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TA Instruments DSC Cooling Accessories

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ABSTRACT
This technical bulletin describes the various Q Series accessory modules available from TA Instruments for quench and programmed cooling experiments in differential scanning calorimetry (DSC).

INTRODUCTION
TA Instruments Tzero™ technology has provided a significant technical advance in DSC operation and in the quality of the results obtained. As previously documented (1,2,3), it provides a much more accurate fundamental measurement of heat flow, which results in the flattest baselines, and superior sensitivity and resolution. At its highest level, it is also capable of direct measurements of heat capacity. Figure 1 shows a schematic of the TA Instruments Tzero DSC cell, which is the first cell designed for excellence in both heating and cooling operation. The schematic displays the identically positioned sensor platforms, the centrally placed Tzero thermocouple, the silver furnace, and the 54 symmetrically spaced, nickel cooling rods that connect to the flange of the cell. This flange is the point of contact for the various cooling devices offered by TA Instruments.

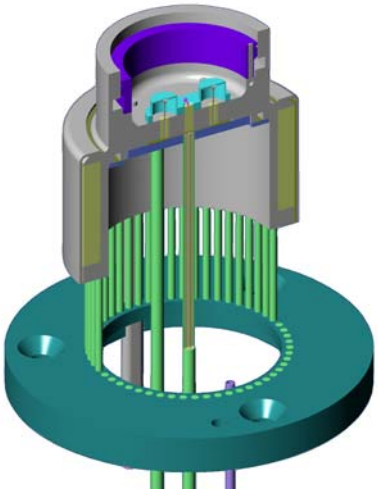


Figure 1. Tzero DSC Cell

TEMPERATURE CONTROL DEVICES
The capability for quench and programmed cooling has long been a requirement for successful operation of differential scanning calorimeters (DSCs). TA Instruments offers

five accessory modules to meet different applications. These include a Quench Cooling Accessory (QCA), a Finned Air Cooling Accessory (FACS), two Refrigerated Cooling Systems (RCS90 and RCS40) and a Liquid Nitrogen Cooling System (LNCS). This document provides descriptive and performance data for each device. Table 1 shows the overall temperature range of operation of the five cooling devices.

Cooler Type	Lower Limit	Upper Limit
QCA	-180 °C	400 °C
FACS	Ambient	725 °C
RCS90	-90 °C	550 °C
RCS40	-40 °C	400 °C
LNCS	-180 °C	550 °C

Table 1. Cooling Device Operating Ranges

QUENCH COOLING ACCESSORY

The Quench Cooling Accessory (QCA) is a manually operated cooler that is the lowest cost alternative to the automated RCS or LNCS for many applications. The QCA is the simplest device, and consists of a stainless steel vessel with an internal reservoir that fits snugly over the DSC Cell. The reservoir is easily filled with ice water, liquid nitrogen, dry ice, or other cooling media. The primary QCA use is with the Q20 DSC or earlier Q10 model to quench cool a sample to a sub-ambient temperature prior to heating to a final upper limit. Since active cooling is not present in QCA experiments, the T1 signal is the only available signal. The recommended temperature of operation of the QCA is from –180 to 400 °C.

FINNED AIR COOLING SYSTEM

The Finned Air Cooling System (FACS) is an innovative cooling accessory for all the Q Series DSC modules, except when using the Pressure DSC Cell. It offers a cost-effective alternative to the refrigerated and liquid nitrogen cooling systems described later. A schematic of the FACS mounted on the DSC Cell is shown in Figure 2. The FACS can be used for controlled cooling experiments, thermal cycling studies, and to improve sample turnaround time. It uses compressed air to cool the DSC cell. Stable baselines and linear heating and cooling rates can be achieved between ambient and 725 °C. A special version of the Quench Cooling Accessory is available for use with the FACS to speed cooling of the DSC cell to ambient temperatures.

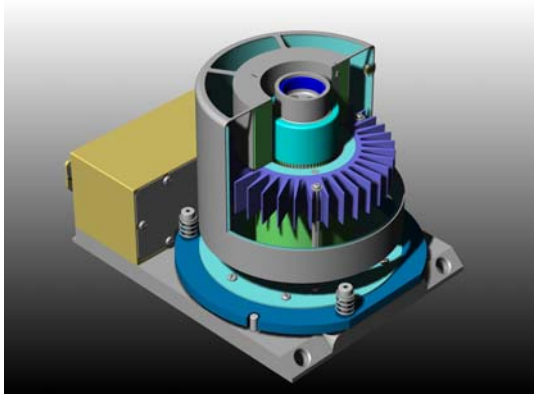


Figure 2. FACS Mounted on a Tzero DSC Cell

REFRIGERATED COOLING SYSTEM (RCS)

