OPERATING INSTRUCTIONS
FOR THE JANIS RESEARCH
MODEL ST-500
SUPERTRAN SYSTEM

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SECTION 1

⚠ WARNING

- Read all instructions before use in order to avoid injury or property damage.
- Keep this manual for future reference.

GENERAL SAFETY PRECAUTIONS

1.1 SAFETY LABELS

⚠ WARNING The **WARNING!** label indicates a hazard. It calls attention to a procedure, practice, or condition that, if not observed or adhered to, could result in serious injury or even death.

NOTICE The **NOTICE!** label indicates a hazard. It calls attention to a procedure, practice, or condition that, if not observed or adhered to, could result in damage to the equipment.

1.2 SAFETY SUMMARY

⚠ WARNING All safety pressure relief valves are installed to provide protection to the equipment and operating personnel. Do not tamper with any pressure relief valve.

⚠ WARNING During a sample change, it is possible for the radiation shield to remain cold even after the sample has warmed to room temperature. Use gloves when handling a cold radiation shield to avoid low temperature burns.

⚠ WARNING During system shutdown, the storage dewar leg of the transfer line will be extremely cold upon removal from the storage dewar. Do not touch the transfer line with bare hands. Always wear insulating gloves when handling the transfer line after use.

NOTICE Only the sample mount of the ST-500 is designed to operate in excess of 325 K (~ 50 °C). Care should be taken not to heat the exterior portion of the cryostat or the transfer line above room temperature, as this will degrade the o-ring seals which seal the vacuum space of the cryostat from the room environment.

NOTICE Do not bend the flexible section of the transfer line to a radius of less than 12 inches, to prevent possible damage to the inner line.

NOTICE Do not close the flow regulator valve on the transfer line more than hand tight. Never use a wrench or any tool on the flow regulator knob, to prevent damage to the needle valve and seat.

NOTICE The stainless steel liquid helium inlet and exhaust legs are attached to the aluminum main body of the ST-500 cryostat by bolted stainless steel flanges. These flanges must never be loosened or removed. Loosening the bolts that secure the flanges will result in damage to the internal components of the ST-500, requiring factory repair to restore the system to service. (This damage is not covered under warranty.)

NOTICE Do not tamper with the heat exchanger, the gold plated piece which connects to the inlet and outlet ports. The heat exchanger is a delicate instrument and may be damaged if tampered with.

NOTICE If using the ST-500 in the vertical position, with the exhaust leg pointing upwards, a drain tube must be used to lead any condensation away from the outlet port. Without using a drain tube, it is possible that water will drip back down into the heat exchanger and then freeze during operation, damaging internal parts.

NOTICE Always use the supplied transfer line when operating with LHe or LN_2. Never connect the cryostat inlet port directly to the outlet of a pressurized LN_2 tank without using the supplied transfer line. High pressure from the LN_2 tank will result in damage to the internal structure of the ST-500, requiring factory repair to restore the system to service. (This damage is not covered under warranty.)

NOTICE When operating above 325 K, the vacuum space of this cryostat should be evacuated <u>continuously</u> to prevent contamination due to heater outgassing.

NOTICE The maximum operating temperature of the Model ST-500 cryostat is 475 K. Choosing a setpoint greater than 475 K will result in damage to the ST-500 system, requiring factory repair to restore the system to service. (This damage is not covered under warranty.)

SECTION 2

INTRODUCTION

2.1 GENERAL DESCRIPTION

The Janis Research Supertran ST-500 System is a continuous flow research cryostat that can be used to perform a wide variety of experiments in the temperature range from 3.5 K to 475 K. Liquid helium or nitrogen is continuously transferred through a high efficiency superinsulated line to a copper sample mount inside the cryostat vacuum jacket. A needle valve is incorporated in the transfer line, and is used to regulate the flow of cryogens to the sample mount.

2.2 SYSTEM COMPONENTS

The Janis ST-500 system includes the following components:

1. Sample mount: This is the gold-plated copper base of the system on which samples can be mounted. The test sample is typically held in good thermal contact with the sample mount using Apiezon Type N (or similar) vacuum grease. The sample mount bolts to a heat exchanger which includes a temperature sensor and a heater to control the temperature of the sample.

