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Thermal Analysis & Rheology

A Subsidiary of Waters Corporation

Refrigerated Cooling System

Manual Addendum

For use with the following:
DSC 10, 910, 912, 2010, 2910 and
2920 single & dual sample cells

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Notice

The material contained in this manual is believed adequate for the intended use of this instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. This publication is not a license to operate under or a recommendation to infringe upon any process patents.

Addendum:
Refrigerated Cooling System

Safety

♦ ***CAUTION:*** **Due to the size and weight of the cooling accessory, the RCS should always be lifted by two people to prevent injury.**

♦ ***CAUTION:*** **The cooling head assembly contains coated fiberfrax material. Excessive handling of this material could cause fiberfrax particles to be emitted into the air. See the MSDS sheet for safety measures to be observed when fiberfrax is used.**

NOTE: The RCS contains hazardous materials (*i.e.*, flammable refrigerants). Contact TA Instruments prior to packaging for shipment.

Water Condensation

The DSC and RCS surfaces get cold during use of the RCS. The cold surfaces can cause condensation and, in some cases, frost to build up. This condensation may drip to the floor. Provisions to keep the floor dry should be made. A slipping hazard may result if the condensation is not cleaned up.

Cold Surfaces

♦ ***CAUTION:*** **Some surfaces of the RCS and DSC system may get extremely cold during the use of the RCS for cooling experiments. This presents a danger to exposed skin coming in contact with and adhering to the cold surfaces. Use forceps or gloves when handling the lids, etc. while the RCS is being used.**

Temperature Range

! WARNING

Do <u>not</u> exceed 400°C with the RCS cooling head installed and the RCS power off. Serious damage and/or injury could occur.

♦ ***CAUTION:***

Do not exceed 400°C with the RCS system installed. If you wish to heat above 400°C, you must first remove the RCS cooling head and gaskets from the RCS-DSC cell, then replace the thermal radiation shield, cell cover, silver lid, and bell jar. Even after these steps have been taken, do not exceed 600°C with the RCS-DSC cell. Follow the directions in this manual for RCS usage.

Declaration of Conformity

In order to comply with the Electromagnetic Compatibility standards of the European Council Directive 89/336/EEC (EMC Directive) and Directive 73/23/EEC on safety, as amended by 93/68/EEC, the following specifications apply to the TA Instruments 230-Volt Refrigerated Cooling System:

- EN 55022, 1995 Limits and methods of measurement of radio disturbance characteristics of Information Technology Equipment, class B.

- EN 50082-1, 1992 Electromagnetic Compatibility - Generic immunity standard - Part 1: Residential, commercial, and light industry

  — IEC 801-2 Electrostatic Discharge 8 kV direct air discharge and indirect discharge
  — IEC 801-3 Radiated, radio frequency, electromagnetic field immunity test
  — IEC 801-4 Fast Transients common model 1 kV

- EN 61010-1, 1993 Safety requirements for electrical equipment for measurement, control and laboratory use, and Amendment 2, 1995.

Introducing the RCS

Description

The Refrigerated Cooling System (RCS) consists of a two-stage, cascade, vapor compression refrigeration system with an attached cooling head. The cooling head fits over the RCS-DSC cell for use with the DSC 10, 910, 912, 2010, 2910 and 2920 single and dual sample cells.

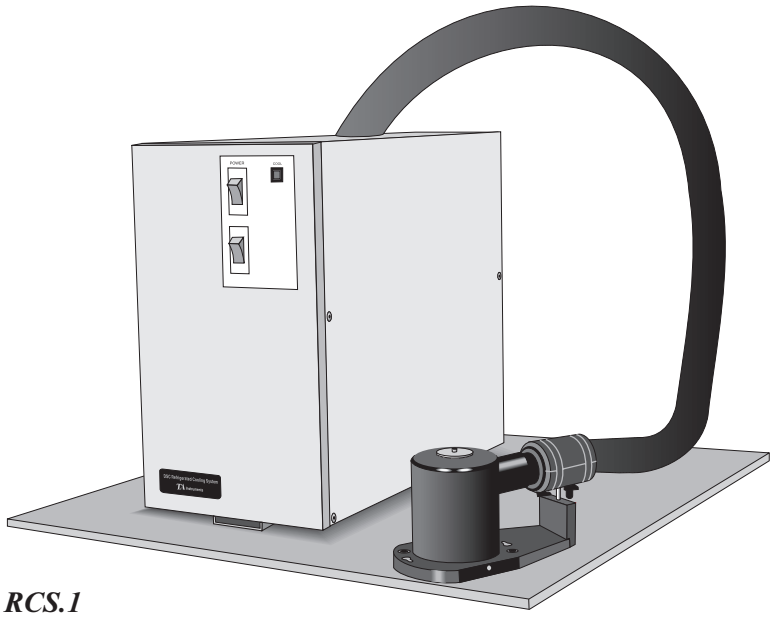

Figure RCS.1
Refrigerated Cooling System

The RCS can be used for experiments requiring cooling within an operating range of -70°C to 400°C. The maximum rate of cooling depends on the temperature range of your experiment. (See the "Specifications" section for maximum cooling rates.)

The housing of the cooling head is heated to prevent condensation and frost build-up on the exterior of the head. Purge gases are supplied to the cooling head from the refrigeration cabinet. The rear panel of the RCS cabinet has two separate gas connection ports: one for nitrogen (used with most experiments) and one for helium (used to enhance cooling performance for quicker cooling, but will hinder the cell's ability to achieve higher temperatures [> 250–300°C], due to its efficiency as a heat transfer medium.) You can choose between two purge gases by programming the switching of the gas supply via the Gas segment in your method. The cooling head purge gas acts as a heat transfer medium between the DSC cell and the heat exchanger in the cooling head and also reduces moisture accumulation in the cooling head.

The RCS has two modes of operation, which are selected by using a switch on the front panel. In the MANUAL mode, the RCS runs continuously. When you select the EVENT mode, the RCS can be turned on by **Event/On** selected from the **Control** menu in the DSC instrument control window. The RCS can be turned off by the Event segment in a method, by the method-end conditions selected, or by the DSC Autosampler sequence settings.

