TECHNICAL NOTE

Thermal Cooling Accessories

Differential Scanning Calorimetry

Introduction

All Differential Scanning Calorimeters (DSC), Simutaneous Thermal Analyzers (STA), and Thermomechanical Analyzers (TMA) require cooling. A DSC with no cooling accessory relies on ambient temperature to cool its furnaces. Similar TMAs, STAs, and TGAs require some way to remove excessive heat from the system. In addition, many materials studied in TMA need to be measured at subambient temperatures. Whether conducting heating, cooling, sub-ambient, or above-ambient experiments, the best results are gained through a system configured with the appropriate cooling accessory. All cooling accessories ensure controlled, stable, reproducible analyses and choosing the appropriate cooling accessory enables the instrument to meet all its capabilities of operating at sub-ambient temperatures and achieving the fastest cooling rates.

Cooling Accessories

PerkinElmer offers a wide range of cooling options with its DSC line: chillers, refrigerated coolers, and LN_2 systems. Each of these options has certain advantages and disadvantages depending on the applications. Chillers are simple recirculating fluid systems that are easy to operate and maintain, but have limited cooling capabilities. Refrigerated coolers, which are called IntraCoolers, work like the freezer in a home and can cool to lower temperatures. They don't require a lot of maintenance and provide excellent control, but have defined temperature limits. LN_2 cooling provides the lowest possible temperatures and fastest cooling rates, and with the new PerkinElmer LN_2 system, very low N_2 consumption.

Whether you are conducting heating, cooling, sub-ambient, or above-ambient experiments, for best results a cooling accessory should be used. All of our refrigerated cooling accessories use non-chlorofluorocarbon (non-CFC) cooling medium and are all "Green" designs, or harmless to our global environment. The DSC 4000, DSC 6000, DSC 8000, and DSC 8500 do not require a dry box when using any of these cooling devices, which makes operator access easier. The interfaces for each cooling option are the same for each DSC series, allowing you easy exchangeability if you require multiple cooling accessories.

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Which One Do You Need?

When selecting the best cooling accessory, the first thing you need to consider is the lowest temperature you expect to see data. The general rule of thumb is that you should start your run at 20 °C below that expected temperature. For example, your lowest transition, minus 20 °C, gives you the starting temperature. If you are looking at polyethylene with a Tg around -120 °C, you should start collecting data at -140 °C. This new LN_2 system is the best system.

The cooling rate is also important. If you are interested in using the PerkinElmer DSC 8500 for ballistic and controlled cooling at the highest possible rate, then liquid nitrogen cooling is your best choice.

What type of tests you are running is also important. Very exothermic reactions and photo cures, for example, may only be run at room temperature and above. Yet, you would want a colder cooler to get as much stability in the heat sink as possible. This helps get better data. So for photocuring, we normally recommend an IntraCooler II.

Besides the technical requirements, the amount of space you have for the cooler, the availability of LN_2, your capital budget, and the expected annual operating expense can all influence your choice of cooling.

While important, the selection of the cooling accessory may not be final nor does it need to be labored. With modern DSC systems, it is easy to upgrade the cooling accessory and switching back and forth between different accessories is common.

Chillers: Simple, Inexpensive and Easy-to-Operate

The chiller is a recirculating fluid bath of coolant pumped through lines to a recirculation head bolted to the DSC and the TMA. For STA 6000, STA 8000, or TGA 4000, the recirculation lines connect directly to the inlet and outlet ports of the analyzers. On the DSC 8X00 and the TMA4000, the lines attach to a special recirculation chamber which then attaches to the instrument. (A different fixture is used with the Pyris 1 TGA.) It can be set at various temperatures from -20 °C to above-ambient for your specific application to provide for maximum stability. The cooling medium must be adjusted to achieve the desired temperature range. The recommended cooling media are water or a water-ethylene glycol mix.

Cooling media	Lowest chiller operating temperature
Distilled H_2O	10 °C
30 % Ethylene Glycol/70 % Distilled H_2O	10 °C
50 % Ethylene Glycol/50 % Distilled H_2O	-20 °C

Most scientists choose one cooling medium mixture for the majority of their work and infrequently change from there. Easy-to-install and operate, chillers are a convenient and inexpensive way of achieving sub-ambient temperatures and a stable heat-sink temperature while still allowing operation to the instrument's maximum temperature.

Chillers are simple to operate and inexpensive, but they are limited in performance.

Figure 1. Chiller for the DSC 4000, DSC 6000, DSC 8000, DSC 8500, and STA 6000. Chiller temperature range = -20 °C to above-ambient Chiller/ DSC system range = -10 °C to 750 °C.