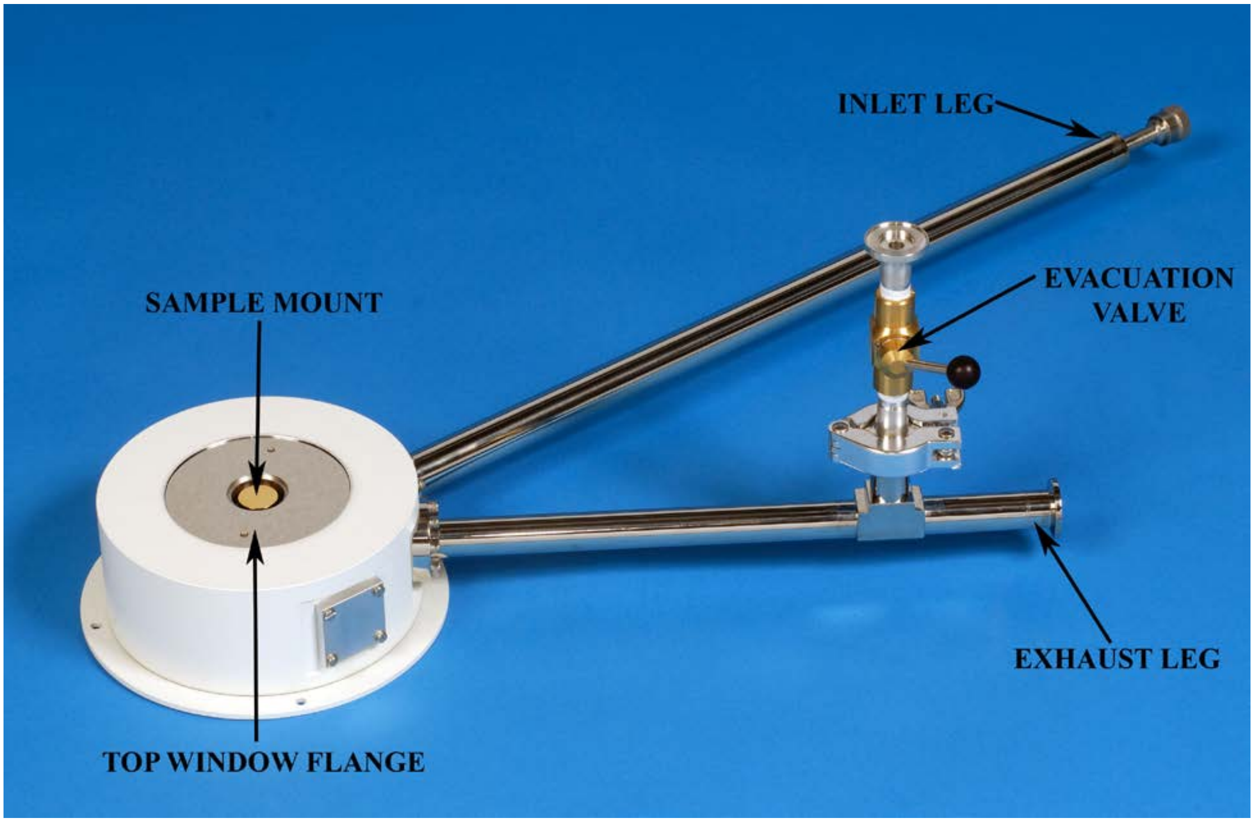
2. Radiation shield: This shield, fabricated from gold-plated copper, is mounted to a thermal anchor in the cryostat. It intercepts room temperature radiation, thereby reducing the heat load on the sample mount. This helps the system achieve lower sample temperatures.

3. Vacuum jacket: The vacuum jacket is the outer layer of metal which surrounds the sample mount and radiation shield. The vacuum jacket can be evacuated through the pumping port evacuation valve located on the exhaust leg of the cryostat.

