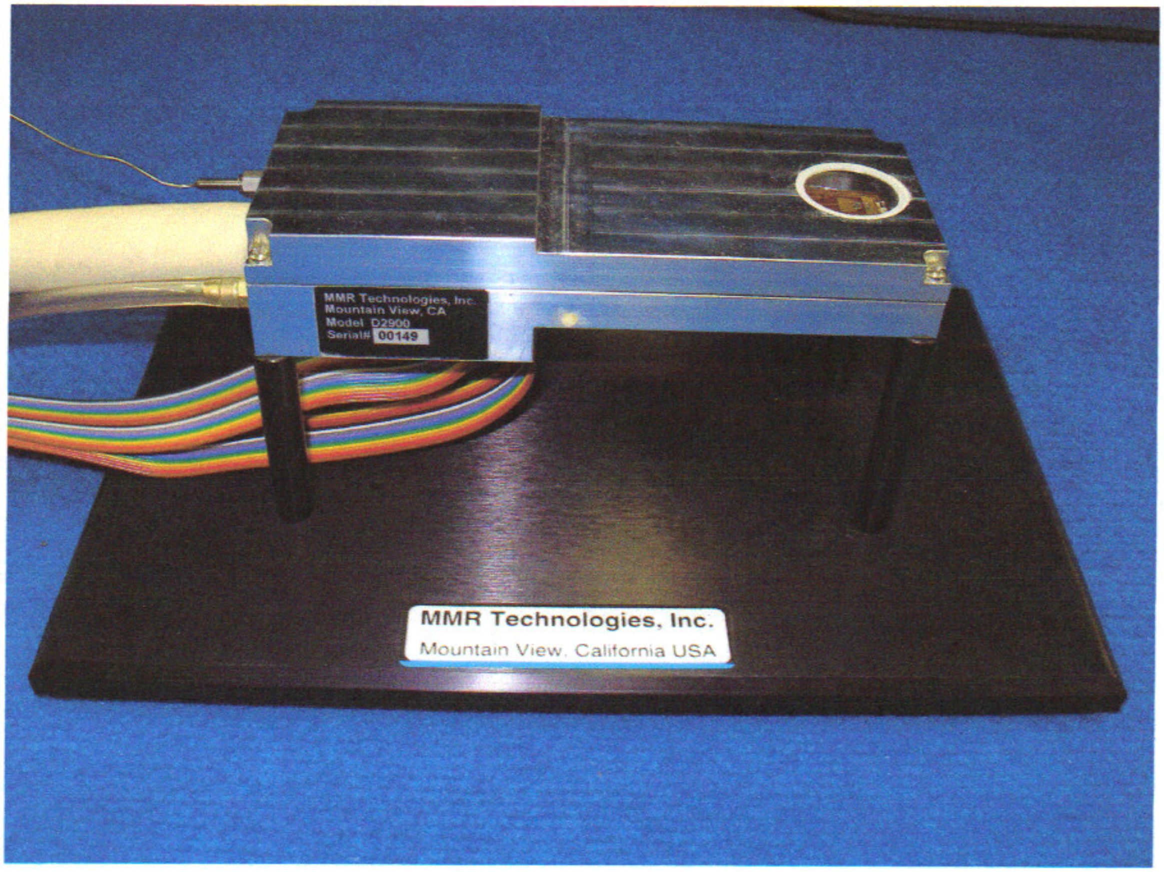
Variable Temperature Seebeck System
Dewar



USER'S MANUAL

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Warning

NEVER DISCONNECT CABLES TO, HANDLE OR WORK ON AN MMR REFRIGERATOR/HOTSTAGE WHEN IT IS CONNECTED TO THE K-20/SB-100 WHILE THE POWER TO THE K-20/SB-100 IS ON!

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REFRIGERATOR: GENERAL INFORMATION

1.1 GENERAL DESCRIPTION

This miniature cryogenic Joule-Thomson (J-T) Refrigerator cooling system is designed to make low temperature material characterization an inexpensive and simple operation.

