OPERATING INSTRUCTIONS FOR THE JANIS RESEARCH SHI-950 REFRIGERATOR SYSTEM

ь Ж

> Janis Research Company 2 Jewel Drive P.O. Box 696 Wilmington, MA 01887-0696

> > (978) 657-8750

ADDENDUM TO OPERATING INSTRUCTIONS FOR SHI-950T SYSTEM WITH SERIAL NUMBER 10,655

The following manual describes the installation and operation of Janis closed cycle refrigerator systems in general. The SHI-950T high temperature system has additional operation requirements as noted below.

OPERATION

For operation below room temperature, the cryostat should be operated per the instructions in the general manual, i.e. by controlling with the heater and sensor mounted on the sample tube. These are wired to the 19-pin electrical feedthrough mounted on the top flange.

For operation above room temperature, the sample tube must be continuously evacuated and the temperature controlled using the sensor and heater mounted on the sample positioner. The maximum operating temperature of the cryostat in this mode is 500 K.

WARNING!

Do not operate above room temperature unless the sample tube is evacuated and the refrigerator is operating. High temperature operation without the refrigerator providing cooling will result in the sample tube overheating and damage to the refrigerator and sensor.

IF THE COLD HEAD EXCEEDS 300 K IT WILL BE DAMAGED!

It is recommended that the relay feature of the temperature controller be used as a safety measure. The controller should be configured to monitor the temperature of the sample tube. If the sample tube exceeds 300 K, the relay should open and cut the power to the heater.

SHUTDOWN

In order to prevent possible overheating during shutdown, follow the steps listed below:

- 1. If operating at 325 K or below: shut off the heater power, followed by the temperature controller and refrigerator power.
- 2. If operating above 325 K:
 - A) Turn off the heater power.
 - B) Wait until the system temperature reaches 325 K or lower.
 - C) Shut off temperature controller and refrigerator.

INTRODUCTION

The Janis model SHI-950 (optical) and SHI-950T (non-optical) are closed cycle refrigerator systems designed to operate from below 5 K (SHI-950) or 4.5 K (SHI-950T) to room temperature. The cold head and compressor are manufactured by Sumitomo Heavy Industries (SHI); operating information and specifications can be found in the accompanying SHI manual.

The sample mount is suspended within an exchange gas sample tube, which in turn is connected by copper straps to the refrigerator second stage. Helium exchange gas forms a thermal link between the refrigerator and the sample; the exchange gas transfers heat from the sample to the refrigerator, cooling the sample in the process. A heater and thermometer are provided on the sample tube for varying the exchange gas temperature. A second thermometer and heater may also be provided on the sample mount, for monitoring or controlling the sample temperature more directly. The control heater and any thermometers are usually connected to an automatic temperature controller, which can be used to both display and control the sample temperature.

An aluminum radiation shield surrounds the sample area, and is cooled by the refrigerator first stage heat station. A vacuum shroud isolates the cold regions from room temperature, and may include optical access ports (SHI-950 only.) The top flange includes an electrical feedthrough for control wiring, a safety pressure relief, and a bellows sealed high vacuum valve, used to evacuate and seal the vacuum region.

An exchange gas valve is located near the top of the sample tube, and is used to introduce or remove helium exchange gas. The sample mount is suspended on a stainless steel tube, which exits through an o-ring compression seal on the sample positioner header. This aluminum header assembly also includes electrical feedthroughs for sample wiring.

INSTALLATION

MOUNTING

The SHI-950 refrigerator cold head should be mounted with the refrigerator motor and the sample positioner assembly at the top. An optional adjustable position mounting flange may be clamped to the outer vacuum jacket, and includes several mounting holes for support legs. If the refrigerator is mounted in any other orientation, the system may not achieve satisfactory minimum temperatures. The separate compressor assembly must remain upright at all times. Water-cooled compressors require cooling water hoses to be installed as described in the accompanying SHI manual.

GAS LINES

Interconnecting helium supply and return gas lines should be installed between the cold head and compressor. Tighten each fitting securely with the appropriate sized wrench. Be sure that supply and return lines do not become crossed during installation.

ELECTRICAL CONNECTIONS

Connect any thermometry cables from the cryostat electrical feedthroughs to the automatic temperature controller. If no temperature controller is supplied with the system, mating connectors are provided for attaching to a user supplied controller and cable. Plug the cold head's control power cord into the jack located on the SHI compressor back panel. Connect the temperature controller and the compressor to the appropriate AC outlets. The compressor requires 3 phase, 200VAC power, and is phase protected.

