not connected to TMA 7.
		Blinking	Blinking or Off	Blinking or Off	Blinking or Off	Hardware component in TAC may be malfunctioning. Call Service Representative.

The TMA 7 is presented in the following topics:

- **Safety Precautions**
- **Calibration**
- **Sample Handling**
- **Maintenance**
- **Part Numbers**

Safety Precautions

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

The following precautions must be observed when using the TMA 7 Thermomechanical Analyzer:

- Never turn off the power to the computer until the appropriate shutdown message is displayed.
- Never remove the outer instrument cover or side panels on the TMA 7 without turning the instrument off and disconnecting its power cord from the line power.
- Always observe the precautions indicated for the use of the various cooling devices.
- Only high-quality purge gases should be used with the TMA 7. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory, such as the **Drierite Compressed Air Filter Dryer**, is recommended for the removal of any moisture from the purge gases.
- Always observe the startup or shutdown procedures with the TMA 7 and all related instruments.
- For superior temperature accuracy, use helium as the purge gas.
- Prior to performing any experiment or calibration, be sure that the thermocouple currently installed in the TMA 7 is functioning properly. The thermocouple can be checked easily by programming the TMA 7 to a temperature of 100°C and checking the temperature displayed in the status panel. (The temperature should read a value close to 100°C if the instrument is calibrated.)
- Make sure that the furnace is programmed to between 20°C and 40°C between runs and if you leave the TMA 7 unattended for a period of time when using a low-temperature cooling accessory (below –20°C).
- If you are using an Intracooler or liquid nitrogen as a cooling device, raise the furnace temperature to 150°C before shutting down the cooling device when finished with normal operation. Leave the TMA 7 on and the furnace at 150°C for at least 2 hours or overnight. This will expel any moisture and prevent it from refreezing and damaging the furnace.

WARNING: **TMA tests are typically performed with forces that are less than 50 mN. Using larger forces can result in damage to the quartzware and personal injury.**

Calibration

All calibrations of the TMA 7 are performed by a Perkin Elmer Service Engineer upon installation, so it will not be necessary for you to perform a complete calibration. A periodic check of the calibration is all that is necessary to verify accurate calibration. Calibration of the TMA 7 should remain unchanged for an extended period of time provided there are no changes in the operating conditions. Even when the system is shut down, the calibration values are stored so that the next time the analyzer is powered on, it will still be calibrated. Some of the changing operating conditions that could affect the temperature or the eigendeformation calibration of the TMA 7 are as follows:

- If the operating temperature range of your experiments changes, temperature calibration may be necessary. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
- If the purge gas or purge gas flow rate changes, the temperature calibration should be checked.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new thermocouple is installed or the thermocouple's position is changed, the temperature calibration should be checked.

- The eigendeformation calibration should be performed if you change the sample tube or probe and if the samples being analyzed are very stiff (i.e., they have a modulus greater than 10 GPa).

There are five calibration routines used to calibrate the TMA 7:

Height Calibration	This calibrates the displacement transducer that is used to measure the position of samples.
Force Calibration	This calibrates the force motor that is used to apply the static force to the sample.
Eigendeformation Calibration	This calibrates the very small movement of the analyzer itself when large forces are applied. After performing the calibration, the compliance of the system is subtracted from the probe position signal. Therefore, increasing force with no sample present will yield zero displacement. Perform the Eigendeformation calibration after Height and Force calibrations.
Temperature Calibration	This allows you to run one or more reference materials to match thermocouple temperature and sample temperature.
Furnace Calibration	This is a nine-point temperature calibration between operator-selected upper and lower temperature limits. The thermocouple temperature will be matched to the programmed furnace temperature when this calibration is complete.

Perkin Elmer ships reference materials and standards in the TMA 7 Spares and Accessories Kit (P/N N519-0600) . Additional standards and reference materials are available through PE XPRESS.

For a summary of the steps involved in calibrating the TMA 7, see **Calibrating the TMA 7**.

Height Calibration

To perform a Height Calibration for the TMA 7, follow the procedure below:

NOTE: A sapphire height displacement standard (P/N N519-1714) is provided in the TMA 7 Spares Kit. It is suitable for height calibration over the full range of the system. However, you can use other materials of known height if you wish. These materials should be very hard (i.e., have a high modulus) and should have flat, parallel surfaces. Use a micrometer (P/N 0990-7281) to measure the height of the displacement standard. The measured height is used in the procedure that follows.

1. Restore the Height calibration values by first selecting **Calibrate** from the View menu and then **Height** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Height** tab in the Calibration window.
4. Enter your name in the Operator field and the height of the displacement standard that you are using in the Ref. Height field. (The current calibration reference material height value is displayed in the Measured field.) Click on **Begin Calibration**.

5. The Height Calibration dialog box displayed instructs you to prepare the analyzer for the zero reading. Carefully lower the furnace assembly by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
6. Press the **Probe Up** button on the front panel of the instrument to raise the probe.
7. Make sure there are no samples on the sample platform.
8. Press the **Probe Down** button on the front panel of the instrument to lower the probe so that it rests on the empty sample platform. Click on **OK** to clear the dialog box. It will take from 20 to 30 seconds for the Y signal value to be displayed in the Read Zero dialog box once the probe tip rests on the platform.
9. When the Y signal stabilizes, select **OK** if you want the current Y value entered as the new zero value. The Y signal will automatically set to zero. The calibration sets the location of the empty sample platform as 0 mm.
10. The next dialog box gives you further instructions. Press the **Probe Up** button on the front panel of the analyzer. Place the sapphire height displacement standard on the sample platform. Press the **Probe Down** button to lower the probe so that it rests on the displacement standard. Click on **OK** to clear the dialog box.
11. It will take 20 – 30 seconds for the Y signal value to be displayed in the Read Value field in the next dialog box. When the Y signal stabilizes, click on **OK** to enter this value as the height calibration value and clear the dialog box.
12. Click on **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.

    The Height Calibration is now complete.

13. Press the **Probe Up** button on the front panel and remove the displacement standard.

You can now perform the next calibration, **Force Calibration.**

Force Calibration

NOTE: Locate the 50-g-force calibration weight (P/N 0990-8825) and weight platform (P/N N519-0392) provided in theTMA 7 Spares Kit.

To perform a Force Calibration:

1. Restore the Force default calibration values by first selecting Calibrate from the View menu and then **Force** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Force** tab in the Calibration window.
4. Enter your name in the Operator field.
5. Click on **Begin Calibration**.
6. Remove the dust cover, remove any sample from the sample platform, install the weight tray in the analyzer, and lower the probe. Click on **OK**. The next dialog box tells you to place the standard 50-g weight onto the tray. After a short time, the calibration will be finished.
7. Remove the 50-g weight and the weight tray from the analyzer at the same time. Replace the dust cover.

The force calibration value is automatically saved to the instrument.

You can now proceed to perform the **Eigendeformation Calibration**.

Eigendeformation Calibration

NOTES: A 9.5-mm (0.375-in.) steel eigendeformation calibration cylinder (P/N N519-1469) is provided in the TMA 7 Spares Kit and must be used for the second part of the Eigendeformation Calibration.

You **must** perform a Height Calibration and a Force Calibration before proceeding to an Eigendeformation Calibration.

Perform an Eigendeformation Calibration as follows:

1. Restore the Eigendeformation default calibration values by first selecting **Calibrate** from the View menu and then **Eigendeformation** from the Restore menu.
2. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
3. Select the **Eigendeformation** tab in the Calibration window.
4. Enter your name in the Operator field and click on Begin Calibration.
5. Remove any sample from the platform, lower the probe by pressing the **Probe Down** button on the front panel of the DMA 7e, wait 1 minute, and then proceed.
6. Raise the probe by pressing the **Probe Up** button on the instrument, insert the steel reference material, and then lower the probe by pressing the **Probe Down** button.
7. After a short time, the calibration is completed. Raise the probe by pressing the **Probe Up** button on the analyzer and remove the steel reference material.
8. Click on **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.

You can now proceed to perform the **Temperature Calibration.**

Temperature Calibration

To perform a Temperature calibration of the TMA 7 using indium as the standard, perform the following steps:

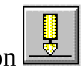
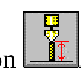
1. **Set up a run and collect data for indium.**
2. **Perform an onset calculation.**
3. **Perform the calibration.**

Indium Run and Data Collection

Set up the indium run for calibrating the TMA 7 as follows:

1. Install the TMA 7 Penetration Probe (P/N N519-0376) according to its installation instructions, and, if necessary, the standard furnace tube.
2. While in the Instrument Viewer or the Method Editor, select **Calibrate** from the View menu.
3. Restore the default temperature calibration values by selecting Temperature from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the All command.
4. Click on **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
5. Click on **Close** to close the Calibration window.

> **NOTE:** If the furnace assembly is not already locked in place at the base of the analyzer, carefully lower it by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base.

6. If there are any samples on the sample tube platform, press the **Probe Up** button on the front panel of the TMA 7 and remove them using tweezers (P/N 0990-8400) provided with the TMA 7.
7. Select calibration method IndCal.tmm provided with Pyris by selecting Open Method from the File menu and choosing the method from the dialog box.
8. Press the **Probe Down** button to lower the probe so that it rests on the sample tube platform. The height signal will be displayed in the Status Panel; it will stabilize several seconds later.
9. Click on the **Zero Height** button  in the Control Panel. This **zeros the probe** for height. The Y value reading displayed in the Status Panel should now be set to 0.000 mm.
10. Press the **Probe Up** button to raise the probe.
11. Using tweezers, prepare a small amount of the indium reference material (P/N 0319-0033) (3-mm square x 0.5-mm thick). Use the tweezers' handle to flatten the indium.
12. Place the indium in the center of a sample platform so that it lies flat.
13. Inspect the thermocouple position (see Installing the Thermocouple).
14. Press the **Probe Down** button to lower the probe. Press **Probe Hold** just before the probe tip touches the calibration reference material.
15. Check that the reference material and probe are positioned properly. The reference material should be in the center of the platform. The probe tip should be over the center of the reference material. Reposition the reference material if necessary.
16. Press the **Probe Down** button so that the probe rests on the calibration reference material. In approximately 30 seconds, the Y signal value will appear in the Status Panel and stabilize several seconds later.
17. Click on the **Read Height** button  in the Control Panel. The TMA 7 will automatically read the height of your calibration reference material and it will appear in the Sample Height entry field in the Method Editor. The reading will be stored in memory.
18. Carefully raise the furnace assembly all of the way up, making sure that it locks in place. Place the diffusion cap around the sample tube at the top of the furnace.
19. Select the **Instrument Viewer** button from the toolbar to display the real-time curve for your indium run.
20. Enter 100°C in the Go To Temp entry field in the Control Panel and click on the **Go To Temp** button.

> **NOTE:** Do not begin the run until the temperature signal stabilizes in the Status Panel.

21. Click on the **Start** button in the Control Panel to start the run.

    The system begins to collect data immediately. The following status indicators on the front of the analyzer should be illuminated: Control, Ready, Data, and Heating. These indicators show that the instrument is in temperature control, data is being collected, and the furnace is heating. The Status Panel should indicate that the TMA 7 is heating and other signals should be updating.

Onset Calculation for Indium

The data file you just collected should be displayed in the Data Analysis window. Perform an onset calculation on the data you just collected as follows:

1. Select **Onset** from the Calc menu. The Onset Calculation dialog box appears.
2. Enter the Left Limit and the Right Limit. You can also click on the two red **X**'s that appear on the curve and drag them to the desired left and right limits. The Left Limit and the Right Limit fields will be filled in on the dialog box.
3. Click on the **Calculate** button.

   The Onset result is used in the temperature calibration. You can now perform the temperature calibration.

Performing a Temperature Calibration

The measured onset value for indium is used to calibrate the temperature of the analyzer. A temperature calibration consists of the following steps:

NOTE: You should have restored the default Temperature calibration value before you performed the indium run.

1. Select **Calibrate** from the View menu.
2. Select the Temperature tab in the Calibration window. Enter your name in the Operator field.
3. Enter the name of the reference material used. (Indium should be displayed as the Ref. Material for Run 1.)
4. Enter the expected onset temperature for the reference material in the Expected Temperature field.
5. In the Measured Temperature field, enter the onset temperature you obtained when you performed the onset calculation.
6. Click on the check box in the Use column.
7. Select **Save and Apply**; select the file to which to save the new calibration values or enter a new file name. This sends the new calibration value to the analyzer and saves the calibration file specified.
8. Select **Close** to exit from the Temperature Calibration box.

Furnace Calibration

NOTE: Perform a Furnace Calibration **after** a Temperature Calibration.

NOTES: Prior to performing the nine-point Furnace Calibration, verify that the sample temperature is at or near the program temperature and that the sample platform is empty. Also, make sure that the current sample temperature is at or below the intended minimum calibration temperature.

You **must** use some form of coolant that will maintain a constant block temperature below 45°C throughout the entire calibration. This can be achieved by using ice water or other coolant in the dewar, or by using a circulator or an Intracooler 1 or 2 as a cooling device.

1. Select **Calibrate** from the View menu.
2. Select the **Furnace** tab in the Calibration window.
3. On the DMA 7e, press the furnace locking mechanism and raise the furnace until it locks in place.
4. Enter your name in the Operator field.
5. In the Minimum field, enter the temperature where the calibration will begin (e.g., 50).
6. In the Maximum field, enter the temperature where the calibration will end (e.g., 250).

NOTES: The difference between the minimum and the maximum temperatures must be greater than 100°C.

Excellent calibration can be achieved for both very low temperatures (down to –170°C) and very high temperatures (up to 500°C) when a circulator or ice in the dewar is used as a constant temperature heat sink and the calibration is performed between these limits.

7. Click on the **Begin Calibration** button.

   The Furnace Calibration is performed automatically. The program and sample temperature values are matched at nine different points over the temperature range selected. When the calibration is complete, the furnace automatically cools to 30°C.

8. To accept the Furnace Calibration values, click on **Save and Apply**. Select an appropriate filename in which to save the calibration.
9. Click on **Close** to exit from the Furnace Calibration window.

Upon completion of the Furnace Calibration, the analyzer is fully calibrated.

Calibration Steps for the TMA 7

A typical TMA 7 calibration consists of the following steps:

1. **Restore the default calibration values.**
2. **Perform a Height Calibration.**
3. **Perform a Force Calibration.**
4. **Perform an Eigendeformation Calibration.**
5. **Perform a Temperature Calibration using indium as the reference materials.**
6. **Perform a Furnace Calibration.**

NOTE: Be sure to perform the calibration procedures in this order.

Restore Default Calibration Values

You can restore the default calibration values for the analyzer by performing the following steps:

1. Select **Calibrate** from the View menu.
2. Click on the **Restore** option on the menu bar to display the Restore menu.

   Select **Height** to restore the default height calibration values.

Select **Temperature** to restore the default temperature **and** furnace calibration values.

Select **Force** to restore the default force calibration values.

Select **Eigendef** to restore the default eigendeformation calibration values.

Select **All** to restore all of the default calibration values.

Sample Handling

In Thermomechanical Analysis the object of an experiment is to record the change in dimension of a sample as a function of temperature or as a function of time at a selected isothermal temperature. The suggestions given in the following topics illustrate some of the techniques that can be used to obtain the most accurate TMA data in a minimum amount of time.

- **Tools for Preparing Samples for TMA Experiments**
- **Sample Preparation for Penetration Testing**
- **Sample Preparation for Expansion Testing**
- **Zeroing the Probe**

After preparing your sample, it needs to be mounted in the TMA 7. This is described in the topic

- **Mounting the Sample**

Tools for Preparing Samples for TMA Experiments

Depending on the type of experiment to be performed and the state of the sample as it is received, different tools may be required for preparing the sample.

WARNING: Always use safety glasses and observe all safety precautions when handling or preparing samples.

Razor blades, knives, scissors, sandpaper, and a standard paper punch are the most commonly used tools for preparing TMA samples. With these tools, many samples such as plastic films and pellets, coatings on thin substrates, and finished products can be cut and prepared for use on the TMA 7. A heavy-duty die punch is often used to cut out samples of thick materials, highly filled materials, laminates, and coatings on thick substrates.

NOTE: In a large number of instances the substrate does not interfere with the analysis. Delamination is difficult and tedious and can destroy the properties that are being tested.

A wire cutter is useful in the study of insulator coatings on wires. Usually it is not necessary to remove the insulator coating from the wire for analysis.

A hacksaw or band saw can also be used to cut out sample materials. A hammer can be used in some instances to chip away a small sample for analysis.

Sample Preparation for Penetration Testing

While there are no rigid requirements in sample preparation for penetration testing, it is preferable to have a fairly small sample so that its center does not lag too far behind the furnace and thermocouple temperature. The sample probe should be lowered onto a section of the sample that is relatively smooth and flat.

The penetration probe with the flat tip (P/N N519-0376) and the penetration probe with the round tip (P/N N519-0377) often can be used interchangeably. For some uses, however, particularly with thin films, one probe may be more effective than the other. When doing a penetration experiment, try both probes and see which works better.

Sample Preparation for Expansion Testing

Sample surfaces that are flat and parallel are required in expansion testing. Therefore, a more careful technique in sample preparation is required. Relatively large samples are required, since any dimensional change will be a small fraction of the total thickness; a sample thickness of 6.5 mm (0.25 in.) will suffice in general. Minimum sample thickness will depend on the expansion coefficient of the sample, the sensitivity of the instrument, and the accuracy requirements. If necessary, sample pieces can be “stacked” to obtain an expansion coefficient.

If the sample surfaces are not flat, the sample may show a penetration effect caused by localized loading. For example, the probe may be making a point contact on the sample. Should this contact condition be observed, it can be improved by placing a thick layer of finely powdered silica on the sample, lowering the probe to distribute and pack the powdered silica, and blowing away any excess powder. Also, the sample diameter can be made smaller than the probe tip diameter. Powdered silica can then be used with the smaller-diameter sample.

Zeroing the Probe

To zero the probe means to set the LVDT to zero height. To zero the probe height:

1. Set the current static force to 100 mN in the Initial Values page.
2. Make sure that there is no sample on the sample platform.
3. Press the **Probe Down** button on the analyzer.
4. Click on the **Read Zero** button on the Control Panel. The Zero Height value is displayed in the Zero field in the Method.

The analyzer is now zeroed for height. The sample zero reflects the difference between calibration zero and the current probe position. When a sample is mounted in the analyzer, the reported height should be close to the sample height.

When & Where to Read Height and Zero to Get Meaningful Results

Type of Analysis	Oscillating DMA	Static DMA	TMA	Sample Height
Type of DMA Mode / Type of DMA Geometry	Temperature Scan Time Scan Frequency Scan Dynamic Stress Scan	Creep-Recovery Static Stress Scan	TMA	For each measuring system, all modes.
Flexure 3-Point Bending 4-Point Bending Dual Cantilever Single Cantilever	ZERO NOT USED The sample height is typed in.	ZERO on the Top of the Sample so the sample height and deflection are in the same direction. BASE	BASE ZERO on the Bottom of the Sample	Always type in the sample height. The automatic height is not used
Tensile Extension Tension	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	Sample height is automatically read. The typed value is not used.
Compressive Parallel Plate Cup and Plate Plate and Tray Sintered Plates	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	BASE ZERO on the Bottom of the Sample	Automatically read. Typed-in values are not used.
How Displacement is Calculated	Amplitude (micrometers) Start, Y, ΔY, Time	Displacement (millimeters) Start Run, End Run, Y, ΔY, Time	ΔY is calculated using "SELECT CALC"	

Mounting the Sample

For most standard DMA 7e and TMA 7 experiments, mount the sample following the steps below. Standard experiments include penetration, expansion, and compression tests. For performing experiments using the dilatometer cell, the extension analysis kit, or the flexure kit, refer to the specific instructions that come with the accessory.

1. Carefully lower the furnace assembly to the base of the analyzer by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
2. Press the **Probe Up** button on the front panel to raise the probe assembly and lock it into place.
3. Make sure that all traces of previous samples have been removed.
4. Cover the furnace opening with a business card or some other suitable material.
5. Using the tweezers provided with the instrument, place the sample to be analyzed on the sample tube platform.

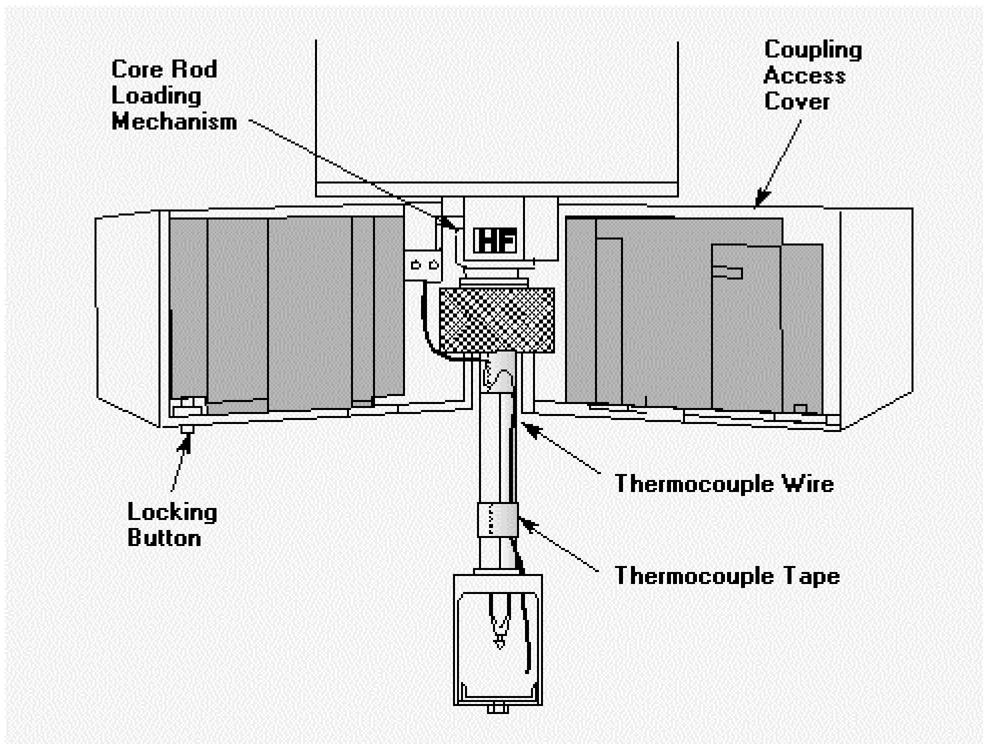
NOTE: If you are running very soft samples in the subambient range (i.e., < 20°C), you should perform step 6 after steps 7 and 8.