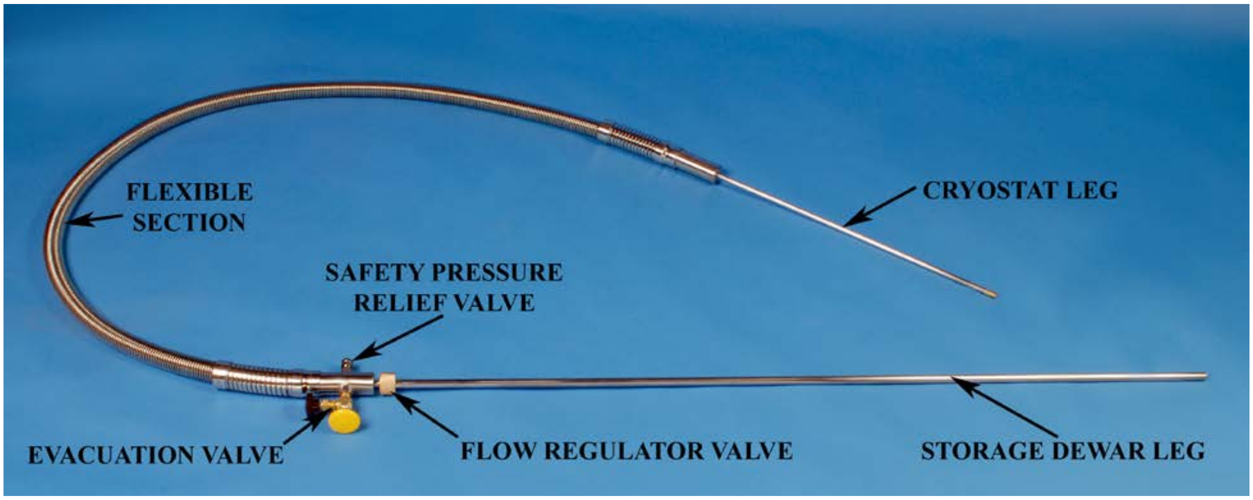
4. Top and bottom window flange: The top and bottom window flanges provide optical access to the sample in the cryostat. The bottom flange is held in place with screws, while the top flange simply sits on an o-ring seal. The top flange acts as a method of pressure relief for the vacuum jacket in the event of a leak. In addition, the top flange provides user access to the sample mount, as detailed in Section 4.1.

5. High efficiency transfer line: The ST-500 transfer line combines vacuum insulation with multilayer superinsulation to provide low cryogen losses during transfer. A needle valve flow regulator is built-in to the transfer line. One end of the transfer line is inserted into the cryogen storage dewar, while the other end is inserted into the cryostat.

6. Optional temperature controller: Many types of automatic temperature controllers are available for use with the ST-500 system. These controllers allow the user to select a desired control temperature while adjusting several other control parameters, including proportional and integral values. The temperature controller then maintains the desired temperature by applying power to the control heater. Controllers purchased through Janis Research include cabling and are tested with the cryostat.



The system components of the standard ST-500 cryostat.



The system components of the cryogen transfer line.

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NOTICE Do not tamper with the heat exchanger, the gold plated piece which connects to the inlet and outlet ports. The heat exchanger is a delicate instrument and may be damaged if tampered with.

SECTION 3

INSTALLATION

3.1 MOUNTING

The standard ST-500 cryostat includes four clearance holes on the periphery of the bottom flange for mounting. These holes may be used for securing the cryostat directly to an optical bench or to a translation stage. To prevent unwanted sample motion, be sure to secure the cryostat firmly to the bench or stage, particularly when mounted vertically. The cryostat may be mounted horizontally or vertically.

NOTICE If using the ST-500 in the vertical position, with the exhaust leg pointing upwards, a drain tube must be used to lead any condensation away from the outlet port. Without using a drain tube, it is possible that water will drip back down into the heat exchanger and then freeze during operation, damaging internal parts.

A very simple drain tube could be a rubber hose that is stretched over the outlet leg and then allowed to hang over the side of the outlet leg. Condensation will appear somewhere further down the rubber tube such that when water drips out, it will drip harmlessly to the floor.

