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# Chapter 3

## Pyris 6 DSC

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### 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- $\mu$ L 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 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 $\mu$ L	0.1 mm	sealed aluminum pans (400)
B014-3016	30 $\mu$ L	0.1 mm	sealed aluminum pans (400)
B014-3017	50 $\mu$ L	0.1 mm	sealed aluminum pans (400)
B014-3018	30 $\mu$ L	0.1 mm	vented aluminum pans (400)
B014-3019	50 $\mu$ L	0.1 mm	vented aluminum pans (400)
B014-3024	50 $\mu$ L	0.1 mm	sealed gold pans (10)

### 2 bar (200 kPa) maximum internal pressure

B014-3020	25 $\mu$ L	0.15 mm	sealed aluminum pans (400)
B014-3021	50 $\mu$ L	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- $\mu$ L internal volume (400 pans and covers)
B016-9321	Aluminum sample pans and covers, 50- $\mu$ L 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- $\mu$ L 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- $\mu$ L 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.

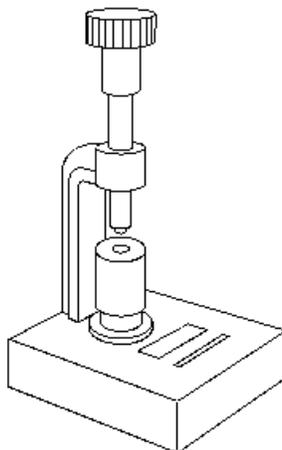
## Crimpers

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

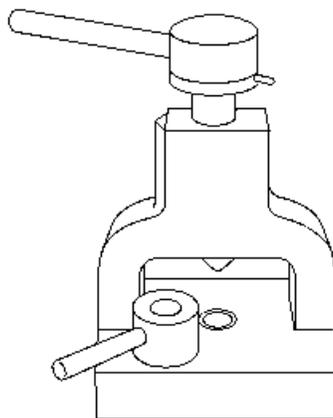
### Standard Sample Pan Crimper Press

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

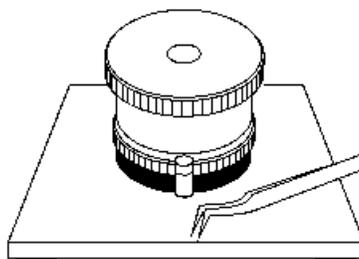
The Volatile Sample Sealer (P/N 0219-0061) is used to seal volatile sample pans. The pans and sealer are used with volatile solid or liquid samples that exert significant vapor pressure in the temperature range of interest. For example, aqueous solutions can be scanned up to and through 100°C to observe 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**

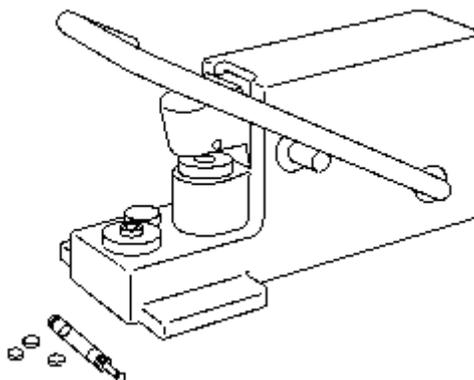
### **Vapor Pressure Sample Pan Kit (P/N N519-0788)**

This kit includes 100 volatile aluminum pans and 100 pierced covers. A 50- $\mu$ m-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**

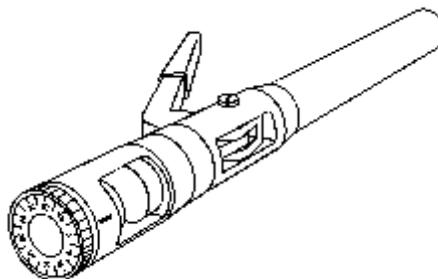
The Universal Crimper Press (P/N B013-9005) is used for sealing all of the robotic system sample pan types as well as most of the other Perkin Elmer DSC sample pans when used with the appropriate optional sealing insert. Other pans that can be crimped by a universal crimper press are listed in the table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

<b>Part No.</b>	<b>DSC Sample Pan Type</b>
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.