6. Press the **Probe Down** button to lower the probe assembly so that it rests on the sample.
7. Carefully raise the furnace assembly all the way up, making sure that it locks in place.
8. Enter the desired temperature in the Go To Temp field in the Control Panel and click on the **Go To Temp** button.

9. When the height signal (mm) displayed in the Status Panel stabilizes, select the **Read Height** button from the Control Panel to automatically read the height of the sample and store it in memory.
10. Place the diffusion cap around the sample tube at the top of the furnace.

Maintenance

Maintenance topics discuss maintaining the TMA 7. The TMA 7 analyzers that were shipped prior to July 1, 1993, are the older analyzers. The new analyzers have a core rod labeled "HF."



The standard shipping version of the TMA 7 includes a turbulent chamber, a small (15 mm) furnace, a flat tip penetration measuring system, a quartz expansion measuring, and a dewar. In order to install the Dewar Kit onto the TMA 7, the turbulent chamber must be removed. If you have only one furnace, you will have to remove the furnace from the turbulent chamber and install it into the dewar.

In order to change from the small (15 mm) furnace to the large (28 mm) furnace on your TMA 7 analyzer, follow the disassembly and assembly instructions inserting the appropriate furnace.

A Dewar Retrofit Kit is available from Perkin Elmer for those users who wish to upgrade their TMA 7 to use the new dewar. The new dewar provides extended LN2 residence time, allowing you to perform longer experiments. The kit must be installed by a Perkin Elmer Service Representative.

WARNING: **Be sure the analyzer is turned off and unplugged before using the instructions below.**

See the following topics for maintenance of the TMA 7:

Dewar Installation

NOTE: The instructions for installing a new dewar into a TMA 7 that has been retrofitted with a new dewar.

The dewar in the TMA 7 analyzer allows you to obtain data in the ambient to subambient temperature range. Liquid nitrogen may be used in the dewar for operation from –170°C. Ice water may be used for operation from 25°C. The standard furnace size for the DMA 7e is the large (28 mm) furnace which gives the analyzer a temperature of –170°C to 500°C, while the TMA 7 analyzer uses a small (15 mm) furnace for a temperature range –170°C to 1000°C. It is possible to use the small furnace in the DMA 7e and to use the large furnace in the TMA 7.

The dewar is easily installed and removed from the analyzer, allowing the turbulent chamber and other environmental systems to be used. It is recommended that you purchase a second furnace if you will be switching between the dewar and the turbulent chamber often. This way you can keep a furnace in each assembly, making the switch faster.

Installation of the dewar into the TMA 7 consists of the following steps:

Turbulent Chamber Removal

NOTE: If you have only one furnace, you will have to remove it from the turbulent chamber and install it into the dewar. This is described in the next subsection.

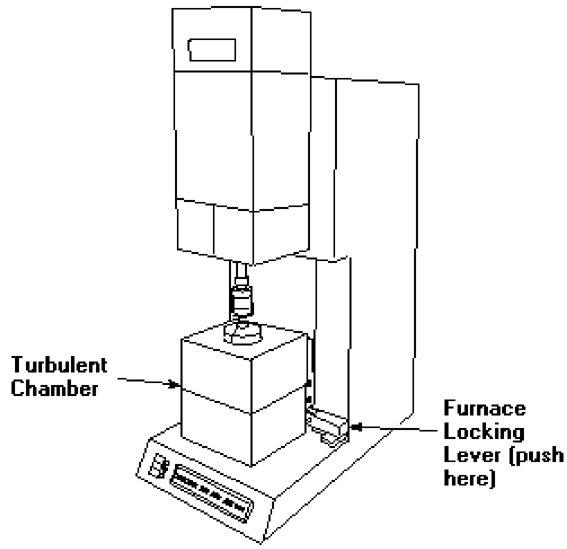
We recommend that you purchase a second furnace if you intend to use both devices. You may want to create and store calibration sets in conjunction with the furnace change.

CAUTION: If you are using a Quartz measuring system and no sample is mounted, you should place the septum (P/N 0009-0652) on the base of the sample tube directly under the probe to prevent damage to the probe or the sample tube.

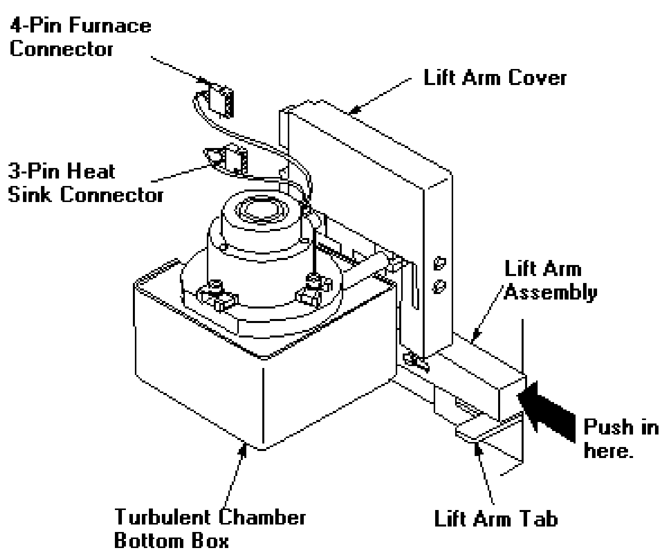
1. Turn off the power to the TMA 7 and the TAC 7/DX.

CAUTION: Be sure to turn the TAC 7/DX off, especially if a new calibration set is going to be used.

2. Remove the diffusion cap.
3. Completely lower the furnace. Make sure that the latch locks the furnace in the fully lowered position.

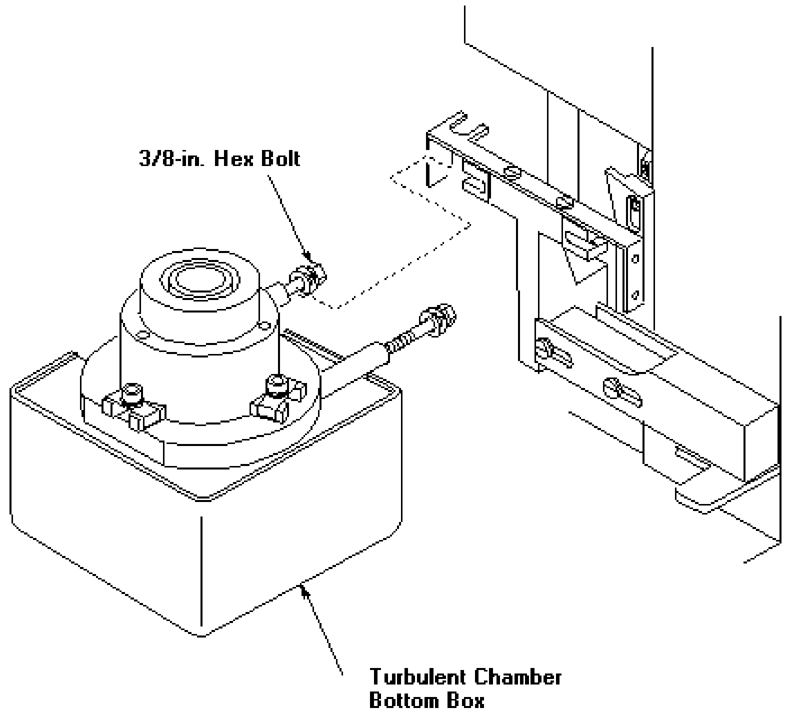


4. Remove the top furnace cover carefully to avoid damaging the measuring system. (If necessary, tilt the turbulent chamber cover toward you.)
5. Unplug the top white (4-pin) furnace connector which is on the left side of the furnace lift arm housing cover.
6. Unplug the bottom white (3-pin) heat sink connector which is also on the left side of the furnace lift arm housing cover.



7. Remove the three flat head screws on the furnace lift arm cover (see figure above). Make sure that the screws do not drop inside the analyzer.
8. Remove the furnace lift arm cover by lifting it straight up and to one side. Make sure that all wires have been cleared out of the way.

For the next five steps refer to the figure below.

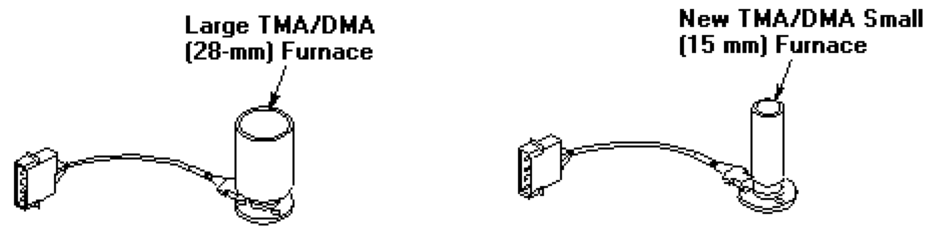


9. Locate the two 3/8-in. mounting box hex bolts that are attached to the mounting ring and furnace assembly lift arm.
10. Completely loosen and remove the leftmost bolt by turning the wrench clockwise.
11. Loosen the remaining bolt to the right by turning the wrench clockwise.
12. Gently move the turbulent chamber bottom box, which contains the circulator and furnace, to the right and remove it from the analyzer.
13. Carefully remove the rightmost bolt from the analyzer and place it into the mounting ring for storage.

Now that the turbulent chamber has been removed from the analyzer, you can remove the furnace from the turbulent chamber.

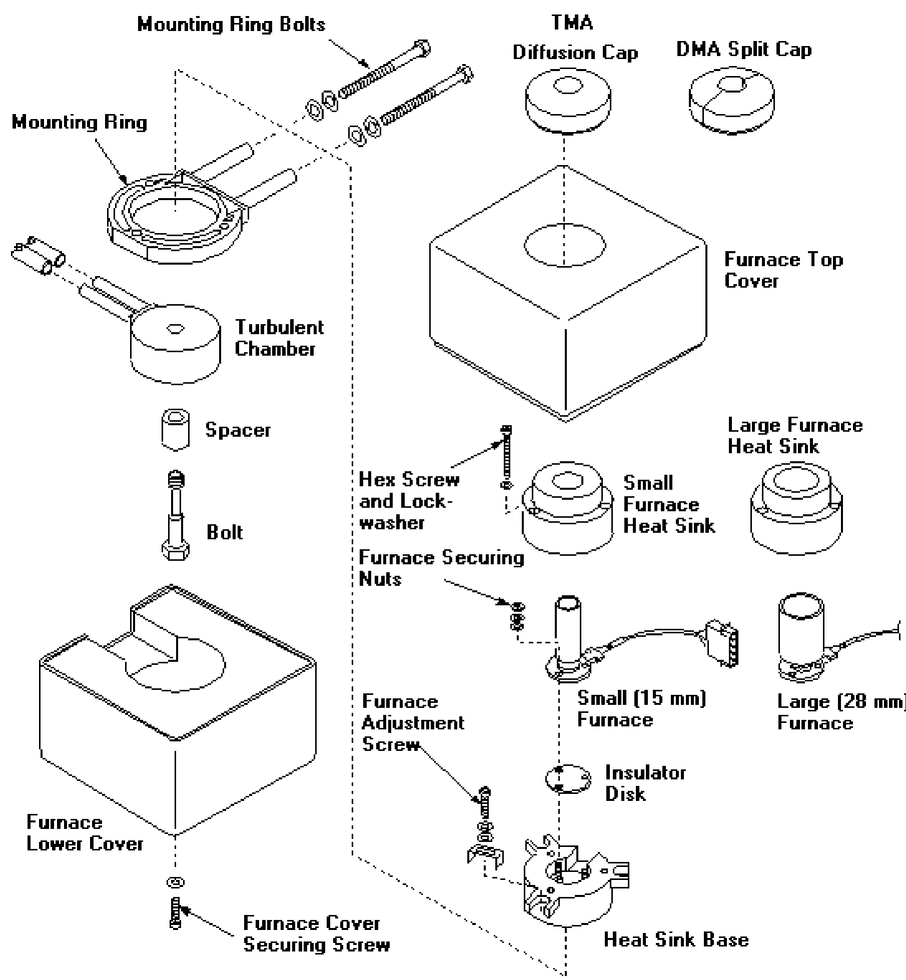
Furnace Removal from the Turbulent Chamber

If you do not have a second furnace to install into the dewar, the furnace in the turbulent chamber will have to be removed and installed in the dewar. Follow the steps here to remove either a **large (28 mm) furnace** or a **small (15 mm) furnace**.



Refer the schematic below and use it to follow the instructions below.

Turbulent Chamber Disassembly Schematic



1. Remove the heat sink by loosening the three 9/64-in. hex head screws using the Allen wrench provided.
2. Carefully lift off the heatsink from the heat sink base; do not bump the sides of the furnace. **The furnace is fragile; it can be broken easily.** Keep the heat sink upright to prevent losing the screws and lockwashers.

The furnace is now exposed.

3. Use a 1/4-in. open-end wrench to loosen the three nuts that hold the furnace in place.

NOTE: The size of the furnace determines the type of mounting screw used. To determine the furnace size, measure the inner diameter of the furnace element.

4. Gently lift the furnace straight up and off of the three setscrews.
5. Replace the three furnace securing nuts onto the setscrews for storage.

Now that the furnace has been removed, it can be installed into the dewar.

Furnace Installation into the Dewar

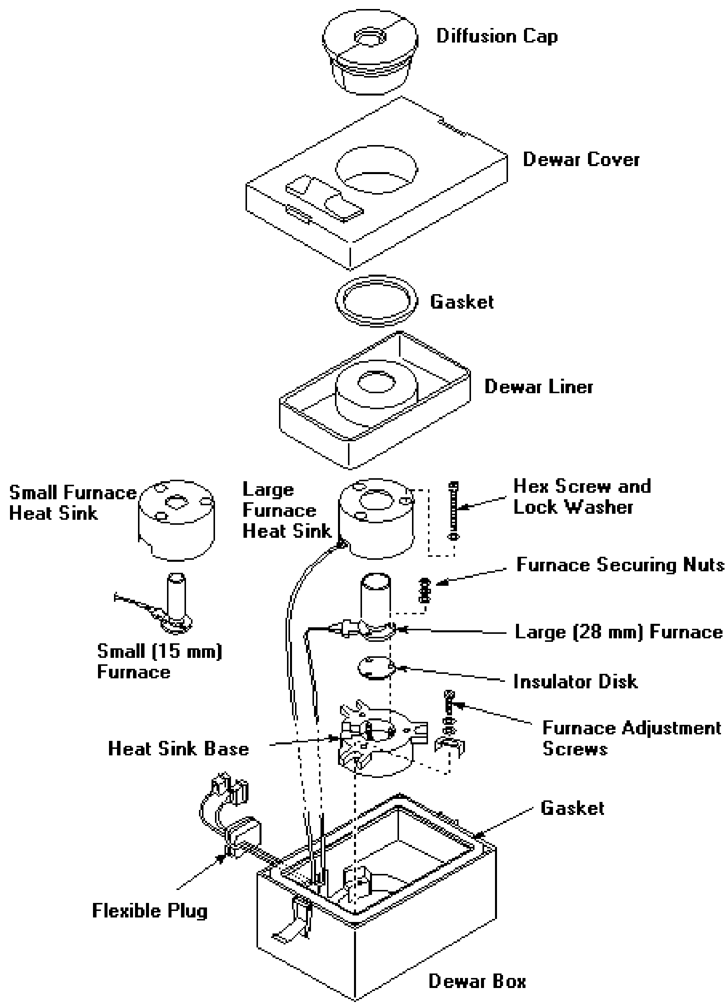
You now can install that furnace into the new dewar. The same instructions can be used for installing a large (28 mm) furnace or a small (15 mm) furnace. Furnace installation consists of the following three steps:

- **Disassembling the Dewar**
- **Installing the Furnace**
- **Assembling the New Dewar**

Disassembling the Dewar

Use the schematic below while following the instructions below.

Dewar Disassembly Schematic



1. Remove the dewar cover by lifting the two latches on the sides of the dewar box.
2. Remove the two gaskets. One is on top of the dewar and the other is on top of the dewar box.
3. Lift the dewar liner out of the dewar box, exposing the heat sink.
4. Loosen the three 9/64-in. hex screws from the heat sink.
5. Lift the heat sink out of the dewar box, taking care to keep the heat sink upright to prevent losing the washers.

   The heat sink base is exposed. If a furnace is installed in the dewar box, it will also be exposed.
6. Remove the three flathead screws from the heat sink base and lift the base out of the dewar box.
7. Place the insulator disk over the three setscrews of the heat sink base.

Now you can install the furnace.

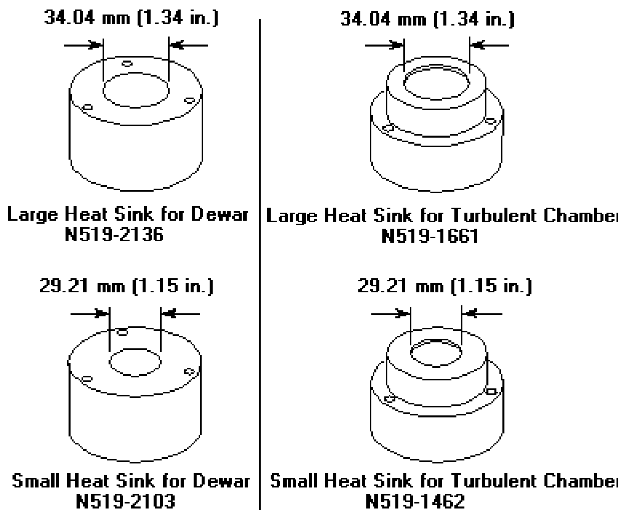
Installing the Furnace into the New Dewar

1. Place the furnace onto the heat sink base by lining up the three holes on the furnace with the three setscrews on the heat sink base.
2. Place a flat washer, a lock washer, and 1/4-in. hex nut onto each setscrew. Tighten the hex nut carefully until it is snug.

CAUTION: DO NOT overtighten the hex nuts; it could cause the furnace to break.

NOTE: You may want to have a second furnace so that the dewar and the turbulent chamber can each have a dedicated furnace.

3. Place the heat sink over the furnace and onto the heat sink base. Be sure to select the appropriate heat sink for the furnace you are using.



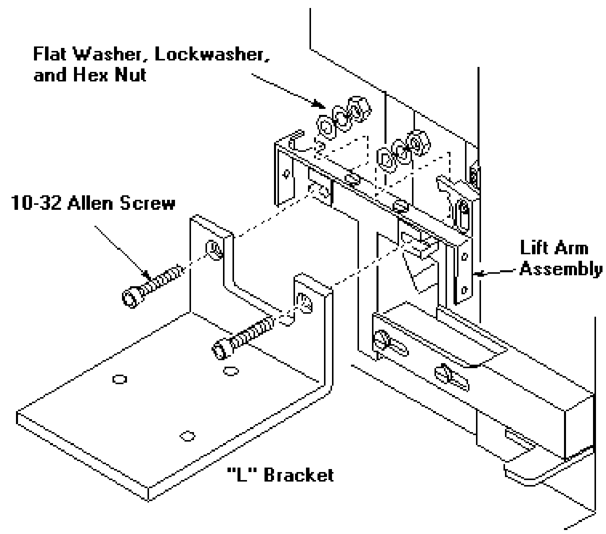
4. Place the three lock washers and 9/64-in. hex screws into the heat sink and tighten them.

Finally, you can assemble the new dewar.

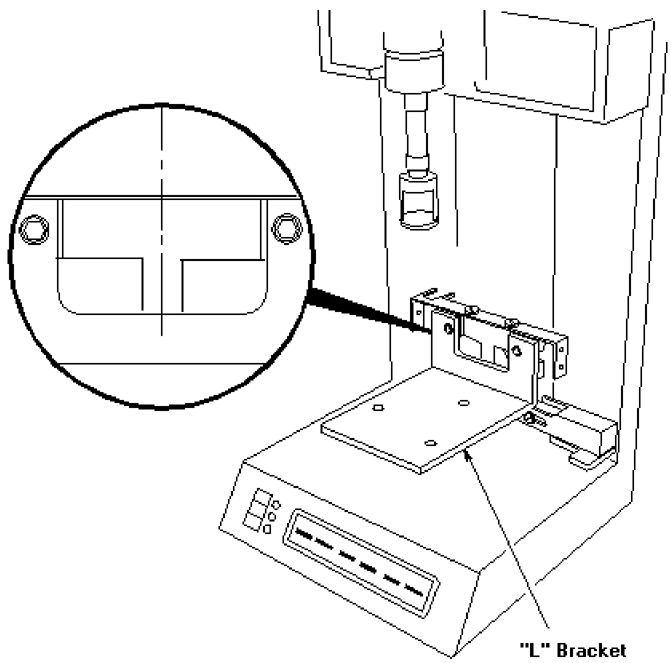
Assembling the New Dewar

At this point a furnace has been installed into the heat sink assembly of the dewar. In the instructions here, when heat sink is mentioned, the furnace is included.

1. Thread the electrical connectors for the furnace and the heat sink through the hole in the back of the dewar box.
2. Place the heat sink base (with furnace and heat sink on top) into the bottom of the dewar box.
3. Install the three brackets, washers, and flathead alignment screws. Make sure to secure them in place. **DO NOT OVERTIGHTEN**. The assembly will be moved for alignment later.
4. Place the flexible plug over the wires in back of the dewar box, pressing the plug into the hole at the back of the box. When the plug is properly seated, it will snap into place.
5. Attach the "L" bracket to the lift arm assembly by placing a 10-32 Allen screw into the left-hand hold of the bracket. Secure the screw by placing a lockwasher, a washer, and a hex nut on the back of the screw and tighten the hex nut.



6. Place a 10-32 Allen screw in the right-hand hole of the "L" bracket. Secure the screw by placing a lockwasher, a washer, and a hex nut on the back of the screw and tighten the hex nut.
7. Adjust the bracket so that it is centered. Once the bracket is centered, tighten the two Allen screws.