3.2 ELECTRICAL CONNECTIONS

If an optional automatic temperature controller has been supplied with the ST-500 system, refer to the accompanying controller manual and connect it to the appropriate AC outlet. Automatic temperature controllers operate by using a feedback control loop, in which the controller sends output to the control heater based upon the signal from the control thermometer. For this reason, it is important that the control heater and thermometer both be attached to the same piece of copper, in the ST-500, this is the heater ring (installed on the heat exchanger). When thermometers are supplied at both the heater ring (for control) and the sample mount (for monitoring the sample temperature), be sure the thermometer at the heater ring is designated as the control channel. In most cases, the control thermometer will be assigned to Channel A of the controller. Connect the supplied thermometry cable from the 10-pin electrical feedthrough on the side of the cryostat to the temperature controller. The dual “banana plug” should be connected to the heater output; Hi and Low terminals. Refer to the accompanying wiring diagram at the end of this manual for specific sensor and wiring details.

If no temperature controller has been supplied with the system, refer to the accompanying wiring diagram for pin assignments of all feedthroughs installed on the dewar. Mating connectors are supplied which allow the user to attach cables as needed.

SECTION 4

OPERATION

4.1 SAMPLE MOUNTING

Access to the sample is provided by removing the top window flange. Before removing the top window flange, vent the vacuum space by opening the evacuation valve, located on the exhaust leg of the cryostat. Two tapped holes are provided to assist in removing this flange. Thread a screw into each of the two provisions, and use them to lift the flange off the cryostat.

The sample can now be mounted to the gold-plated copper sample mount. A thin film of thermal grease such as Apiezon-N Grease can also be used to improve the thermal contact between the sample and sample mount. (However, do not use Apiezon N grease for operation above 325 K.) It is important to establish good thermal contact with the sample mount to ensure cooling of the sample

The ST-500 system includes provisions for additional electrical feedthroughs for customer wiring of the samples. Small gauge wires (32AWG) should be used to minimize heat leak into the sample, and the wires should be thermally anchored in several spots to the cold finger by using Stycast epoxy, varnish, by tying with nylon string or floss, or by using mylar or aluminum tape.

Once the sample is mounted in the system, reinstall the top window flange. Any visible dirt or lint on the sealing o-ring is sufficient to cause a vacuum leak, so be sure the o-ring and flange are clean and lightly greased before reinstalling the top window flange.

4.2 EVACUATION

The Model ST-500 system must be evacuated for proper performance. The system includes two independent vacuum spaces. One vacuum space insulates the high efficiency transfer line, while the vacuum space inside the cryostat surrounds the sample region. Both vacuum spaces utilize a low leak rate evacuation valve, which allows evacuation and sealing.

Prior to operation of the ST-500 cryostat, connect a turbomolecular or diffusion pump to the pumping port evacuation valve, (located on the exhaust leg of the cryostat), and evacuate to a pressure of 1.0×10^{-4} Torr or less. The vacuum space must be pumped for several hours, and preferably overnight, to remove all of the air and reduce the out-gassing from the bellows and other parts of the cryostat. Better vacuum levels provide greater insulation, resulting in shorter cooldown times and lower temperatures at the sample mount. A mechanical vacuum pump with a liquid nitrogen cold trap can be used instead; however, this will not produce the best vacuum possible and may result in higher temperatures. The cryostat evacuation valve should also be closed once evacuation is complete, to avoid backstreaming of oil from the vacuum pump into the cryostat. Outgassing and o-ring permeation will cause the pressure to rise slowly over time, therefore periodic re-evacuation will be necessary. Re-evacuation of the cryostat is required whenever a new sample is installed, or when the minimum temperature obtained begins to increase.

The vacuum space of the liquid helium transfer line may occasionally require evacuation. The same type of vacuum pumping station and pressure should be used as discussed above. The vacuum space of the transfer line should be pumped overnight to ensure the best vacuum possible. The rigid leg of the transfer line incorporates an activated charcoal getter, to help maintain good vacuum levels when the leg is inserted into a cryogen storage dewar. For this reason, it is preferable to maintain vacuum in the transfer line at all times, and to never allow helium gas or moist air to enter this vacuum space. (In the event moisture or helium gas accidentally contaminates the transfer line vacuum space, re-evacuate the line for several days before operating again.) Once the vacuum space of the transfer line has been pumped overnight, the transfer line vacuum valve should be closed, and the transfer line is ready for operation.