IntraCoolers: Low Maintenance Systems for Cooling

Intracoolers are fully sealed refrigeration units which, unlike chillers, have their cooling medium sealed inside the system. Using a green coolant similar to Freon, these units provide low-term, low maintenance cooling with low operating expenses. Since Intracoolers only require electrical power, they are an excellent option for those without LN_2 access who need low temperature or fast cooling rates.

With a fixed cooling media, the cooling capability is increased by additional refrigeration stages in the system. Those most useful to DSC measurements are those that offer 2- stage refrigeration.

IntraCooler II	
Instruments	DSC 4000, DSC 6000, DSC 8000, DSC 8500, TMA 4000
IntraCooler II Lowest Puck Temperature	-96 °C
IntraCooler II/DSC System Range	-70 °C to 750 °C

The IntraCooler II uses a dual stage heat exchanger and is a very popular cooling choice because its temperature range encompasses most DSC applications work. It allows faster cooling rates than a chiller or a one-stage intercooler (IntraCooler I) with little increase in cost or size.

Liquid Nitrogen Cooling Systems (LN_2): Maximum Performance

For the lowest possible operating temperatures as well as the fastest cooling rates, an LN_2 system is required. LN_2 systems require a source of liquid nitrogen and tend to have a larger footprint than other systems. However, LN_2 is readily available and relatively safe to handle. Because of their consumption of LN_2, these systems do tend to be more expensive to operate. In addition, for operation below -100 °C, you will need to run with He or a He blend, which also increases cost.

Despite these disadvantages, LN_2 systems are very popular as no other cooling system can allow you to reach temperatures of -180 °C nor can any other system give you the cooling rates that LN_2 can. For these reasons, people needing the ultimate in low-temperature performance or in fast-scan cooling rates choose LN_2 systems for their laboratories.

Figure 2. IntraCoolers for use with the DSC 8000 series. Available for operation at temperatures of -40 and -70 °C.

Portable Cooling Device	
Instruments	DSC 4000, DSC 6000
Portable Cooling Device/DSC System Range	-110 °C to 450 °C

The Portable Cooling Device is a specially designed liquid nitrogen cooling system that enables customers to add the full power of LN_2 cooling to a DSC 4000 or DSC 6000 used in conjunction with their existing cooling system. Those that do not need to use LN_2 regularly can use another cooler and still have the capability to go to -100 °C when needed.

You no longer need the commitment of space and finances to a permanent Liquid Nitrogen cooling system when it is only an occasionally requirement. This approach reduces LN_2 consumption as you only cool when needed. The dewar fits directly in the DSC 4000 and DSC 6000 furnaces and therefore can be used while the DSC has a chiller or IntraCooler accessory attached. Simply install it in the DSC furnace and fill it with liquid nitrogen. After a stable instrument temperature is reached, the run can be started. Please note, this system cannot be used with an autosampler.

Cryofill	
Instruments	DSC 6000 with autosampler LN_2 cooler for autosampler
Cryofill LN_2/DSC System Range	-170 °C to 300 °C

The Cryofill LN_2 system allows a DSC 6000 with autosampler to operate between -170 °C and 300 °C under He purge. Low temperature operation is simple and automated, allowing the analyst to do other tasks.

CLN_2	
Instruments	DSC 8000, DSC 8500 Full range cooling with controlled set points
CLN_2/DSC System Range	-170 °C to 750 °C

The CLN_2/DSC System enables the fastest cooling rates possible in the industry. The CLN_2 is a controlled refrigeration unit that employs liquid nitrogen as the cooling medium. This cooling device extends the sub-ambient range and enables the fastest cooling of all the cooling choices. Unlike previous LN_2 systems, where you were required to operate at -170 °C, the CLN_2 allows you to choose a higher block temperature to conserve LN_2.

The CLN_2 is considered a controlled liquid nitrogen cooling device rather than a simple LN_2 system because simple user controls allow you to change the heat exchanger temperature from -190 °C up to 35 °C. This novel approach can dramatically reduce costs if experiments are not always run at the extremely low temperatures.

- If the heat-exchanger is set at -100 °C or above, a Nitrogen purge can be used. This is a much cheaper option than using the alternative Helium or Helium blends.
- By setting the heat-exchanger to higher temperatures significant cost saving can be made in terms of Liquid nitrogen usage. Less LN_2 is required to maintain the heat sink at -100 °C or -130 °C than at -170 °C, which reduces your costs.

Instrument Cooling Accessories Chart	
Lowest Block Temperature	**Cooling Accessory**
Ambient	No Cooler
-20 °C	Chiller
-70 °C	IntraCooler II
-100 °C	Portable Cooling Device*
-180 °C	CLN2**, Cryofill***

* DSC 4000 and 6000 manual systems only
** DSC 8000 and 8500 systems only
*** DSC 6000 with autosamler only

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