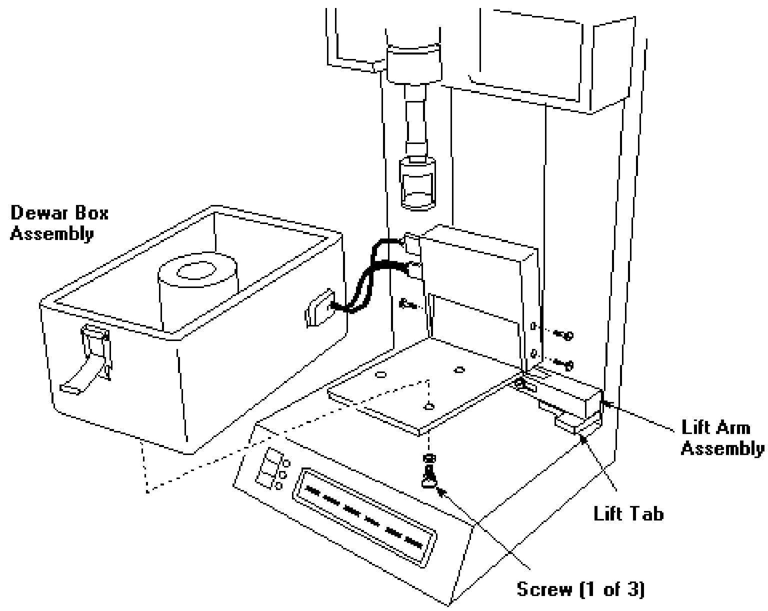


8. Replace the furnace lifting arm cover with the three flathead screws.

Now you can connect the dewar.

Connecting the Dewar

1. Plug the heat sink's 3-pin connector into the left side of the analyzer.



2. Plug the furnace's 4-pin connector (P/N N519-0781) into the left side of the analyzer above the 3-pin connector.
3. Slide the dewar into place on the furnace "L" bracket.
4. Holding the dewar in place with your left hand, unlock the furnace latch with your right hand and raise the dewar and furnace into position. Make sure that it locks into position.

CAUTION: DO NOT let the furnace assembly rise too quickly and impact the measuring system since the furnace or the measuring system may be damaged.

5. Insert the knurled head screws into the base of the dewar, going through the bottom of the "L" bracket. Tighten the screws; however, do not overtighten.
6. Align the furnace by moving either the heat sink assembly or the dewar box so that the measuring system is centered inside of the furnace.
7. Tighten the knurled nut on the base of the dewar box.
8. Lower the base.
9. Tighten the three flathead alignment screws that are on the heat sink base.
10. Insert the dewar liner.
11. Place the rectangular gasket inside the dewar box and around the ridge between the dewar liner and the dewar box.
12. Place the circular flat gasket on top of the heat sink cover in the dewar liner.
13. Replace the dewar lid.

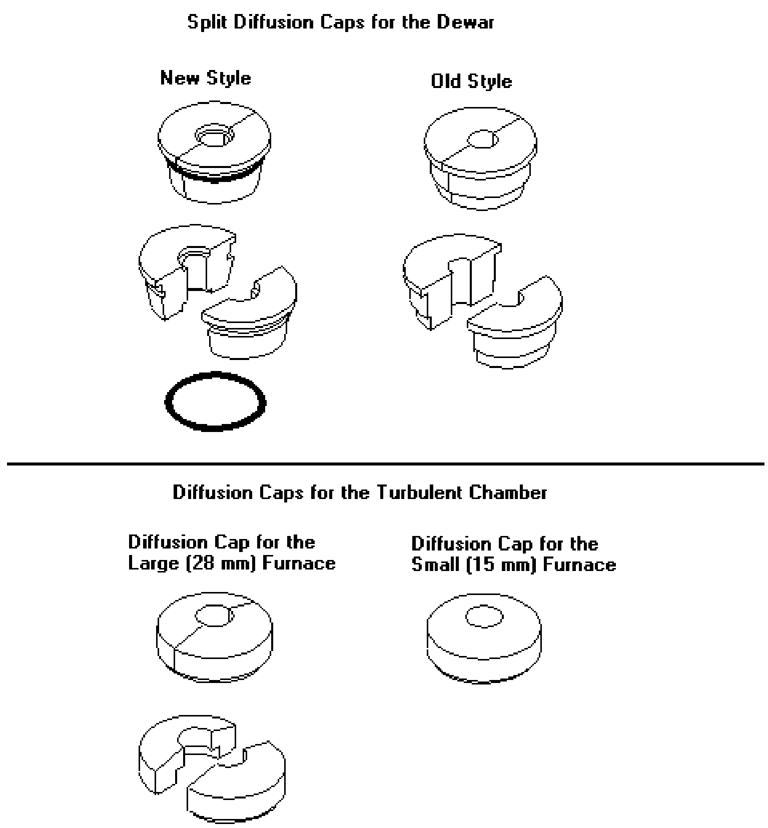
14. Visually inspect the circular gasket so that it does not extend toward the furnace. A good seal is necessary to sustain the low temperature for an extended period.
15. Lock the dewar lid into place using the latches on the side of the dewar box.
16. Insert the funnel into the dewar opening if you are going to fill it with liquid nitrogen, or put the filler cover in place if you do not intend to fill the dewar immediately.

Finally, you can prepare the dewar for use.

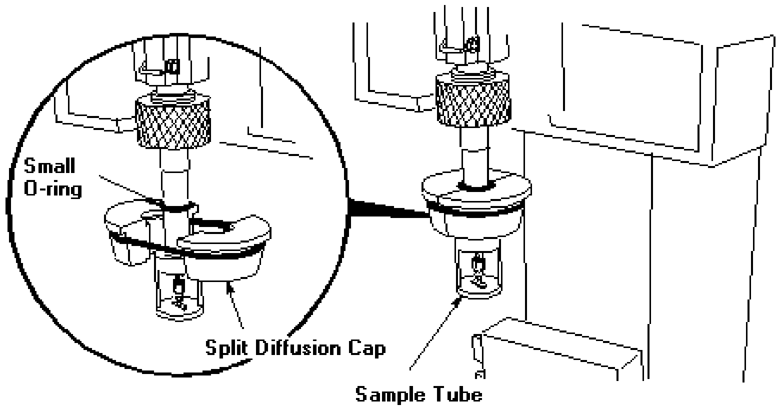
Preparing the Dewar for Use

The final step in installing the new dewar into a TMA 7 is preparing the dewar for experiments.

1. Slide an O-ring over the two halves of the split diffusion cap. The O-ring should slip into the groove under the lip of the cap.



2. Place a small O-ring over the measuring system and slide it up the shaft of the universal sample tube.
3. Pull the two halves of the split diffusion cap open so that they go around the measuring system. Slide the split diffusion cap up the universal sample tube so that the cap rests on top of the tube.



4. Roll the small O-ring down into the diffusion cap.
5. If the coupling access cover is open, close it.
6. Raise the furnace and, if necessary, press the O-rings into place.

The system is now ready to use. Power up the system. The Ready and Down lights on the front panel should light up. If any other lights come on, there is an improper connection. Check the cables and connections. If necessary, disassemble the dewar and reassemble it, making sure to follow all of the steps in the procedure.

Furnace Replacement in the Dewar

These instructions are for replacing the furnace in the dewar.

NOTE: The older DMA 7/TMA 7 analyzers can be retrofitted with the new style dewar by having a Perkin Elmer Service Representative install the Dewar Retrofit Kit onto the analyzer. The new style dewars provide extended LN2 residence time, allowing longer experiments to be performed. Contact your local Perkin Elmer Representative for details.

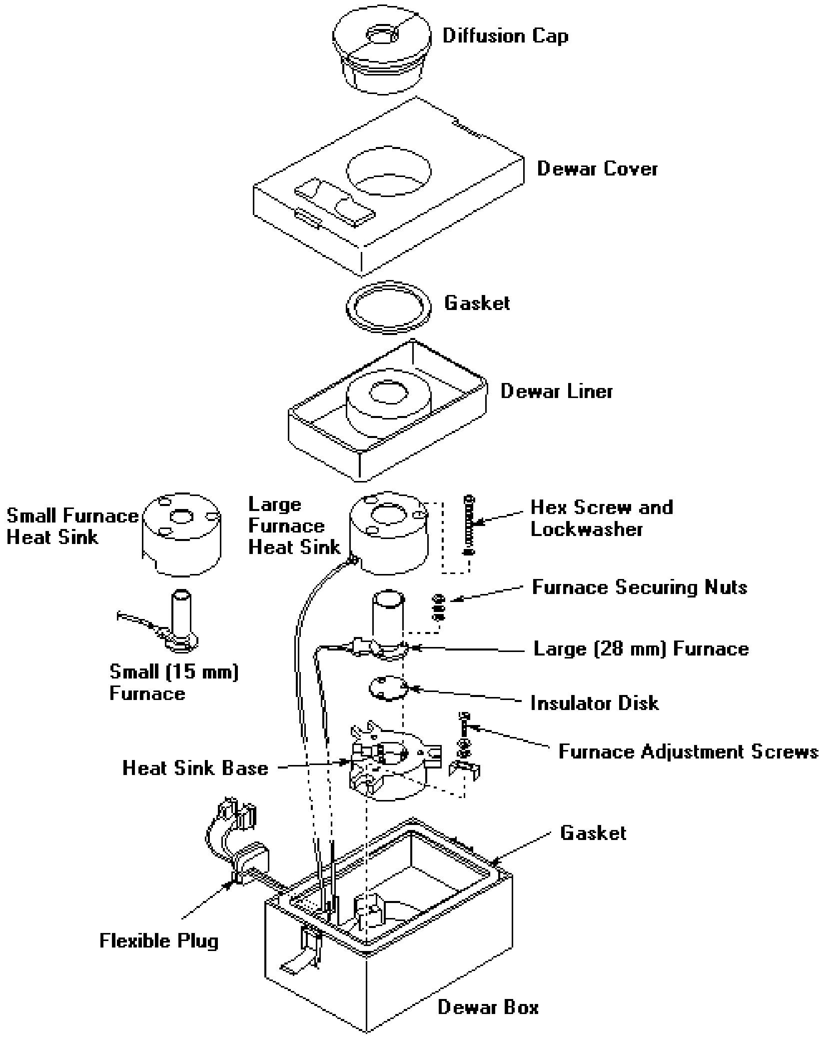
Replacing the furnace in the new dewar consists of three major steps:

- **Remove the Furnace from the Dewar**
- **Install the Furnace in the Dewar**
- **Align the Furnace in the Dewar**

Furnace Removal from the Dewar

See a schematic of the dewar with furnace below and use it to follow the instructions below.

Dewar Assembly with Furnace



1. Lower the dewar assembly and lock it into place at the base of the analyzer.
2. Remove the dewar cover by lifting the two latches on the sides of the dewar box and disconnecting them from the top cover.
3. Remove the two gaskets. One is on top of the dewar liner and the one is on top of the dewar box.
4. Lift the dewar liner out of the dewar box, exposing the heat sink.
5. Remove the three hex screws from the heat sink.
6. Lift the heat sink out of the dewar box, taking care not to lose the washers.
7. Unplug the furnace’s 4-pin connector and the heat sink’s 3-pin connector from the analyzer.
8. Remove the flexible plug from the rear of the dewar and guide the connectors through the hole.
9. Remove the three hex screws from the heat sink base and lift the base out of the dewar box.

10. Remove the three furnace securing nuts, lockwashers, and flat washers from the setscrews using a 1/4-in. open-end wrench.
11. Lift the furnace straight up and off of the setscrews.

NOTE: DO NOT remove the insulator disk. However, if it is broken, replace it.

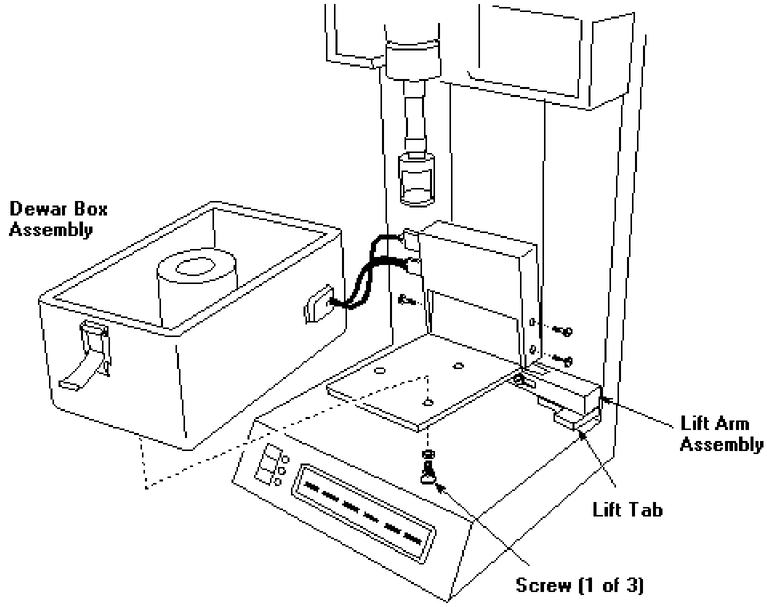
The next step is installing the furnace into the new dewar.

Furnace Installation into the Dewar

1. Examine the insulator disk that is on top of the heat sink base. If the disk is broken, replace it with a new one.
2. Place the furnace onto the heat sink base by lining up the three holes on the furnace base with the three setscrews on the heat sink base.
3. Place a flat washer, a lockwasher, and a 1/4-in. hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since you could damage the furnace.

4. Thread the connectors for the furnace and the heat sink base through the hole in the back of the dewar box.
5. Place the heat sink base (with furnace attached) into the bottom of the dewar box.
6. Insert the three hex head alignment screws and secure them in place. Do not overtighten the screws since later on you will be moving the assembly to align the furnace.
7. Carefully place the heat sink onto the heat sink base. Take care not to bump the furnace; it is extremely fragile.
8. Place the three hex screws into the heat sink and tighten them.
9. Place the flexible plug over the wires in back of the dewar box. Press the plug into the hole at the back of the dewar box. When the plug is properly seated, it will snap into place.
10. Plug the heat sink's 3-pin connector into the left side of the analyzer above the lift arm cover.
11. Attach the furnace's 4-pin connector (P/N N519-0781) into the left side of the analyzer above the 3-pin connector.



12. While holding the dewar in place with your left hand, unlock the furnace latch with your right hand and raise the dewar and furnace into position. Make sure that it locks into position.

The sample tube should be in the center of the furnace. Proceed with aligning the furnace.

Furnace Alignment in the Dewar

The final step in replacing the furnace in a dewar is aligning the furnace.

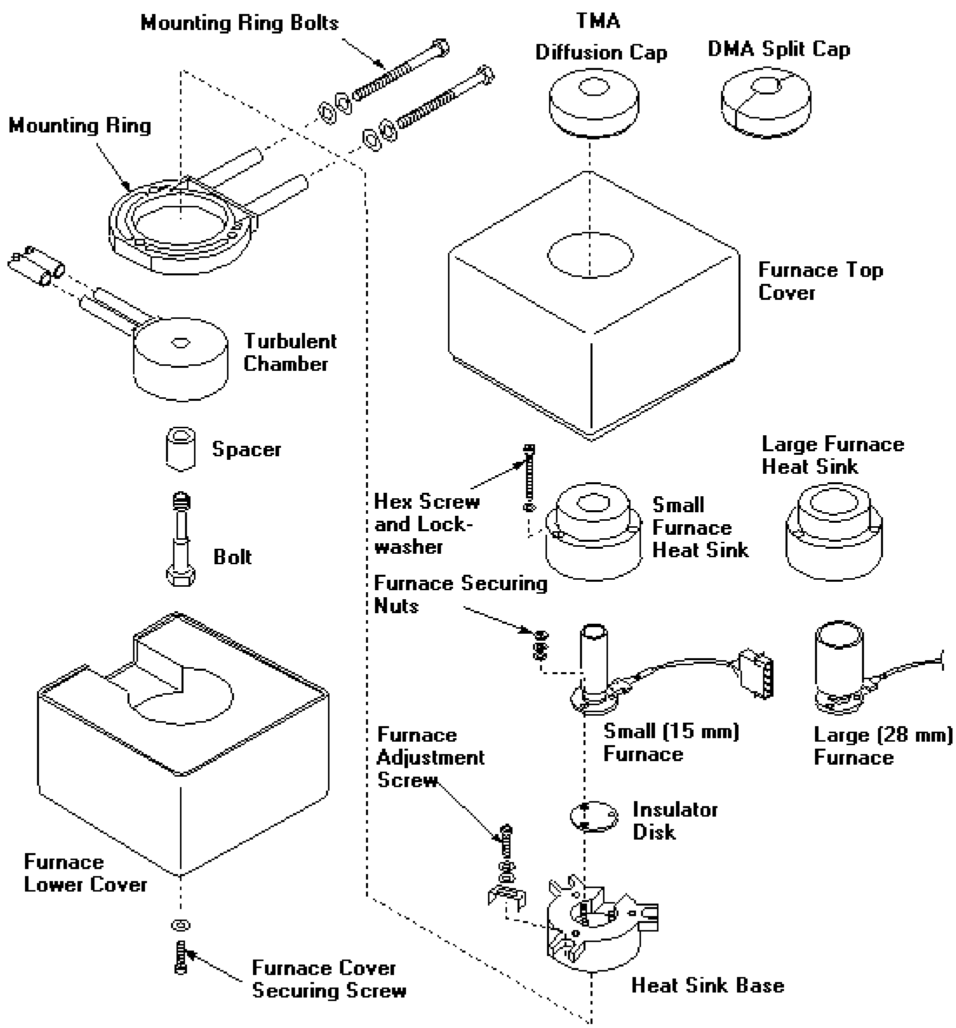
1. Loosen the three furnace adjustment screws that secure the heat sink base to the dewar box assembly.
2. Slowly elevate the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the tip of the sample tube but does not come into contact with it. Make sure that the furnace assembly is locked into the elevated position.
3. Continue making final adjustments to the furnace/heat sink assembly. Make sure that the sample tube is centered inside the furnace. When centering is complete, lock the furnace/heat sink assembly into place by tightening the three furnace adjustment screws.
4. Insert the dewar liner.
5. Place the rectangular gasket inside the dewar box and around the ridge between the dewar liner and the dewar box.
6. Place the circular flat gasket on top of the heat sink cover (which is part of the dewar liner).
7. Replace the dewar cover.
8. Visually inspect the circular gasket so that it does not extend outward toward the furnace. A good seal is necessary to sustain the low temperatures for an extended period.
9. Lock the dewar cover into place with the latches on the side of the dewar box.
10. Insert the funnel into the dewar opening if you are going to fill it with liquid nitrogen, or place the filler cover in place if you do not intend to fill the dewar immediately.

Furnace Replacement in Turbulent Chamber

The instructions below are for replacing the furnace in the turbulent chamber of a TMA 7.

- **Remove the Furnace from the Turbulent Chamber**
- **Install the Furnace in the Turbulent Chamber**
- **Align the Furnace in the Turbulent Chamber**

Furnace Assembly with Turbulent Chamber



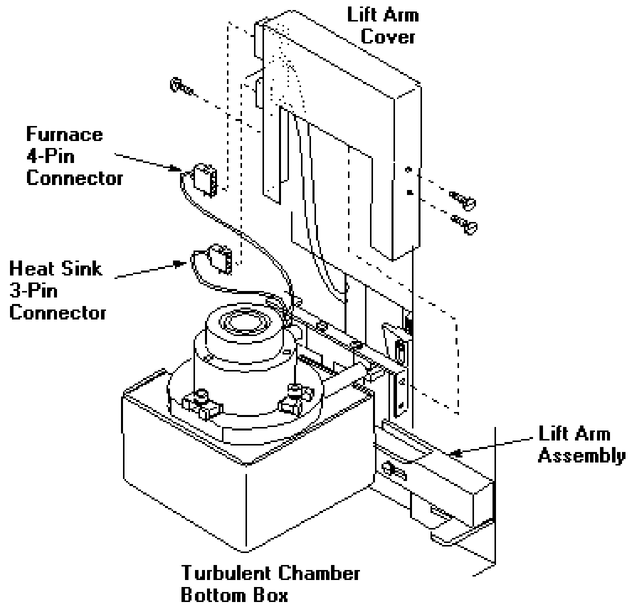
Furnace Removal from the Turbulent Chamber

See the display above of the furnace assembly with the turbulent chamber to refer to as needed as you perform the steps below.

The first step in replacing the furnace in the turbulent chamber is to remove the existing furnace:

1. Lower the furnace assembly and lock it in place at the base of the analyzer.
2. Remove the top furnace cover from the furnace assembly.

3. Unplug the 4-pin furnace connector from the analyzer.



4. Remove the three hex screws and the three lockwashers from the heat sink and lift off the heat sink.
5. Remove the three furnace securing nuts on the base of the furnace.
6. Lift the furnace straight up and off of the setscrews.

NOTE: Do not remove the insulator disk. If the disk is broken, replace it.

The next step in replacing the furnace in the turbulent chamber is installing the furnace.

Furnace Installation in the Turbulent Chamber

Install the new furnace into the turbulent chamber as follows:

1. Examine the insulator disk that is on top of the heat sink base. If it is broken, replace it.
2. Place the furnace over the three setscrews, with the furnace connector facing the rear of the analyzer.
3. Place a flat washer, a lockwasher, and hex nut on each setscrew. Use a 1/4-in. open-end wrench to tighten the hex nuts.

NOTE: DO NOT overtighten the hex nuts since it can cause the furnace to break.

4. Attach the 4-pin furnace connector to the left side of the analyzer.
5. Install the heat sink. Use the three hex screws and lock washers that you removed from the heat sink earlier to secure the heat sink in place.

The final step in replacing the furnace in the turbulent chamber is alignment.

Furnace Alignment in the Turbulent Chamber

1. Loosen the three furnace adjustment screws which secure the heat sink base to the furnace lift arm.
2. Slowly raise the furnace assembly, adjusting the furnace/heat sink so that the furnace surrounds the tip of the sample tube but does not come into contact with it. Make sure that the furnace assembly is locked into place in the highest position on the analyzer.
3. Continue to make final adjustments to the furnace/heat sink assembly. Make sure that the sample tube is centered inside the furnace. When centering is complete, lock the furnace/heat sink assembly into place by tightening the three furnace adjustment screws.
4. Place the cover over the turbulent chamber.

Replacing Large Furnace with Small Furnace

To replace a large furnace with a small furnace in a newer TMA 7, see the appropriate subtopic for your system setup:

- **Furnace Replacement in Turbulent Chamber**
- **Furnace Replacement in the Dewar**

Old Dewar Installation into the TMA 7

For instructions on installing the old dewar into TMA 7, see *Chapter 7, DMA 7/7e Analyzer*. The instructions for installing the dewar into the DMA 7 apply to the TMA 7 as well.