NOTICE When operating above 325 K, the vacuum space of this cryostat should be evacuated continuously to prevent contamination due to heater outgassing.

4.3 INITIAL COOLDOWN (LHe)

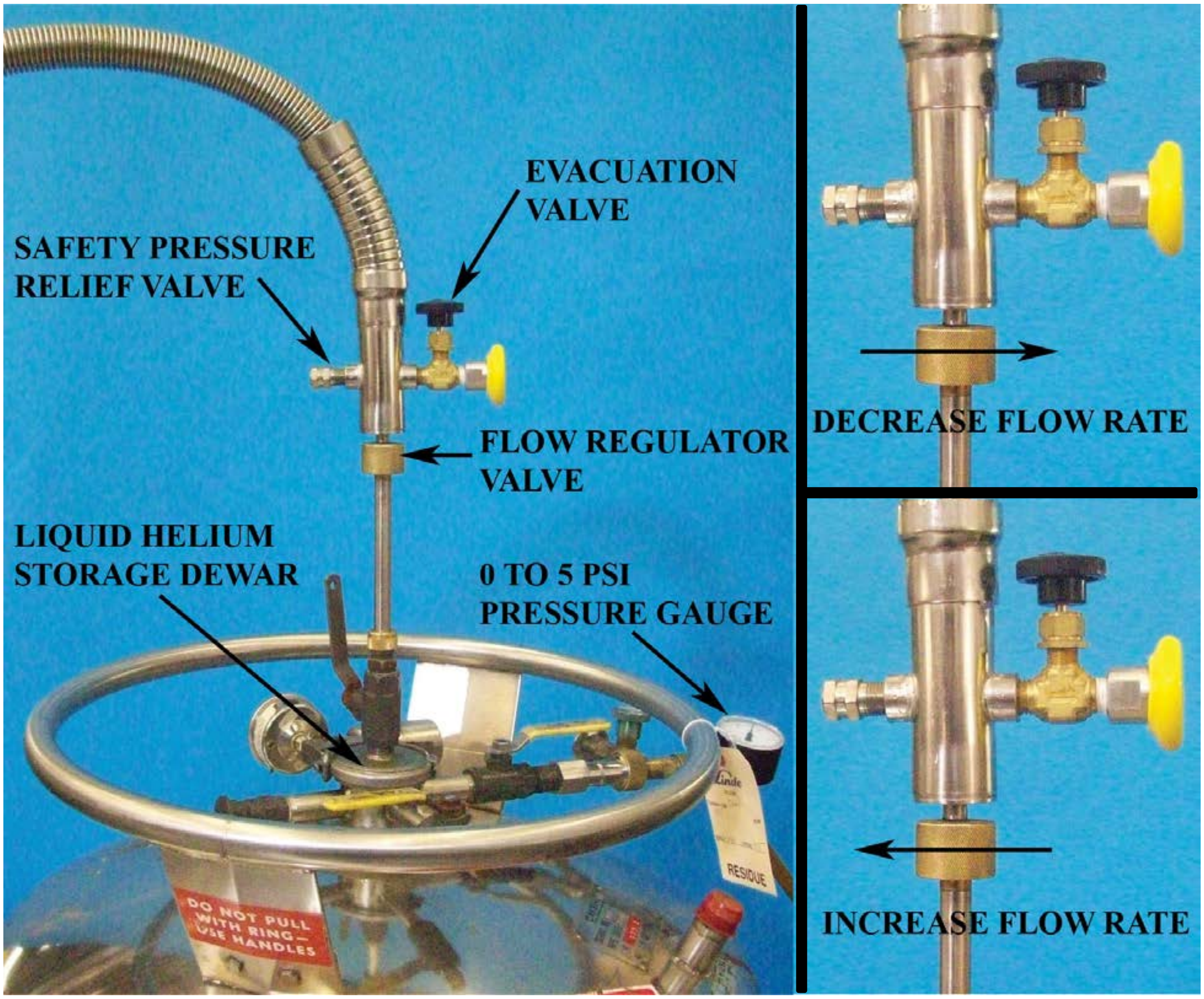
NOTICE Do not close the flow regulator valve on the transfer line more than hand tight. Never use a wrench or any tool on the flow regulator knob, to prevent damage to the needle valve and seat.