These refrigerators are designed to cool small samples to about 80K within 15 minutes using nitrogen gas at 1800 psi input pressure. Once minimum temperature is achieved, 250mW of power can be dissipated indefinitely, with about a 4°K to 5°K increase in minimum temperature.

When this system is combined with the K-20 Programmable Temperature Controller, any temperature in the range of +300°C to -203°C may be selected as an operating temperature, provided the appropriate refrigerator or Wide Temperature Thermal Stage is used in the system.

1.2 PRINCIPLES OF OPERATION

When a gas such as nitrogen is allowed to expand through a porous plug or fine capillary tube at high pressure, the gas cools. This is known as the Joule-Thomson effect. The magnitude of the effect is small being about 0.1K/atm for nitrogen at ambient temperatures.

This figure can be magnified by allowing the expanded, cooled gas to pass through a countercurrent heat exchanger, precooling the incoming high pressure gas. This regenerative cooling continues until the gas liquefies or the temperature drop is limited by the heat load to the cooled end of the heat exchanger. For example, high pressure nitrogen at ambient temperature normally enters the heat exchanger at a pressure of 120 atmospheres. As it passes down the heat exchanger, it is cooled to 150K at nearly constant pressure. The gas now expands through a fine capillary.

This reduction of pressure results in cooling (the Joule-Thomson effect) and the formation of liquid. Heat dissipated by the device being cooled is absorbed by the liquid nitrogen as it vaporizes. The vapor then passes back up the heat exchanger, precooling the incoming gas. Finally, it vents at 1 atmosphere and at a temperature which is a little below ambient.

1.3 SYSTEM REQUIREMENTS

System operation requires the following items:

- 1800 psi nitrogen gas source (99.998% pure).

- N_2 regulator capable of 500 - 1800 psi delivery pressure.
- Vacuum pump capable of 5 millitorr.

1.4 SUGGESTED ACCESSORIES

Vacuum Grease:		Apiezon Typon "M" High Vacuum Grease
Thermal Grease:	For systems monitored by a Mass Spectrometer	Apiezon Type "M" High Vacuum Grease
	For all other cases	Dow Corning 340 Thermal Grease
Thermally Conductive Epoxy:		Ablestik Laboratories' Silver Filled Epoxy Ablebond #88-1

1.5 IMPORTANT SYSTEM INFORMATION

1.5.1 Refrigerators

The glass refrigerators are fragile, so careful handling is a must. When heating them to cure cements or epoxies, do this slowly, and do not exceed 50°C as such temperatures could damage the glass-to-aluminum seal at the base of the refrigerator. When operating a refrigerator in conjunction with the temperature controller, cold end temperatures up to 100°C may be set. The cold end heater is too small to heat the refrigerator base above 50°C.

1.5.2 Nitrogen Gas

To avoid clogging of the refrigerators by foreign gasses such as CO and H_2O which condense at temperatures above the minimum operating temperature of the refrigerator, use a nitrogen gas supply which is 99.998% pure or better.

DO NOT EXCEED 1800 PSI IN THIS SYSTEM!

1.5.3 Filters

MMR's filters clean the nitrogen gas supply, which should be at least 99.998% pure to start with, to the very high purity levels required for clog-free operation of the refrigerators. When the filter is not in use, the stainless steel end caps should be in place to hermetically seal the filter and to avoid contamination of the adsorbent.

1.5.4 Gas Lines

When shipped the two ends of each gas line are joined with a threaded barrel to form a hermetic seal and to keep internal contamination of the line to a minimum. When not in use, the gas line should be stored with the two ends joined by the threaded barrel. When the end of any gas line is disconnected, a stainless steel end cap and threaded barrel should be used to hermetically seal the gas line.The shorter the gas line downstream of the filter, the less chance there is of contaminating the rest of the system.

Do not over tighten the connector nuts. Overtightening can damage the connections. Also, avoid making sharp bends in the tubing. Sharp bends may block gas flow, and will eventually weaken the tubing.

1.5.5 Vacuum Chamber

Do not use any sharp objects (fine tip tweezers, razors blades, etc.) on the sealing surfaces of the vacuum chamber. Scratches will cause the O-ring to leak.

