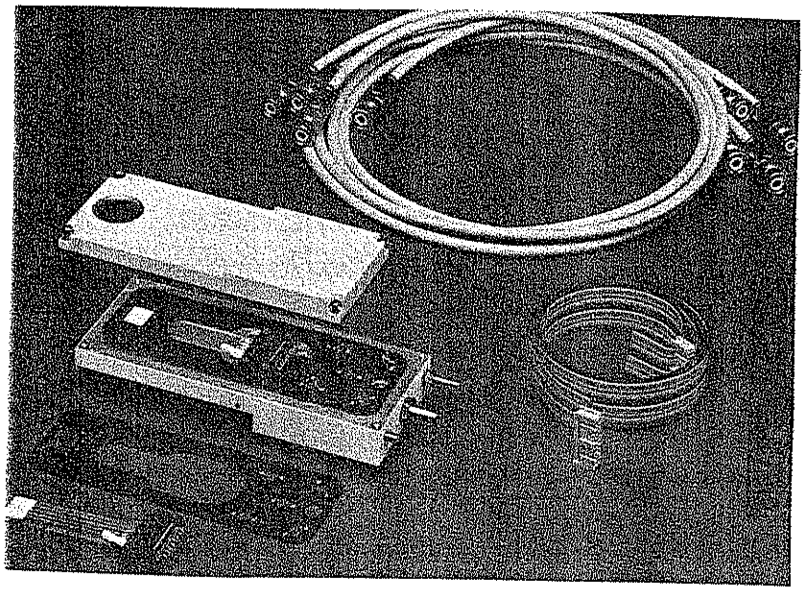
LOW TEMPERATURE HALL SYSTEM DEWAR



USER'S MANUAL

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N E V E R !

NEVER HANDLE OR WORK ON AN MMR REFRIGERATOR WHEN IT IS CONNECTED TO THE H-50 WITH THE POWER ON!

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REFRIGERATOR: GENERAL INFORMATION

General Description

This miniature cryogenic J-T 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° increase in minimum temperature.

When this system is combined with the K-20 Programmable Temperature Controller, any temperature in the range +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 0.1K/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 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 items:

- -1800 psi Nitrogen gas source (99.998% pure).
- -N_2 regulator capable of 500 - 1800 psi delivery pressure (see Reorder Parts List, Section VI).
- -Vacuum pump capable of 5 millitorr

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

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 gasses 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 should be 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