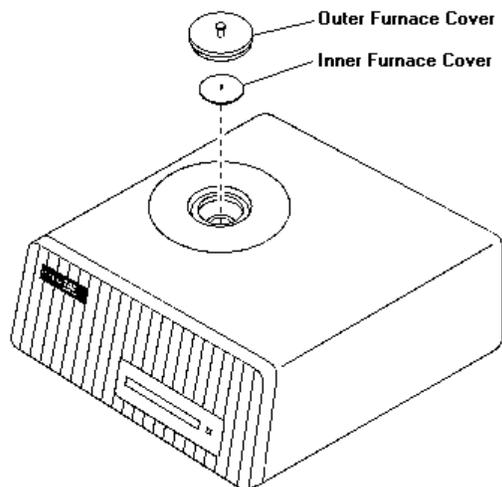
Generally, slower heating rates improve the peak resolution while faster heating rates improve the apparent sensitivity. Heating rates greater than 40°C/min are generally used for rapidly heating or cooling the Pyris 6 DSC to the beginning temperature of the next segment or to selected isothermal temperatures 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.

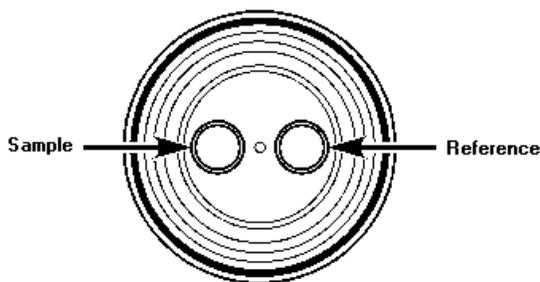
You load the sample into the Pyris 6 DSC as follows:

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  $20^{\circ}\text{C}/\text{min}$  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  $\Delta H$  for heat flow calibration. If you performing a heat flow calibration, you will need the  $\Delta H$  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  $\Delta H$  result, or use the  $\Delta H$  result recorded for the reference material when you performed this step for temperature calibration.

The measured  $\Delta H$  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  $\Delta H$  for the reference material used (e.g., 28.45 for indium).
5. Enter the measured  $\Delta H$  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  $-120^{\circ}\text{C}$  to  $200^{\circ}\text{C}$ . 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).

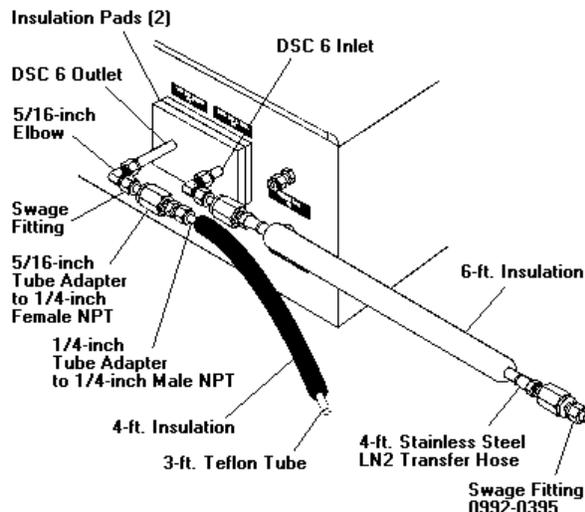
We recommend that you use the following items to set up the analyzer for subambient operation:

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.