DEWAR SYSTEM ASSEMBLY

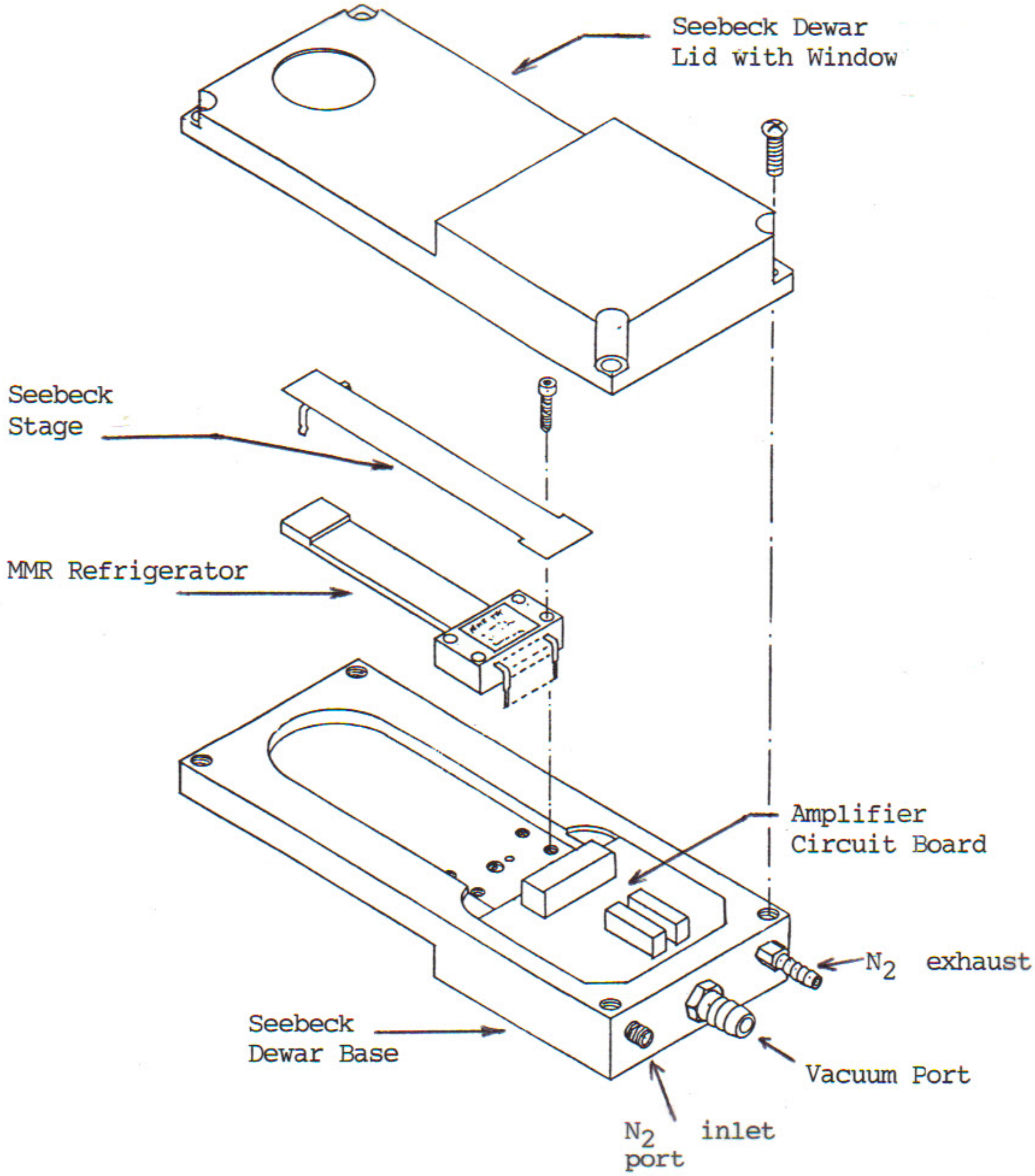

Figure 1. Seebeck System Dewar - Top Side

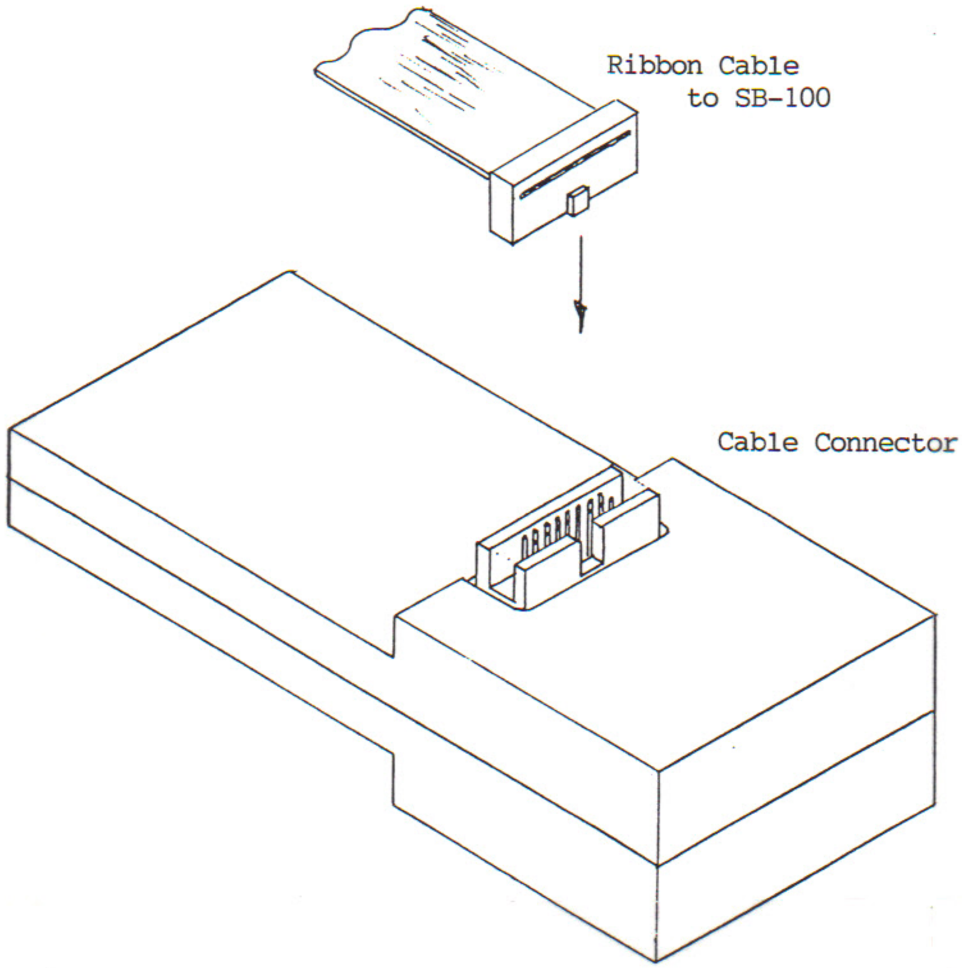

Figure 2. Seebeck System Dewar - Underside

2.1 INITIAL SET UP

1. Recharge N_2 gas filter if required - filter contents should be changed about every third tank of nitrogen (see Section 2.3, Filter Refilling Instructions).

2. Connect a vacuum hose between the vacuum port and vacuum pump. The shorter the hose the better.

3. Connect either the 3' or 5' stainless steel gas line between the N_2 gas regulator and the INTAKE end of the filter. Connector nuts need only be finger tight. If this is not sufficient, tighten only an additional 1/4 turn with a small wrench.

4. Connect the 6" gas line between the filter and N_2 intake port. Again, connector nuts need only be finger tight.

5. Connect a convenient length of Tygon tubing between the N_2 exhaust port and flow meter to monitor the gas flow rate.

6. Make all electrical connections between the user's instruments and the refrigerator harness. Plug the refrigerator harness into the 20 pin connector on the vacuum chamber base.