SAMPLE INSTALLATION

Remove the refrigerator top clamp, then pull the sample positioner assembly vertically out of the sample tube to gain access to the sample mount. Several types of holders are available, including optical, resistivity, and blank holders. Since the sample is cooled by exchange gas, special care in thermally anchoring the sample to the holder is not needed. Tapped holes are provided for mounting the sample or holder to the sample mount.

EVACUATION

The model SHI-950 is equipped with a bellows sealed evacuation valve, which allows evacuation and sealing of the insulating vacuum jacket. Prior to cooldown, connect a turbomolecular or diffusion pump to the valve and evacuate the shroud to a pressure of 10⁻⁵ Torr or less. Better vacuum levels provide greater insulation, resulting in shorter cooldown times and lower final temperatures. A cold-trapped mechanical vacuum pump can be used instead; however, this may limit the lowest temperature attainable. After evacuation is complete, seal the vacuum valve firmly. Outgassing and o-ring permeation will cause the pressure to rise slowly over time, therefore periodic re-evacuation will be necessary. Re-evacuation is required whenever the minimum temperature obtained begins to increase.

EXCHANGE GAS INTRODUCTION

Connect a mechanical roughing pump to the exchange gas valve, located near the top of the sample tube. Evacuate the sample tube for several minutes to a pressure of about 10^{-3} Torr, then close the valve. Connect a source of helium gas to the valve, and introduce gas through the valve until the safety pressure relief valve begins venting. Close the valve, trapping the exchange gas within the sample tube.

OPERATION

START-UP

Turn on any automatic temperature controller and observe the room temperature readings. All sensors should display values between about 285 K and 300 K, depending on the actual temperature of the surroundings. Turn on the compressor and cold head, referring to the SHI manual for proper start-up sequence. The cold head will begin cooling immediately, and the sample mount should achieve 10 K in about five hours. In order to reach the lowest temperatures the amount of exchange gas in the sample tube may be adjusted.

TEMPERATURE CONTROL

An automatic temperature controller may be used for operation in the temperature range between <5 K and 300 K. There are two heaters installed on the cryostat, either of which may be designated for use as the control heater. One is installed on the sample tube (for control of the exchange gas temperature), and the other is installed on the sample positioner (for control of the sample temperature directly). Three modes of operation are possible, as follows:

1. If the sample tube heater is chosen as the control heater, the sample is heated indirectly through contact with the helium exchange gas. For operation in this mode, connect the controller heater output to the electrical connector on the vacuum jacket top flange, and choose this thermometer as the control sensor.

Advantage: Thermal contact between the sample and the sample holder is not important for cooling the sample. The sample is surrounded by an isothermal gas, therefore the temperature is more uniform.

Disadvantage: It can take a long time for the temperature to stabilize, particularly at high temperatures.

- 2. If the sample positioner heater is chosen as the control heater, the sample is heated directly by conduction of the heater through the copper sample holder. The heater and temperature sensor are wired to a feedthrough on the top sample positioner assembly. Advantage: More rapid temperature change is possible. Disadvantage: There can be some uncertainty in the temperature measurement since the sample is surrounded by gas that is at a different temperature from the sample mount. Good thermal contact between the sample and holder is important.
- 3. Some controllers are equipped with dual heater outputs. If this type of controller is available, it is recommended that the main heater loop be connected to the sample tube heater and the secondary loop be connected to the sample positioner heater. This method combines the advantages of the above methods, while reducing the disadvantages.

Once the heater configuration has been decided upon, choose a temperature setpoint within the range 5 K - 300 K, and enter values for proportional, integral, and derivative controls. Some experimentation is required to optimize these settings for a particular application. In general, when operating at the lowest temperatures, (where the heat capacities are smallest), the proportional value should be low, and the integral values should be high. Rate control can usually remain zero throughout the operating range. As the control temperature is increased, larger proportional and smaller integral values will be required. Some temperature controllers include an autotuning function that selects appropriate proportional, integral, and rate values automatically. This function is only useful for temperatures above about 50 K, however. For complete discussion of this feature, as well as comprehensive controller operating procedures and specifications, refer to the temperature controller manual.

When operating at temperatures above 5 K, the helium exchange gas pressure will increase. This is normal, and the excess gas pressure will vent out the sample chamber safety pressure relief valve. In general, additional helium gas does not need to be introduced, unless return to the lowest possible temperature is desired.