♦ ***CAUTION:*** **Rapid on/off cycling of the RCS will damage the refrigerant compressors.**

To achieve the highest cooling rate, use helium gas to purge the cooling head. Once the RCS is installed on your DSC cell, you can easily access your samples by removing the lids from the cooling head housing and the heat exchanger, exposing the lid of the DSC cell.

Specifications

Table RCS.1
Cooling Rate Specifications

Cooling Rate	Minimum Temperature
20°C/min	25°C
10°C/min	-25°C
5°C/min	-50°C

Cooling rates are obtained using 150 ml/min helium cooling head purge and 25 ml/min nitrogen DSC cell purge.

Cooling rate will usually be improved by increasing the helium flow or by using helium instead of nitrogen for the cell purge.

Table RCS.2
Accessory Specifications

Only values with tolerances or limits guaranteed data. Values without tolerances are for information only.

Dimensions Width Depth Height	 10 in. (25.4 cm) 20 in. (50.8 cm) 18.5 in. (47 cm)	
Weight	70 lbs (32 kg)	
Temperature Range	-70°C to 400°C	
Voltage Current Power on VA	120 V 7 A 840 VA	230 V 5 A 1100VA
Fuse Types	120-volt RCS 250 V, 3/4 amp Fast acting glass	230-volt RCS 250 V, 1/2 amp Fast acting glass
Refrigerants	Ethane, propane, propylene	

Preparing the DSC

In order to allow the RCS cooling head to mount correctly to the DSC cell and obtain the best cooling performance, a specialized DSC cell must be used with the RCS. This cell includes a flanged metal sleeve as seen in the illustration below.

If you have the *original* DSC cell currently installed on your instrument, you will need to prepare your instrument for RCS use by following the steps in this section to mount the RCS-DSC cell.

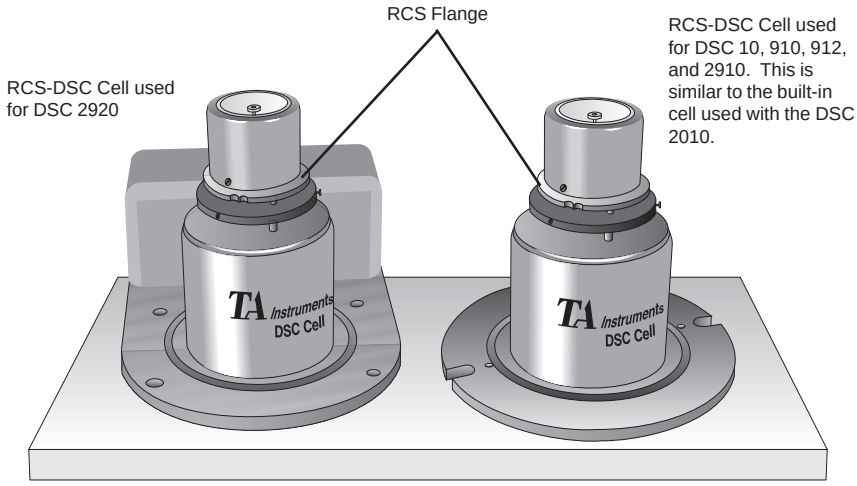

Figure RCS.2
Specialized RCS-DSC Cells

♦ ***CAUTION:*** **Allow the DSC to cool to ambient temperature before attempting to perform the steps below.**

1. Remove the bell jar, cell cover, and silver lid from the DSC cell currently installed on your instrument. Then remove the original cell from the instrument.

2. Remove the RCS-DSC cell from its shipping container.

3. Remove the three (3) screws that secure the radiation shield to the RCS-DSC cell assembly and remove the radiation shield, refer to Figure RCS.3.

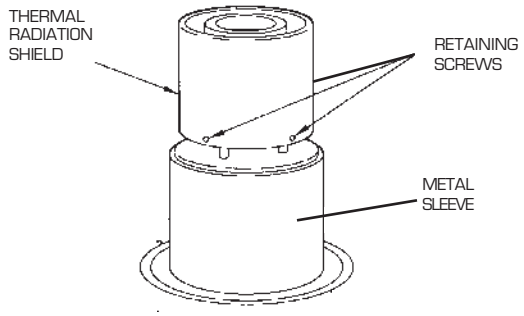

Figure RCS.3
Parts of the DSC Cell

4. Mount the RCS-DSC cell on your instrument as you would mount the original DSC cell. Refer to your *DSC Operator's Manual* for instructions, if needed.

You are now ready to install the RCS assembly.

Installing the RCS

When you receive the RCS, place it near the DSC instrument, leaving at least 10 cm (four inches) clearance on all sides of the refrigeration cabinet for air flow.

1. Install the large rubber gaskets over the RCS-DSC cell as required for your instrument type: three 3.2 mm (1/8-inch) thick gaskets are required for 2010 and 2920 instruments, and one (1) gasket is required for all other DSC instruments. An extra gasket, measuring 1.6 mm (1/16-inch) thick, is also supplied in your RCS kit. This is an optional gasket that can be used as needed to seat the cooling head properly over the cell as described in step 3.

2. Orient the refrigeration cabinet so that the refrigerant line forms a loose loop and the cooling head slides easily over the cell without twisting the refrigerant line.

♦ ***CAUTION:*** **Do not twist the refrigerant line so that the cooling head is forced down onto the cell. Readjust the position or the orientation of the refrigerant cabinet so that the cooling head slides easily over the cell.**

3. Install the cooling head, after the gaskets have been installed, as follows:

    a. Press down lightly on the cooling head and observe the gap between the bottom of the cooling head baseplate and the top of the gasket stack. If this gap is

approximately 1.0 mm (0.04 inch), continue the installation with step 3b. If the gap is appreciably less, lift the cooling head off the cell and replace the 3.2 mm (1/8-inch) top gasket with the supplied 1.6 mm (1/16-inch) gasket. Slide the RCS cooling head over the cell and continue with step 3b.

b. Press down on the cooling head assembly and look down into the top of the cooling head assembly to make sure that the inside diameter of the heat exchanger (the copper colored ring) is concentric with the inside diameter of the top of the RCS-DSC cell. If it is not, repeat steps 1 and 2.