7. Before mounting the refrigerator, run 500 psi nitrogen through the system for about 30 seconds to purge the lines of any moisture that may have collected during nonuse.

8. Clean off any dust or particles on the mini O-ring and apply a very thin film of vacuum grease to it. Position it in the depression around the N_2 intake hole.

2.2 REFRIGERATOR PREPARATION

9. Clean off any dust or particles on the exposed surface of the O-ring in the refrigerator base and apply a thin layer of vacuum grease to it.

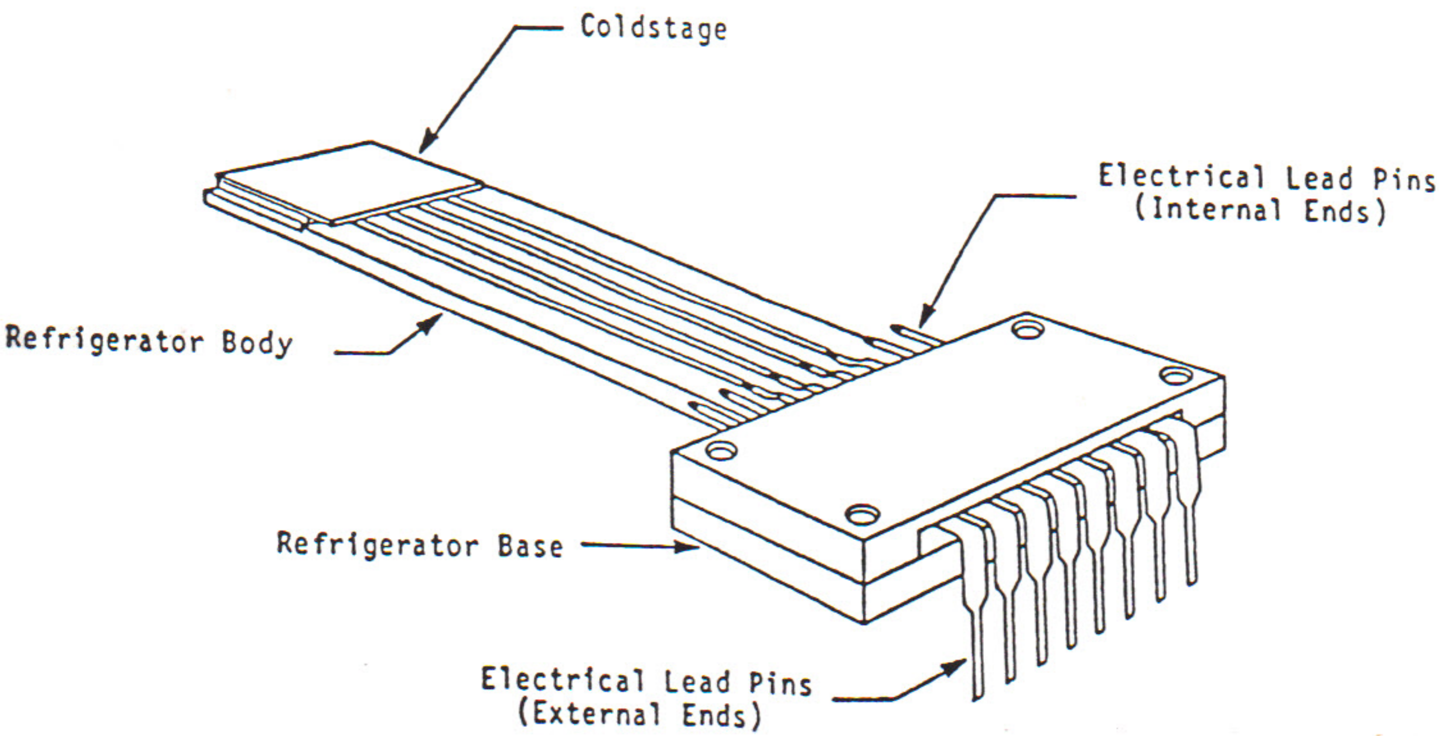

Figure 3. Seebeck Refrigerator

2.3 FINAL ASSEMBLY

10. Position the refrigerator base over the N_2 intake and exhaust holes, being careful to align the electrical lead pins (external ends) with the 8 pin holes (in the DIP socket) closest to the N_2 intake and exhaust holes. Be sure all 8 lead pins are fully inserted into the pin holes.