1. For the fastest cooldown and best results, attach a mechanical vacuum pump to the exhaust port (NW-25 flange) of the cryostat, and evacuate this space continuously during the first phase of cooldown. This removes any air or moisture remaining in the line from previous operation and shortens the cooldown time by several minutes.

2. Close the flow regulator valve at the storage dewar leg of the transfer line, by turning the knurled stainless steel operator knob counter-clockwise (as viewed from above). The cryostat inlet port includes an o-ring compression fitting, while the transfer line delivery leg ends in a brass tip. For best efficiency, insert the transfer line completely into the inlet port of the cryostat, and once the transfer line reaches the bottom, tighten the brass compression fitting securely. After cooling the cryostat, push the transfer line firmly into the inlet port to be sure that the brass tip makes a snug connection.

Follow step 3 if your storage dewar leg has a hole in it (near the regulator valve), otherwise go directly to step 4

3. Place your thumb over the small hole in the storage dewar leg and slowly insert the leg of the transfer line into the liquid helium storage dewar. (For easiest adjustment of the transfer rate, the storage dewar should be equipped with a 0 - 5 psi pressure gauge, a venting valve, and a hose adapter for adding helium gas pressure if necessary.) Once the hole in the storage dewar leg is below the O-ring compression seal of the storage dewar, open the flow regulator valve about 2 – 3 full turns, and monitor the pump pressure or exhaust for a few seconds to confirm that the flow is not restricted. (The pump pressure should increase slightly, and the exhaust sound of the vacuum pump will typically change. The flow rate is quite small during the initial phase of the transfer, until the entire inner line becomes cooled.) Close and open the flow regulator valve several times during the first few minutes of transfer to be sure the valve does not freeze shut during cooldown, and that the flow is not restricted. Once the leg is fully inserted into the storage dewar, raise the leg about 1 cm from the storage dewar bottom to minimize the chance of frozen debris entering the line (an inlet filter is incorporated in the transfer line to minimize this possibility).



Insert the transfer line into the storage dewar, then open the flow regulator valve.

<u>Ignore step 4 if your storage dewar leg has a hole in the storage dewar leg (near the regulator valve)</u>

4. Slowly insert the leg of the transfer line into the liquid helium storage dewar. (For easiest adjustment of the transfer rate, the storage dewar should be equipped with a 0 - 5 psi pressure gauge, a venting valve, and a hose adapter for adding helium gas pressure if necessary**.)** Once the storage dewar leg is below fully inserted into the storage dewar, open the flow regulator valve about 2 – 3 full turns, and monitor the pump pressure or exhaust for a few seconds to confirm that the flow is not restricted. (The pump pressure should increase slightly, and the exhaust sound of the vacuum pump will typically change. The flow rate is quite small during the initial phase of the transfer, until the entire inner line becomes cooled.) Close and open the flow regulator valve several times during the first few minutes of transfer to be sure the valve does not freeze shut during cooldown, and that the flow is not restricted. Once the leg is fully inserted into the storage dewar, raise the leg about 1 cm from the storage dewar bottom to minimize the chance of frozen debris entering the line (an inlet filter is incorporated in the transfer line to minimize this possibility).

5. In general, heat introduced by conduction down the transfer line leg is sufficient to maintain positive pressure in the storage dewar. Adjust the storage dewar vent valve to maintain a

storage dewar pressure of 2 - 3 psi. (In some cases, helium gas will need to be added to maintain constant pressure in the storage dewar.)

6. After a short wait (typically 5 - 10 minutes) the sample mount will begin to cool rapidly. At this time, the vacuum pump should be disconnected from the system, and the escaping helium vapor vented into the atmosphere or a gas collection system. (Be sure to allow the pump to reach atmospheric pressure before disconnecting, to avoid introducing air and moisture into the line.)

