TATE WARREN

Cryogenic Microminiature Refrigeration System IIB

Model K2205

USER'S MANUAL

MMR Technologies, Inc.
1400 North Shoreline Blvd., Suite A5
Mountain View, CA 94043
(415)962-9620

October 1984

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Safety Precautions

Always wear protective glasses when operating this refrigeration system.

Always cover the refrigerator with the vacuum chamber lid before turning on the high pressure gas.

Handle the refrigerator with care - it is made of glass.

Avoid any restrictions in the exhaust gas line - back pressure build-up can burst the refrigerator.

Never heat the refrigerator above 50°C except when operating in conjunction with a temperature controller.

Never use pressurized oxygen in the refrigerator system.

Do not overtighten connections.

Do not make sharp bends in the tubing.

Read the User's Manual carefully before using this refrigeration system.

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SECTION I
GENERAL INFORMATION

General Description

This miniature cryogenic J-T cooling system is designed to make low temperature material characterization an inexpensive and simple operation. The system is used in both research and Q/A Testing labs and can be customized for production-line testing needs. Applications include temperature characterization of materials, biological samples, optoelectronic detectors and lasers, low noise amplifiers, and most other electronic devices. MMR's system can be incorporated into OEM systems for localized component cooling, providing stable peak operating temperatures. It is also used in certain thin film fabrication techniques that require low and stable temperatures.

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, 250 mW of power can be absorbed indefinitely, with about a 4 degree increase in minimum temperature.

When this system is combined with the K-77 Temperature Indicator/Controller or the K-20 Programmable Temperature Controller, +100°C to -196°C may be selected as an operating temperature.

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 0.1 K/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 enters the heat exchanger at 100 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 nitrogen as it vaporizes. The vapor then passes back up the heat exchanger, precooling the incoming gas. Finally, it vents at 1 atmosphere at a little below ambient temperature.

Kit Notes

Kit operation requires the following user supplied items:

- Minimum 1800 psi nitrogen gas supply (99.998% pure).
- Nitrogen regulator capable of 500 to 1800 psi delivery pressure (see Reorder Parts List, Section VI).
- Vacuum pump capable of 5 millitorr (see Reorder Parts List, Section VI).

Important System Information

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.

Nitrogen Gas

To avoid clogging of the refrigerators by foreign gases such as CO_2 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 THE SYSTEM.

Filters

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

Gas Lines

The two ends of each gas line are joined with a threaded barrel to keep internal contamination of the line to a minimum. When not in use, the gas lines should be stored with the two ends joined by the threaded barrel. The shorter the gas line downstream of the filter, the less chance there is of contaminating the rest of the system.

Do not overtighten 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.