11. Secure the refrigerator base to the vacuum chamber base with the four screws provided by partially tightening each screw about 1/4 turn in sequence until the refrigerator base is flush and snug with the surface of the vacuum chamber base. Do not overtighten. ALWAYS USE ALL FOUR SCREWS.

12. Mount your device or specimen onto the refrigerator coldstage (see Section 4.2, Thermal Contact) using thermal grease or thermally conductive epoxy (see Section 1.4, Suggested Accessories). Be sure there is sufficient clearance between the mounted device and the vacuum chamber lid ceiling. Coldstage-to-ceiling lid distance is about 3mm.

13. Clean off any dust or particles on the captured O-ring in the vacuum chamber lid and apply a thin layer of vacuum grease to it. Secure the lid to the base with the four screws provided. Be sure the device on the coldstage does not contact the chamber lid ceiling. The lid is reversible, that is, it may be mounted so that the window is either over the coldstage or over the refrigerator base.

14. **Important** To prevent channeling of the incoming gas through the filter contents, the filter should be positioned VERTICALLY, with the INTAKE end up, during system operation.

2.4 FILTER REFILLING INSTRUCTIONS

The procedure for refilling the filter is described below. It should be completed quickly because the adsorbent begins to degrade as soon as it is exposed to the atmosphere. Save the stainless capnuts originally supplied at the time of shipment of the end fittings of the filter. Reinstall these capnuts whenever the filter is removed for the system set-up. This will prevent needless contamination of the adsorbent, and help protect the threads of the fittings. Refill the filter as follows:

1. Secure filter in a vertical position with the end labeled INTAKE up.

2. Remove the fitting at the INTAKE end and check the condition of the O-ring . Replace the O-ring if it is damaged, (MMR #A910030-000)

3. Discard the expended contents of the filter.

4. Open Replacement Pack #1 and pour its contents into the filter.

5. Lightly tap the filter to settle the contents.

6. Open Replacement Pack #2 and begin to pour its contents into the filter. (The contents of Replacement Pack #2 are more than enough to refill the filter.) Stop the filling procedure when the level of the contents is at the bottom of the threads. Alternately tap and refill the filter until this level is maintained.

7. Reinstall the end fitting, being sure that the O-ring is properly seated in place. The end fitting is properly installed when it bottoms out against the filter barrel - finger tightness will usually do this.

8. Purge the filter for about 3 minutes with 500 psi gas before using it in the refrigerator system. To purge the filter, run a gas line from the nitrogen gas source to the fitting at the INTAKE end of the filter, and attach a gas line to the fitting opposite the INTAKE end to prevent the teflon insert in the fitting from being dislodged by the flow of gas.

NOTE: The direction of the gas flow through the filter contents is important. When the filter is properly filled, as described above, the incoming gas will first pass through a sieve from Pouch #2 (4A) which will remove water vapor, and then through a sieve from Pouch #1 (13X) which will remove hydrocarbons. If this order is reversed, hydrocarbons caught by sieve 13X may be displaced by incoming water vapor and exhausted out the filter and into the refrigerator system.

SYSTEM OPERATION

1. Run 500 psi nitrogen through the system for about 30 seconds to purge it of any moisture that may have collected during nonuse.

2. Turn on the vacuum pump and allow enough time for the vacuum level to reach 10 millitorr before turning on the high pressure nitrogen. (See Section 4.3, Vacuum).

3. Turn on the high pressure nitrogen (1800 psi MAXIMUM - see Section 4.1, Gas Pressure) and allow the refrigerator to reach minimum temperature before turning on power to your device on the coldstage.