DEWAR SYSTEM ASSEMBLY

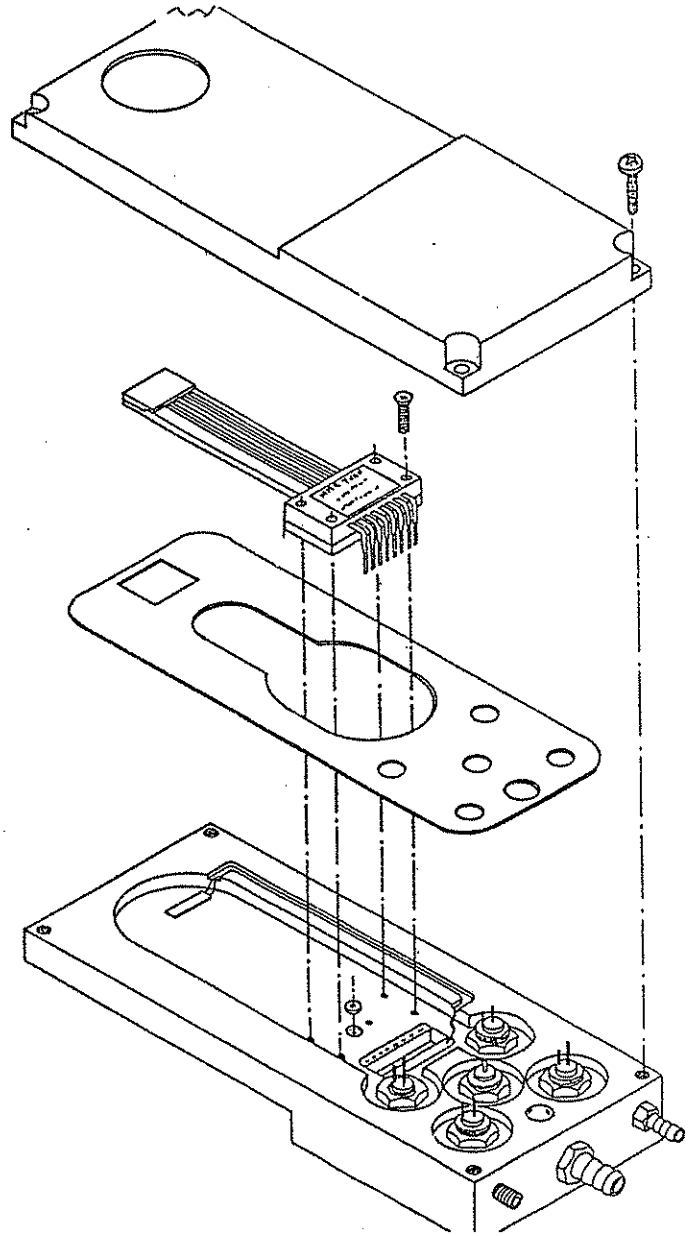

Figure 1 - Hall System Dewar - Top Side

Initial Set Up

1. Recharge N_2 gas filter if required - filter contents should be changed about every third tank of nitrogen (see "Filter Refilling Instructions", page 10).

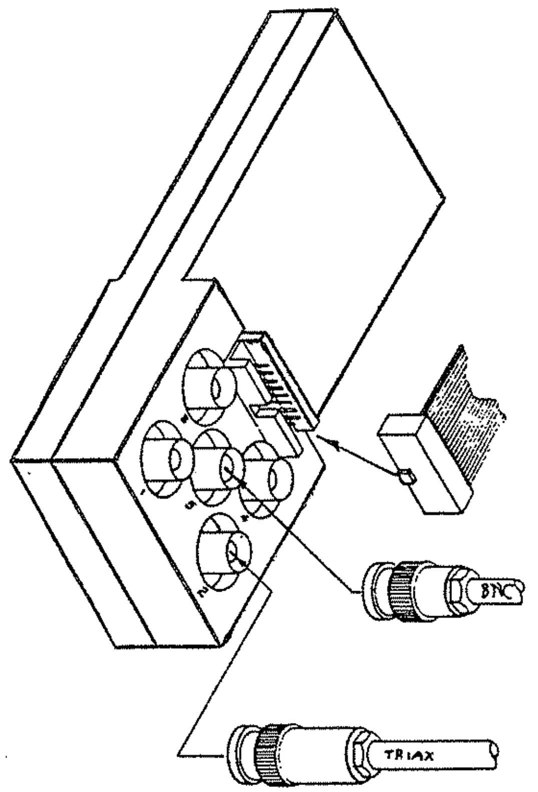

Figure 2 - Hall System Dewar - Underside

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.

Refrigerator Preparation

9. Mount your device or specimen onto the refrigerator coldstage (see "Thermal Contact", page 13) using thermal grease or thermally conductive epoxy (see "Suggested Accessories", page 2). Be sure there is sufficient clearance between the mounted device and the vacuum chamber lid ceiling. Coldstage-to-ceiling lid distance is about 3mm.