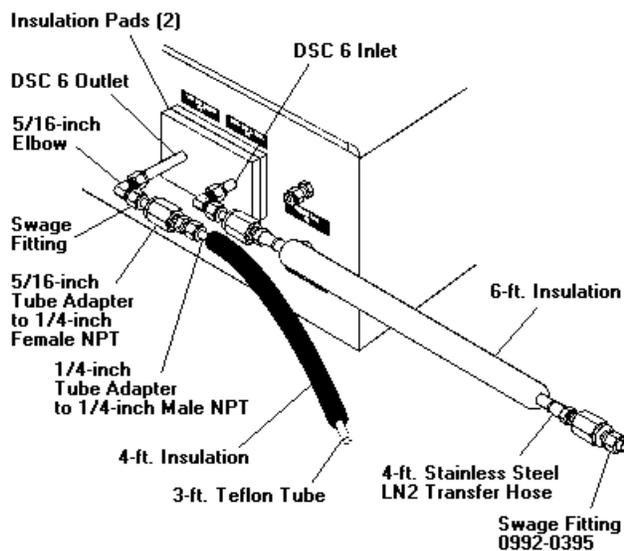
- [Preparing the Pyris 6 DSC for Subambient Operation](#)
- [Assembling the Regulator](#)
- [Connecting the Pyris 6 DSC to the Dewar](#)

## 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.

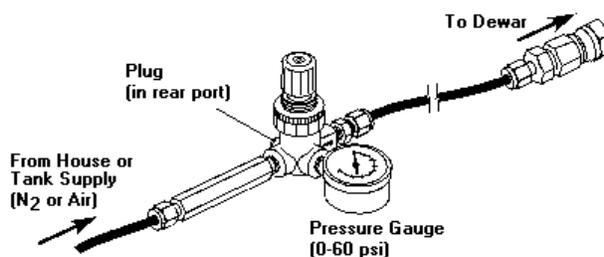
1. Place both insulation pads (P/N N519-2149) over the tubes labeled Cooling Liquid Out and Cooling Liquid In so that they are flush with the back of the analyzer.
2. Swage one of the 5/16-in. elbows (P/N 0992-0146) to the tube labeled Cooling Liquid Out.
3. Swage the other 5/16-in. elbow (P/N 0992-0146) 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 N<sub>2</sub> 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 LN<sub>2</sub> 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



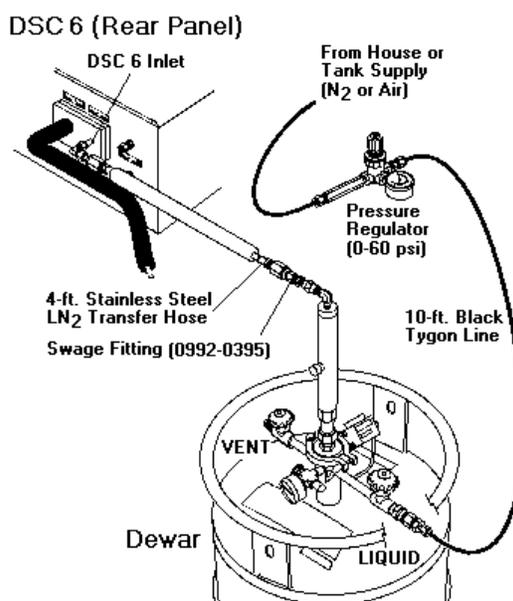
1. Remove the regulator and retaining ring (P/N 0990-7085) and plug (P/N 0990-3666) from the packaging.
2. Place the retaining ring over the top portion of the regulator (i.e., push/pull knob) and screw it onto the threads.
 

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.

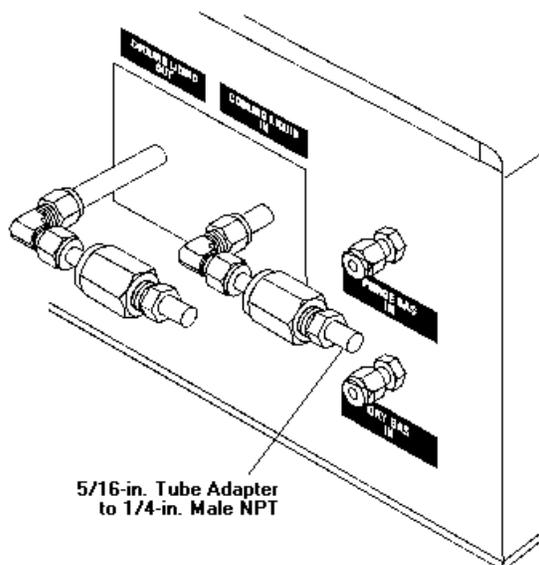
To set up the Pyris 6 DSC for **ambient operation only**, see the full installation instructions given in the online Installation Help.

