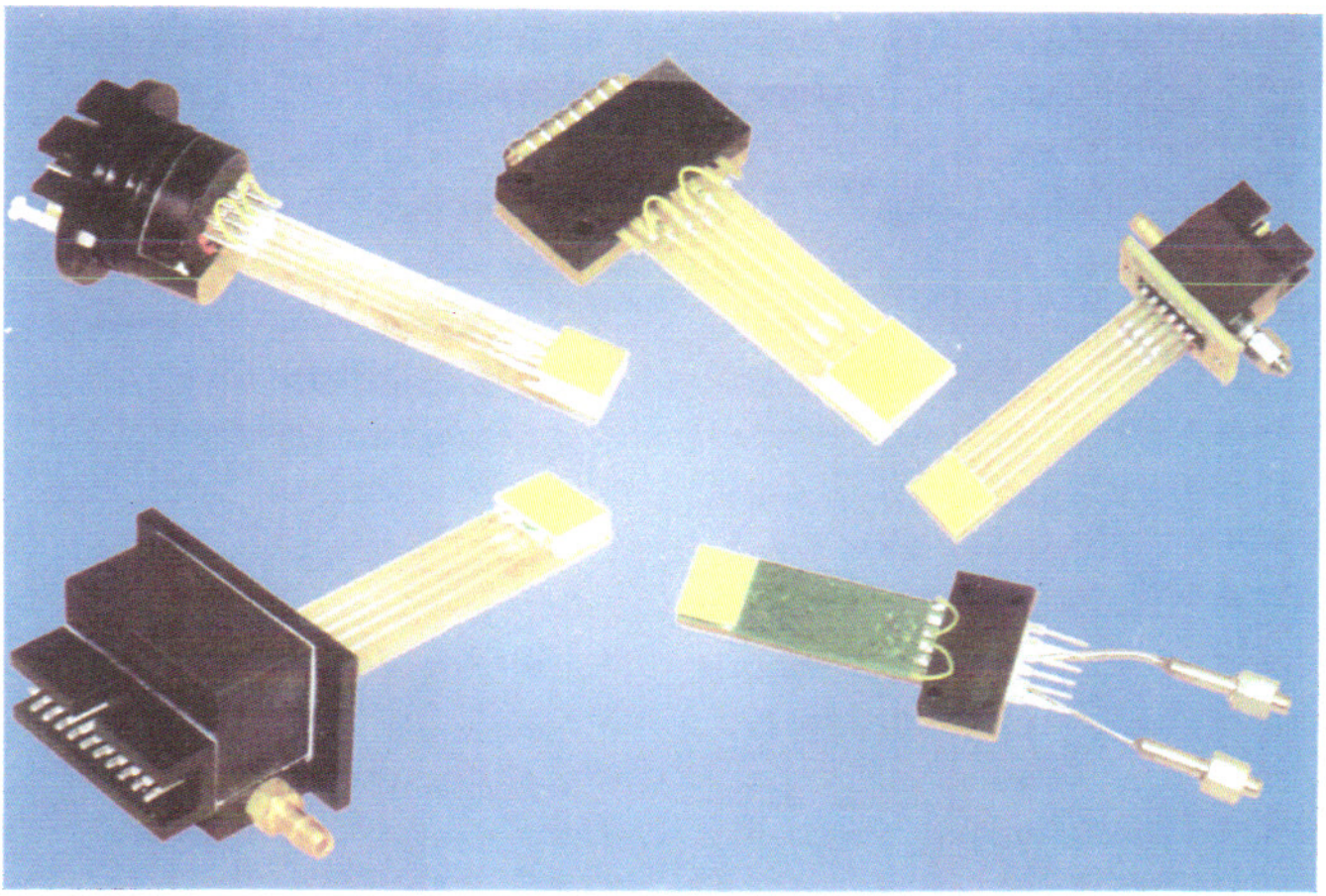
Operation of
Joule-Thomson Refrigerator



USER'S MANUAL

MMR Technologies, Inc.
1400 North Shoreline Blvd., Suite A5
Mountain View, CA 94043
(650) 962-9620
www.mmr-tech.com

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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/H-50 WHILE THE POWER TO THE K-20/SB-100/H-50 IS ON!