4. Look inside the top of the cooling head assembly again after you have made sure that it meets the criteria in step 3b. There should be a small uniform gap (~1.0 mm [~0.040 inch]) between the underside of the copper heat exchanger and the top of the cell and between the bottom of the anti-frost sleeve and the top of the copper heat exchanger. If the gaps are too large, repeat steps 1, 2, and 3. See the arrows on Figure RCS.4, on the next page, for the location of the gaps.

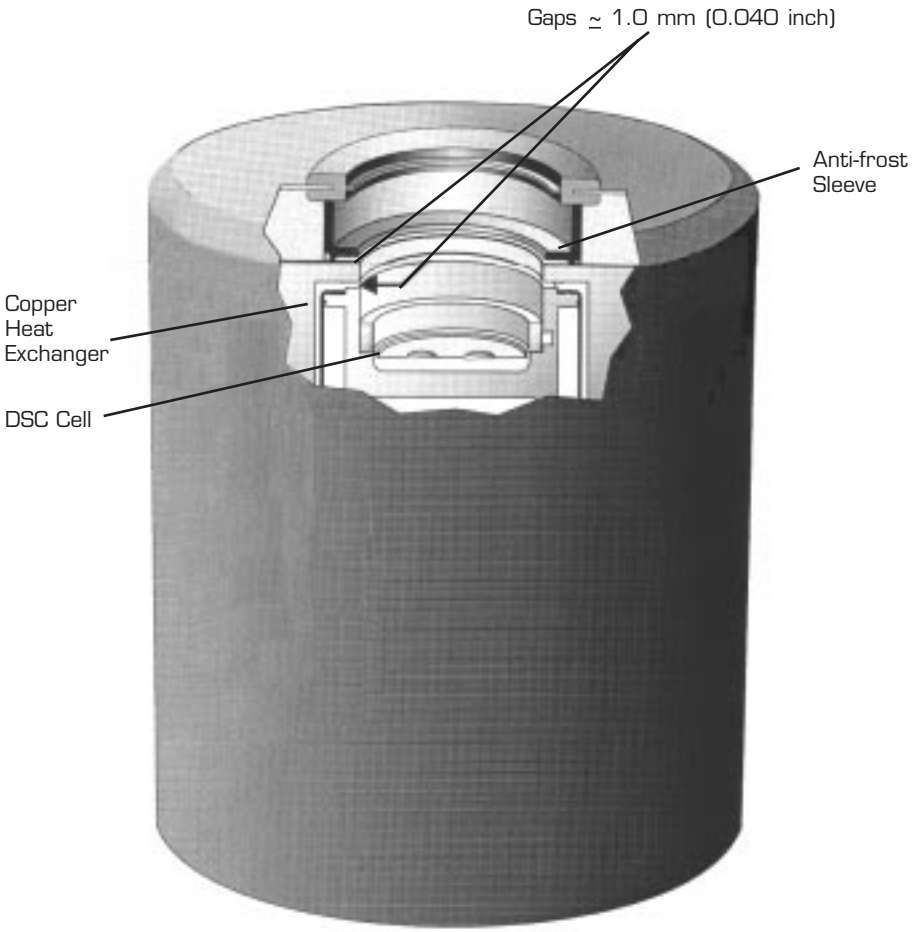

Figure RCS.4
Relationship of DSC Cell to Copper Heat Exchanger

5. Locate the screws (included in the RCS Accessory Kit) that are needed for your type of DSC instrument. Refer to Figure RCS.5 to decide which screws you will need.

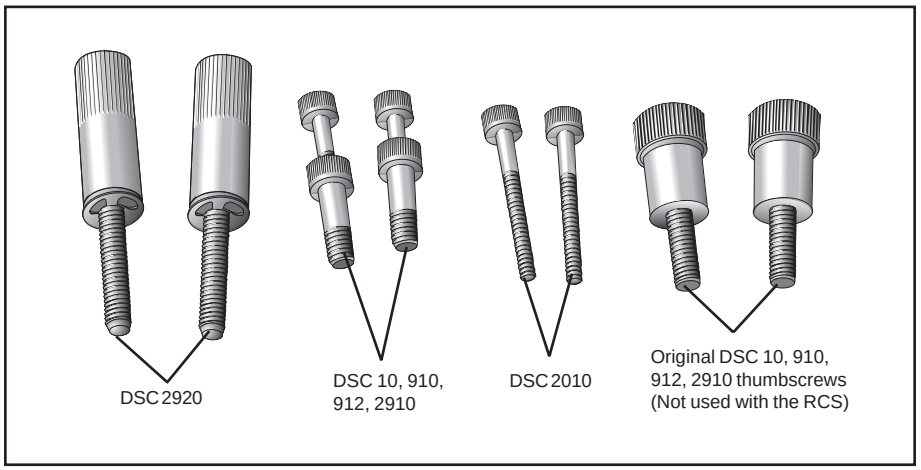

Figure RCS.5
Screws

6. While pressing down on the cooling head assembly, install two (2) screws through the baseplate of the cooling head assembly.

   - *DSC 2920 and 2910:* Tighten the thumbscrews evenly until they are hand-tight.

   - *DSC 10, 910, 912, 2010:* Tighten the screws using a hex key until they are just snug.

   The baseplate of the cooling head should be tight against the gasket(s) on the DSC cell. If it is not, check to be sure the rubber gaskets are installed correctly over the DSC cell as described in step 1 and repeat steps 3 and 4.

   See Figures RCS.6, 7 and 8 for correct installation of the RCS Assembly on the DSC 2010, 2910 and 2920.

7. Look down into the top of the cooling head assembly to make sure that the inside diameter of the heat exchanger (the copper colored ring) is concentric with the inside diameter of the top of the DSC cell. There should be a small uniform gap (~1.0 mm [0.040 inch]) between the underside of the copper heat exchanger and the top of the DSC cell. If proper alignment is not obtained, remove the thumbscrews and repeat steps 3 through 7.