Furnace Replacement in Turbulent Chamber of Older TMA 7

For instructions on replacing the furnace in the turbulent chamber in an older TMA 7, see *Chapter 7, DMA 7/7e Analyzer*, the subsection "Furnace Replacement in Turbulent Chamber of DMA 7/TMA 7."

Replacing Large Furnace with Small Furnace in Older TMA 7

To change from the large furnace to the small furnace in the older TMA 7, see *Chapter 7, DMA 7/7e Analyzer*, the subsection "Furnace Replacement in Old Dewar" or "Furnace Replacement in Turbulent Chamber of the DMA 7/TMA 7."

Upgrade TMA 7 with New Dewar

The TMA 7 analyzer shipped prior to July 1, 1993, is considered an older TMA. A Dewar Retrofit Kit is available from Perkin Elmer for those users who want to upgrade their TMA 7 for use with the new dewar. The new style dewar provides extended LN2 residence time, allowing you to perform longer experiments. The kit must be installed by a Perkin Elmer Service Representative.

Replacing the Thermocouple

A thermocouple is a device that generates a differential voltage as temperature is increased or decreased. The voltage is generated by fusing wires made from two different metals at the tip. When this device is repeatedly flexed, the point at which the two wires are fused is stressed and can occasionally fail. Restricting the movement of the thermocouple can greatly improve its service life. The high-temperature tape, provided in the Spares Kit, restricts the movement of the thermocouple when placed at the top and bottom of the sample tube. This tape also serves as an insulator.

Although the calibration software can compensate for thermocouple placement, careful routing and placement of the thermocouple can improve thermal performance of the analyzer, especially when stainless steel measuring systems are used.

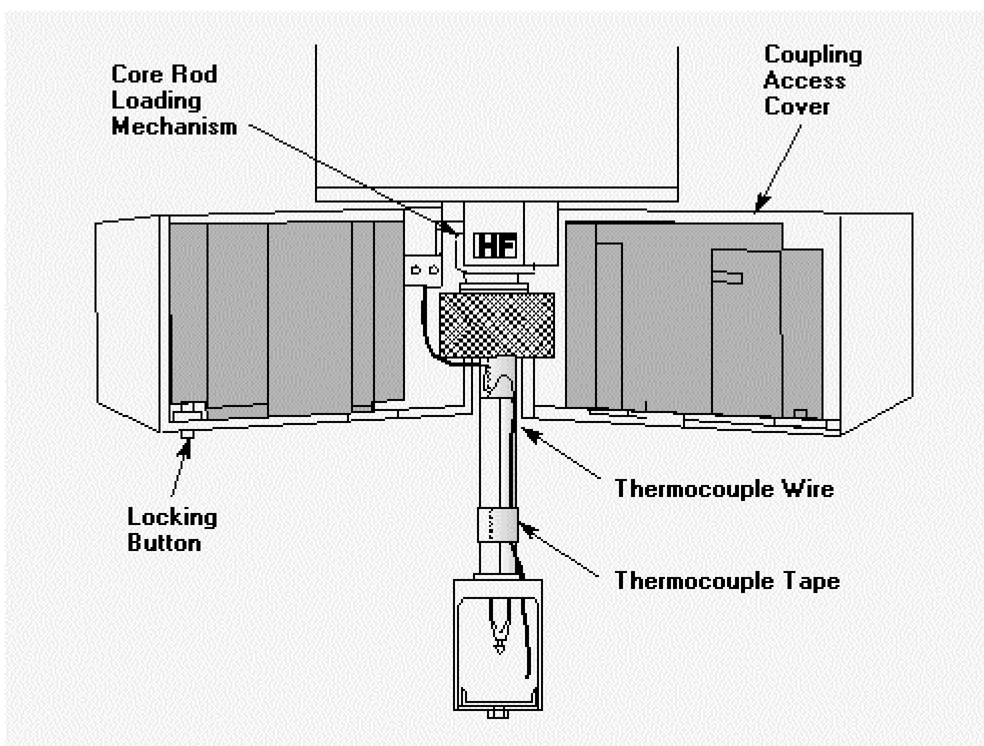
CAUTION: The thermocouple should not touch any metal parts.

To replace the sample thermocouple, you must first remove the old thermocouple and then install the new one.

Removing the Thermocouple

NOTE: Before performing this procedure, shut the system down and turn off the analyzer. Remove the line power from the analyzer. Make sure that the furnace, sample tube, and probe are sufficiently cooled so that you do not burn yourself.

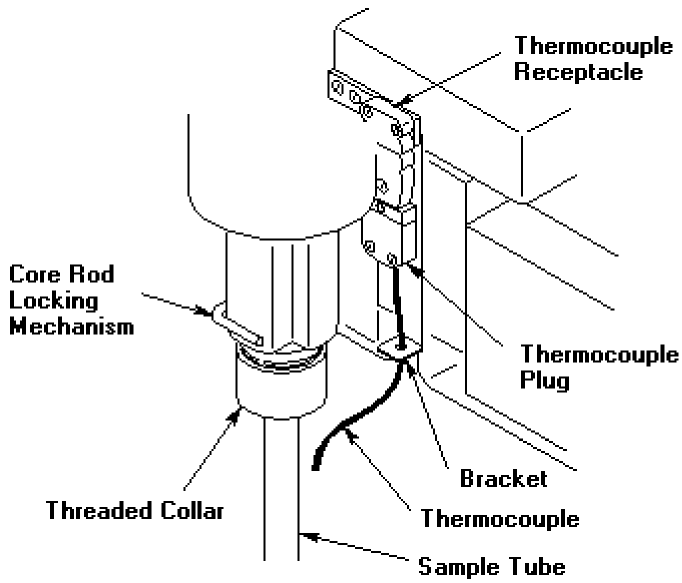
1. Remove the furnace diffusion cap and carefully lower the furnace assembly to the base of the analyzer by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the analyzer.
2. Press the locking button that secures the coupling access covers and separate the doors.



3. Remove the thermocouple from the sample tube by grasping the thermocouple just above the point where it enters the sample tube and then pulling it straight out.

NOTE: The thermocouple may be taped to the sample tube. This is usually done to provide consistent thermocouple positioning in the measuring system.

4. Unplug the thermocouple from the receptacle at the back of the probe assembly by gently grasping the sides of the thermocouple connector and pulling the connector straight down.
5. Pull the thermocouple up through the bracket.



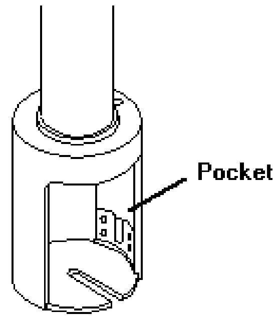
Installing the Thermocouple

Follow the instructions below when installing for the first time or replacing the TMA 7 sample thermocouple.

1. Take the new thermocouple and plug it into the receptacle at the back of the probe assembly. Observe the polarity (+ or –) as marked on the plugs.
2. Apply a piece of tape along the length of the sample tube. This insulates the thermocouple from the sample tube.
3. Route the thermocouple along side of the sample tube on top of the tape, making sure to include the loop at the top to prevent tip movement.

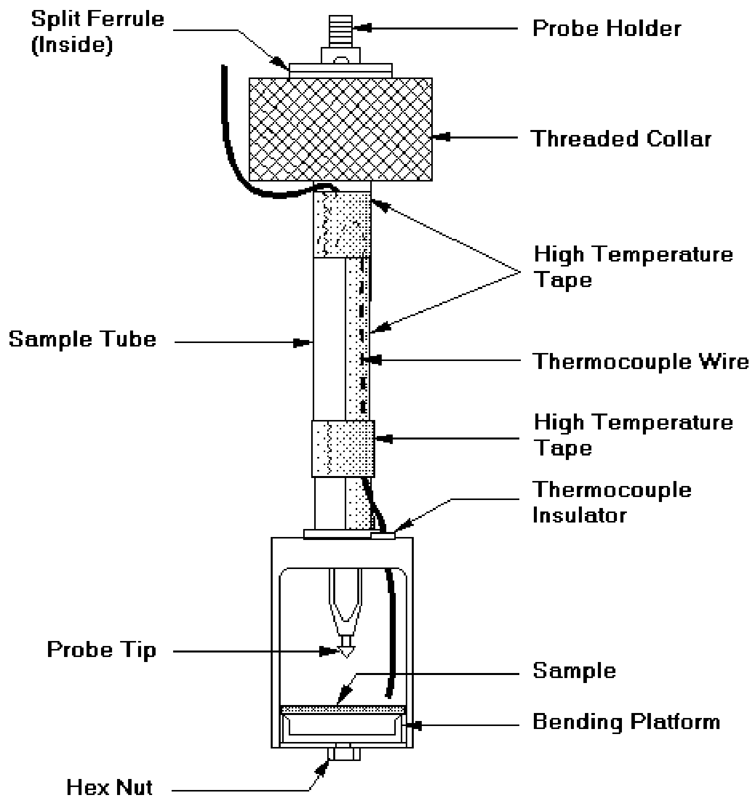
CAUTION: Do not route the thermocouple through the mounting bracket. This allows you to remove the sample tube from the analyzer without disturbing the thermocouple route or position. Remember, do not make any sharp bends in the thermocouple. This may damage the thermocouple.

4. Place the thermocouple insulator (a white ceramic sleeve about 2 mm long and 1 mm wide) in the hole in the top of the base of the sample tube.
5. Route the tip of the thermocouple through the insulator.
6. If you are using the stainless steel sample tube (P/N N539-0107), place the tip of the thermocouple all the way down into the bottom of the thermocouple pocket on the inside side wall of the sample tube.



If you are using the standard quartz sample tube, the thermocouple should extend straight down the tube's sidewall and rest near the platform surface without touching the sample, the probe, or the sample holder.

7. Apply another piece of tape over the thermocouple and over the first piece of tape. This will hold the thermocouple in place.
8. Apply a piece of tape around the thermocouple at the top and bottom of the sample tube. This will secure the thermocouple.



NOTE: The top of the sample tube does not reach extreme temperatures during normal use. However, the adhesive on the high-temperature tape may age over time or if very high temperatures over long periods of time are used. Positioning the lower tape wrap approximately 1 cm from the thermocouple insulator should reduce the aging of the tape adhesive. Replace the tape if discoloration or other signs of wear appear.

9. Slowly close the coupling access covers, making sure that you do not pinch the thermocouple in the doors as they are closed.
10. The thermocouple should exit through the rear of the access covers. Notice that a space is present at the back of the access covers where the thermocouple can pass without being bent or pinched.

The thermocouple installation is now complete.

Maintenance of Sample Tubes and Probes

Maintenance of the sample tube and probe of the TMA 7 entails:

- **Removing the Sample Tube and Probe**
- **Cleaning the Sample Tube and Probe**
- **Replacing the Sample Tube and Probe**

These are topics in Pyris Multimedia Presentations Help.

Cleaning Sample Tubes and Probes

To clean the quartz sample tube and probe, follow the instructions below:

WARNING: **Wear protective gloves and safety glasses when cleaning the sample tube and probe. Use a fume hood when working with solvents or cleaning solutions.**

1. For organic contaminants, insert the tip of the probe or the base of the sample tube over a Bunsen burner for several seconds.

CAUTION: Heat the quartz glassware for only a few seconds. Prolonged heating could damage the glassware.

2. In some cases, it may be necessary to dissolve contaminants from the glassware using suitable solvents. Dip the glassware in the solvent to dissolve the contaminant.

Accessories, Replacement Parts, and Parts Provided

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

Parts Provided

TMA 7 with Dewar Assembly and Turbulent Chamber

N519-0090	100 V, 50 Hz/60 Hz
N519-0091	115 V, 60 Hz
N519-0092	230 V, 50 Hz

Spares and Accessories Kit (N519-0600)

Part No.	Description	Quantity
N519-0376	Flat Tip Penetration Probe	1
N519-0378	Expansion Probe	1
N519-1549	Sample Deformation Tube	2
N519-0417	Thermocouple	1
N539-1051	Height Calibration Standard	1
0990-8825	Force Calibration Standard (50 g)	1
N519-1469	Eigendeformation Calibration Standard	1
0990-7278	Allen Key	1
0990-8400	Tweezers (nonmagnetic)	1
0319-0033	Indium Calibration Standard	1
0319-0036	Zinc Calibration Standard	1
N519-0392	Weight Platform	1
0998-1614	2A Fuse	4
0998-1626	3A Fuse	2
0998-1616	4A Fuse	2
0154-1498	Type “H” Purge Gas Restrictor	1
0990-3906	Female Connector	2
0250-6483	Teflon Tubing 1/8-in. o.d. x 1/16-in. i.d.	24 ft
0990-8134	Thread Sealant Tape	1
0992-0008	Hose Clamp	10
0250-6519	Tygon Tubing (1/2-in. o.d., 3/8-in. i.d.)	24 ft
0419-1434	Liner Top	1
0990-8521	Plastic Box	1
N519-1663	Liner	1

N519-1664	Card	1
N519-1665	Label	1

NOTE: Parts provided are subject to change and may not be reflected here. The parts provided are those listed in the current price list.

TAC 7/DX Interface

Part No.	Description	Quantity
N539-0070 N519-0310	TAC 7/DX (100 V) Interface TAC 7/DX to Analyzer Cable	1
N519-0071 N519-0310	TAC 7/DX (115 V, 50/60 Hz) Interface TAC 7/DX to Analyzer Cable	1
N519-0072 N519-0310	TAC 7/DX (230 V, 50 Hz) Interface TAC 7/DX to Analyzer Cable	1

Accessories and Replacement Parts

The following accessories and replacement parts can be ordered for use with the TMA 7:

Furnace and Furnace Accessories

Part No.	Description
N519-0370	Furnace Assembly, small, high-temperature, 15-mm i.d. (to 1000°C)
N519-1431	Platinum Furnace Shield
N519-1580	Furnace Diffusion Cap
N519-0417	Sample Thermocouple

Quartz Probes and Probe Kits

Part No.	Description
N519-0376	Penetration Probe, 1-mm-diameter flat tip
N519-0377	Compression Probe, 0.5-mm-radius (0.020-in.) spherical tip
N519-0378	Expansion Probe, 3.7-mm-diameter flat tip
N519-0416	Compression Probe, 1.5-mm-radius (0.06 in.) spherical tip

Flexure Analysis Kit (N519-0399)

Includes:

Part No.	Description
N519-0393	Flexure Probe
0219-0215	Flexure Platform, 5-mm
0993-7156	Instruction Manual

Extension Analysis Kit (N519-0619)

Includes:

Part No.	Description
N519-0609	Quartz Extension Probe
N519-1700	Quartz Extension Sample Tube
0993-7243	Instruction Manual
N519-0611	Clamp Assembly
N519-0615	Sample Load Fixture

Small Quartz Dilatometer Kit (0319-0461)

0.1-mL capacity to be used with the 15-mm-diameter furnace.

Includes:

Part No.	Description
0319-1705	Small quartz dilatometer barrel and plunger
0419-0197	Aluminum oxide powder, vial of 28 g
0993-9318	Instruction Manual

Large Quartz Dilatometer Kit (N519-0763)

1-mL capacity to be used with the large 28-mm-diameter furnace.

Includes:

Part No.	Description
N519-2094	Large quartz dilatometer barrel and plunger
0419-0197	Aluminum oxide powder, vial of 28 g
N519-1837	Large Quartz Sample Tube
0993-8821	Instruction Manual

Calibration Materials

Part No.	Description
0219-1269	Sapphire Disk for Ordinate Calibration
0319-0033	Indium Temperature Calibration Reference Material
0319-0036	Zinc Temperature Calibration Reference Material

Purge Gas Accessories

Part No.	Description
0154-1496	Type "A" Purge Gas Restrictor (provides a flow of ~10 cc/min purge per lb of pressure)
0154-1498	Type "H" Purge Gas Restrictor (provides a flow of ~ cc/min purge per lb of pressure)
0990-3906	Female Connector
0250-6483	Teflon Tubing, 1/8-in. o.d. x 1/16-in. i.d. (24 ft)
0990-8134	Thread Sealant Tape
0212-1127	Pressure Regulator for Gas Line Use (secondary regulator 0 – 100 psi)
0240-0084	Oxygen Pressure Regulator
0240-0085	Helium/Nitrogen Pressure Regulator
0290-1624	Float Displacement Flow Meter
0023-0522	Two-Stage, Soap Bubble Gas Flow Meter
0319-0039	Filter Dryer Kit

Cooling Accessories

Part No.	Description
0319-0205	Intracooler 1 (115 V)
0319-0206	Intracooler 1 (230 V)
0329-0207	Intracooler 2 (115 V)
0329-0208	Intracooler 2 (230V)
N519-0514	TMA 7 Liquid Nitrogen Dewar Assembly
0319-0101	TMA 7 Turbulent Chamber

Miscellaneous Accessories

Part No.	Description
0419-0197	Aluminum Oxide Powder (vial of 28 g)
0990-8353	Teflon Spray Lubricant (for probe coupling)
0990-7280	Outside Micrometer Caliper (0 – 1 in.)

0990-7281	Outside Micrometer Caliper (0 – 25 mm)
N519-1643	TMA 7 Dust Cover
0990-8400	Tweezers, nonmagnetic

Automatic Gas Selector Accessories

The GSA 7 and the TAGS are automatic gas switching accessories that can be used with the TMA 7. Directly controlled from Pyris Software for Windows, the GSA 7 and the TAGS permit you to select the time into a run when a purge gas change is to be made. There are two separate purge gas inlets and one purge gas outlet on the GSA 7; there are 4 purge gas inlets on the TAGS.

Part No.	**Description**
N519-0270	GSA 7 115 V, 60 Hz
N519-0271	GSA 7 230 V, 50 Hz
N519-0269	GSA 7 100 V, 50/60 Hz
N520-2019	TAGS, 110/120 and 220/240 V

Helium/Nitrogen Pressure Regulator

Two-stage regulator for precise outlet pressure control. Brass body and stainless steel diaphragm for long life and minimum maintenance. Maximum inlet pressure of 20,700 kPa. Supplied with CGA-580 adapter.

Float Displacement Flow Meter

Float Displacement Flow Meter allows accurate control and conservation of purge gas, measures nitrogen flow from 0 to 100 cc/min, includes 1/8-in. NPT female connections.

Oxygen Pressure Regulator

Guarantees constant delivery pressure even when cylinder pressure varies. Two-stage regulator with brass body and stainless steel diaphragm. Maximum inlet pressure of 20,700 kPa. Supplied with CGA-540 adapter. Includes a Swagelok 1/4-in. NPT to 1/8-in. fitting which is used to attach the regulator to the purge gas tubing.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the plug attached to the N519-0310 analog cable at the back of the TAC 7/DX Thermal Analysis Instrument Controller. The TAC 7 then connects to the analyzer.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Instructions for installing TAGS are given in the online Installation Help.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 1/4-in. pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

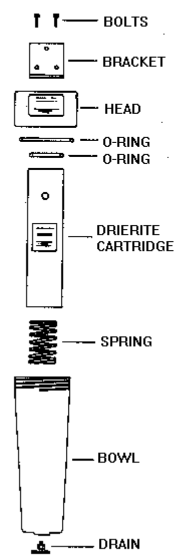
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4-in. pipe fitting using Teflon tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4-in. pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury. If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2-1/2" o.d. O-ring on top of cartridge.
8. Place the 4" o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD SOAP AND WATER. DO NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

Chapter 9
DTA 7 Differential Thermal Analyzer

Overview

The Perkin Elmer DTA 7 is a computer-controlled differential thermal analyzer which measures the temperature difference between a sample and a reference material as a function of temperature as they are heated, cooled, or held at a constant (isothermal) temperature. The DTA 7 is connected to your computer via the **Thermal Analysis Instrument Controller (TAC)**. The TAC 7/DX controls the analyzer and digitizes the analog output from the detector before sending it on to the computer. Through control of Pyris software, the DTA 7 is programmed from an initial to a final temperature through transitions in the sample material such as melting, glass transition, solid-state transitions, or crystallization.

Usually, the DTA 7 is programmed to scan a temperature range by heating or cooling at a linear rate over 150 temperature ramps for the study of endothermic and exothermic reactions. The DTA 7 can also be used to gather and analyze data from isothermal reactions such as crystallization or curing.

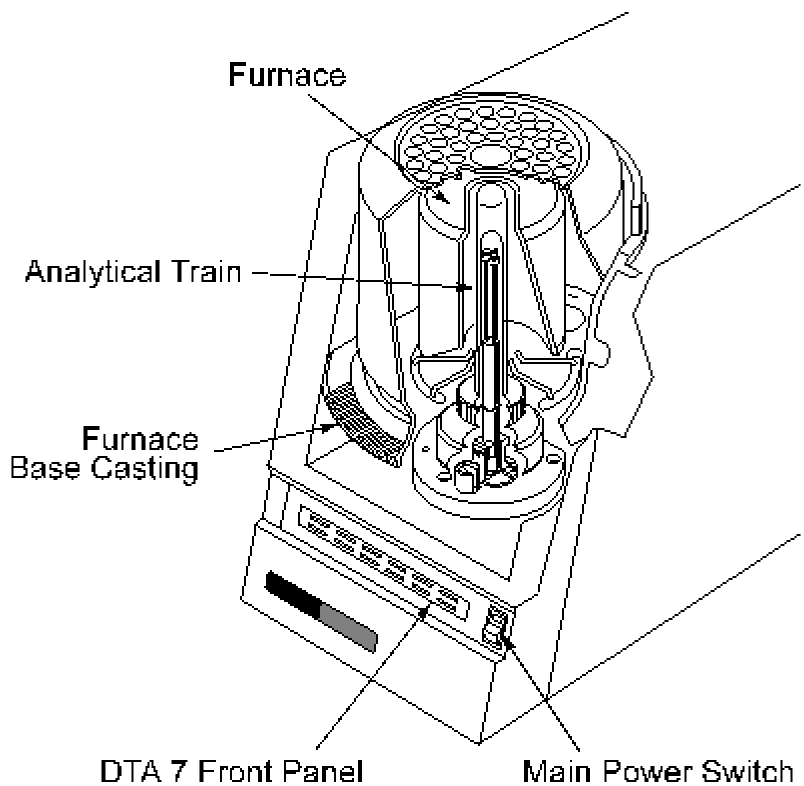
High sensitivity, automation, broad temperature range, rapid cooling, simplicity of operation, small size, and powerful software are a few of the DTA 7's many features. They provide the thermal analyst with the means to perform direct quantitative measurement of the endothermic and exothermic behavior of a host of sample materials at high temperatures.