Vacuum Chamber

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

SECTION II

SYSTEM ASSEMBLY

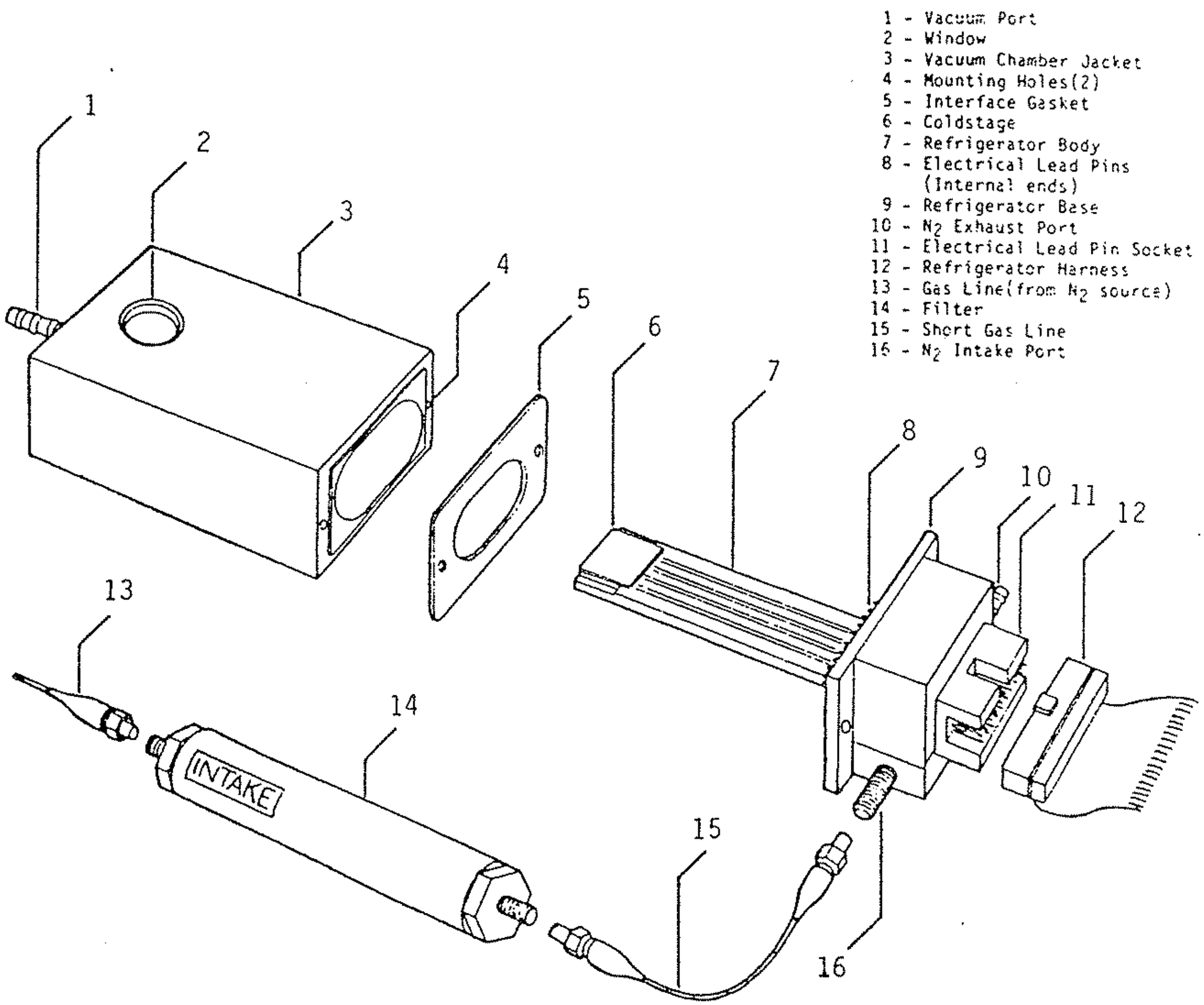

Figure 1 - System Assembly

Initial Set Up

1. Recharge nitrogen gas filter if required - filter contents should be changed about every third tank of nitrogen (see "Filter Refilling Instructions", page, 9).

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 nitrogen 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.

Refrigerator Preparation

4. Clean off any dust or particles on the interface gasket, apply a thin film of vacuum grease to it on both sides, and slip it carefully over the refrigerator body, positioning it against the refrigerator base. The gasket may be held in place against the refrigerator base by sliding the two mounting screws through the refrigerator base holes and threading them into the gasket holes.

5. Mount your device or specimen onto the coldstage (see "Thermal Contact", page 13, and "Temperature Sensing" page 14) using thermal grease or thermally conductive epoxy (see "Suggested Accessories", page 8). BE SURE THERE IS SUFFICIENT CLEARANCE BETWEEN THE MOUNTED DEVICE AND THE VACUUM CHAMBER JACKET CEILING. Coldstage-to-ceiling distance is about 13mm.

6. Make all electrical connections (see "Electrical Connections", page 14, and "Suggested Accessories", page 8) between the mounted device on the coldstage and the electrical lead pins (internal ends). If these wires are long they may be wrapped around the refrigerator body (to take up slack) before being soldered to the lead pins. Use a low-wattage soldering iron only.