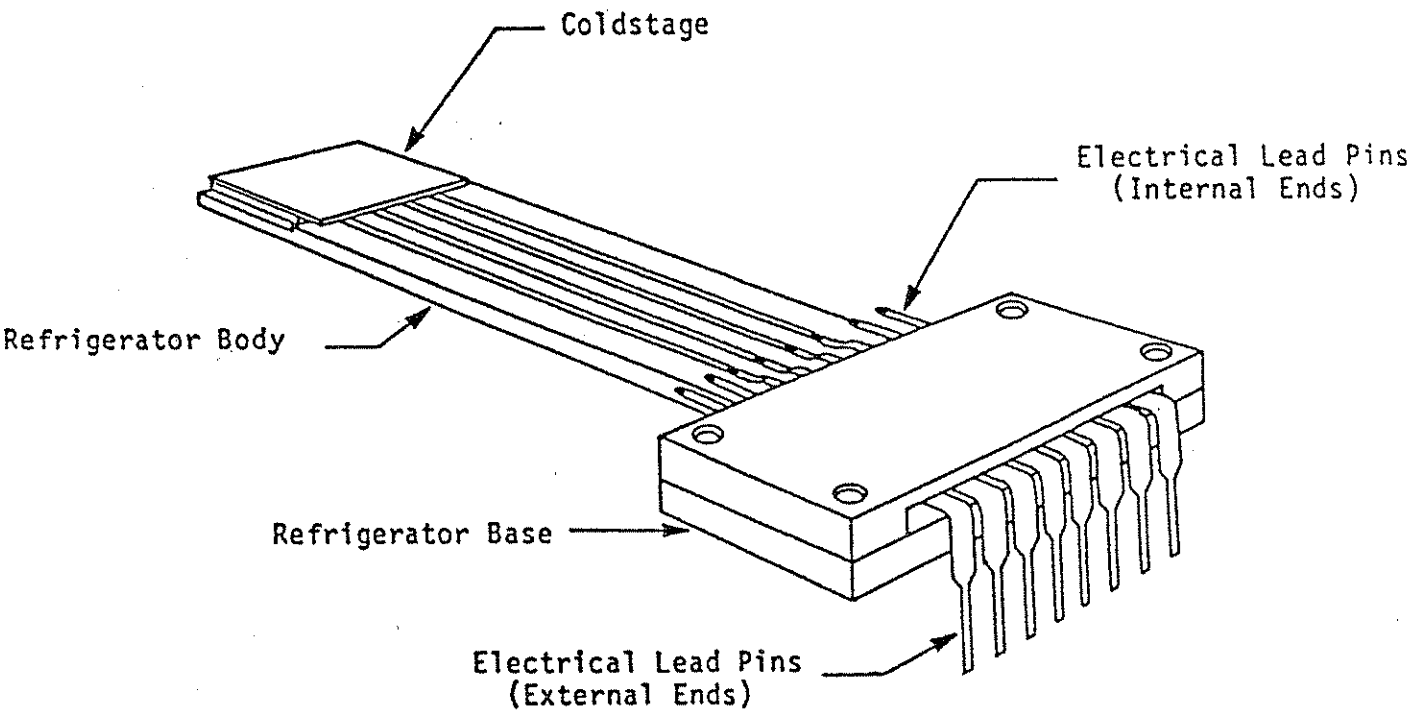

Figure 3 - LTHS Refrigerator

10. 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.

Final Assembly

11. 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.

12. Secure the refrigerator base to the vacuum chamber base with the four screws provided by partially tightening each screw in sequence until the refrigerator base is flush and snug with the surface of the vacuum chamber base. Do not over-tighten. ALWAYS USE ALL FOUR SCREWS.

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. Make all electrical connections between the mounted device and the electrical probe leads on the Kapton harness. Use #34 - #40 gauge copper wires, soldered to the copper terminal pads on the harness, to connect to the contact points on the sample.

15. 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.

Filter Refilling Instructions

Complete the refilling process quickly because the adsorbent begins to degrade as soon as it is exposed to the atmosphere.

Save the nylon cap nuts provided on the end fittings of the filter. Reinstall these cap nuts whenever the filter is removed from the refrigerator system set-up. This will prevent needless contamination of the adsorbent and help to protect the threads of the fittings.

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

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

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 completely 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 1 minute with 500 psi nitrogen gas before using it in the refrigeration system. To purge the filter, run a gas line from the nitrogen gas source to the fitting at the INTAKE end of the filter. Also, 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 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 (4A) which will remove water vapor, and then through a sieve (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 your 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 N_2 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.

SECTION 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, 250 mW of power can be dissipated indefinitely, with about a 4° 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 2). 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 vaccum 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 2) on all O-rings will help prevent leaks.

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 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 thermally conductive grease or epoxy for mounting the device on the refrigerator. Recommendations for a suitable grease or epoxy are provided under "Suggested Accessories".

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 the refrigerator.
f. Cracked refrigerator (see #11, below).

5. Large Heat Capacity of Device

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

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 most applications.

8. 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.

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 LID.
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 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.