Some of the features of the DTA 7 are as follows:

- With high sensitivity, the computerized DTA delivers superior quantitative performance, from ambient to elevated temperatures, for a wide variety of samples.
- Kinematic mounting of the furnace assembly guarantees the exact positioning of the furnace. This provides baseline reproducibility.
- Platinum and platinum/30% rhodium furnace windings ensure long furnace life.
- A furnace interlock mechanism prevents the furnace from being raised while at elevated temperatures. Interlock temperature is user-selectable. This minimizes exposure to any hot surface.

- Large temperature range from ambient to 1600°C allows the analyst to run a wider variety of samples. The range of the differential temperature (ΔT in °C) or the heat flow (ΔQ in mW) is the ordinate value versus temperature.
- Forced-air cooling of the furnace allows rapid cool-down. Also, the DTA 7 can be used to perform controlled cooling experiments.
- Bench space required is minimal — the DTA 7 occupies less than 26 cm (10 in.) of linear bench space.
- The furnace lift mechanism and the hand rest area facilitate sample loading and removal.
- Simultaneous operation with other thermal analyzers.
- Optional sample loading tray allows you to handle and transport samples and facilitates sample loading and removal.
- Optional open furnace tube allows samples that off-gas to be collected for further analysis by other techniques such as infrared spectroscopy and mass spectrometry. You may also connect the tube to an outside vent and/or hood for safe removal of potentially hazardous gases.

Interior View of the DTA 7



The DTA 7 consists of a cell base and a high-temperature furnace. The integral component of the system is a matched pair of thermocouples of platinum and platinum/10% rhodium. The thermocouples are used to monitor the temperature of both sample and reference material. They are also used to measure the differential temperature signal.

The DTA 7 furnace uses platinum and platinum/30% rhodium windings, ensuring increased lifetime. Over the broad temperature range from ambient to 1600°C, the furnace provides a uniform temperature environment for the sample and reference materials. These materials are surrounded by and closely coupled to the furnace.

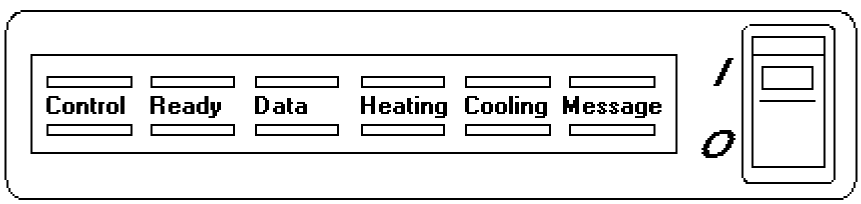
The furnace can be removed easily by first lifting then rotating it out of the way for access to the sample area. Incorporating a three-point kinematically mounted design, the furnace repositions itself exactly each time the furnace is raised or lowered.

Program cooling and fast turnaround times are facilitated by an automatic forced-air cooling system which channels forced air between the internal furnace wall and the furnace tube, resulting in a dramatic decrease in cool-down times. With rapid cooling, more samples can be run in less time, leading to higher productivity.

The DTA 7 has both DTA (differential thermal analysis) and DSC (differential scanning calorimetry) modes of operation. In the DTA mode, the system monitors the signal representing the difference in temperature between the sample and an inert reference material (ΔT) as a function of temperature. In the DSC mode, the normal (ΔT) signal is optimized and conditioned by the Pyris software so that the output is calibrated in units of mW, facilitating peak area measurements. The DSC mode is used for those applications where quantitative analysis based on peak area is desired.

Operating Controls

The DTA 7 has six status indicators on the front panel.



Control

The DTA 7 temperature sensors are in control of the temperature; power is being supplied to the furnace to maintain the program temperature selected in Pyris software. The Control light should always be illuminated during the main section of a temperature program run in order to obtain accurate data. If the Control light goes out during a cooling run, the data after that will not be accurate.

Ready

While blinking, Ready means that the program temperature of the DTA 7 has reached the Load Temperature defined in Pyris. When the Ready light is lit continuously, it means that the power is supplied to the DTA 7.

Upon startup of the system, the Ready indicator should be the only indicator illuminated.

Data

When the Data light is on, data are being collected. The Data light remains unlit at all other times.

Heating

When the Heating light is illuminated, the DTA 7 is heating under temperature program control at the selected rate.

Cooling

When the Cooling indicator is illuminated, the DTA 7 is cooling under temperature program control at the selected rate.

Message

When the Message indicator is blinking, an error or informative message must be acknowledged at the computer.

Power

The power switch is on the right side of the front panel. When illuminated, line power is being supplied to the DTA 7.

Status Indicators

The current status of the DTA 7 is displayed in the Status Panel in the Pyris Software for Windows screen. You can also see the status of the instrument by looking at the front panel.

Summary of the DTA 7 Status Indicators

Indicator	Blinking	On	Off
Control	Does not blink	Power is being supplied to the furnace	Analyzer is not in temperature control Power is not being supplied to the furnace
Ready	Program temperature has reached Load Temperature	Analyzer temperature has reached Load Temperature Power is being supplied to DTA 7	Analyzer temperature has not reached Load Temperature Power is not being supplied to DTA 7
Data	Does not blink	Data are being taken from the analyzer	Data are not being taken from the analyzer
Heating	Does not blink	Analyzer is heating under program control at the selected rate	Analyzer is not heating
Cooling	Does not blink	Analyzer is cooling under program control at the selected cooling rate	Analyzer is not cooling
Message	A message is waiting for you		No message is waiting

The status indicators can be used for **diagnostic troubleshooting**.

Diagnostic Troubleshooting

The status indicators on the DTA 7 panel can be used for diagnostic troubleshooting:

Control	Ready	Data	Heating	Cooling	Message	Cause
On or Off	On	–	–	–	–	Computer, TAC 7, and all analyzer modules functioning properly
On or Off	Blinking	–	–	–	–	Computer, TAC 7/DX, and all analyzer modules functioning properly. Analyzer at Load Temperature
–	On	On	On	On	On	TAC 7/DX for that analyzer module is not turned on or properly connected to the analyzer. Check that all cables are connected properly, TAC 7/DX power cord is connected to line power, and TAC is turned on
–	–	–	Blinking	Blinking	–	The cable connecting the TAC 7/DX to the analyzer is loose or has failed. The furnace cable is broken or not connected to the analyzer.
–	–	Blinking	Blinking or Off	Blinking or Off	Blinking or Off	A hardware component in the TAC 7/DX may be malfunctioning. Call your Service Representative
–	Off	Blinking	On or Off	On or Off	On or Off	A hardware component in the TAC 7/DX may be malfunctioning. Note the lights that are blinking and call your Service Representative.
–	Off	On or Off	On or Off	On or Off	Blinking	A hardware component in the TAC 7/DX may be malfunctioning. Note the lights that are blinking and call your Service Representative
–	Off	Blinking	Off	Off	Blinking	A hardware component in the TAC 7/DX may be malfunctioning. Note the lights that are blinking and call your Service Representative.
–	Blinking	Off	Off	On or Off	On or Off	A hardware component in the TAC 7/DX may be malfunctioning. Note the lights that are blinking and call your Service Representative.

The DTA 7 Hardware Help comprises the following topics:

Safety Precautions

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

The following precautions must be observed when using the DTA 7:

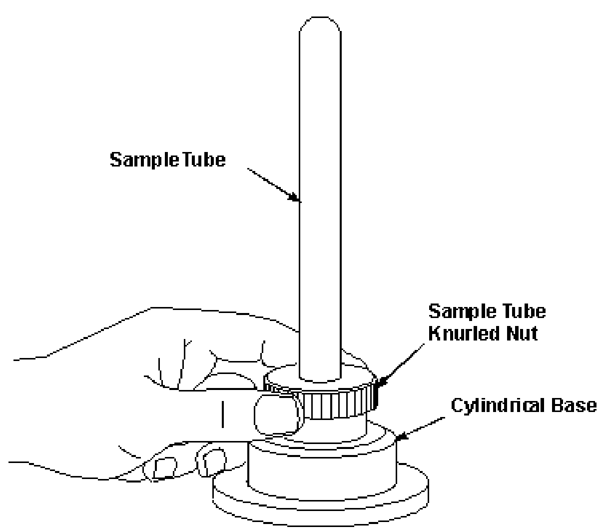
- Never turn the computer off until the following message appears:

  It's now safe to shut off your computer.

- Never press the Reset button on the computer if the software appears to malfunction. Press the **Ctrl–Alt–Del** keys simultaneously and select the Task Manager. From the Task Manager close the Pyris software.
- Always observe the startup and shutdown procedures with the DTA 7 and all related instruments.

WARNING: **Wear insulated gloves when handling the sample tube and set it down on an insulated pad. The sample tube assembly glows red at temperatures above 800°C. Below that temperature it may appear cool but it is still hot enough to cause serious burns.**

- Always set the sample tube on an insulated pad.
- Make sure the furnace is programmed between 20 and 40°C between runs and if you leave the analyzer unattended.
- To prevent damage to the furnace, DO NOT operate the instrument in an isothermal mode at high temperatures (greater than 1000°C) for a long period of time (greater than 30 minutes).
- Make sure that the sample tube is mounted securely before operating the DTA 7 to minimize the possibility of burns.



- Do not permit the temperature of the furnace to increase at a rate greater than 100°C/min.
- During an experiment the furnace can be operating at temperatures greater than 1500°C. Always turn the analyzer off and allow it to cool before touching the furnace or the sample tube assembly.

WARNING: Due to its explosive nature, hydrogen should not be used as a purge gas.

- Use only high-quality purge gases. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for removing any moisture from the purge gases.
- DO NOT put any restrictions such as flow meters into the purge gas exit fitting (exhaust connection). A back pressure can occur and cause damage to the sample area.
- Do not immerse the purge gas exit line in a liquid because the liquid may be drawn back into the sample tube.
- DO NOT use the DTA 7 near combustible gases, solids, or liquids.
- Use only the power cords provided.

CAUTION: The instrument requires a good earth ground that is common to all the components of the DTA 7 system. The grounding must be accomplished by a separate wire in the electrical supply system. The maximum permissible resistance between the ground connections of any two units in the system is 1 ohm.

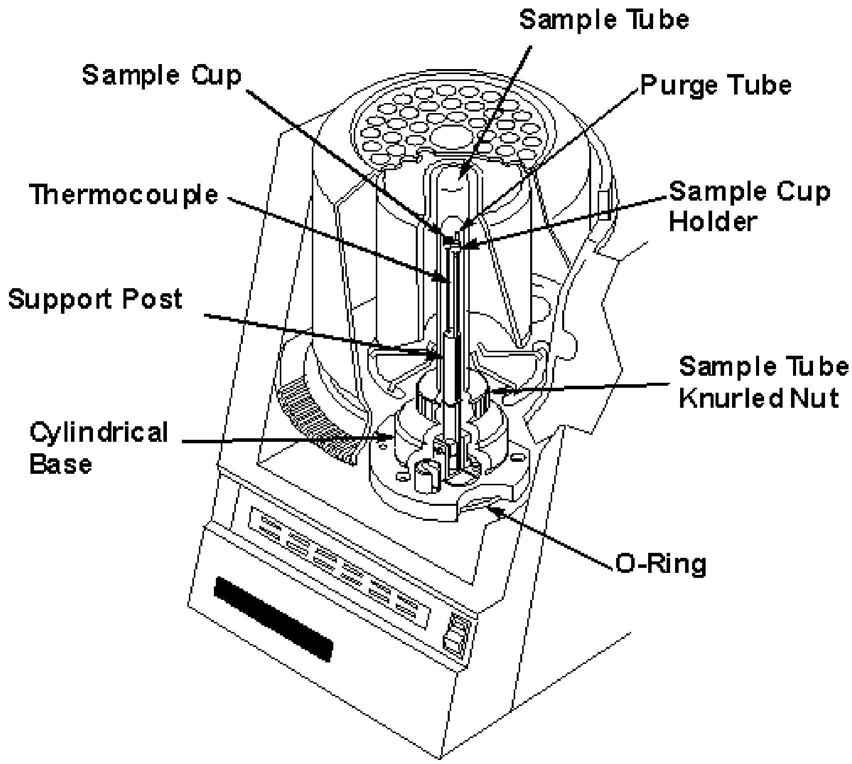
WARNING: Shut down the instrument and disconnect the power cord from the line power before removing the instrument covers.

- Do not remove covers from the instrument or attempt internal adjustments. All necessary internal adjustments are made at the factory. Should readjustment become necessary. It must be performed by a Perkin Elmer Service Engineer.
- Handle the thermocouples with care. To prevent the internal leads from breaking, DO NOT attempt to bend the thermocouples.
- DO NOT use metal samples, or samples that react with platinum, in platinum sample cups. Use aluminum oxide cups instead to prevent the samples from adhering to the cups.
- When using platinum sample cups, it is necessary to cover the thermocouple bead with a small amount of aluminum oxide powder before placing the platinum sample cup into the sample cup holder. Doing so will prevent the platinum sample cup from fusing to the thermocouple bead.
- Do not operate the instrument in a cold room. The instrument was designed to operate in an environment that is between 15°C and 30°C.

Features

Sample Area

The DTA 7 sample area is composed of a matched pair of thermocouples. The left thermocouple is used for sample materials, while the right thermocouple is used for reference materials. A purge tube sits behind the thermocouples and the sample area is enclosed within the sample tube. The sample tube is made of alumina (aluminum oxide). The furnace assembly can be lifted and swung out of the way for access to the sample area. An O-ring seal is used to seal the sample area from the external atmosphere during an analysis.



Furnace Lock

The furnace lock is a safety feature that prevents the furnace from being raised when the lock is in place. This keeps the user from touching the sample area until the sample temperature falls below the furnace lock temperature. The furnace lock temperature is set in the Preferences page; the default is 55°C. The furnace lock is enabled and disabled by clicking on the **Furnace Lock** button on the control panel 

Furnace Cover

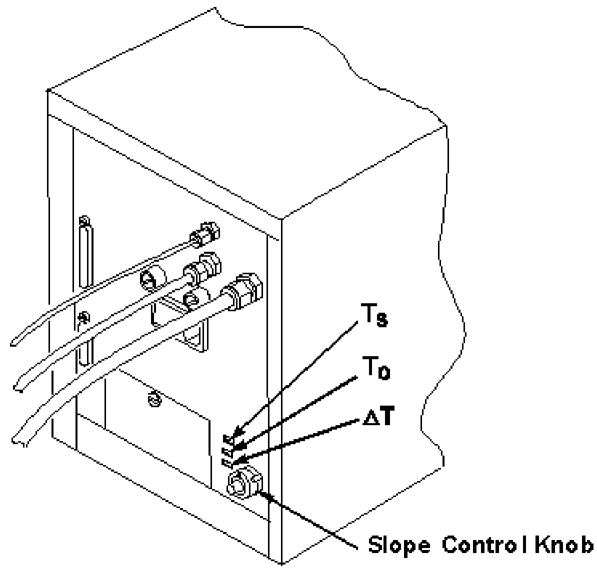
The furnace cover is used to control the heat convection generated by the furnace. The furnace cover can be closed or open by using the **Cover Lock** button  on the control panel. When you enable the cover lock by clicking on the **Cover Lock** button, the furnace cover will be closed when the furnace is heating or holding isothermally and during all run conditions. When the furnace is cooling, the cover will be open to allow the heat to escape more quickly.

When the cover lock is disabled, the furnace cover will remain open at all times. This setting is important if you are using the open sample tube (P/N N538-0062).

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

Trimpots and Slope Control Knob

When the sample and reference thermocouples are replaced or the furnace and/or furnace thermocouple is replaced in the DTA 7, it may be necessary to calibrate the new thermocouples or furnace. In order to do so, use the three adjustable trimpots located at the back of the instrument.



The trimpots are used to calibrate the Sample Temperature Zero (Ts), the Oven Temperature Zero (To), and the ΔT Zero. They should be calibrated in the following order: Ts, ΔT, To.

For instructions on adjusting each of the potentiometers, see **Calibrating Thermocouples**.

The slope control knob (see figure above for location) is used to optimize the slope of the baseline. The baseline needs to be adjusted whenever the sample and reference thermocouples, the furnace thermocouple, or the furnace is changed. For best results, the baseline should be optimized using the same temperature range and scanning rate that you use for most of your analyses.

NOTE: Make sure that the trimpots are optimized ***before*** adjusting the slope control knob.

For instructions on optimizing the DTA 7 baseline using the slope control knob, see **Manual Baseline Optimization**.

NOTE: If the slope is adjusted, you may need to perform a temperature calibration. Therefore, always optimize the DTA 7 baseline ***before*** the instrument is calibrated.

Manual Baseline Optimization

The procedure for manual baseline optimization consists of making an initial baseline run to determine the characteristics of the baseline with the current settings. If adjustment is necessary, calculate the amount of adjustment needed, make the adjustment on the slope control knob, and then rerun the baseline for confirmation.

The baseline needs to be adjusted whenever the sample and reference thermocouples, the furnace thermocouple, or the furnace is changed. For best results, the baseline should be optimized using the most commonly used temperature range and scanning rate.

The procedure below describes the technique for optimizing the DTA 7 baseline using the slope control knob. Make sure that the trimpots are optimized before adjusting the slope control knob.

NOTE: If the slope control is adjusted, you may need to recalibrate the DTA 7 temperature axis. Therefore, always optimize the DTA 7 baseline before the instrument is calibrated.

1. Select the temperature range and scanning rate over which the baseline is to be optimized. It is not necessary to optimize the DTA 7 baseline over the entire temperature range if most of your experiments will be done using a small range.

   For example, if your work involves the analysis of the melting profiles of metals, you will generally be working in the range between 200°C and 1000°C. Therefore, optimize the DTA 7 baseline between those temperatures.
2. Set the purge gas to a flow rate of approximately 20 – 30 mL/min.
3. Check and record the current slope control knob settings.
4. Place a sample cup holder onto each thermocouple if they are not already in place.
5. Place a sample cup in each sample cup holder.
6. In Pyris Software for Windows, select the **Instrument Viewer** button on the toolbar if the Instrument Viewer is not already displayed.
7. In Instrument Viewer, select Auto-Rescale from the Display menu; a checkmark should appear next to the selection in the menu. With Auto-Rescale on, data are automatically optimized on the screen.
8. Select Rescale X from the Display menu. In the dialog box, select Temperature in the Set Axis Units box. Enter Minimum and Maximum values for the lower and upper temperature values on the X axis. Click on **OK**.
9. In the Curves menu, select ΔT. This will be the Y axis.
10. Select Rescale Y from the Display menu. In the dialog box enter Minimum and Maximum values for the lower and upper ΔT values.
11. Select Y Initial to set the Y axis where the data will be displayed.
12. Select the **Method Editor** button from the toolbar.
13. Click on the Program tab. In the Initial Temp field, enter your initial temperature, e.g., 200°C.
14. In the For field, enter the amount of time you want to hold isothermally at the initial temperature.
15. Click on the **Add a step** button and select Temperature Scan. This adds another step to the temperature program.
16. In the Rate field, enter a new heating rate.
17. Enter a value in the To field, the temperature to which the analyzer should heat.
18. Select the **End Condition** button and choose Go To Load Temp. This will cool the analyzer to the load temperature (specified in Preferences) at the end of the run.
19. Click on the **Done** button to close the End Condition section.

20. Click on the Initial State tab and check that there is no entry in the Baseline File box. If there is a file name, select Use Baseline Subtraction to remove the “X” in the box.
21. The remaining items in the method will remain unchanged. They should remain with their default values. Select Save Method from the File menu and enter a file name.
22. In the Control Panel, type in the value of the Initial Temperature into the Go To Temp field and click on the **Go To Temp** button.

    The DTA 7 will now program to the starting temperature and hold there. After a short time, the Ready and Control status indicators should light and the Y axis signal (ΔT) should be displayed in the Status Panel (make sure that one of the Status Panel boxes has ΔT selected for display).
23. After the analyzer has equilibrated, click on the **Start Method** button on the Control Panel. A baseline curve will be generated and displayed in Instrument Viewer.

    Note the shape of the curve. If it is not level, it will have to be adjusted. If its slope is up, turn the slope control knob clockwise. If it slopes down, turn the knob counterclockwise.

    NOTE: Move the black lever on the slope control knob to the left to unlock the knob.

24. Observe the arrow at the left of the dial. This is the current baseline setting. Turn the knob in the appropriate direction to remove the slope of the baseline.
25. Allow the run to finish, making any necessary adjustments to the baseline as it reaches higher temperatures.
26. Perform another run using the same method. The baseline should now be level. If not, use the slope control knob to readjust the baseline then rerun the method again.
27. When satisfied with the baseline, move the black lever on the slope control knob to the right to lock in the new setting.

Calibration

Calibration of the DTA 7 for temperature and energy is accomplished by running high-purity standards or reference materials with known temperature and energy transitions. The data obtained after running the reference materials (i.e., temperature transitions and ΔH values) are used in the calibration routines to automatically calibrate the DTA 7. Once this calibration is performed, the analyzer will be calibrated continuously, even when the system is shut down. Unless major changes to the analyzer’s condition are made, the DTA 7 should remain calibrated.

The DTA 7 is shipped with reference materials in the Spares kit. Additional reference materials and standards are available from *PE XPRESS*.

There are three calibration routines available for the DTA 7:

- **Temperature Calibration**
- **Heat Flow Calibration**
- **Furnace Calibration**

All three calibration routines can be used independently or they can be applied simultaneously. The Furnace calibration ***must*** be performed ***after*** temperature calibration. Heat flow calibration can be performed at any time.

The recommended procedure for calibrating the DTA 7 is to perform a two-standard temperature calibration when the instrument is first installed or when the DTA 7 sample and reference thermocouples have been changed. Temperature calibration should also be performed if either the furnace thermocouple or the furnace itself has been changed. This calibration will normally be performed by a Perkin Elmer Service Engineer upon installation of your DTA 7.

NOTE: If you need to optimize the DTA 7 baseline using the slope control knob, do so before calibrating the analyzer (see Manual Baseline Optimization).