8. Set the module mode on the controller to accommodate the use of mechanical cooling:

   Choose the RCS mode, then you can proceed with the rest of the installation.

NOTE: To run MDSC™ experiments with the DSC 2910, you must use the mode "DSC Modulated." Be careful not to exceed 400°C in this mode.

9. Place a cap over the vacuum and air cool ports on all DSC instruments to prevent moisture from entering through those ports.

   - *DSC 2010, 2910, and 2920:* Do not use compressed air for cooling when you are using the RCS. Most compressed air has a high moisture content and this will cause water and ice to condense around the cell. Laboratory quality dry air may be used for this purpose.

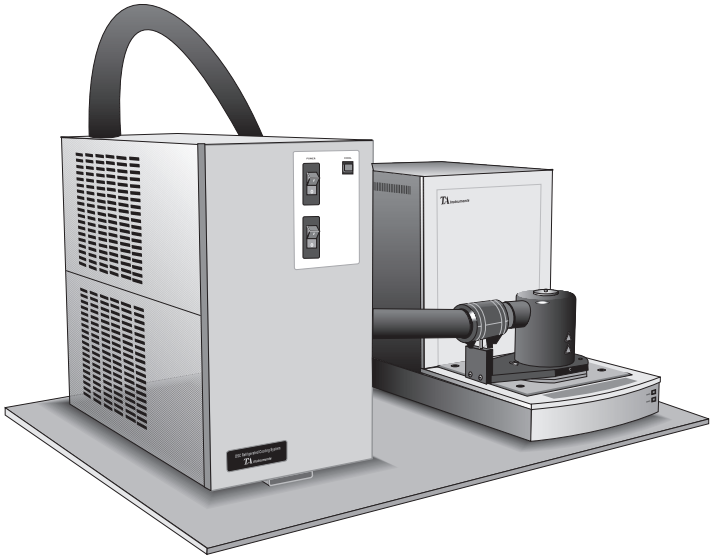

Figure RCS.6
RCS Mounted on the DSC 2010

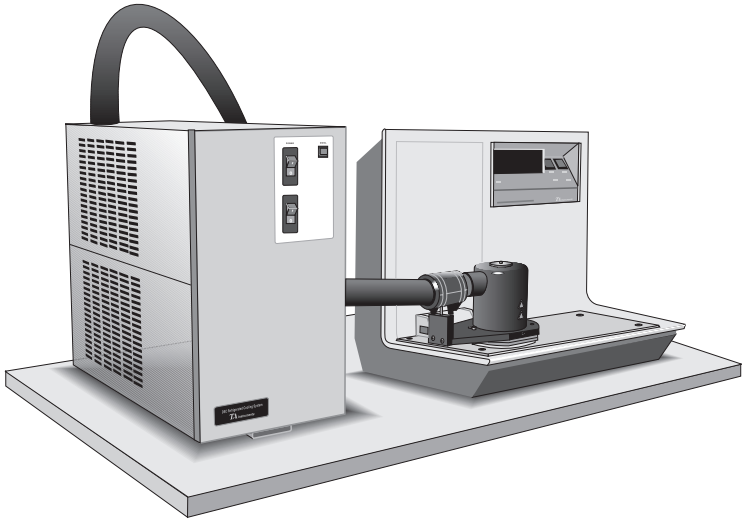

Figure RCS.7
RCS Mounted on the DSC 2910

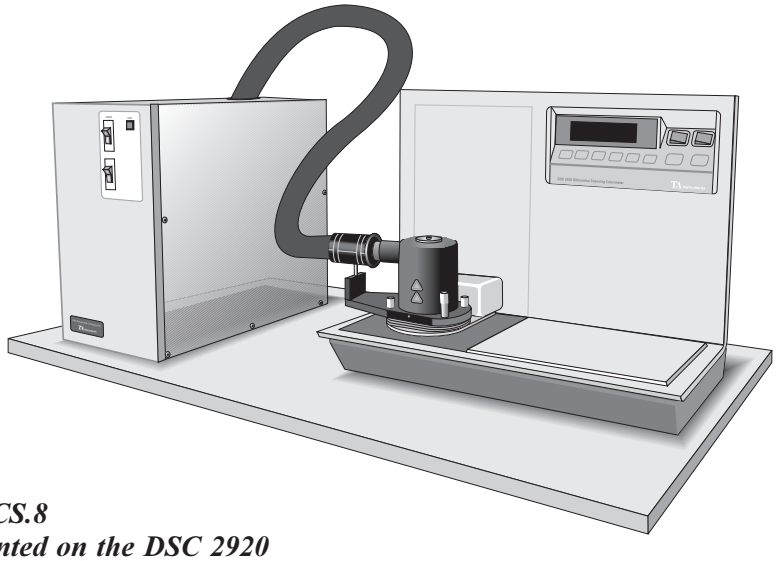

Figure RCS.8
RCS Mounted on the DSC 2920

Connecting Cables and Lines

The DSC Module Accessory Kit contains the cables that you will need to install before the RCS can be operated. Obtain the cables from the kit and follow these instructions to install them.