4. Turn on power to the device, let temperature stabilize, and conduct experiments.

5. After experiment is finished, turn off the N_2 gas and the vacuum pump. If the refrigerator has been operating at a very low temperature, minimize thermal shock by allowing the refrigerator to warm up for a few minutes before venting the vacuum chamber. Be aware of the temperature of the refrigerator and mounted device before touching them.

SYSTEM PERFORMANCE VARIABLES

These refrigerators are designed to cool small samples or devices, not dissipating any power, to about 80K within 15 minutes using nitrogen gas at 1800 psi input pressure. Once minimum temperature is achieved, 250mW of power can be dissipated indefinitely, with about a 4°K to 5°K increase in minimum temperature.

There are a number of variables that affect the cool down rate, minimum temperature, and refrigeration capacity:

4.1 GAS PRESSURE

As the gas pressure is increased, both the flow rate through the refrigerator and the effectiveness of the Joule-Thomson cycle increase. Hence, the cool-down rate will increase as the pressure is raised. We recommend using the maximum input pressure of 1800 psi for the initial cool-down. Once minimum temperature is achieved, the gas pressure can be reduced to match the experiment. This will conserve gas and, consequently, increase the useful life of a cylinder of nitrogen. Reducing the flow in this way will also slightly reduce the temperature of the coldstage.

4.2 THERMAL CONTACT

It is essential to have good thermal contact between the mounting pad and the specimen or device being cooled. The thermal resistance between the mounting pad and a device, properly mounted on the coldstage, is about 60mK/mW, but this will significantly increase with poor thermal contact. A thin layer of thermally conductive grease or epoxy should be used (see Section 1.4, Suggested Accessories). When heating the refrigerators to cure cements and epoxies, do not exceed 50°C.

4.3 VACUUM

The heat leak to the refrigerator, due to the thermal conduction of the residual air in the vacuum chamber, must be kept to a minimum. MMR recommends the use of a vacuum pump capable of a vacuum level of 5 millitorr or better, typically this requires a vacuum pump with a speed of 90 liters per minute. This will allow the vacuum level of the refrigerator to reach the recommended level of 10 millitorr or better for proper refrigerator performance, pump down time is normally a few minutes. A thin film of vacuum grease (see "Suggested Accessories", page 2) on all O-rings will help prevent leaks.

TROUBLESHOOTING GUIDE

Symptoms*	**Possible Causes**
	Once a cause has been determined, refer to the appropriate number under the "Possible Solutions", starting on the next page.
No cooling at all	- Poor thermal bond, #3 - No gas flow, #6 - Inadequate gas pressure, #7 - Excessive heat dissipation, #8
Partial cool-down, -30°C to -100°C, and then warm up	- Clogging, #1 - Poor thermal bond, #3 - Poor vacuum, #4
Slow cool-down:	- Partial clogging, #2 - Poor thermal bond, #3 - Large heat capacity of device, #5 - Inadequate gas pressure, #7 - Excessive heat dissipation, #8 - Cracked or broken refrigerator, #9 - Restriction in gas exhaust line, #10
Broken refrigerator:	- Replace refrigerator (see precautions under "Cracked or Broken Refrigerators", #9).
Connector leak:	- Connector leak, #11
Broken tubing:	- Replace tubing (available exclusively through MMR and its distributors.)

*See "System Performance Variables" in this User's Manual, for a discussion of the various factors influencing the performance of MMR's refrigerators.

Possible Solutions

1. Clogging

The frequency of clogging is a function of both the refrigerator's cool-down rate and the purity of the gas being used. Once the refrigerator cools down, clogging is rarely a problem. If clogging occurs, allow the refrigerator to warm up to room temperature, purge it with 500 psi gas for 30 seconds, then initiate the cool-down cycle again. If clogging persists, one of the following is true:

a. If the cool-down rate is slow, taking over 15 minutes to reach minimum temperature, then the underlying problem is probably not gas purity, but rather one of the following discussed below in #'s 4, 6, 7, 9, 10, 11, or 12.

b. If the cool-down rate is adequate, then the underlying problem is one of the following:

(1) Impurities have collected in the system downstream of the filter during storage and assembly. Remove the refrigerator and the mini O-ring and purge the system with 500 psi nitrogen gas for 30 seconds. Replace the refrigerator and initiate the cool-down cycle again.