Temperature Calibration

For the two-point temperature calibration of the DTA 7, you run two high-purity standards or reference materials (aluminum and gold) and measure the melt onset for each. The measured onset temperature is compared with the expected onset temperature to calibrate the temperature axis over a very broad temperature range. The calibration adjusts the slope of the temperature calibration curve, resulting in linear temperature calibration over the temperature range used. Once the calibration has been performed, the temperature slope correction will remain constant for a long time. It is recommended that temperature calibration be performed only periodically, i.e., every three to six months after initial installation.

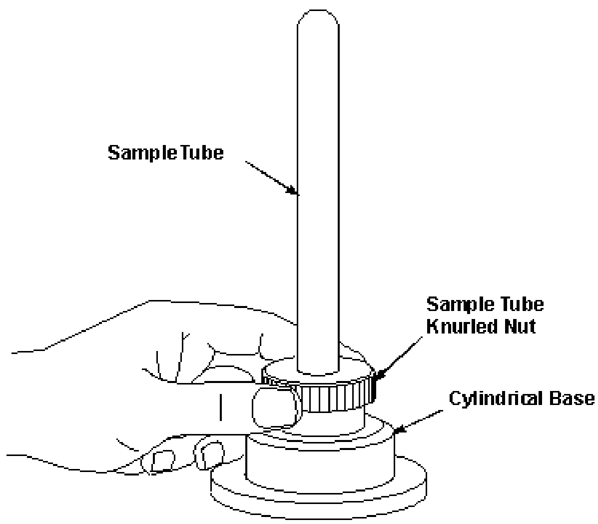
The steps below describe how to perform a temperature calibration experiment on your DTA 7:

1. While using the Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.
2. Restore the default temperature calibration by selecting Temperature from the Restore menu. If you are performing all of the calibration procedures, restore all default calibration values by selecting the All command.
3. Select the **Save and Apply** button; select the file to which to save the current calibration values or enter a new file name.
4. Select **Close** to close the Calibration window and begin using the new calibration values.
5. Using the AlumCal method, **complete a scan for aluminum** under the same conditions that you run your samples.
6. **Perform a Peak Area calculation** and include the Onset temperature. Record the ΔH and Onset results; you will need the Onset result for Temperature calibration and the ΔH for Heat Flow calibration.
7. Using the GoldCal method, **complete a scan for the gold** reference material.
8. Perform a Peak Area calculation with Onset; record the Onset temperature and the ΔH.
9. Select Calibrate from the View menu. The Calibration window appears.
10. Select the Temperature tab. Enter the name of the operator, the name of the reference materials used, the expected Onset values, and the Onset results just measured for each reference material.
11. Select the check box in the Use column for each reference material to be used in the calibration.
12. Select the **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.
13. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

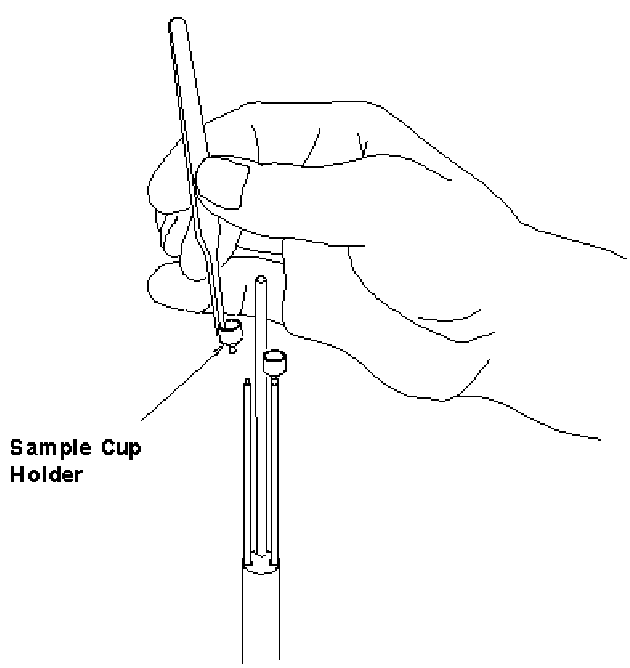
Set Up an Aluminum or Gold Run and Collect Data

To run aluminum or gold reference material as part of a temperature calibration of the DTA 7, set up the run as follows:

1. Cut and weigh a small piece of the aluminum calibration reference material (P/N N538-0057) or gold calibration reference material (P/N N538-0058) supplied in the DTA 7 Spares Kit (P/N N538-0015). Recommended sample size is between 5 and 10 mg. The Perkin Elmer AD-6 Autobalance is ideally suited for weighing reference materials.
2. Lift the furnace assembly and swing the furnace to the right side.
3. Remove the sample tube by unscrewing the knurled nut at the base, then lift the sample tube up and over the thermocouples, being careful not to break the thermocouples or the purge tube.

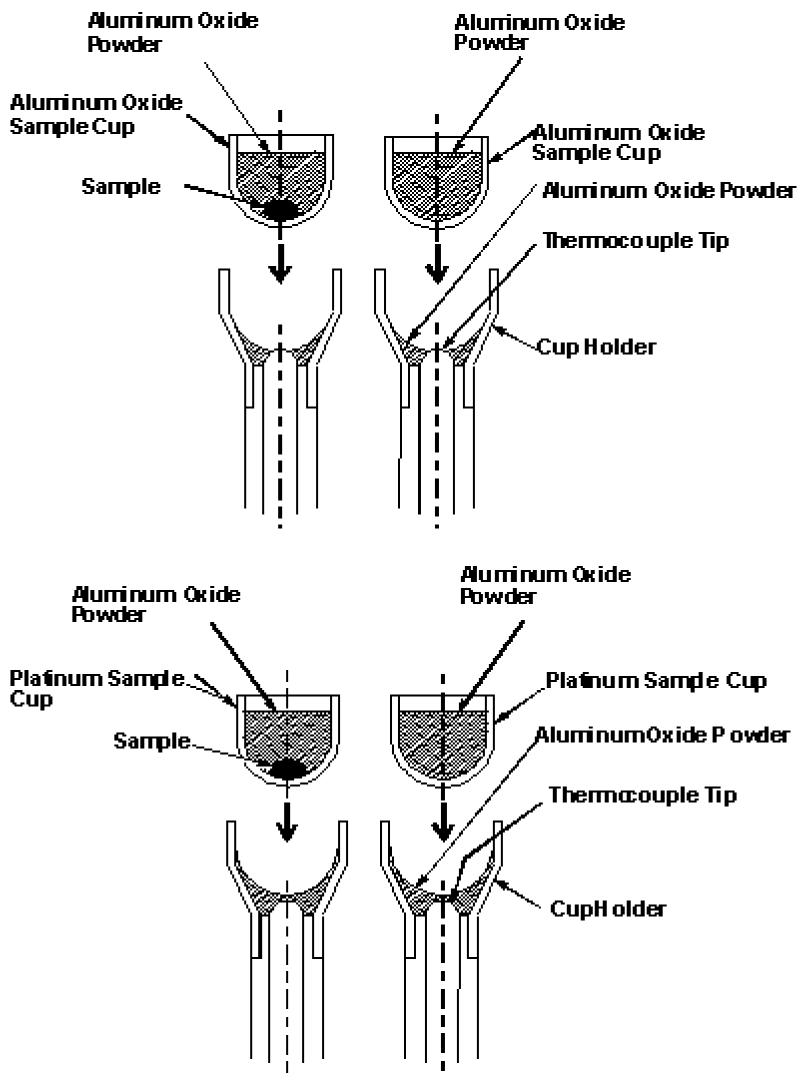


4. If not already in place, use tweezers to place the sample cup holders onto the thermocouples.

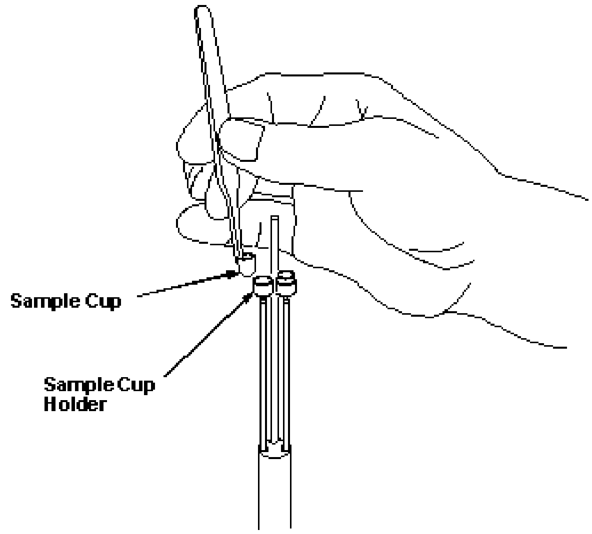


5. Place a small amount of aluminum oxide (alumina) powder, supplied in the Spares kit, into the cup holders. This helps create better thermal contact between the thermocouples and the sample cups.

Proper Filling of Alumina and Platinum Sample Cups



6. Using tweezers, place a sample cup into each of the sample holders.



7. Place a small amount of alumina powder into the cups. This will help prevent the sample from adhering to the sample cup. [A sample loading tray (P/N N538-0061) is available to make loading samples easier.]
8. Place the aluminum or gold reference material into the left sample cup. Cover the sample with more alumina powder.
9. Fill the right sample cup with alumina powder. The reference sample cup should be treated in the same way as the sample material.
10. Remove the sample loading tray if it was used. Replace the sample tube. Ensure that the ring on the sample tube is screwed on tightly.
11. Lift the furnace enclosure assembly and swing it to the center. Lower the assembly into place.
12. If not already displayed, select the Method Editor icon from the toolbar.
13. From the File menu, select **Open Method**. The Open Method dialog box displays the methods available in the default Method directory.
14. Select the **AlumCal.dtm** method for the aluminum run or **GoldCal.dtm** for the gold run.

    These methods are set up to condition the sample by heating it past the melting point, cooling it to reform the sample, then heating to melt the sample. The second melt will then be used for the calibration.
15. In the Sample Info page, fill in the sample information; enter the weight of the aluminum sample, the file name under which the data will be saved, and any comments about the analysis.
16. Select the Program tab if you want to modify the temperature program. Change the Rate value if you want to calibrate at a heating rate other than 10°C. You should select a heating rate that matches the heating rate you will use when running your samples.

    You may also want to change the Initial Temperature or the To value or the time of the run. Select the Initial Temperature to be low enough to allow at least 5 – 10 minutes of run time while scanning before the melt is obtained. Select the beginning and ending temperatures of the run to be far enough apart to obtain the melt.

NOTE: Do not allow metal reference materials to sit for long periods of time at temperatures above their melting point.

17. Click on the **Read Zero** button on the control panel. This zeros the difference in temperature at the sample and reference thermocouples.
18. Enter the initial temperature of the experiment in the Go To Temp field in the control panel and click on the **Go To Temp** button.
19. After the DTA 7 has stabilized, click on the **Start Method** button in the DTA 7 Control Panel to start the run. The Control, Ready, Data, and Heating status indicators on the DTA 7 should be illuminated. These indicators tell you that power is supplied to the analyzer, the DTA 7 is in temperature control, data are being collected, and a heat experiment is being performed.

    The Status Panel should indicate that the analyzer is heating. Both the temperature and Y axis value should be updating continuously in the Status Panel and the curve is displayed in the Instrument Viewer.
20. When the run is complete, the data file is saved with the file name that you entered in the method.

After performing the aluminum run, you must do the same for gold reference material following the steps above.

After performing the gold run, you are ready to perform the Peak Area calculation.

Performing a Peak Area with Onset Calculation

For the aluminum and gold runs that you performed, you now can perform a peak area with onset calculation. The data file for the gold reference sample that you just collected should be displayed in the Data Analysis window. Perform an onset calculation on the data as follows:

1. Select **Onset** from the Calc menu. The Onset Calculation dialog box appears.
2. Enter the Left Limit and the Right Limit. You can also click on the two red **X**'s that appear on the curve and drag them to the desired left and right limits. The left **X** should be on the linear part of the curve and the right **X** should be in the middle of the sloped part of the curve. The Left Limit and the Right Limit fields in the dialog will be filled in automatically.
3. Click on the **Calculate** button. The Adjust Tangents dialog box is displayed.
4. Adjust the tangent lines of each point until they intersect. Use the **Restore** button to return the lines to their original position if you are not satisfied with their position.
5. Click on the **Calculate** button in the Adjust Tangents dialog box. The onset temperature is calculated for the sample and is displayed on the screen.
6. Record the Onset value for temperature calibration and ΔH for heat flow calibration.
7. Select New Data from the File menu and select the data file collected for your aluminum run. The curve should be displayed in the Data Analysis window.
8. Repeat steps 1– 6 for the aluminum.

Once you obtain the Onset values or the ΔH values, you can perform the temperature calibration or the heat flow calibration, respectively.

Heat Flow Calibration

If you want to calibrate the heat flow of the DTA 7, you must accurately weigh the reference material before performing the calibration. Typical weights for reference materials range from 5.000 to 20.000 mg. The accuracy and precision to which you weigh the reference material relates directly to the accuracy and precision of the energy measurements made on the DTA 7. The Perkin Elmer AD-6 autobalance is ideally suited for weighing reference materials and samples that will be analyzed with the DTA 7.

1. While using the Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.
2. Restore the default Heat Flow calibration by selecting Heat Flow from the Restore menu.
3. Select **Save and Apply**; select the file to which to save the current calibration values or enter a new file name.
4. Select **Close** to close the Calibration window.
5. Complete a scan using a reference material or use one that was run for the Temperature calibration.
6. Perform a Peak Area calculation and note the ΔH result. You can also use the ΔH result recorded for one of the reference materials used in the Temperature calibration.
7. While in Method Editor or Instrument Viewer, select **Calibrate** from the View menu.
8. Select the Heat Flow tab. In the Calibration table, enter the name of the reference material used, the expected ΔH value (400.10 for aluminum and 63.73 for gold), the measured ΔH, the expected or theoretical melting point for the reference material (660.10°C for aluminum and 1063.00°C for gold), the weight of the reference material used, and the name of the method file used for the run.
9. Select **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.
10. Go on to the next calibration procedure or select **Close** to close the Calibration window and begin using the new calibration values.

Furnace Calibration

NOTE: Furnace calibration is performed **after** temperature calibration has been performed.

Furnace calibration should be performed when the furnace or the furnace thermocouple has been changed. Performing a furnace calibration calibrates the furnace over a wide temperature range. The DTA 7 should be calibrated using the same temperature range that you will use with your experiments. A nine-point temperature calibration between selected upper and lower temperature limits will be performed:

NOTE: After the gold reference material has been run, allow the analyzer to cool and then empty the sample cups.

1. While in the Instrument Viewer or the Method Editor, select **Calibrate** from the View menu.
2. If applicable, complete the Temperature calibration.
3. Remove the cups from the sample and reference cup holders.
4. Select the Furnace tab in the Calibration window.
5. In the Minimum field, enter a minimum temperature that is below your normal operating region. The minimum temperature limit is 50°C.
6. In the Maximum field, enter a maximum temperature that is above your normal operating region. The maximum temperature limit is 1500°C.
7. Select the **Begin Calibration** button.
8. The Furnace calibration will begin and take about 2.5 hours to complete.

9. Select the **Save and Apply**; select the file to which to save the new calibration values or enter a new file name.
10. Select **Close** to close the Calibration window and begin using the new calibration values.

When to Calibrate

Once the calibration programs are performed, the temperature and heat flow calibrations of the DTA 7 should remain unchanged for long periods of time, provided there are no changes in the instrument operating conditions. Below are some of the conditions that will change the furnace, temperature, or heat flow calibration of the DTA 7 and will require you to recalibrate the instrument:

- If the operating temperature range of your experiments changes. Run a standard in the new range of interest to determine if the current calibration is still valid.
- If the Slope control is adjusted. Always optimize the DTA 7 baseline before the instrument is calibrated.
- If you change the type of purge gas or its flow rate, the calibration should be checked for highest accuracy.
- If a new furnace or furnace thermocouple is installed, both temperature and furnace calibrations should be performed.
- If a new pair of sample and reference thermocouples are installed, a new temperature calibration should be performed.
- If the instrument has been turned off for a long time (weeks or months), the instrument may appear to require recalibration. Condition the DTA 7 by performing several heating and cooling runs with the sample and reference cups empty and then check the calibration by running standard materials.

NOTE: The temperature calibration is slightly dependent on the scan rate; therefore, the final calibration should be performed at the scan rate that you will use for your experiments. As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating and cooling rates, it is better to calibrate at the slowest rate you will be using.

Calibration Standards

Very-high-purity (greater than 99.9%) metal standards, reference materials, or inorganic materials are typically used to calibrate the temperature and energy axes of the DTA 7 when operating in the temperature range of 200°C to 1600°C. Two high-purity metal reference materials have been supplied with your DTA 7 for calibration purposes: aluminum (P/N N538-0057) and gold (P/N N538-0058). The transition temperatures and energies (heat flow) for some reference materials are listed below:

Thermal Capacities of Reference Materials

Reference Material	Melting Point (°C)	Energy (ΔH in J/g)
Zinc	419.51	112.01
Aluminum	660.10	400.10
Silver	960.85	104.73
Nickel	1452.85	297.60
Palladium	1551.85	165.04
Gold	1063.00	63.73

The zinc reference material should not be run more than twice.

The selection of the proper calibration materials is crucial for obtaining optimal performance of the DTA 7 in the temperature range in which you will perform your experiments. Usually, you should select reference materials whose transition temperatures encompass the temperature range of interest. For example, if you operate the DTA 7 from 500°C to 1000°C, perform a temperature calibration using aluminum (melting temperature = 660.1°C) and gold (melting temperature = 1063.0°C) as the reference materials.

Operating Variables

In differential thermal analysis, the object of an experiment is to record the difference between the sample temperature and the reference temperature over a linearly changing temperature range or while the temperature remains constant. This delta temperature data can be analyzed or converted to heat flow for analysis. You wish to obtain accurate data in a minimum period of time. The suggestions in the topics below discuss some of the techniques that can be used to obtain the most accurate DTA 7 data.

- **Sample Preparation**
- **Sample Loading**
- **Sample Cup Holders and Sample Cups**
- **Sample Size**
- **Sample Atmosphere**
- **Temperature Range**
- **Scanning Rate**
- **Automatic Baseline Optimization**

Sample Preparation

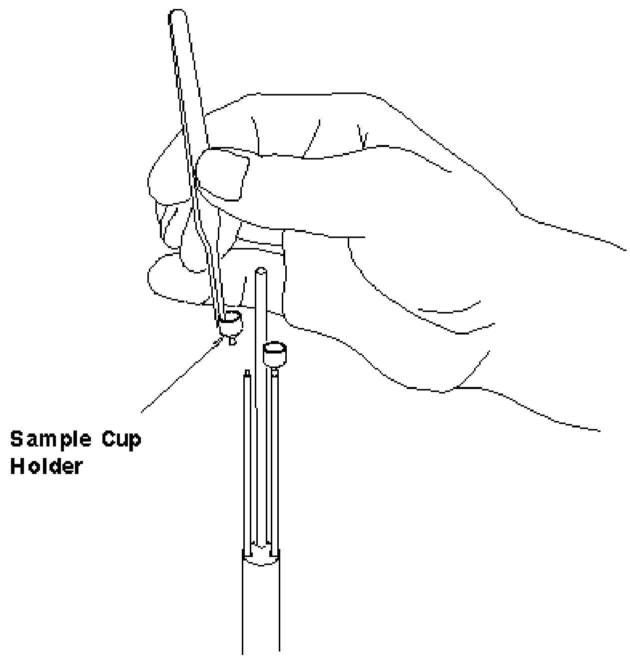
The DTA 7 can be used to analyze solid or liquid samples. Solid samples can be in film, powder, crystal, or granular form. The recommended sample size is 10 – 30 mg. Although quantitative accuracy will remain the same regardless of sample shape, the qualitative appearance of a run may be affected by the sample configuration. Proper sample preparation, which maximizes the contact surface between the cup and the sample, reduces the resistance of the sample to heat flow through the DTA temperature sensors and results in maximum peak sharpness and resolution. In some cases, the DTA 7 should ramp to the sample's melting point, then cool before the actual run. This changes the shape of the sample and, therefore, its thermal characteristics. For example, when running gold leaf or gold wire, the first run melts the sample and forms it into a small ball. The second run is used to obtain the actual data.

Load the sample and reference cups into the DTA 7 as outlined in **Sample Loading**.

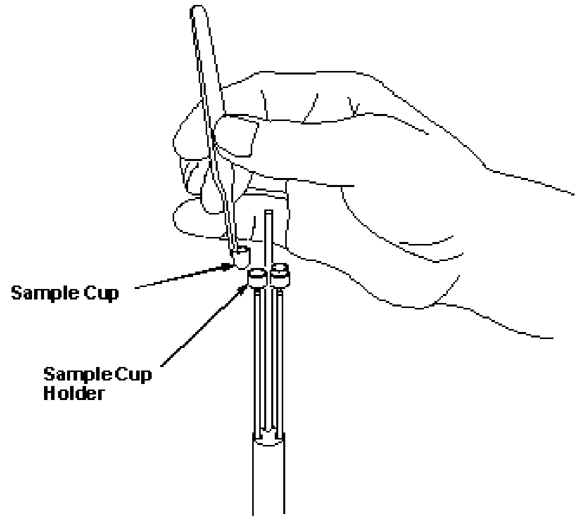
Sample Loading

Load the sample and reference cups into the DTA 7 as follows:

1. Grasp the front lip of the furnace casting with both hands and lift the casting straight up.
2. Swing the furnace casting assembly to the right side of the DTA 7 to access the sample area.
3. Unscrew the knurled ring at the base of the sample tube and lift the ring and sample tube straight up to clear the thermocouples and the purge tube.
4. Using tweezers, place the sample cup holders on top of the sample and reference thermocouples. A sample cup holder is 0.47 cm (0.185 in.) in diameter and is made of platinum/10% rhodium.



5. Place a small amount of aluminum oxide powder into the cup holders. This helps create better thermal contact between the thermocouples and the sample cups.
6. Using tweezers, place a sample cup into each holder. Sample cups are available in platinum and alumina and are 60 and 100 µL (60 and 100 cu. mm), respectively.