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SAFETY PRECAUTIONS

HANDLE THE REFRIGERATOR WITH CARE - it is made of glass. When not in use, store the refrigerators in the foam-lined box provided or in the system vacuum chamber (under vacuum, if possible).

CHECK REFRIGERATOR EXHAUST TUBING periodically for constrictions in the line. Back pressure build-up in the line will burst the refrigerator.

NEVER HEAT THE REFRIGERATOR ABOVE 50 °C except when operating it in conjunction with the temperature controller. Preparation and curing of samples on the Cold Stage can be done using the controller to achieve and maintain needed temperatures. Do not use heat guns or hot plates.

DO NOT USE PRESSURIZED OXYGEN in this system.

DO NOT OVERTIGHTEN CONNECTIONS.

DO NOT MAKE SHARP BENDS in the high pressure gas line tubing.

READ THE USER'S MANUAL thoroughly before using this refrigerator in any MMR system.

USE THE REFRIGERATOR ONLY IN A VACUUM. Use Vacuum Chamber supplied by MMR.

WEAR SAFETY GLASSES when handling or operating the refrigerator.

WARNING!

DO NOT ATTEMPT TO OPERATE A VACUUM-ASSISTED JOULE-THOMSON REFRIGERATOR UNTIL YOU HAVE CAREFULLY READ THESE INSTRUCTIONS.

DO NOT CONNECT OR LEAVE THE REFRIGERATOR EXHAUST PORT CONNECTED TO A VACUUM PUMP WHEN THE PUMP IS TURNED OFF! If gas is flowing through the refrigerator when the pump is turned off, gas pressure may build up inside the refrigerator and the refrigerator may burst.

> **Note:** Operation of vacuum assisted Joule-Thomson refrigerators in an MMR system requires two vacuum pumps: one to evacuate the refrigerator chamber and one to provide vacuum assist at the refrigerator exhaust port.

SECTION 1. VACUUM PUMP REQUIREMENTS FOR VACUUM ASSIST AT REFRIGERATOR EXHAUST PORT

The vacuum pumps used on the exhaust port of the refrigerator must be capable of sustaining a gas flow of 0.1 cubic feet per minute (6 cubic feet per hour) while maintaining a vacuum pressure of less than 100 mm Hg (100 torr).

> **Note:** The vacuum pump should be of a type which allows gas to flow through the pump, i.e. into the pump at the vacuum intake port and out of the pump at the vacuum exhaust port when the pump is turned off. If a pump of the type described above is not used, then a 1 psi pressure relief valve must be installed on the line used to connect the refrigerator exhaust port to the pump intake port.
>
> **Caution:** Failure to use the type of pump described above could result in the refrigerator being over pressurized at the refrigerator exhaust port, causing the refrigerator to explode or to develop a high pressure leak into the vacuum chamber.

SECTION 2. REFRIGERATOR COOL-DOWN PROCEDURE

2.1. Initial Refrigerator Cool-Down

2.1.1. Before connecting the gas line to the refrigerator, purge the gas lines and filter with 500 psi nitrogen (99.998% pure or better) for about 30 seconds to remove any moisture that may have accumulated in the line.

2.1.2. Connect either the 3-foot or the 5-foot stainless steel gas line between the gas regulator and the INTAKE end of the filter. The MMR gas line adapter is necessary to make this connection. Connector nuts need only be finger tight. If this is not sufficient, tighten only an additional 1/4 turn with a 1/4 inch wrench. The filter should be vertical with the INTAKE at the top.