(2) Bad gas supply - use at least 99.998% pure nitrogen from a reputable supplier.

(3) Exhausted filter - replace filter contents after every three cylinders, or as needed. Always cap the filter, the gas lines and the refrigerator when the system is not in use.

2. Partial Clogging

Partial clogging may occur for the same reasons discussed in Clogging, #1 above. This will retard the cool-down rate of therefrigerator. However, by itself, partial clogging will rarely keep the refrigerators from reaching minimum temperature.

3. Poor Thermal Bond

A poor thermal bond between the device being cooled and the refrigerator will cause the temperature of the device to lag that of the refrigerator during cool-down. Also, a poor thermal bond will significantly increase the temperature gradient between the device and the refrigerator once minimum temperature is reached.

To insure a good thermal bond, use as thin a layer as possible of thermally conductive grease or epoxy for mounting the device on the refrigerator. Recommendations for a suitable grease or epoxy are provided under "Suggested Accessories", Section 1.4.

4. Poor Vacuum

5 millitorr or better is required in the vacuum chamber for the refrigerator to operate properly. Maintain the shortest length of tubing possible between the vacuum chamber and the vacuum pump. Vacuum problems may arise from:

a. Dirt or lack of vacuum grease on one of the three O-rings in the system.
b. Excessive moisture in the system.
c. Leak in the vacuum line leading to the chamber.
d. Inadequate or damaged vacuum pump (check oil level in pump).
e. Excessive outgassing of specimen or bonding epoxy or grease used on refrigerator.
f. Cracked refrigerator (see #11, below).

5. Large Heat Capacity of Sample or Device

The heat capacity of the device may exceed the refrigerator's ability to cool it in a reasonable time. Reduce the size of the device or sample.

6. No Gas Flow

a. Is gas turned on?
b. Check for a kink, break, or leak in the gas supply line.

7. Inadequate Gas Pressure

MMR's refrigerators require 1800 psi gas to cool down properly. However, once minimum temperature has been reached, lower pressures, down to 1200 psi, can be used in some applications.

8. Excessive Heat Dissipation

MMR refrigerators have refrigeration of approximately 250 milliwatts when they are at a temperature of 85°K. Their capacities are less than this during the cool-down cycle and when the input gas pressure is below 1800 psi.

9. Cracked or Broken Refrigerator

MMR refrigerators are glass and should be handled with care. If a crack is detected in a refrigerator, DO NOT use it.

Observe the following additional precautions to avoid damage to the refrigerators:

a. DO NOT RAISE INPUT PRESSURE ABOVE 1800 PSI.
b. AVOID RESTRICTIONS IN OUTFLOW LINE.
c. ALLOW FOR SUFFICIENT CLEARANCE BETWEEN THE DEVICE BEING COOLED, ONCE IT IS MOUNTED ON THE REFRIGERATOR, AND THE VACUUM CHAMBER
d. USE ALL FOUR SCREWS IN SECURING THE REFRIGERATOR TO THE VACUUM CHAMBER BASE.

10. Restriction in Gas Exhaust Line

As noted above, restrictions in the gas exhaust line may cause irreparable damage to the refrigerator if the back pressure in the refrigerator's outflow channel exceeds 60 psi. Back pressures below this level may not cause damage, yet will impair the operating performance of the refrigerators.

11. Connector Leak

MMR gas line connectors are designed to provide quick and easy assembly of the refrigeration systems and also allow for safe operation at high gas pressures. Use finger-tight assembly initially. If a leak is detected, use a small wrench to turn the nut only an additional 1/4 turn.

If an audible leak persists, it may be due to a missing teflon washer or due to some dirt on the teflon washer inside the threaded barrel in the base of the vacuum chamber. In this case, carefully wipe the dirt off the surface of the teflon washer with a flat toothpick or other non-scratching material which can be inserted into this area.