CHANGING SAMPLES

During normal operation, the helium exchange gas pressure decreases as the temperature decreases, creating a partial vacuum in the sample tube. To remove the sample positioner assembly, first connect the helium gas source to the exchange gas valve, and raise the gas pressure until it vents through the safety pressure relief valve. Continue admitting gas through the valve, and remove the positioner assembly. Immediately cap off the sample tube to prevent air and moisture from entering and freezing within the tube. To re-install the positioner assembly, once again raise the gas pressure to atmospheric, then uncap the sample tube and insert the positioner assembly. Exchange gas may be added to reach the lowest temperature, as during the initial cooldown. Typical cooldown time to the minimum temperature is 30 to 45 minutes, depending upon the mass of the sample.

SHUTDOWN

To shut down the system, simply turn off the compressor and temperature controller. Heat can be applied to the sample mount or control heater via the temperature controller if desired, to expedite the system warm-up time to room temperature, but do not exceed 300 K or damage to the heater and thermometers may occur.

If the interconnecting gas lines must be removed for any reason, allow the system to completely stabilize at room temperature before disconnecting. Never disconnect the gas lines while the

system is cold, as dangerous pressures can develop inside the cold head. Unless the lines must be disconnected to move the system, it is best to leave them connected at all times.

÷

. .

> ۰ ۲

MAINTENANCE

SCHEDULED MAINTENANCE

The cold head requires periodic maintenance every 10,000 hours of operation. The compressor requires replacement of a charcoal adsorber every 20,000 hours of operation. Contact Janis for details of these maintenance operations. No other scheduled maintenance is required for the SHI-950 system.

UNSCHEDULED MAINTENANCE

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

VACUUM LEAKS

Condensation on the outside of the vacuum jacket and inability of the sample mount to reach 5 K may be indications of a vacuum problem. If these symptoms appear, re-evacuate the shroud as described in the evacuation paragraph above. If the symptoms disappear, no further action may 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.

WIRING

Occasionally a heater or thermometer wire may be broken during sample removal or installation. If this occurs, reconnect the broken wire using 60/40 rosin core solder. Be sure to insulate the joint with shrinkable PVC tubing or Teflon insulation.

The heater located at the sample chamber wall and sample mount is designed to accept 100 watts of power. The sample mount heater is designed to accept 25 watts output from an automatic temperature controller. Occasionally, however, a heater may burn out. Replacement heater kits are available from Janis, and include all materials and instructions necessary for replacement.

REFRIGERATOR/COMPRESSOR FAILURE

Compressor and refrigerator failures are characterized either by an inability to operate, or by an increase in the minimum achievable temperature. In either case, refer to the troubleshooting section in the SHI operating manual for suggested corrective actions. If satisfactory operation is still not achieved, contact Janis Research for further recommendations.

CRYOSTAT SERIAL NUMBER:10655

19 PIN FEEDTHROUGH LABELED "A"

- LOCATION: VACUUM SHROUD TOP FLANGE
- PIN A Ph.Br WIRE
- PIN B -Ph.Br WIRETG-120-SD CALIBRATED GaAIAs DIODE S/N 11200TO BE INSTALLED ON SAMPLE TUBE BY CUSTOMERPIN C -Ph.Br WIRENOTE: PH. BRONZE WIRE ENDS IN THE SAMPLE TUBE AREAPINS "A" AND "C" ARE TWISTED PAIRS

PINS "B" AND "D" ARE TWISTED PAIRS

- PIN D Ph.Br WIRE
- PINE -

. . .

- PINF -
- PING-
- PINH -
- PIN J -
- PINK -
- PIN L -
- PIN M -
- PIN N -
- PIN P -
- PIN R -
- PINS-
- PIN T -
- PIN U -
- 25 OHM HEATER ON SAMPLE TUBE

PIN V - (2-50 OHM HEATERS WIRED IN PARALLEL)

CRYOSTAT SERIAL NUMBER: 10655

TG-120-SD CALIBRATED GaAlAs DIODE

S/N 11199 ON SAMPLE MOUNT (SAMPLE SENSOR)

8 PIN FEEDTHROUGH LABELED "B"

LOCATION: SAMPLE POSITIONER ASSY

- PIN A POS. CURRENT (I+)
- PIN B POS. VOLTAGE (V+)
- PIN C NEG. CURRENT (I-)

PIN D - NEG. VOLTAGE (V-)

- PINE -
- PINF -
- PING-
- PINH -
- PIN J -
- 25 OHM HEATER ON SAMPLE MOUNT
- PIN K -

10-PIN FEEDTHROUGH LABLELED "C" ON SAMPLE POSITIONER ASSEMBLY SUPPLIED WITH CONNECTOR ONLY FOR CUSTOMER USE