7. Once the sample mount temperature reaches about 5 K, the flow regulator valve can be closed slightly, until the sample temperature begins to increase. Open the regulator just enough to cool the sample mount to 4.2 K. For best operating efficiency, it is best to open the regulator valve just enough to maintain the desired minimum temperature.

If the experimental configuration does not allow the transfer line to be inserted into the cryostat first (due to a low ceiling height or the cryostat mounting geometry), the following steps may be substituted for steps 1 – 3 above.

1. Open the flow regulator valve at the storage dewar leg of the transfer line, by turning the knurled stainless steel operator knob clockwise (as viewed from above).

2. With your thumb over the small hole in the storage dewar leg (or without if no hole is present), slowly insert the storage dewar leg of the transfer line into the liquid helium storage dewar. Once the leg is fully inserted into the storage dewar, raise the leg about 1 cm from the storage dewar bottom to minimize the chance of frozen debris entering the line. Note that you will not be able to feel any gas exiting the transfer line bayonet.

3. Insert the transfer line completely into the inlet port of the cryostat, and once the transfer line reaches the bottom, tighten the brass compression fitting securely.

4. Close the flow regulator valve at the storage dewar leg of the transfer line, by turning the knurled stainless steel operator knob counter-clockwise (as viewed from above). Attach a mechanical vacuum pump to the exhaust port of the cryostat, and pump for several minutes. This will evacuate the area inside the sample mount and the transfer line capillary, and provide a path for the liquid helium that is free from air and moisture. Open and close the flow regulator valve several times during the first few minutes of transfer, and listen as the exhaust sound of the vacuum pump changes. This that the valve does not freeze shut during cooldown, and that the flow is not restricted.

NOTICE Do not close the flow regulator valve on the transfer line more than hand tight. Never use a wrench or any tool on the flow regulator knob, to prevent damage to the needle valve and seat.

4.4 INITIAL COOLDOWN (LN_2)

Cooldown with LN_2 is very similar to operation with LHe, and the procedure above can be used with the following exceptions.

1. Because LN_2 will freeze when it is pumped, it is best not to initiate transfer with a vacuum pump. Instead, use a storage dewar pressure of about 4 psi, and open the flow regulator

valve about 3 - 5 turns. It will take 5 - 10 minutes before the sample mount temperature begins to drop.

2. It is often necessary to add gas pressure to the storage dewar to maintain constant pressure of 5 psi. Either helium or nitrogen gas can be used for this purpose.

3. Once the temperature reaches about 100 K, close the flow regulator valve completely, and wait until the sample temperature stabilizes or begins to increase. As in Step 6 above, open the flow regulator just enough to reach 78 K. Too large a flow will cause liquid to collect inside the sample mount, making temperature control at higher temperatures difficult or impossible.

4.5 OPERATION

For operation at 5 K (or 78 K with LN_2), the flow regulator valve should be opened just enough to maintain this temperature at the sample mount. The flow rate required is a function of the heat load into the sample mount, and will vary depending on configuration and experimental application.

For operation above 5 K (or 78 K with LN_2), use an automatic temperature controller, as described in Section 4.6 below. During operation above 20 K, the flow regulator valve can be partially closed, reducing the LHe consumption significantly. Adjustments in the flow regulator setting can be made at any time, either to increase the cooling power or reduce the cryogen consumption. In general, the smallest flow rate should be chosen that will cool the sample to the desired temperature. For elevated temperatures, choose a setpoint using the automatic temperature controller. The temperature controller will supply power to the control heater, and will stabilize the system at the selected temperature setpoint.

Continuous operation below 5 K is achieved by reducing the pressure at the helium vent port with a mechanical vacuum pump. The flow regulator valve can be partially opened, continuously flowing liquid helium through the cryostat, and resulting in a constant temperature of 3.5 K or less (as measured at the heat exchanger).

4.6 TEMPERATURE CONTROL

Standard ST-500 systems are supplied with a silicon diode thermometer and 50 ohm control heater. Options include other diode or resistance thermometers and different heater resistances. The actual configuration of your system thermometry can be found on the wiring diagram at the end of this manual.