2.1.3. Insert or mount the refrigerator assembly into its vacuum chamber. Make sure the refrigerator O-ring(s) has a "wet" look. If it does not, apply a very thin film of vacuum grease on the sealing surface of the chamber and on the O-ring. Make sure the refrigerator is mounted per the instructions in your MMR System User's Manual.

2.1.4. Connect the 6-inch gas line between the filter and the gas intake port on the refrigerator. Again, connector nuts need only be finger tight.

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

> **Note:** Vacuum-assisted Joule-Thomson refrigerators will subsequently have the tubing from the refrigerator exhaust port connected to the gas intake port of a vacuum pump. Tygon tubing supplied with MMR standard refrigerators may not have sufficient wall thickness to prevent the tubing from collapsing on itself under vacuum. Replace the Tygon tubing with vacuum hose in this case. Connect this tubing as specified in 2.1.5 above. Do not connect tubing to vacuum pump at this point.

2.1.6. Evacuate the system and allow enough time for the vacuum level to reach 5 millitorr before turning on the high pressure gas.

2.1.7. Purge the entire system with gas at 500 psi for about 30 seconds.

2.1.8. Adjust the high pressure gas regulator to increase the gas pressure to the refrigerator from 500 psi to 1800 psi MAXIMUM and allow the refrigerator to reach minimum temperature.

> **Note:** Vacuum-assisted Joule-Thomson refrigerators will subsequently be connected to a vacuum pump to obtain temperatures below 80K. Follow the procedures outlined below, starting with step 2.1.9.

2.1.9. Low Temperature Operation of Vacuum-Assisted Joule-Thomson Refrigerators:

(a) Disconnect the Tygon tubing between the exhaust port of the refrigerator and the flow meter at the exhaust port of the refrigerator.

(b) Connect a short length of vacuum hose to the refrigerator exhaust port.

DO NOT CONNECT THE OTHER END OF THE VACUUM HOSE TO THE VACUUM PUMP AT THIS TIME!

(c) Turn on the vacuum pump being used to provide the vacuum assist to the Joule-Thomson refrigerator. Do this before connecting it to the vacuum hose from the refrigerator exhaust port.

THIS IS A DIFFERENT PUMP THAN THE ONE BEING USED TO EVACUATE THE REFRIGERATOR CHAMBER.

OPERATION OF THE VACUUM-ASSISTED JOULE-THOMSON REFRIGERATOR IN AN MMR SYSTEM REQUIRES TWO VACUUM PUMPS: ONE TO EVACUATE THE REFRIGERATOR CHAMBER AND ONE TO PROVIDE VACUUM ASSIST AT THE EXHAUST PORT OF THE REFRIGERATOR.

(d) Connect the other end of the vacuum hose to the intake port of the vacuum pump.

(e) Monitor refrigerator temperature and reduce input gas pressure to the refrigerator from 1800 psi to achieve lowest refrigerator temperature (to as low as 1100 psi).

(f) Upon completion of cool-down prepare to shut the system down using the following procedure.

2.2. Refrigerator Warm-Up Procedure

Vacuum-assisted Joule-Thomson refrigerators start with Step 2.2.1 below. Standard refrigerators start with Step 2.2.3 below.

2.2.1. Disconnect the hose from the vacuum pump providing the vacuum assist to the refrigerator. This leaves the vacuum pump sucking air from the room.

2.2.2. Turn off the vacuum pump providing the vacuum assist to the refrigerator.

2.2.3. Turn off the power to any device mounted on the refrigerator cold pad. Set the temperature controller to 300 °K and wait for the refrigerator to reach 300 °K.

2.2.4. Turn off the gas to the refrigerator.

2.2.5. Let the refrigerator warm up in the vacuum chamber, under vacuum, for 10 minutes.

2.2.6. Slowly vent the vacuum chamber using clean, dry backfill gas (Nitrogen) to bring the chamber up to atmospheric pressure.