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 NOT accept 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



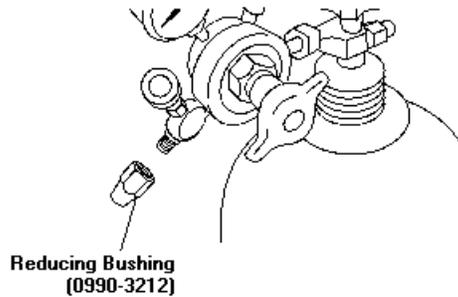
**WARNING:** When making connections, first turn the fitting until it is **fingertight**, then turn it an additional 1/2 to 3/4 turn with a wrench. Use a second wrench to keep the connection at the Pyris 6 DSC stationary. Turn preswaged fittings 1/8 turn past **fingertight**. **DO NOT OVERTIGHTEN.**

### Connect the Purge Gas Line to the Gas Supply

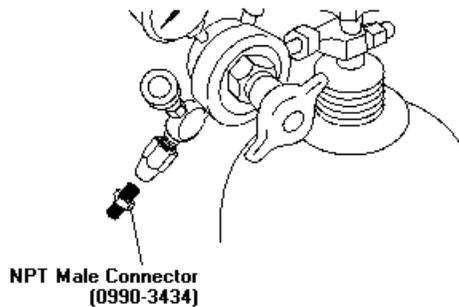
Perform the following procedure to make the connections at your purge gas supply cylinder or cylinders if you intend to use a **Thermal Analysis Gas Station (TAGS)**:

**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.

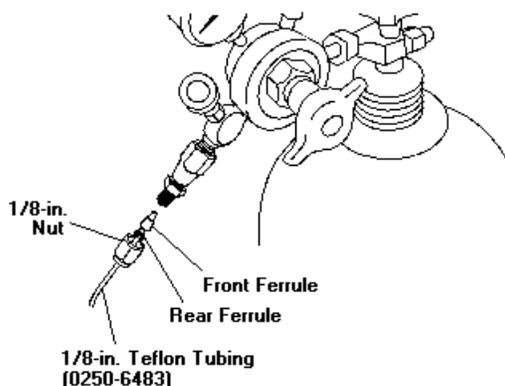
1. Wrap the threads of the shutoff valve fitting with Teflon tape, going around twice. Be sure to start wrapping one or two threads in from the end of the fitting so that the full width of the tape is on the threads.
2. Connect a reducing bushing to the taped shutoff valve fitting.



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 **connect 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

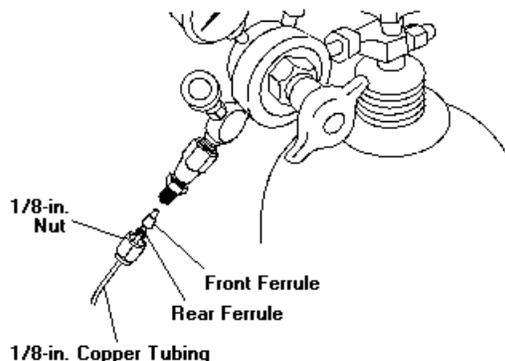
If you are going to use a Filter Dryer Accessory (P/N 0319-0039), install it in the purge gas line as described in the following procedure:

**NOTE:** The Filter Dryer Accessory is optional and may be purchased from Perkin Elmer. Contact your local Perkin Elmer Sales Representative.