1. Turn the RCS cabinet around so that you can easily see the back panel. Locate the connections shown in Figure RCS.9.

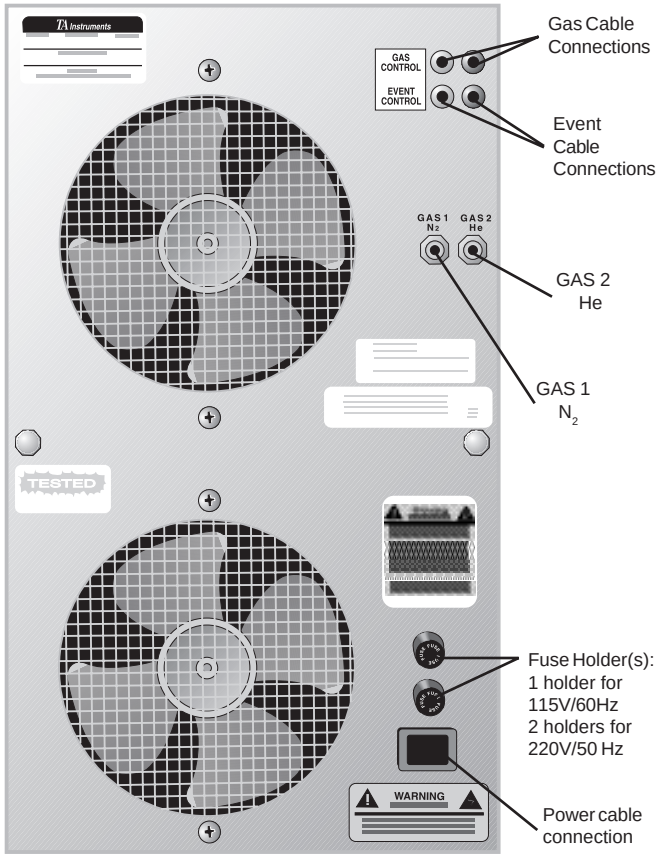

Figure RCS.9 RCS Cabinet Back Panel

2. Each purge gas should have a flowmeter installed in the supply line to the RCS cabinet. Connect the nitrogen and helium supply sources (gas lines not supplied) to the appropriate connections on the rear panel of the RCS cabinet (see Figure RCS.11).* The recommended flow range is 0–150 ml/min.

* Make sure that the gas line connections are leak free, as leaks will reduce the purge gas flow, with a resulting loss in cooling performance.

Recommendations:

- **Heating: Nitrogen at GAS 1.**

NOTE: If you are only using one gas, it is recommended that it be connected to GAS 1.

- The cooling head purge gas is used to keep a dry environment and to mediate heat exchange between the cooling head and the DSC cell. It does not eliminate the need to also use a purge gas for the DSC cell.

- The gas connection labeled GAS 1/N_2 is normally open and should be connected to the gas that is used the most. This gas will flow when the Gas method segment is in the 1 state or the RCS is turned off.

- The gas line connected to the connection labeled GAS 2/He will flow when the Gas segment is switched to state 2, and the RCS is turned on.

- **Cooling: Helium at GAS 2**. For maximum cooling performance, helium must be used for one of the cooling head purge gases. Please note that the maximum temperature that can be achieved, when using helium as the cooling head purge, is approximately 200°C. You can conserve helium by using nitrogen for the cooling head purge gas during the heating portion of the method or when cooling requirements are not so great.

3. Locate the gas cable in the DSC Module Accessory Kit.

4. Connect the gas cable to the terminals on the rear of the RCS cabinet and the other end to the DSC or Module Interface using the appropriate connector (see Figure RCS.10 on the next page).

5. Check that the power switch is turned off, then plug in the power cable—first to the RCS cabinet, then to the outlet.

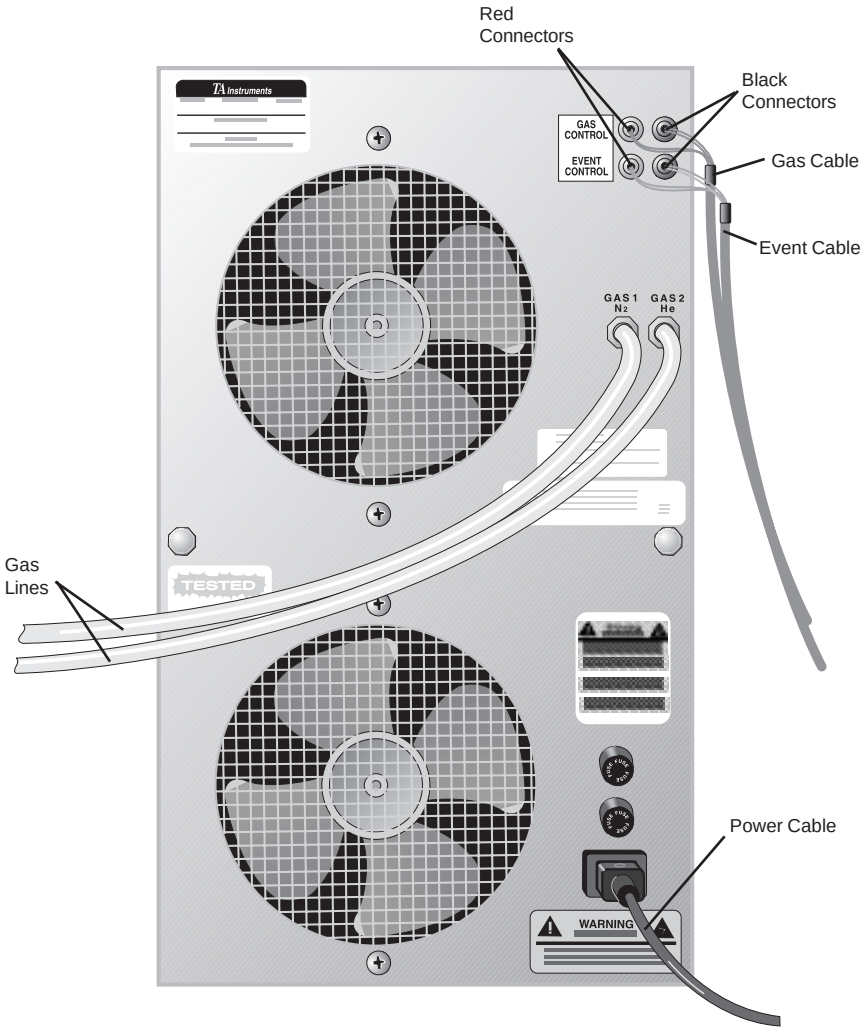

Figure RCS.10
Rear Panel of RCS
Cabinet with Connections

Using the RCS-DSC Cell Without the RCS

Once the RCS-DSC cell is installed on the instrument you can perform experiments using the Refrigerated Cooling System, as well as other types of experiments. However, there are some experiments that will require you to place your original DSC cell on the instrument.

LNCA

Please note that the LNCA can only be used if you modify the RCS-DSC cell. This is because the LNCA heat exchanger will interfere with the flanged metal sleeve.