An RCS is often the cooling device of choice for trouble-free, unattended DSC / MDSC® operation over a broad temperature range. It is a compressor based refrigeration system to which a cooling head is attached via an insulated tube. The head mounts snugly over the Tzero DSC Cell and mates to the cooling flange (see Figure 3). As a sealed system requiring only electrical power, the RCS is highly cost effective compared to cooling systems based upon liquid nitrogen. TA Instruments offers two complementary models; the RCS90 and the new RCS40. Both use the same cooling head, which encases the Q DSC Cell and eliminates frosting issues typical of other designs. The user identifies the RCS to the Advantage software by check-box selection. Cooling rates depend upon the temperature range selected for the thermal experiment. Ballistic cooling between two temperatures is easily achieved using the RCS.

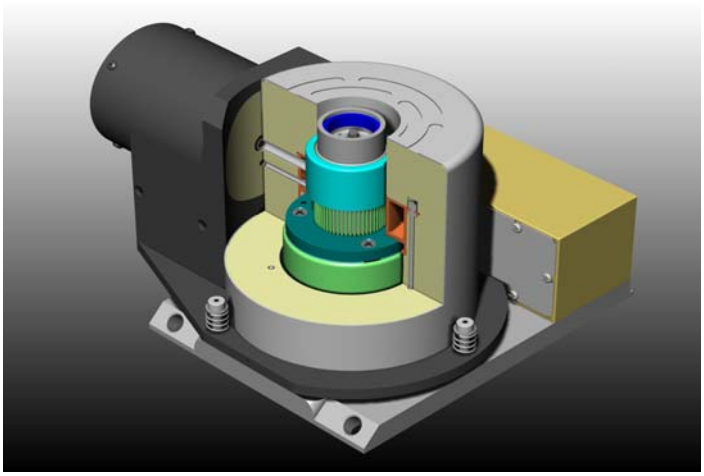


Figure 3. RCS Cooling Head Mounted on a Tzero DSC Cell

RCS90

The RCS90, shown coupled to a Q2000 DSC in Figure 4, is a two-stage refrigeration system, which permits DSC / MDSC operation from -90 °C to 550 °C. Its stable and reliable programmed cooling rate performance over various temperature ranges is summarized in Table 2. Rapid ballistic cooling is also a powerful feature. The RCS90 has the ability to cool the Tzero DSC Cell from 500 to 25 °C in around 7 minutes. It is compatible with all Q Series DSC modules, except when using the Pressure DSC Cell.



Figure 4. Q2000 / RCS90

Controlled Rate	To Lower Temperature
100°C/min	300°C
50°C/min	120°C
20°C/min	-20°C
10°C/min	-50°C
5°C/min	-75°C
2°C/min	-90°C

Table 2. RCS90 Controlled Cooling Rates, from 550°C (upper limit)
Performance may vary slightly, depending on laboratory conditions.

RCS40

The RCS40, shown with a Q200 DSC in Figure 5, is a compact, single-stage refrigeration system, which allows similar trouble-free DSC / MDSC operation from -40 °C to 400 °C. Table 3 shows cooling rate performance achievable to temperatures as low as –40 °C. Ballistic cooling from 400 to 25 °C can be accomplished in about 7 minutes and from ambient to –40 °C in 9 minutes. It shows remarkable stability in both ballistic and controlled cooling operation. The RCS40 is compatible with all second-generation Q Series DSC modules, except when using a Pressure DSC Cell.



Figure 5. A Q200 DSC with an RCS40

Controlled Rate	To Lower Temperature
65°C/min	250°C
50°C/min	175°C
20°C/min	40°C
10°C/min	0°C
5°C/min	-15°C
2°C/min	-40°C

Table 3. RCS40 Controlled Cooling Rates, from 400°C
Performance may vary slightly, depending on laboratory conditions.

Table 4 displays the impressive stabilization times to attain a set heating rate with a Q2000 / RCS40 system.

Heating Rate	Stabilization Time
2 ºC / min	9 sec
5 ºC / min	11 sec
10 ºC / min	12 sec
20 ºC / min	13 sec
50 ºC / min	14 sec

Table 4. RCS40 Heating Rate and Stabilization Times

A comparison of the RCS90 and RCS40 in controlled cooling performance from their maximum temperatures of use is shown in Table 5. It provides a convenient way to select the device best suited to a particular temperature range.