The Model ST-500 system operates from 3.5 K to 475 K. Choose a temperature setpoint from within the appropriate range, and enter values for Proportional (P), Integral (I), and Derivative (D) parameters. Some experimentation may be required to optimize these settings for a particular application. In general, when operating at the lowest temperatures, (where the heat capacities are smallest), the (P) value should be low, and the (I) value should be high. Derivative (D) control can usually remain zero throughout the operating range. As the control temperature is increased, larger proportional and smaller integral values can improve temperature stability and response time. Note that the sensitivity of silicon diode thermometers change significantly at about 25 K. The (P) value can be increased 5 – 10 times when controlling the temperature above 25 K.

Some controllers include an autotuning function that selects appropriate PID values automatically. This function is most useful only for temperatures above 50 K. For complete discussion of this feature, as well as comprehensive controller operating procedures and specifications, refer to the temperature controller manual.

NOTICE The maximum operating temperature of the Model ST-500 cryostat is 475 K. Choosing a setpoint greater than 475 K will result in damage to the ST-500 system, requiring factory repair to restore the system to service. (This damage is not covered under warranty.)

4.7 CHANGING SAMPLES

Before changing samples, the sample mount should be warmed to room temperature. This can be accomplished in either of two ways.

1. The flow regulator valve can be closed, and the system allowed to warm up for several hours. Use the control thermometer to determine when the system is approaching room temperature.

2. The flow regulator valve can be closed, and the temperature controller set for 295 K. Once the thermometer reaches 295 K, wait until the heater power approaches 0%. The evacuation valve can then be opened and the sample changed as described in Section 4.1. (Dry nitrogen or argon gas can be used to break the vacuum if the sample is particularly sensitive to water vapor.)

⚠ WARNING During a sample change, it is possible for the radiation shield to remain cold even after the sample has warmed to room temperature. Use gloves when handling a cold radiation shield to avoid low temperature burns.

4.8 SYSTEM SHUTDOWN

To shut down the system, simply close the flow regulator valve and turn off the temperature controller. If available, mount a pressure relief valve on the exhaust leg of the cryostat. This will permit any cryogen remaining in the inner line to vent safely to atmosphere, while preventing any air or moisture from entering the cryostat. The storage dewar should then be vented to atmospheric pressure, and the transfer line storage dewar leg removed from the storage dewar.

⚠ WARNING During system shutdown, the storage dewar leg of the transfer line will be extremely cold upon removal from the storage dewar. Do not touch the transfer line with bare hands. Always wear insulating gloves when handling the transfer line after use.

SECTION 5

MAINTENANCE

5.1 SCHEDULED MAINTENANCE

The Model ST-500 system requires no regularly scheduled maintenance. The exterior surfaces of the vacuum jacket can be cleaned with a spray household cleaner periodically as necessary. The transfer line should be evacuated periodically, as discussed in Section 4.2.

5.2 UNSCHEDULED MAINTENANCE

Unscheduled maintenance may be occasionally required to repair problems arising during the course of operation. These problems may be related to vacuum leaks or wiring failure.

5.3 VACUUM LEAKS

Condensation on the outside of the vacuum jacket, and inability to reach 4 K are indications of a vacuum problem. If these symptoms appear, re-evacuate the shroud as described in Section 4.2. If the symptoms disappear, no further action should be required. If the symptoms remain, or reappear quickly, a vacuum leak may be present. Contact Janis Research to obtain further direction in this case.

5.4 WIRING

Occasionally a heater or thermometer wire may be broken during sample removal or installation. If this occurs, reconnect the broken wire using high melting point solder (500 K melting point). One suitable brand of solder is HMP solder from Multicore/Loctite, part number MM01007, available from Newark. Be sure to insulate the joint with shrinkable PVC tubing or Teflon insulation.

The heater located at the sample mount is designed to accept the normal output of most temperature controllers. Occasionally, however, a heater may burn out. Replacement heater kits are available from Janis, and include all materials and instructions necessary for replacement.