Final Assembly

7. Slip the refrigerator into the vacuum chamber jacket and secure it with the two screws provided by partially tightening each screw until the refrigerator base is flush and snug against the chamber. Do not overtighten. ALWAYS USE BOTH SCREWS. BE SURE THE DEVICE ON THE COLDSTAGE DOES NOT CONTACT THE CHAMBER LID CEILING.

8. Connect one end of the 6" gas line to the filter and run 500 psi nitrogen through the lines for about 30 seconds to purge them of any moisture that may have collected during nonuse. Connect the other end of the 6" gas line to the nitrogen intake port. Again, connections need only be finger tight.

9. Connect a convenient length of Tygon tubing between the nitrogen exhaust port and flow gauge to monitor the gas flow rate.

10. Make all electrical connections between the user's instruments and the refrigerator harness. Plug the harness into the electrical lead pin socket.

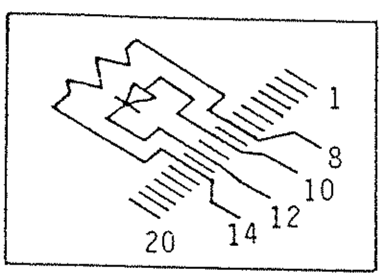

Figure 2
Internal Connections Between Refrigerator Harness and Coldstage Diode and Resistor Heater

11. 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.

Suggested Accessories

Vacuum Grease:

Apiezon Type "M" High Vacuum Grease

Thermal Grease:

For systems monitored by a Mass Spectrometer:

Apiezon Type "M" High Vacuum Grease

All other cases:

Dow Corning 340 Thermal Grease

Thermally Conductive Epoxy:

Ablestik Laboratories' Silver Filled Epoxy

Ablebond #88-1

Electrical Connections:

Wire	Size	Resistance*	Thermal Conductance*
Manganin	#30	1.0 ohm	6.5 mW
Copper	#40	0.2 ohm	7.5 mW

* For a pair of wires, each 6 cm long.

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 steel capnuts originally supplied at the time of shipment on the end fittings of the filter. Reinstall these cap nuts whenever the filter is removed from 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 nitrogen 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 Pouch #2 (4A) which will remove water vapor, and then through a sieve 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.

SECTION III
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 "Vacuum", page 14).

3. Turn on the high pressure nitrogen (1800 PSI MAXIMUM - see "Gas Pressure", page 13) and allow the refrigerator to reach minimum temperature before turning on power to the device on the coldstage.

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

5. After the experiment is finished, turn off the nitrogen gas and the vacuum. 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 IV

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 absorbed indefinitely, with about a 4 degree increase in minimum temperature.

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

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 refrigeration capacity of the refrigerator to that required by 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.

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 60 mK/mW, but this will significantly increase with poor thermal contact. A thin layer of thermally conductive grease or epoxy should be used (see "Suggested Accessories", page 8). When heating the refrigerators to cure cements and epoxies do not exceed 50°C.

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. This will allow the vacuum level at the refrigerator to reach the recommended level of 10 millitorr or better for proper refrigerator performance. A thin film of vacuum grease (see "Suggested Accessories", page 8) on the interface gasket will help prevent leaks. The system is not compatable with a vacuum of 10^{-6} torr or better.

Electrical Connections

Use the smallest wire size possible for any electrical leads to the device being cooled. This will minimize the heat leak to the device from the leads. Although low thermal conductance wires such as manganin can be used, they have a high resistance and are slightly difficult to solder. Copper wires are easy to solder and have low electrical resistance and low thermal conductance if long lengths and small diameters are used, but are fragile in these small diameters (see "Suggested Accessories", page 8).

Temperature Sensing

Use either a platinum resistance thermometer or a silicon diode for accurate temperature sensing. However, if a thermocouple must be used, bring the reference junction for the thermocouple outside the vacuum chamber for more accurate readings. This is necessary because the electrical lead pins in the base of the refrigerator, and even the vacuum chamber to a limited extent, will be cooled slightly when the refrigerator is operating. Hence, soldering the thermocouple wires to the lead pins will result in reference junction induced errors.