- If you can remove the RCS-DSC cell from your instrument, it is best to take it off and place your original DSC cell back on the instrument to run the LNCA.

- If your cell is permanently attached, such as the DSC 2010, you will need to modify the RCS-DSC cell with insulation tape. Refer to the *DSC 2010 Operator's Manual* for directions, or the installation instruction sheet that comes with the insulation tape kit.

NOTE: Once you have installed the insulation tape on the RCS-DSC cell to use it with the LNCA, this becomes a permanent change. The cell cannot be reconfigured as a standard RCS-DSC cell.

DSC Cooling Can

The DSC Cooling Can may be used with the RCS-DSC cell, if you place the aluminum spacer (supplied with the RCS Accessory Kit) on top of the DSC cell before installing the cooling can.

Above Ambient Only (No Cooling Required)

RCS-DSC cells may be used for above ambient experiments that do not require the RCS:

- *Temperatures from ambient to 400°C:* The RCS-DSC cell may be used with the RCS cooling head in place and the RCS turned off. It is preferable, however, that the RCS head be removed prior to running above ambient experiments.

- *Temperatures above 400°C:* You must remove the RCS cooling head and gaskets from the cell. Then place the thermal radiation shield, cell cover, silver lid, and bell jar back on the cell before running experiments above 400°C.

NOTE:

The RCS-DSC cell may only be used up to 600°C.

!WARNING

Do not exceed 400°C with the RCS cooling head installed and the RCS power off. Serious damage and/or injury could occur.

♦ ***CAUTION:***

Do not exceed 400°C with the RCS system installed. If you wish to heat above 400°C, you must first remove the RCS cooling head and gaskets from the RCS-DSC cell, then replace the thermal radiation shield, cell cover, silver lid, and bell jar. Even after these steps have been taken, do not exceed 600°C with the RCS-DSC cell. Follow the directions in this manual for RCS usage.

Using the RCS

When in use the RCS runs continuously, at a fairly constant temperature, creating a low temperature heat sink surrounding the RCS-DSC cell. Heat flows from the cell to the heat sink, cooling the cell. There is no control over the temperature of the RCS.

Start-Up Time

The RCS will not begin to cool the RCS-DSC cell until the second stage compressor begins to run, this is indicated by the green light on the front panel of the refrigeration cabinet. When starting from room temperature, it will normally take approximately 15 to 20 minutes for the RCS to reach operating temperature after the green light goes on. Purge gas must be flowing through the DSC cell and the RCS cooling head during initial cool down to prevent condensation and subsequent frost build-up.

To operate the RCS, first set the gas flow rates as explained in the next section.

Setting the Flow Rates

1. Set the flow rate of the DSC cell purge to 25 ml/min.

NOTE: When using the RCS with the DSC 2920 Autosampler, the recommended DSC cell purge rates are 25 ml/min for nitrogen and 25 ml/min for helium.

2. Turn on the power switch on front of the RCS cabinet.

3. Set the flow rates of the RCS purge gases as follows:

   a. Set the flow rate for GAS 1 (usually nitrogen connected to gas connection GAS 1/N_2), using the flowmeter, to 150 ml/min.

   b. Switch to Gas 2 using **Control/Gas/2** from the Instrument Control menu.

   c. Set the flow rate for GAS 2 (usually helium connected to gas connection GAS 2/He), using the flowmeter, to 150 ml/min.

NOTE: For maximum cooling rates, helium should be used as the GAS 2 purge gas.

Setting Up an RCS Experiment

General Guidelines to Prevent Condensation

You can load samples into the DSC cell while the RCS is running. To prevent condensation in the DSC cell during sample loading, warm the DSC cell to a temperature above ambient using one of the following procedures.

- Start your thermal methods with an Initial temperature segment of 20–30°C. When the DSC cell is ready, as indicated by the READY message on the controller screen, load the sample and select **Resume** to move to the next method segment.

or

- Keep the DSC cell warm by inserting the Initial temperature method segment at the end of the previous thermal method. To start the new experiment, first select STOP, then START.

NOTE: Loading samples when the DSC cell is below ambient temperature will almost always cause condensation within the DSC cell, resulting in erroneous heat flow data.

Experimental Procedure

1. Remove the RCS outer lid, middle lid and the DSC cell silver lid.

2. Load your sample and reference pans, then replace the lids.

3. Set up the thermal method with the following guidelines (see example experiments on pages RCS-35 to 38):

   - During *cooling* segments, use helium to purge the RCS cooling head.

   - During *heating* segments, use nitrogen to purge the RCS cooling head.

   - Use the Gas segment to switch between nitrogen and helium during a thermal method. Switch the gas at the beginning of a heating or cooling method segment.

   - Use an Initial temperature segment at the end of each method to set the cell temperature to room temperature and minimize condensation while removing the sample and loading the next sample. (Note: Be sure to end the current experiment by pressing STOP, then you can begin the next experiment.)

NOTE: When using the RCS with the DSC 2920 Autosampler, air cool can be activated automatically at the end of the run to quicken the cell's return to ambient temperature. It is recommended that you use only laboratory quality dry air for this purpose to minimize moisture and frost condensation around the cell and prevent contamination of the cell by particulates and oil found in the compressed air.

4. Start the experiment.

Controlling the RCS
Using the Event Feature

The RCS can be controlled using one of the two switches found on the front panel—the MANUAL switch or the EVENT switch.

- When the switch on the front panel of the RCS is set to MANUAL, the RCS will operate in the standard mode and will remain ON at the end of a method.

- When the switch on the front panel of the RCS is set to EVENT, the instrument will use the event feature of the DSC.

To use the event feature follow the instructions found in this section.

1. Make sure that the RCS purge gas is flowing, then turn on the RCS power.

2. Turn the toggle switch on the front of the RCS panel to EVENT.

   This allows the RCS to be turned on when **Event On** is selected from instrument control program on the controller and to be turned off the RCS when **Event Off** is selected. The default setting for the Event function is to be automatically turned off at the end of a method. This feature will let you set up a long experiment to run unattended and the RCS will shut down automatically at the end of the experiment.