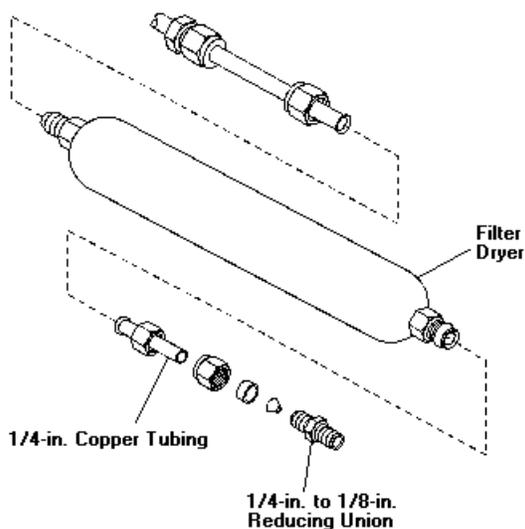
If you have a Drierite Compressed Air Filter Drier (P/N 0992-3454), its installation instructions follow these instructions.

**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.

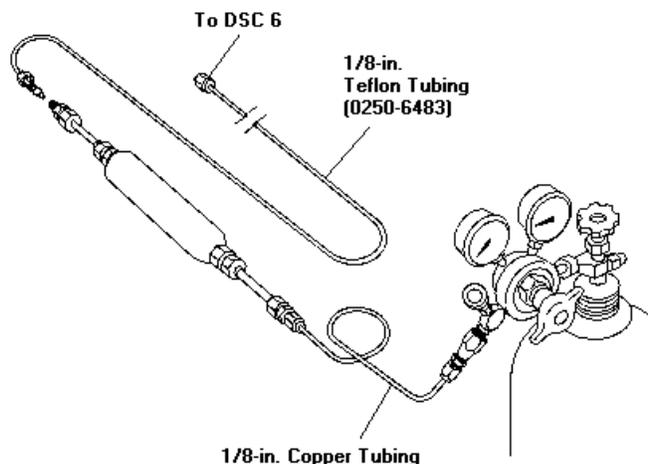
1. Connect one end of the 1/8-in. copper tubing supplied with the Filter Dryer Accessory to the NPT male connector which was installed on the regulator shutoff valve.



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

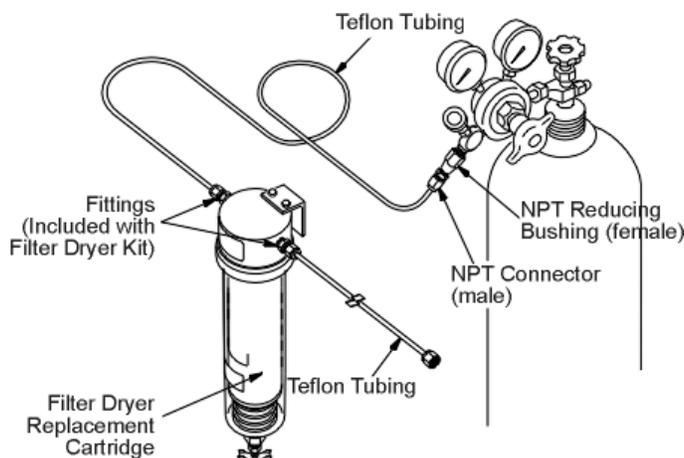
If you are going to install the Drierite Compressed Air Drier Model 207 (P/N N537-0103), follow the instructions below.



**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.

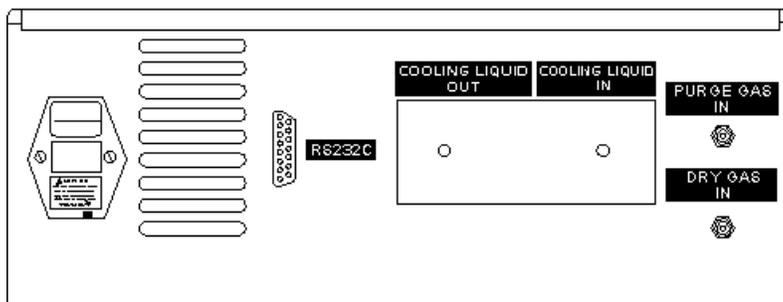
**NOTE:** When making the connections, first turn the fitting until it is fingertight, then turn it an additional 1/2 to 3/4 turn with a wrench. Use a second wrench to keep the connection at the TAGS stationary. Turn preswaged fittings 1/8 of a turn past fingertight. Do not overtighten.