SECTION V

TROUBLESHOOTING GUIDE

Symptoms*	Possible Causes Once a cause has been determined, refer to the appropriate number under "Possible Solutions", starting on the next page.
1. No cooling at all:	°Poor thermal bond, #3 °No gas flow, #8 °Inadequate gas pressure, #9 °Excessive heat dissipation, #10
2. Partial cool-down, -30°C to -100°C, and then warm up:	°Clogging, #1 °Poor thermal bond, #3 °Poor vacuum, #4
3. Slow cool-down:	°Partial clogging, #2 °Poor thermal bond, #3 °Large electrical leads, #6 °Large heat capacity of device, #7 °Inadequate gas pressure, #9 °Excessive heat dissipation, #10 °Cracked or broken refrigerator, #11 °Restriction in gas exhaust line,#12
4. Inadequate temperature (above 85K):	°Poor thermal bond, #3 °Poor vacuum, #4 °Poor temperature sensing, #5 °Large electrical leads, #6 °Excessive heat dissipation, #10 °Cracked or broken refrigerator, #11 °Restriction in gas exhaust line,#12
5. Broken refrigerator:	°Replace refrigerator (see precautions under "Cracked or Broken Refrigerators", #11)
6. Connector leak:	°Connector leak, #13
7. Broken Tubing:	°Replace tubing (available exclusively through MMR and its distributors).

*See "System Performance Variables", page 13, 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. Disconnect gas line from nitrogen intake port and run 500 psi gas through the filter and gas line for 30 seconds. Reconnect the gas line to the nitrogen intake port 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 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 the refrigerator. 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 achieved.

To insure a good thermal bond, use as thin a layer as possible of a thermally conductive grease or epoxy for mounting the device on the refrigerator. Recommendations for a suitable grease or epoxy are provided under "Suggested Accessories", page 8.

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 the interface gasket.
b. Excessive moisture in the system.
c. A 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 the refrigerator.
f. Cracked refrigerator (see #11 below).

5. Poor Temperature Sensing

Avoid using thermocouples if possible (see "Temperature Sensing", page 14). Provide a good thermal bond between the temperature sensor and the mounting pad.

6. Large Electrical Leads

#40 copper wire or #30 manganin wire should be used to minimize the heat leak to the device being cooled.

7. Large Heat Capacity of Device

The heat capacity of the device exceeds the refrigerator's ability to cool it in a reasonable time.

8. No Gas Flow

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

9. 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 most applications.

10. Excessive Heat Dissipation

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

11. 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 GAS 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 LID.

d. USE BOTH SCREWS IN SECURING THE REFRIGERATOR TO THE VACUUM CHAMBER JACKET.

12. Restriction in Gas Exhaust Line

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

13. Connector Leak

MMR Gas line connectors are designed to provide quick and easy assembly of the refrigeration system 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 some dirt on the teflon washer inside the threaded barrel in the refrigerator base. 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.

SECTION VI

REORDER PARTS LIST

Description	Part #
Vacuum Chamber Jacket	B000005
Refrigerator Model R2205	B000001
System IIT User's Manual	A920002
Filter	B000086
Filter Refill Pack	A910029
Refrigerator Harness	B000040
Connector Harness for use w/K77	B000011-020
5' Stainless Steel Gas Line	B000097-030
3' Stainless Steel Gas Line	B000097-020
6" Stainless Steel Gas Line	B000097-010
10-32 x 1/8" Gas Line Adapter	B000113
Interface Gasket	B000008-000
Flow Meter	A930066
Carry Case with Foam Insert	C000099

Required Additional Parts

Description	Part #
N_2 Regulator (Delivery Pressure 100 to 2500 psi)	A930065
Vacuum Pump - 5 Millitorr	
115 VAC/60 Hz	A930032
230 VAC/50 Hz	A930041