NOTE: The default setting for the Event feature cannot be changed when using the RMX or OS/2 instrument control software. If you want the RCS to remain on at the end of a run, you must set the toggle switch on the front panel of the RCS to MANUAL.

3. Select **Control/Event/On** from the instrument control program.

4. Allow 15 to 20 minutes for the temperature of the RCS to stabilize before you begin any experiments.

♦ ***CAUTION:*** **We do not recommend that you turn the RCS on and off rapidly after short experiments.**

Changing the Event Default Setting

When using the TA Instruments *Thermal Solutions/Advantage* software, you can change the default setting for the event feature for the DSC or the DSC Autosampler using one of the following procedures.

Event Control with the DSC

To control the event feature when you are using the DSC, follow these steps:

1. Switch the RCS to the EVENT mode.

2. Select the **Instrument Parameters** window (TS) or the **Post Test** window (Advantage) for the DSC instrument.

3. Check **Event (RCS) Unchanged** box in the Method-End Conditions.

When you perform these steps, the RCS will remain on at the end of an experiment. This allows you to perform a series of experiments and the RCS remains on at the end of each method. For the last experiment, the RCS can be turned off automatically at the end of the method by including an Event Off segment in the method or by unchecking the **Event (RCS) Unchanged** box before you start the last experiment. This procedure typically applies when you are not using the DSC Autosampler.

Event Control with the DSC Autosampler

To control the event feature when you are using the DSC Autosampler, follow these steps:

1. Switch the RCS to the EVENT mode.

2. Select the **Instrument Parameters** window (TS) or the **Post Test** window (Advantage) for the DSC Autosampler instrument.

3. Check the **Event (RCS) Unchanged** box in the Method-End Conditions so that the RCS will remain on after each method in the sequence.

4. *For Thermal Solutions:* Select **Autosampler/Settings** from the AutoDSC menu. Select the **Stop Page**, then check the **Event (RCS) Off** box, which controls the condition of the RCS at the end of the sequence.

   For Thermal Advantage: Select **User Preferences — Auto Sampler** Page from the AutoDSC menu. Check the **Event (RCS) Off** box, which controls the condition of the RCS at the end of the sequence.

Checking this box turns off the RCS compressor at the end of the Autosampler sequence. If **Event (RCS) Off** is not selected, then the RCS compressor remains on at the end of the sequence.

When you perform these steps, the RCS will remain on at the end of each method in the sequence in order to be used for the next method, but will turn off at the end of the Autosampler sequence. This allows you to run autosampler cooling methods using the RCS unattended and it will not continue to run when not needed.

Example RCS Experiments

DSC Heating Method

Method Segments	Purpose
1 Data Storage: OFF	Turns data collection off
2 Initial temperature*: 30.00°C	Nitrogen is default cooling head purge gas Minimizes condensation while loading the sample
3 Select Gas: 2	Switch to helium for rapid cooling
4 Ramp 10°C/min to -50°C	Cool to start temperature
5 Isothermal for 5 min	Temperature stabilization
6 Select Gas: 1	Switch to nitrogen for heating
7 Data Storage: ON	Turns data collection on
8 Ramp 10°C/min to 300.00°C	Heating ramp
9 Data Storage: OFF	Turns data collection off
10 Initial temperature: 30.00°C	Minimizes condensation while removing the sample or loading the next sample.

(continued on next page)

DSC Cooling Method

Method Segments	Purpose
1 Data Storage: OFF	Turns data collection off
2 Initial temperature*: 30.00°C	Nitrogen is default cooling head purge gas Minimizes condensation while loading the sample
3 Ramp 25°C/min to 300°C	Heating ramp
4 Isothermal for 5 min	Temperature stabilization
5 Select Gas: 2	Switch to helium for rapid cooling
6 Data Storage: ON	Turns data collection on
7 Ramp 10°C/min to -25.00°C	Cooling ramp
8 Data Storage: OFF	Turns data collection off
9 Select Gas: 2	Switch to nitrogen for heating
10 Initial temperature: 30.00°C	Minimizes condensation while removing the sample or loading the next sample.

NOTE:

* When using the Initial temperature segment at the beginning of an experiment, it is necessary for the operator to select RESUME under Instrument Control in order to go to the next segment in the method.

DSC Method to Dry the RCS

NOTE: To dry the RCS cooling head assembly, first turn off the RCS.

Method Segments	Purpose
1 Data Storage: OFF	Turns data collection off
2 Equilibrate at 200°C	Nitrogen is default cooling head purge gas Eliminates any condensation
3 Isothermal for 60.00 min	Brings heat exchanger to temperature
4 Initial temperature 30.00°C	Brings cell to room temperature

MDSC™ Method

Method Segments	Purpose
1 Data storage: Off	Nitrogen is default cooling head purge gas Save disk space
2 Initial temperature*: 30.00°C	Minimizes condensation while loading the sample
3 Select Gas: 2 (See NOTE on next page.)	Switch to helium for increased cooling efficiency
4 Ramp 10°C/min to -50.00°C	Cool to start temperature
5 Modulate ± 0.5°C for 60 sec	Start modulation
6 Isothermal for 5.00 min	Modulation stabilizes
7 Data storage: On	Start data collection
8 Ramp 5.00°C/min to 200.00°C	Ramp during modulation
9 Data storage: Off	Stop data collection
10 Select Gas: 1	Switch to nitrogen
11 Modulate ± 0.000°C for 60 sec	Turns off modulation
12 Initial temperature: 30.00°C	Minimizes condensation while removing the sample

* See note about Initial temperature segment on page RCS-36.

NOTE:

The maximum temperature that can be achieved, when using helium as the cooling head purge, is approximately 200°C. Use nitrogen from the beginning of the experiment, if it is necessary to exceed 200°C.

Troubleshooting Guide

The problems most often encountered when using the Refrigerated Cooling System are usually related to improper installation and setup. This section lists the type of problem and possible solutions to fix the situation.

Problem:

Poor baseline performance, including excessive noise, extraneous thermal events, and poor reproducibility.