7. Place a small amount of aluminum oxide powder into the cups. This helps prevent the sample from adhering to the sample cups. A sample loading tray (P/N N538-0061) is available to make loading samples easier.
8. Place the sample material into the left sample cup. Cover the sample with aluminum oxide powder or place the sample cup cover onto the sample cup. The reference sample cup should be prepared the same way.
9. Remove the sample loading tray, if used. Replace the sample tube. Make sure that the ring on the sample tube is screwed on tightly.
10. Lift the furnace enclosure assembly and swing it to the center; lower the assembly into place.

Sample Cup Holders and Sample Cups

Four sample cup holders (P/N 0419-1246) are supplied in the Spares and Accessories kit. The cups are 0.47 cm (0.185 in.) in diameter and made of platinum and 10% rhodium.

There are two sample cups available: platinum and alumina. The alumina sample cups are available in two sizes: 60 and 100 µL (60 and 100 cu. mm). They come packaged in different amounts:

Sample Cup P/N	Description	Quantity
0419-1290	60 µL	1
0419-0166	60 µL	6
0419-0387	60 µL	25
0419-1291	100 µL	1
0419-0217	100 µL	6
0419-0388	100 µL	25

Determining the Heat Capacity of Sample Cups

Use the following equation to determine the **heat capacity** of the sample cup used with the DTA 7:

$$Cp(\text{cup}) = Tc \times wt$$

where *Cp*(cup) is the heat capacity of the sample cup at constant pressure (J/°C), *Tc* is the **thermal capacity** (J/g/°C) of the sample cup, and *wt* is the weight (g) of the sample cup and lid.

For example, a typical weight of an empty platinum cup with a lid is 0.026 g:

$$Cp\text{(cup)} = 0.1326 \times 0.026 = 0.0034 \text{ J/°C at } 25\text{°C}$$

Below are the thermal capacities of sample cups at various temperatures:

Sample Cup Material	25°C	327°C	1027°C	1327°C	1527°C
Alumina	0.7719	1.1040	1.2682	1.3129	1.3410
Platinum	0.1326	0.1410	0.1602	0.1682	0.1741

Heat Capacity

The quantity of heat required to increase the temperature of a system or substance one degree. Heat capacity is usually expressed in calories per degree centigrade (cal/°C) or joules per degree centigrade (J/°C). Pyris Software for Windows uses J/°C.

Thermal Capacity

The quantity of heat necessary to produce unit change of temperature in unit mass. Thermal capacity is expressed in joules per gram per degree centigrade (J/g/°C). It is numerically equivalent to specific heat.

Sample Size

The quantity of samples that can be analyzed with the DTA 7 is limited only by the volume of the sample cup that you use. However, the sample size, in conjunction with the scanning rate, will affect the quality of your results. With the DTA 7, as with all thermal analyzers, running a large sample at a fast scanning rate will improve the usable sensitivity. Running a small sample at a slow scanning rate will improve the resulting peak resolution. Most samples run on the DTA 7 will be in the 0.5 – 30-mg range.

NOTE: To perform decomposition studies and other analyses in which the sample may tend to contaminate the sample holders, use a small amount of sample (1 – 5 mg or less). We also suggest that an open sample tube be used in such analyses.

Sample Atmosphere

You can control the atmosphere in which the sample is run by using a purge gas to displace or introduce reactive gases in the sample area. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another, always check the temperature and energy calibration. A purge gas flow rate of between 20 and 30 cc/min is recommended. This flow rate will be realized by an inlet purge gas rate of between 20 and 30 psi and the appropriate flow restrictor.

You can switch from one purge gas to another if you use the **GSA 7 Gas Selector Accessory**. The **Thermal Analysis Gas Station (TAGS)** allows you to switch among four purge gases. You can change the purge gas from the control panel or in the Method Editor.

GSA 7 Gas Selector Accessory

The GSA 7 Selector Accessory is a computer-controlled gas-switching device that permits the automatic switching between two sample purge gases at an operator-selected time or temperature during an analysis.

Electrical Connections

The cable that exits the back of the GSA 7 connects directly to the plug attached to the N519-0310 analog cable at the back of the TAC 7/DX Thermal Analysis Instrument Controller. The TAC 7 then connects to the analyzer.

Purge Gas Connections

The rear panel of the GSA 7 has five connectors for inlet and outlet of purge gases.

GAS A IN	Connects the inlet purge line from GAS A
GAS B IN	Connects the inlet purge line from GAS B
A/B OUT	Connects directly to the analyzer. It carries GAS A or GAS B to the analyzer, depending upon the gas selected.
GAS A VENT	Connects to a purge line that vents GAS A. When GAS B is the selected purge gas, GAS A is vented through this connector.
GAS B VENT	Connects to a purge line that vents GAS B. When GAS A is the selected purge gas, GAS B is vented through this connector.

WARNING: **Do not use toxic or corrosive gases for purge gases. It is a good laboratory practice to run the lines from the GAS A VENT and GAS B VENT to a fume hood or other suitable container.**

TAGS (Thermal Analysis Gas Station)

The Thermal Analysis Gas Station (TAGS) (P/N N520-2019) is a very flexible instrument with which you can fully control up to four purge gases used in thermal analysis. The gases controlled by the TAGS are selected in the Purge Gas page of Preferences. In addition to gas switching via a gas program in the Pyris method, you can also control the flow rate of each gas. A gas program consists of up to 10 gas steps per temperature program step. Each gas step is characterized by the type of the gas, its flow rate, and the condition at which to change to the next step.

Instructions for installing the TAGS are given in the online Installation Help.

Drierite Compressed Air Filter Dryer Accessory (N537-0103)

WARNING: **FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.**

The features of the Drierite filter dryer accessory are

- Dries air to a dew point of –100°F
- Maximum working pressure 125 PSIG
- Flow rates up to 10 SCFM
- Disposable cartridge indicator window
- Clear polycarbonate bowl
- Anodized aluminum head
- 1/4" pipe connections

The DRIERITE Model 207 Compressed Air Drier will dry up to 10 SCFM of air saturated at 90°F and 125 PSIG to a dew point of –100°F for intervals of 20 to 30 minutes. The drier will dry air flows up to 5 SCFM continuously. The disposable cartridge has an indicator window that turns from blue to pink when the cartridge is exhausted. The cartridge is easily replaced by depressurizing the drier and unscrewing the polycarbonate bowl from the aluminum head.

The compressed air enters the DRIERITE Model 207 Compressed Air Drier through a port in the left side of the head and passes downward between the cartridge and the bowl and beneath the cartridge, where condensed water is trapped. The air then passes upward through the cartridge which contains Du-Cal DRIERITE and Indicating DRIERITE and is dried to –100°F. The air exits the drier through a port in the right side of the head. Water that is trapped below the cartridge must be periodically removed by opening the condensate drain valve.

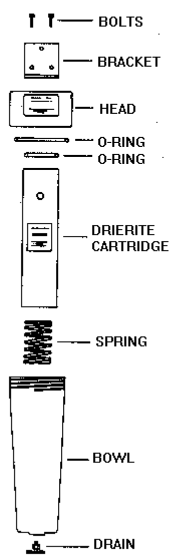
NOTE: Do not use this drier in the presence of vapors or liquid containing phosphate esters, synthetic lubricants, hydrocarbon solvents, methanol, acetone or lacquer solvents.

Installation

CAUTION: Read all warnings and safety precautions before installing this drier.

1. Install the drier in an upright position as near to the point of use as practical.
2. Mount bracket in desired location.
3. Attach aluminum head to bracket with 1/4-20 bolts provided.
4. Connect air supply to the inlet side of the aluminum head (marked IN) with suitable 1/4" pipe fitting using Teflon tape on the threads.
5. Connect the outlet side of the aluminum head (marked OUT) to the point of use with a suitable 1/4" pipe fitting using Teflon tape.
6. Valves should be located near the drier in the supply line and in the outlet line to facilitate changing cartridges.
7. The cartridge shipped with the drier must be punctured in both ends before use. (Follow instructions for changing cartridges.)

NOTE: If the drier is located in an area where it may become damaged or exposed to chemicals incompatible with polycarbonate, the use of a bowl guard is recommended.



Operation

CAUTION: The polycarbonate bowl, being tough and transparent, is ideal for use with driers. It is suitable for use in normal industrial environments, but should not be subjected to direct sunlight, an impact blow, nor temperatures outside of the rated range. As with most plastics, some chemicals can cause damage. The polycarbonate bowl should not be exposed to chlorinated hydrocarbons, ketones, esters and certain alcohols. It should not be used in air systems where compressors are lubricated with fire- resistant fluids such as phosphate esters or di-ester types. These chemicals can weaken the bowl and possibly cause the bowl to burst causing eye or other serious injury.

If fogging or crazing of the polycarbonate bowl occurs, the use of the drier must be discontinued. This is an indication of chemical attack and the bowl must be replaced before the use of the drier can resume.

WARNING: FAILURE OR IMPROPER SELECTIONS OR IMPROPER USE OF THIS PRODUCT CAN CAUSE DEATH, PERSONAL, INJURY AND PROPERTY DAMAGE.

1. Drain any water that is trapped beneath the cartridge by opening the condensate drain valve.
2. Replace the cartridge with new one when the window turns from blue to pink.

Changing Cartridges

1. Close supply valve and outlet valve.
2. Release air pressure by opening the condensate drain valve.
3. Unscrew and remove the bowl by turning counterclockwise by hand. (Use no tools.)
4. Remove exhausted cartridge.
5. Puncture both ends of a new cartridge with an opener or sharp instrument.
6. Place the new cartridge in the bowl on the spring support with the indication window toward the top.
7. Place the 2-1/2" o.d. O-ring on top of cartridge.
8. Place the 4" o.d. O-ring inside the aluminum head. (The O-rings should be clean and dry. Use no grease.)
9. Secure the bowl to the head by screwing clockwise until contact with the O-ring is made (hand tight only - do not overtighten).
10. Close the condensate drain valve.
11. Open the supply valve and the outlet valve.

The drier is now ready for use.

Cleaning

To clean the polycarbonate bowl use ONLY MILD SOAP AND WATER. DO NOT use cleaning agents such as acetone, benzene, carbon tetrachloride, gasoline, toluene, etc., which are damaging to plastic.

Temperature Range

The temperature range used for your analyses depends on the sample and the specific application. The DTA 7 can be used to analyze samples in a range from ambient to 1600°C. Isothermal analyses also can be performed at any selected temperature in the range of the instrument.

CAUTION: The platinum/10% rhodium sample cup holders have a melting point of 1810°C. The platinum sample cups, however, have a melting point of 1769°C and generally should not be used at temperatures above 1500°C.

Alumina sample cups should be used when metal samples are being run and when the DTA 7 is programmed to run at temperatures above 1500°C.

Scanning Rate

The DTA 7 allows scanning rates from 5.0°C/min to 100°C/min in steps of 5.0°C. Proper selection of the scanning rate will increase the efficiency of your analysis at the desired sensitivity. Generally, slower scanning rates improve the peak resolution while faster scanning rates improve the usable sensitivity.

NOTE: Scanning rates greater than 40°C/min are normally used only for rapidly heating or cooling the DTA 7 to the starting temperature or to a selected isothermal temperature. Typical experimental scanning rates range from 2°C/min to 20°C/min.

Automatic Baseline Optimization

An automatic baseline optimization feature allows you to use a previously saved baseline file to automatically correct sample runs performed with the DTA 7. The data of the file entered in the Baseline File Name field on the Initial State page of the Method Editor will be subtracted from the data as it is collected if the Use Baseline Subtraction box is checked on the same page. Below is information to help you determine if a new baseline file should be used for baseline optimization. If you are not sure that the current baseline is adequate, the easiest way to assess its validity is to do a run with an empty sample cup on both the sample and the reference thermocouples. Enter the baseline file name in the DTA 7 method, click on Use Baseline Subtraction, and start the run. A fairly straight, flat curve should be obtained. If a reasonable data curve is not obtained, you should create a new baseline file (with no baseline file entered in the method) and use it for future runs.

1. If you change the temperature range of operation, the baseline file that you use may not encompass this new range. You may have to create a new baseline file.

NOTES: If you created a baseline file that has heating and cooling ramps at different scanning rates, the software will try to select and use the best segment (heating vs. cooling and closest rates). However, we recommend that the temperature program of the baseline file match exactly the temperature program of the method used for your experiments. You may save several baseline files. Alternatively, you can run a baseline over a very wide temperature range and use it for all data sets.

If you are running a multiramp experiment and each heating cycle is the sample rate and temperature range, then only one identical baseline heating scan is required. This baseline will be subtracted from each heating cycle. This also applies to cooling experiments.

2. If you do an experiment that uses a very different heating or cooling rate than the rates used with the current baseline file, perfect optimization may not be achieved. You may have to create a new baseline file that uses the new scan rates.

3. If you change the settings of the slope control or the trimpots, you may have to create new baseline files.
4. If you change the type of sample purge gas or if the purge gas flow rate is changed, you may want to create new baseline files for optimal performance.
5. If you change any of the thermocouples or the furnace and/or furnace thermocouple, you will have to create new baseline files.
6. If the sample cup holders, sample cups, or sample tube become very contaminated, the baseline optimization routine may appear to be working incorrectly. Clean the sample holder, change the sample cups, and clean or change the sample tube before deciding if a new baseline file must be used.

Maintenance

The DTA 7 needs little routine maintenance other than proper treatment as a sensitive electronic device. Occasionally the furnace control or sample and reference thermocouples may require changing, or the furnace and other accessories such as the purge tube may need to be replaced. When the thermocouples are replaced, they must be recalibrated. This involves adjusting the three trimpots at the back of the analyzer.

Maintenance of the DTA 7 involves the following topics:

NOTE: Tools needed to perform the maintenance steps below are a posidrive screwdriver for M3 screws (size #1) and a posidrive screwdriver for M4x screws (size #2).

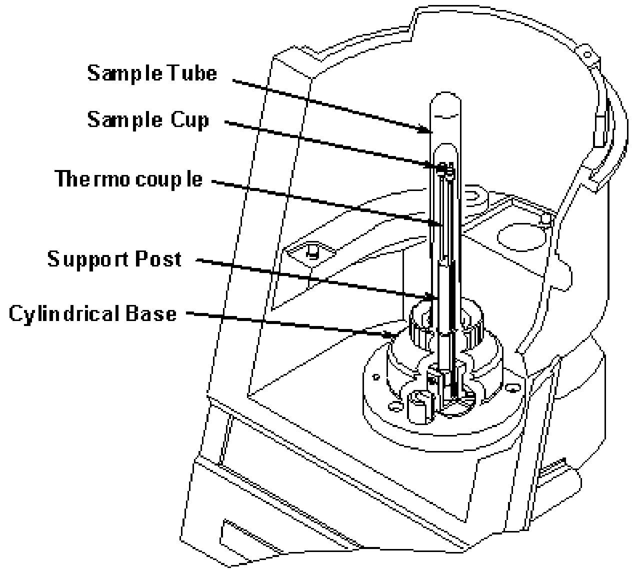
- **Replace the Sample and Reference Thermocouples**
- **Replace the Purge Tube**
- **Replace the Furnace Control Thermocouple**
- **Replace the Furnace**
- **Replace Fuses**
- **Calibrating Thermocouples**

Replace the Sample and Reference Thermocouples

The sample and reference thermocouples are brittle and can break easily. Always handle the thermocouples with care and avoid making any sharp bends that might damage them. The sample and reference thermocouples are a matched pair (P/N N538-0052). Thus, it is important that both sample and reference thermocouples are replaced at the same time, even if only one is damaged.

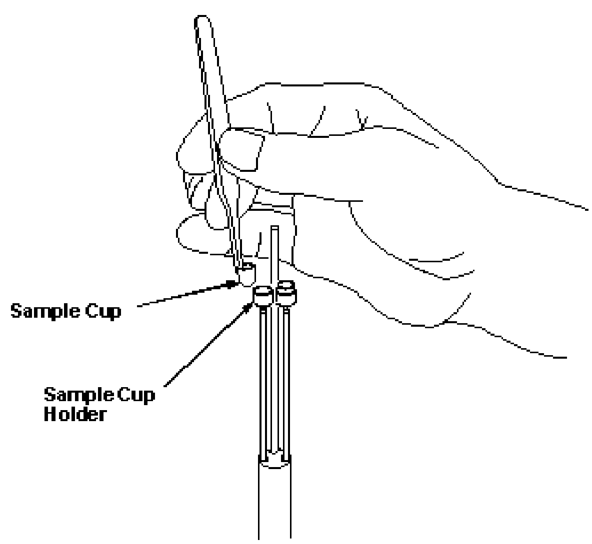
NOTE: Before performing this procedure, shut down the system in the following order: printer, TAC, DTA 7, computer. Remove the line cord from the analyzer and make sure that the furnace has cooled sufficiently.

Follow the procedure below to replace the sample and reference thermocouples.

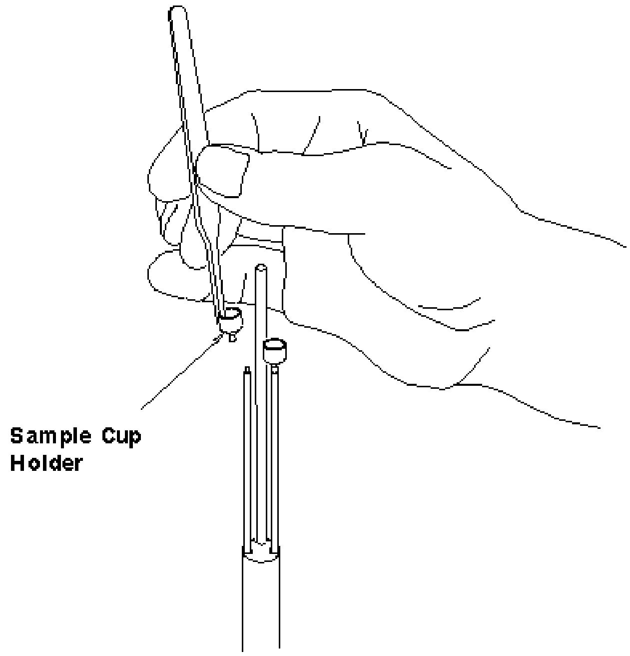


NOTE: If the purge tube breaks while you are replacing the sample and reference thermocouples, you will need to replace the purge tube also.

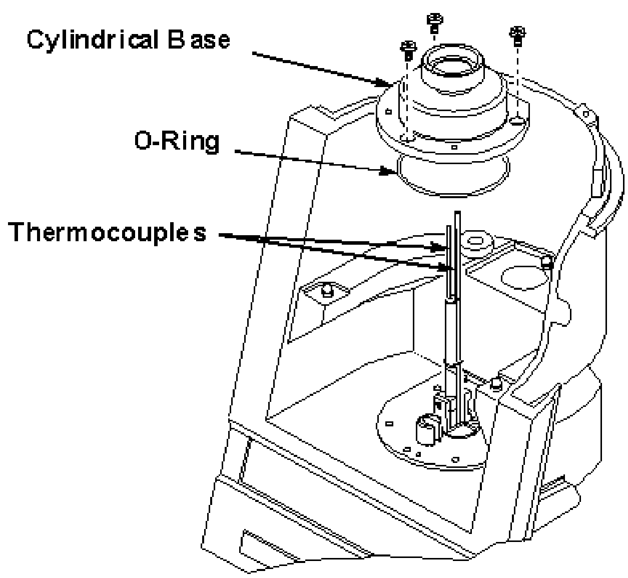
1. Shut off the power to the DTA 7 and the TAC 7/DX.
2. Raise the furnace assembly and swing it to the right side.
3. Unscrew the sample tube knurled nut, then carefully lift the sample tube straight up and off of the base.
4. Using tweezers, lift the sample cups up and off the sample cup holders.



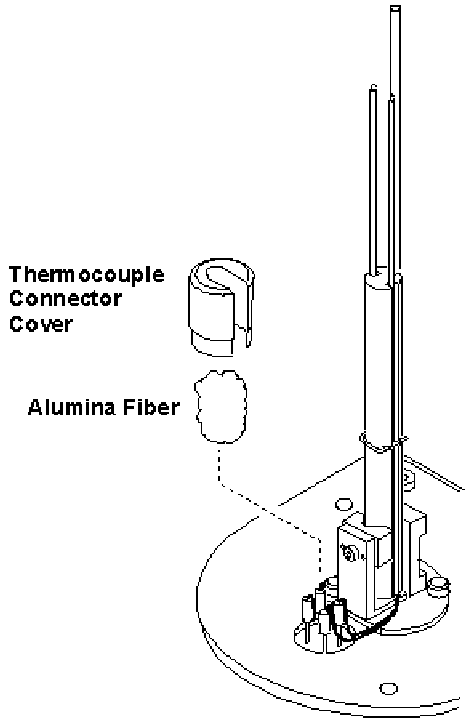
5. Using tweezers, lift the sample cup holders up and off the thermocouples.



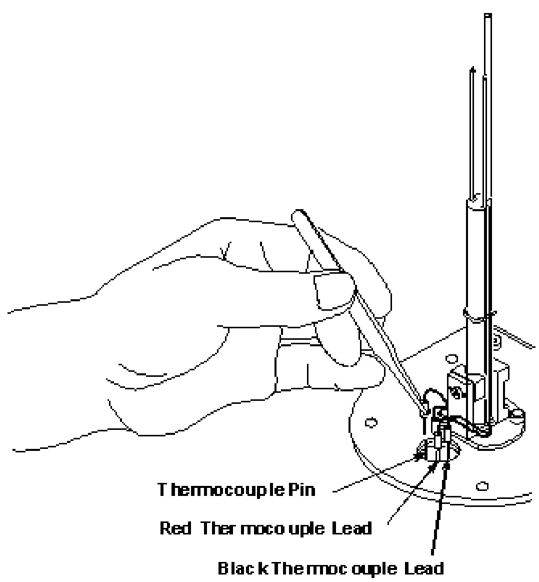
6. Locate and loosen the three posidrive screws on the cylindrical base. Carefully lift the base straight up and off the front casting, over the thermocouples and the purge tube.