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 Pyris 6 DSC in subambient mode.

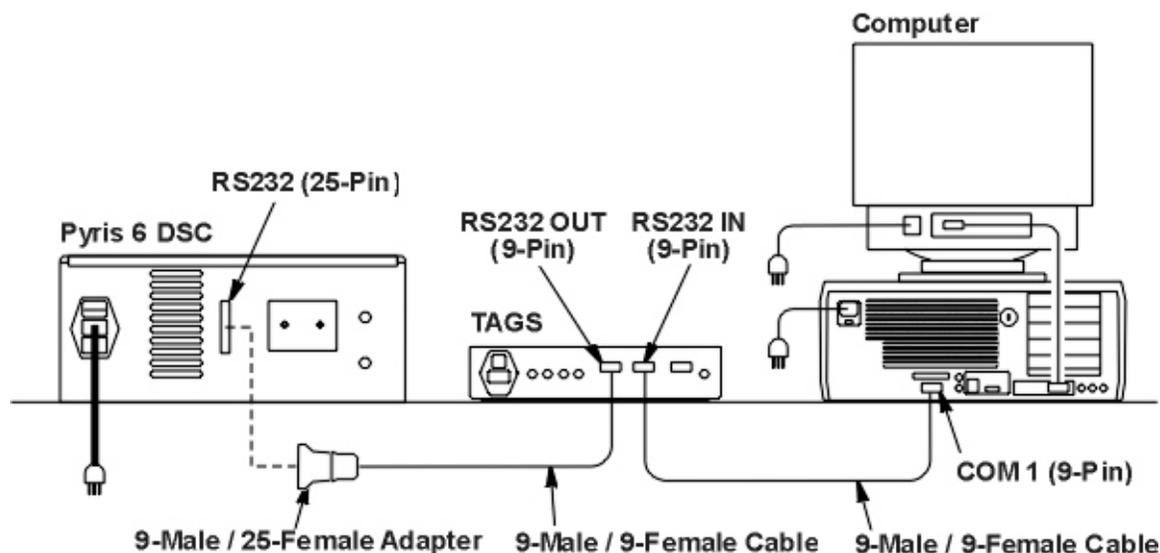


1. Place the 1/8-in. Teflon tubing from your purge gas line or TAGS onto the inlet labeled Purge Gas In.
2. Place the 1/8-in. Teflon tubing from your dry gas line onto the inlet labeled Dry Gas In.

After finishing the connection of purge gas(es) and dry gas supplies, you 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  $-170^{\circ}\text{C}$ . 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. ( $^{\circ}\text{C}$ )	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

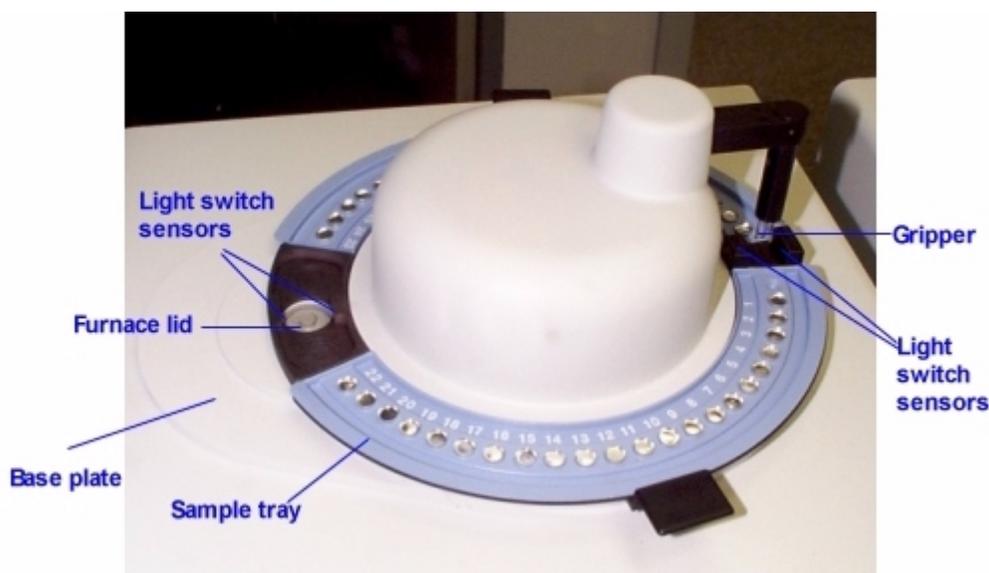
1. Tygon tubing (P/N 0250-6519) for connection of liquid coolant
2. hose clamps (P/N 0992-0044)
3. Teflon tubing (P/N 0250-8059) for purge gas connection
4. RS-232 cable for communicating to the computer
5. Power cable