Controlled Cooling Rate	RCS90	RCS40
100 ºC / min	300 °C	NA
65 ºC / min	170 °C	250 °C
50 ºC / min	120 °C	175°C
20 ºC / min	-20 °C	40 °C
10 ºC / min	-50 °C	0 °C
5 ºC / min	-75 °C	-15 °C
2 ºC / min	-90 °C	-40 °C

Table 5. RCS90 and RCS40 Cooling Comparison

The RCS90 is well established for delivering controlled cooling conditions needed for excellent MDSC operation when coupled to a Q-Series MDSC module. The RCS40 is also a capable cooling device for use in high quality MDSC materials studies. Figure 6 shows that a modulation amplitude of +/- 1 ºC can be readily maintained from 0 to 400 ºC at normal periods from 60 to 100 seconds, and from 80 to 400 ºC at short modulation periods of 40 sec. The “green” area of the plot indicates the “safe” choice of conditions for successful MDSC work with the RCS40.

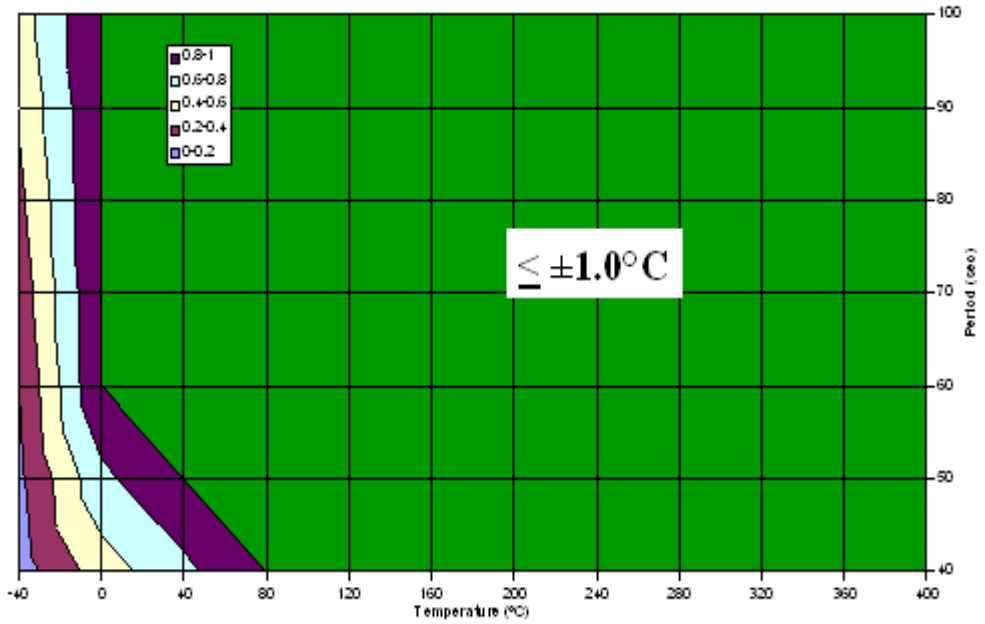


Figure 6. Achievable MDSC Amplitude (+/-ºC)

LIQUID NITROGEN COOLING SYSTEM (LNCS)

The LNCS provides the highest performance and greatest flexibility in cooling. It has the lowest operational temperature (to -180 °C), greatest cooling rate capacity (to 140 °C/min), and an upper temperature limit of 550 °C. It is ideal for isothermal crystallization studies. The LNCS uses liquid nitrogen efficiently, thus reducing operating costs. Its autofill capability allows the LNCS to be automatically refilled from a larger liquid nitrogen source for continuous DSC operation. The LNCS (Figure 7) is available for all Q Series DSC modules, except when using the Pressure DSC Cell.



Figure 7. Q2000 with LNCS

SUMMARY

TA Instruments has a very complete line of DSC cooling accessory to meet the needs of thermal analysts. They range from the low cost QCA designed for quench cooling to the more advanced programmed cooling capability from RCS and LNCS models. All provide excellent value in use in DSC materials analysis. Table 6 summarizes their relative capabilities and also provides, for TA Instruments' users, a listing of the signals available for use with each cooling device.

Cooling Device	Mode Quench Cool	Mode Program Cool	Cooling Medium	Temp Range	Q DSC Signal
LNCS		X	Liquid N2	-180 to 550 °C	T_{4P}, T_4, T_1
RCS90		X	Refrigerant Gas	-90 to 550 °C	T_{4P}, T_4, T_1
RCS40		X	Refrigerant Gas	-40 to 550 °C	T_{4P}, T_4, T_1
FACS	X	X	Compressed Air	Amb to 725 °C	T_{4P}, T_4, T_1
QCA	X		User specified	180 to 400 °C	T_1

Table 6. Comparison of TA Instruments Cooling Devices

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KEY WORDS
DSC, QCA, FACS, RCS, LNCS

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