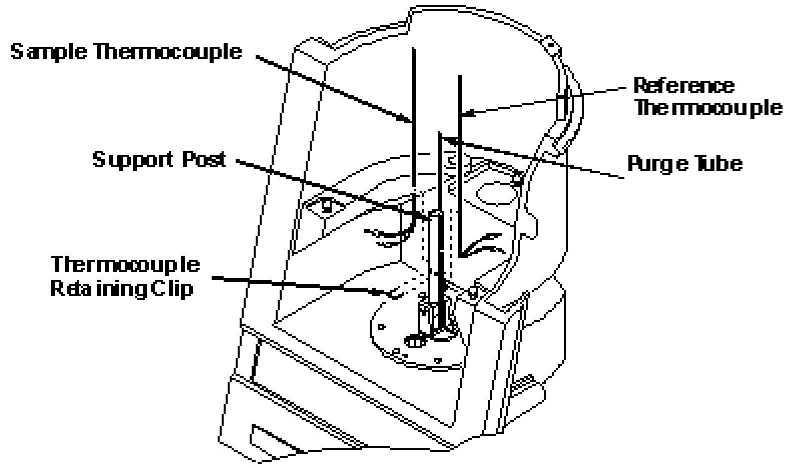
7. Remove the thermocouple connector cover by lifting it up and off. The cover is packed with alumina fiber, which is used to insulate the connectors. Some of the insulation will remain on the baseplate around the connector pins.



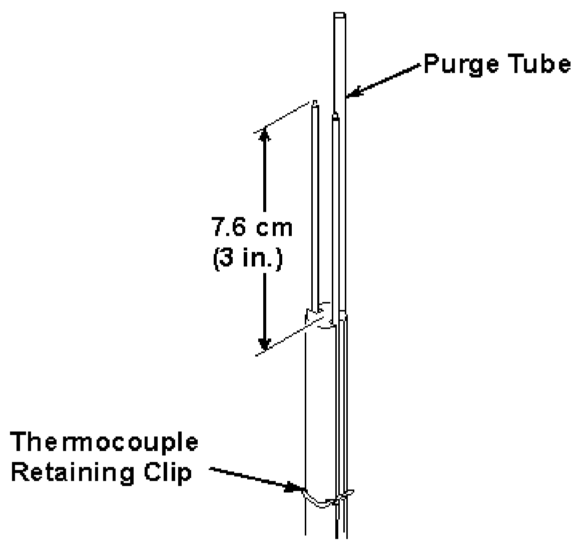
8. Remove all of the alumina fiber from around the thermocouple pin connectors and place it in the thermocouple connector cover. Brush off all remaining insulation or dust from the baseplate and from around the thermocouple pins.
9. Unplug the thermocouple pin connectors from the sockets using tweezers. The black wires are in the front and the red wires are in the back.



10. Remove the thermocouple retaining clip and set it aside.
11. Remove the old thermocouples.



12. Place the new thermocouples in the grooves on either side of the support post.
13. Replace the thermocouple retaining clip.
14. Gently slide each thermocouple so that each one extends 7.6 cm (3.0 in.) above the support post.



15. Plug in the thermocouple connectors. Be careful not to get any alumina fiber in between the receptacles and connectors. Remember to plug the black connectors into the front and the red connectors into the back.
16. Replace the alumina fiber around the connectors, pack the thermocouple connector cover with alumina fiber, and replace the thermocouple connector cover.
17. Seat the O-ring into the groove in the bottom of the cylindrical base. Lower the cylindrical base into position. Make sure that it does not touch the thermocouples.
18. Replace the posidrive screws on the cylindrical base and tighten.
19. Using tweezers, place the sample cup holders onto the thermocouples.
20. Place a small amount of alumina powder into the sample cup holders. This is done to increase energy transfer. However, when using alumina sample cups, the tips of the thermocouples should not be covered with the powder and the thermocouple tip should make contact with the

sample cup. When using platinum sample cups, the tips of the thermocouples should be covered with alumina powder.

21. Using tweezers, place the sample cups into the sample cup holders.
22. Carefully lower the sample tube assembly into place and tighten the sample tube knurled nut.

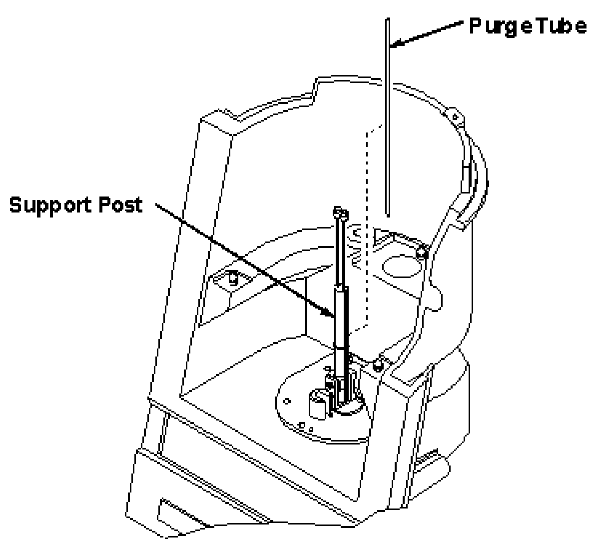
NOTE: The bottom of the sample tube has a notch that corresponds to a pin in the cylindrical base. This notch helps orient the sample tube.

Replace the Purge Tube

CAUTION: The purge tube is brittle. Always handle the purge tube with care to avoid damaging it.

NOTE: Before performing this procedure, shut down the system in the following order: printer, TAC, DTA 7, computer. Remove the power cord from the analyzer and make sure that the furnace has cooled sufficiently.

1. Shut off the power to the DTA 7.
2. Raise the furnace assembly and swing it to the right side of the instrument.
3. Unscrew the sample tube knurled nut; carefully lift the sample tube straight up and off the module.
4. Locate and loosen the three posidrive screws on the cylindrical base; carefully lift the base straight up and off the front casting, over the sample and reference thermocouples and the purge tube.
5. Locate the purge tube and lift it off the assembly. It is a snug fit but the purge tube can be removed.



6. Locate the flat spring steel clip in the back of the support post clamp.

7. Push the flat spring clip away from the clamp while sliding the new purge tube into position along the back of the support post. The clip will block the tube if it is not pushed away from the clamp.
8. Push the new purge tube down into its seat underneath the tube support assembly. The seat has a small O-ring that forms a seal around the purge tube. (You will feel some resistance from the O-ring as the purge tube is pushed into it.) When the tube touches the bottom of the baseplate, it is installed securely.
9. Make certain that the purge tube bottoms properly so that it is held securely and is exactly vertical.
10. Seat the large O-ring into the groove in the bottom of the cylindrical base. Lower the base into position; take care not to touch the thermocouples.
11. Replace the posidrive screws in the cylindrical base and tighten.
12. Carefully lower the sample tube assembly into place and tighten the sample tube knurled nut.

NOTE: The bottom of the sample tube has a notch that corresponds to the pin in the cylindrical base. This helps orient the sample tube.

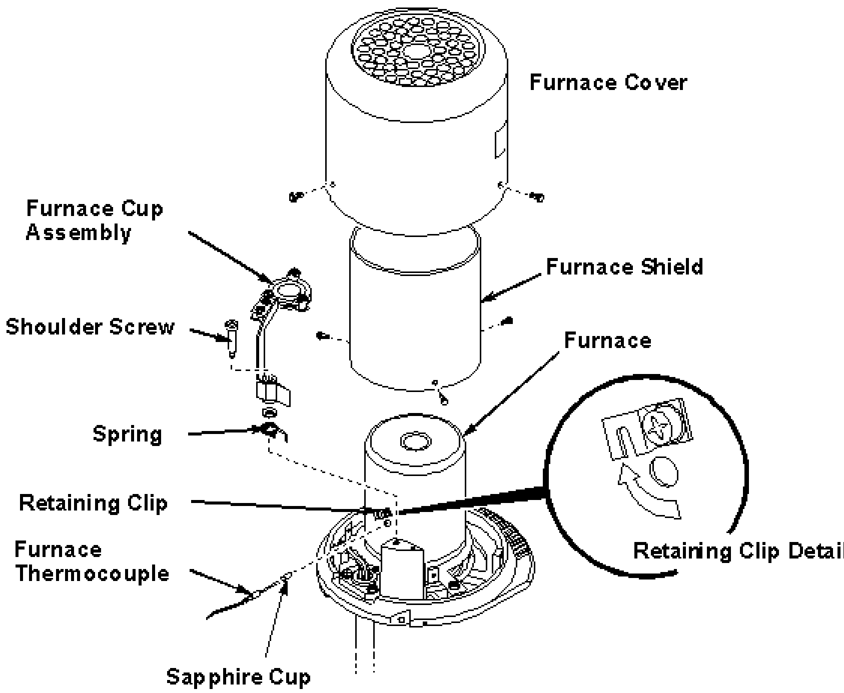
Replace the Furnace Control Thermocouple

The furnace control thermocouple should be replaced whenever the monitored temperature becomes erratic or when the furnace is replaced.

NOTE: Before performing this procedure, shut down the system in the following order: printer, TAC, DTA 7, computer. Remove the line cord from the analyzer and make sure that the furnace has cooled sufficiently.

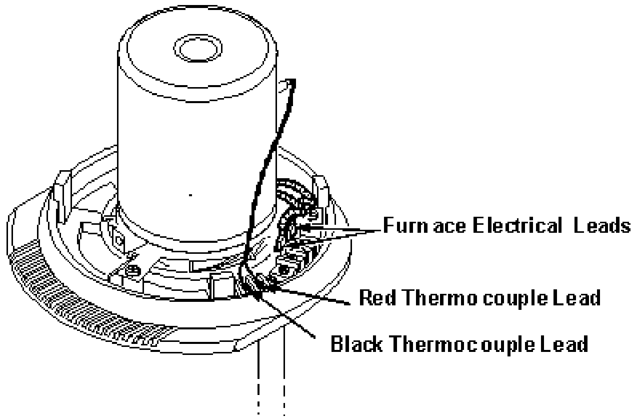
Follow the procedure below to replace the furnace control thermocouple.

Furnace Assembly Disassembly Schematic



1. Shut off the power to the DTA 7.
2. Disconnect the power cord from the analyzer.
3. Raise the furnace assembly and swing it to the right side.
4. Unscrew the three posidrive screws on the furnace outer cover then lift the cover off of the assembly.
5. Unscrew the shoulder screw on the furnace cap assembly. Remove the furnace cap assembly while noting the position of the spring.
6. Remove the three screws that secure the furnace shield. Lift the shield of the furnace.
7. Locate the retaining clip that holds the furnace thermocouple in place. Loosen it but **do not** remove the flat screw on the clip.
8. Swing the retaining clip up for clearance and pull the furnace thermocouple straight out.

NOTE: The sapphire cup at the end of the thermocouple may have become fused to the thermocouple. In this case, the cup (P/N N519-1875) will need to be replaced.



9. Write down the position of the black and red furnace thermocouple leads. Disconnect the leads from the connectors.
10. Place the sapphire cup onto the new thermocouple and insert them into the furnace. Swing the retaining clip over the new thermocouple and tighten the flat screw. Make sure that the thermocouple is held snugly in place. It should not be loose.
11. Connect the thermocouple lead to the connectors.
12. Replace the furnace shield and tighten the three screws that secure it in place.
13. Replace the furnace cap assembly, carefully loading the spring in its proper position. Tighten the shoulder screw.
14. Replace the furnace outer cover and tighten the posidrive screws.

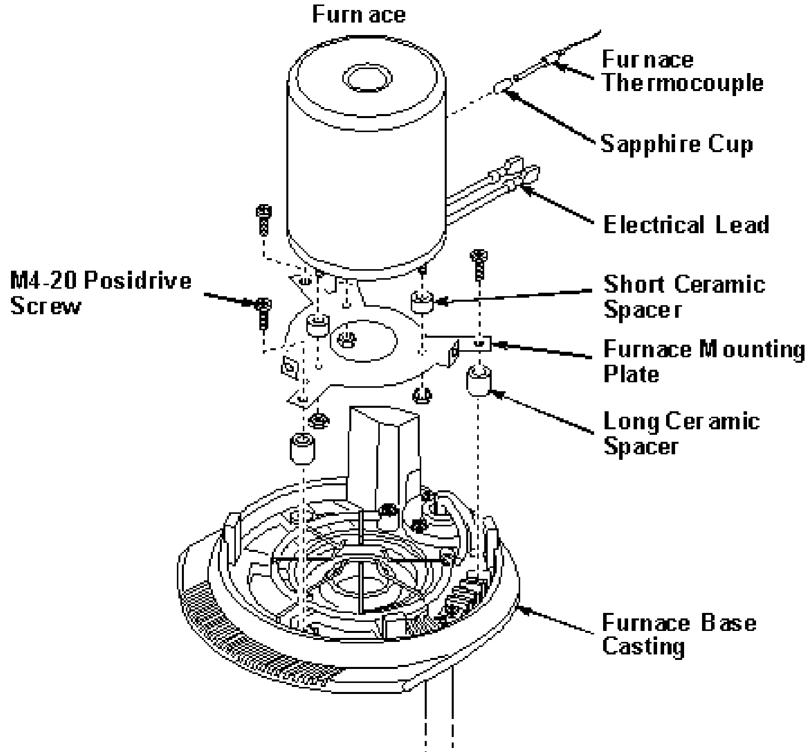
Replace the Furnace

NOTE: Before performing this procedure, shut down the system in the following order: printer, TAC, DTA 7, computer. Remove the line cord from the analyzer and make sure that the furnace has cooled sufficiently.

Refer to the figure in the subsection above when performing the first nine steps of replacing the furnace assembly according to the procedure below:

1. Shut off the power to the DTA 7.
2. Disconnect the power cord from the analyzer.
3. Raise the furnace and swing it to the right side.
4. Unscrew the three posidrive screws from the furnace outer cover. Lift the cover off the assembly.
5. Unscrew the shoulder screw on the furnace cap assembly; remove the assembly, noting the position of the spring.
6. Remove the three screws that secure the furnace shield and lift the shield off of the furnace.
7. Locate the clip that holds the furnace thermocouple in place on the furnace. Loosen but **do not** remove the flat screw on the clip.
8. Swing the clip up to clear it away from the thermocouple and pull the furnace thermocouple, with the sapphire cup, straight out.
9. Disconnect the furnace’s electrical leads, noting their location.

Furnace Disassembly



10. Remove the three posidrive screws that secure the furnace mounting plate and lift the furnace assembly with the mounting plate off the furnace base casting.

    NOTE: There are three long ceramic spacers located directly below the mounting plate. They are held in place by M4-20 screws when assembled.

11. Place the furnace assembly upside-down on the bench so that the mounting plate faces up and remove the three nuts and washers.
12. Lift the mounting plate off the furnace assembly and note the orientation of the short ceramic spacers.
13. Remove the three short ceramic spacers from the studs.
14. Put the short spacers onto the new furnace assembly's studs.
15. Place the mounting plate on the new furnace and secure it with the nuts and washers.
16. Place the long ceramic spacers into position and turn the new furnace assembly with mounting plate right-side up so that the leads face the pivot shaft. Mount the assembly onto the furnace base casting with the M4-20 posidrive screws.
17. Set the torque on the screws so that the assembly is snug but the position of the furnace can be adjusted by hand.
18. Connect the new furnace's electrical leads.
19. Swing the furnace base casting into position and carefully lower it over the sample tube.

20. While looking down into the furnace assembly's hole, move the furnace element to center it around the sample tube. Once positioned properly, tighten the M4-20 screws.
21. Lift the furnace assembly up and swing it to the right.
22. Replace the furnace shield and tighten the three screws.
23. Ensure that the sapphire cup in on the furnace thermocouple. Insert the thermocouple with sapphire tip into the furnace. Swing the clip over the thermocouple and tighten the flat screw.
24. Replace the furnace cap assembly, carefully loading the spring in proper position. Tighten the shoulder screw.
25. Replace the furnace outer cover and tighten the posidrive screws.

Replace Fuses

The main fuses of the DTA 7 are located in the power module and can be different according to the voltage that is to be used. The fuses to be used for the specific voltage rating are as follows:

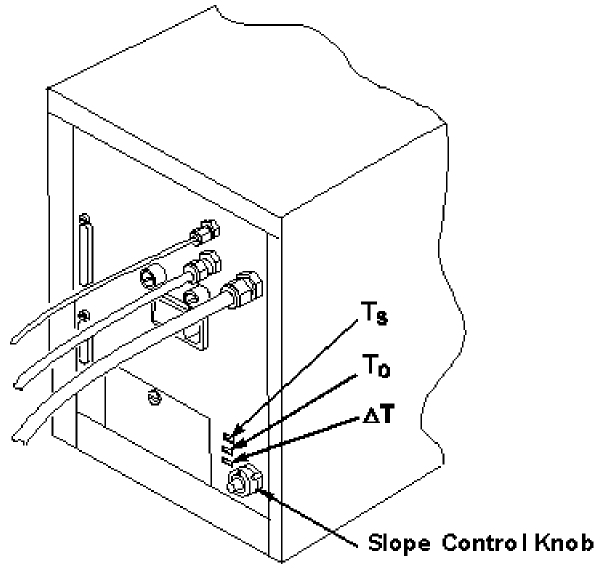
Voltage	**F1**	**F2**	**F3**	**F4**
100 and 120 VAC	Short	12A 0999-1640	Short 0991-1536	1A 0998-1611
220 and 240 VAC	12A 0999-1640	12A 0999-1640	0.63A 0999-1646	0.63A 0999-1646

Use the following procedure to replace the main fuses:

1. Turn off the main power switch on the front panel of the DTA 7 and disconnect the power cord.
2. Open the fuse cover of the voltage selector assembly (on the back of the instrument next to the line cord input).
3. Inspect the fuses and replace them if necessary.
4. Replace the fuse cover of the voltage selector assembly.
5. Connect the power cord and turn on the main power switch.

Calibrating Thermocouples

Once any of the three thermocouples or the furnace has been replaced, it may be necessary to calibrate the new thermocouple(s) or the furnace. Calibration is done by using the trimpots located on the rear of the analyzer.



The trimpots are used to calibrate Ts (Sample Temperature Zero), To (Oven Temperature Zero), the ΔT (ΔT Zero). The order in which these should be calibrated is Ts, ΔT, To.

Calibrate Sample Temperature Zero (Ts)

The purpose of the Ts trimpot is to calibrate the output of the sample thermocouple at one point, i.e., room temperature. With the furnace equilibrated at ambient temperature and the Control light out, adjust the Ts pot using the small screwdriver that came in the Spares kit) until the Status Panel displays the correct temperature.

Calibrate ΔT Zero

The purpose of this adjustment is to make certain that the sample and reference thermocouples' differential signal is balanced to zero. With the furnace equilibrated at ambient temperature, and the screen displays at –1.0 and +1.0 on the Y axis, adjust the ΔT trimpot until it reaches zero.

NOTE: One complete turn of the pot is approximately equal to 0.8°C. Turning **clockwise** will reduce the ΔT reading while turning it **counterclockwise** will increase the ΔT reading.

Calibrate the Oven Temperature Zero (To)

The purpose of adjusting this trimpot is to ensure that the sample temperature thermocouple and the oven control temperature thermocouple agree at one temperature. This is usually ambient or room temperature. In the Control panel, enter a temperature in the Go To Temperature field that is 6°C – 8°C above the actual temperature. Adjust the oven trimpot until the Control light on the DTA 7 comes on.

Part Numbers

Your analyzer should be one of the following part numbers:

N538-0064 100 V

N538-0065 120 V

N538-0066 220 V

Spares and Accessories Kit (N538-0015)

Part Number	Description	Quantity
0154-1498	Restrictor “H”	1
0240-1286	Micro Hayman Spatula	1
0250-6483	Teflon Tubing – 0.125 in.	4
0250-6515	Tygon Tubing – 0.25 in. o.d. black	4
0303-1358	Label	1
0419-0197	Alumina Powder – 28 g	1
0419-0287	Bulk Fiber – Alumina	1
0419-1231	Clip — Thermocouple Spring	4
0419-1235	Purge Tube	1
0419-1246	Cup Holder – Platinum	3
0419-1290	Sample Cup – Alumina (60 cu. Mm)	6
0419-1431	Liner Top	4
0419-1432	Liner Bottom	4
0419-1433	Liner Bottom	1
0419-1434	Liner Top	1
0990-2245	O Ring for Purge Tube	3
0990-3094	Insert – Tubing	6
0990-3172	Fitting – Plug	1
0990-3906	Female Connector 1/4 NPT – 1/8 Tubing	1
0990-7236	Allen keys – V Block	1
0990-7273	Screwdriver	1
0990-8134	Teflon Tape	1
0990-8400	Tweezers	1
0990-8406	Plastic Box	2
0990-8521	Plastic Box	1
0992-1018	O Ring – 2.7 i.d. x 0.07 wd	2
0992-1019	O Ring – 0.87 i.d. x 0.07 wd	2

0998-1608	Fuse – 0.2 A	2
0998-1611	Fuse – 1 A 250 V Slo Blo	3
0998-1640	Fuse – 12 A	4
0999-1646	Fuse – 0.63 A	4
N538-0057	Aluminum Wire Calibration Std	1
N538-0058	Gold Wire Melt Calibration Std	1

TAC 7/DX Interface

N519-0070	100 V
N519-0071	115 V, 50/60 Hz
N519-0072	230 V, 50 Hz

All TACs use N519-0310, the TAC 7/DX to analyzer cable.

Accessories and Replacement Parts

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

- If you are located within the U.S., call toll free at 1-800-762-4002, 8 a.m. – 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside of the U.S., call your local Perkin Elmer sales office.

You can also see the PE XPRESS Catalog in Adobe® Acrobat® .pdf format by selecting **PE Express Catalog** from the Pyris Software for Windows menu and then selecting **Thermal and Elemental Analysis**. You can then print the catalog out for future use.

The following accessories and replacement parts can be purchased for use with the DTA 7:

DTA 7 Sampling Accessories

Sample Loading Tray	N538-0061
Open Sample Tube	N538-0062
Sample Cup Covers (2 per pkg.)	N538-0063

DTA 7 Replacement Parts

Support Post	0419-1228
O-Ring Seal (Bottom)	0990-2218
Button MTG Nylon	0994-0661
Thermocouple Connector Cover	0419-1406
Furnace Thermocouple	N538-0051
Sapphire Cup	N519-1875

Retaining Clip (for Furnace Cap Assy.)	0419-1230
Spring (for Furnace Cap Assy.)	N538-1058
Shoulder Screw	0991-9185
Short Ceramic Spacer	0419-1234
Long Ceramic Spacer	N538-1001

Index

E

F

G

H

Q

R

S

T