## 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 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 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:

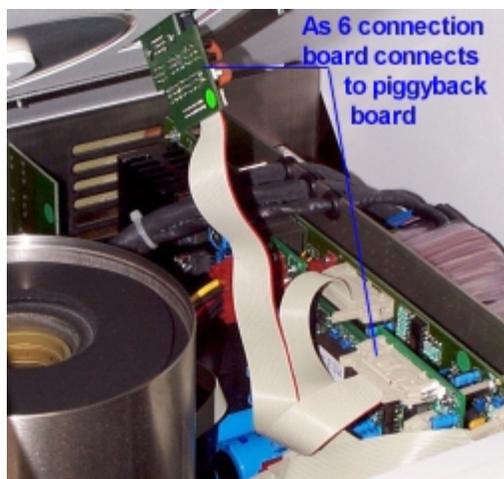


**WARNING:** To remove the furnace lid(s) of the parent instrument, always use tweezers. The furnace lid(s) might be hot.

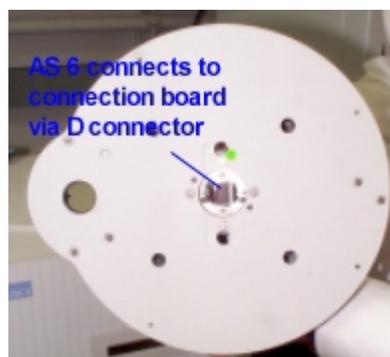
**CAUTION:** Before powering up the parent instrument, make sure that the AS 6 is properly attached to the base plate on the parent instrument.

## 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 or 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 has 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 if 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  $\mu\text{L}$ . 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.



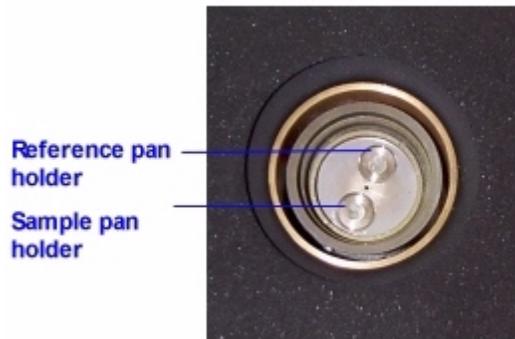
**Base plate lid holder with the upper furnace lid removed.**

6. Remove the lower furnace lid and the lower furnace lid holder.

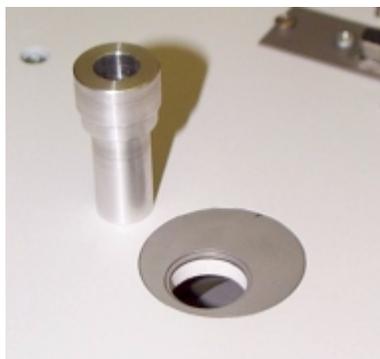


**Lower furnace lid holder with lower lid removed.**

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.

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