Solution:

Check the following items:

1. Make sure that an RCS-DSC cell (not a standard DSC cell) has been installed on the instrument. The RCS cooling head can be installed on any DSC cell, but will not work due to improper alignment with the standard DSC cell.

2. Check the alignment of the RCS cooling head on the RCS-DSC cell to make sure that it is properly installed as described in this addendum.

3. Make sure that the correct thumbscrews are installed and tightened, and that the base-plate of the RCS cooling head is tight against the rubber gaskets.

4. Make sure there is no accumulation of ice within the cell or inside the cooling head. Purging the RCS-DSC cell and the RCS housing helps prevent the accumulation of ice. Follow these suggestions to correct this problem, if you suspect an ice accumulation:

   a. Check to make sure that the cell purge gas is connected, and that the flow rate is correct. Also check to make sure that the cooling gases are connected to the RCS, there are no leaks in the lines, and that the gas flows are correct.

   b. Check to make sure that the vacuum and air cool ports on the cell base have caps.

   c. If all of the gas connections and flow rates are found to be correct, the cell and RCS cooling head should be dried as follows to eliminate moisture:

      To dry the system, turn the RCS off, but maintain the flow of purge gas to the cell and cooling gas to the RCS. (Note that with the RCS turned off, the cooling gas connected to GAS 1 port will continue to flow.) Now execute a thermal method which will hold the cell at 200°C for one hour (see example drying method).

Problem:

Cooling rates are too low or cannot be maintained to a low enough temperature.

Solution:

Check the following items:

1. Make sure that the cooling gases are properly connected to the RCS cabinet, and that the flow rates are correct.

2. Use a high thermal conductivity gas (*e.g.,* helium) as the cell purge gas to achieve maximum cooling rates:

    a. Make sure that the high thermal conductivity gas is connected to the GAS 2 port on the RCS cabinet.

    b. Ensure that the thermal method includes a Gas 2 segment to switch to the high thermal conductivity gas before executing cooling segments.

    c. Try increasing the helium flow, this will usually improve your cooling rates.

3. Check the installation of the RCS cooling head on the cell to make sure that the heat exchanger is properly aligned and has the 1.0 mm (~0.40-inch gap).

4. Make sure that the correct thumbscrews are installed and tightened. Check the baseplate of the RCS cooling head to make sure it is tight against the rubber gaskets.

5. Make sure that the vacuum and air cool ports on the cell base have caps.

Problem:

Cooling light on the RCS does not illuminate.

Solution:

1. If you are beginning a thermal method with the RCS-DSC cell at an elevated temperature, the green cooling light on the front panel of the RCS cabinet may take longer than usual to illuminate. It may go on and off several times during cooling before finally remaining illuminated. This is normal and does not indicate a fault. It is occurring because the heat exchanger in the RCS cooling head is hot.

2. If the RCS is turned on while the RCS-DSC cell is being maintained at a high temperature, the cooling light may continue to cycle or may not illuminate. Reduce the cell temperature before starting the RCS. The RCS cannot start when running a thermal method that holds the cell at a high temperature.

3. If none of the situations above is in effect, and the green cooling light fails to illuminate, it indicates fault with the refrigeration system and the RCS unit must be returned for service.

Problem:

Specified minimum temperature cannot be reached.

Solution:

Check the following items:

1. Make sure that the cooling gases are properly connected to the RCS cabinet, and that the flow rates are correct.

2. Use a high thermal conductivity gas (*e.g.,* helium) as the cell purge to achieve maximum cooling rates:

    a. Make sure that the gas is connected to the GAS 2 port on the RCS cabinet.

    b. Ensure that the thermal method includes an Gas 2 segment to switch to the high thermal conductivity gas before executing cooling segments.

    c. Try increasing the helium flow, this will usually improve your cooling performance.

3. Check the installation of the RCS cooling head on the cell to make sure that the heat exchanger is properly aligned and has the 1.0 mm (~0.40-inch gap).

4. Make sure that the correct thumbscrews are installed and tightened. Check the baseplate of the RCS cooling head to make sure it is tight against the rubber gaskets.

5. Make sure all gas connections are free of leaks.

6. Make sure that the vacuum and air cool ports on the cell base have caps.

Problem:

Cannot achieve 400°C temperature.

Solution:

Make sure that nitrogen is being used by selecting Gas 1 (Nitrogen). The maximum temperature that can be achieved when using helium as the cooling head purge is approximately 200°C.

Problem:

Cannot equilibrate at a temperature (low or high).

Solution:

Use a Ramp segment in your method instead of the Equilibrate segment.

Problem:

Condensation forms on the top of the cooling head and/or on the inner stainless steel lid.

Solution:

Verify that a 1-mm (0.04-icnh) gas exists between the bottom of the anti-frost sleeve and the top of the copper heat exchanger. See Figure RCS.4 on page 16.

Parts List

Part Number	Description
990697.001	Cooling Accessory Gasket 3.2 mm (0.12-inch) thick
990697.002	Cooling Accessory Gasket 1.6 mm (0.06-inch) thick
991115.001	Middle Lid #2 (stainless steel)
990686.001	Outer Lid #3 (aluminum)
990674.001	Sockethead Cap Screw (Modified) - Bottom (DSC 10, 910, 912, 2910)
990674.002	Sockethead Cap Screw (Modified) (DSC 10, 910, 912, 2910)
205085.058	Sockethead Cap Screw (DSC 2010)
915033.901	Shoulder Thumbscrew with Retainer Ring (DSC 2920)
270305.001	Hex Key 4.8 mm (3/16 inch)
270406.001	Hex Key 4.0 mm (5/32 inch)
991118.001	Cooling Can Spacer
253827.000	Power Cord - 120 V/60 Hz
270469.001	Power Cord - 230 V/50 Hz
991123.001	Silver Lid #1 for RCS-DSC Cell
205225.020	Fuse, Fast Acting, Glass, 0.75 Amp/125 V (1 each required for 120 V/60 Hz)
205225.018	Fuse, Fast Acting, Glass, 0.50 Amp/250 V (2 each required for 230

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