

CRYO INDUSTRIES

OPERATING INSTRUCTIONS

MODEL RC152

CRYOGENIC WORKSTATION

INTRODUCTION

The model RC 152 cryogenic WORKSTATION is a highly versatile variable temperature optical cryostat featuring sample in vapor flow. The RC 152 cryogenic system transfers liquid helium from a storage dewar through a highly efficient transfer line to the cryostat heat exchanger.

This RC152 has been modified to direct the flow in a donut shaped annulus. The sample is top loaded into the center allowing sample in vacuum operation. A cone interface is provided in the heat exchanger to cool the sample.

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DESCRIPTION

The typical experimental arrangement will consist of a RC 152 system, a liquid helium storage dewar, helium gas cylinder with pressure regulator, vacuum pump and temperature monitoring or control electronics.

The storage dewar insert leg inserts into the liquid helium in the storage dewar. A small pressure is used to transfer the liquid into the transfer line and directly to the heat exchanger. The flow control valve regulates the flow; adjustment is through the valve control knob. The helium flows into a heat exchanger (vaporizer) assembly and heated to the desired temperature. The sample to be cooled is suspended in the flowing vapor exiting from the heat exchanger.

Pressure inside the storage dewar is adjusted using a helium gas cylinder containing helium gas; gas of 'welding grade' purity is suitable. A gas regulator should be used, with a sensitivity of 0-5 psig. The transfer line 'leg' will seal to the 0.5 inch quick connect, located at the top of most storage dewars. The pressurization connection can be made to the storage dewar through the vent valve located on the top of the storage dewar.

The model RC 152 features quick disconnect for transfer line - WORKSTATION separation, just remove the clamp and withdraw the transfer line.

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The evacuation valve for the transfer system is located above the flow valve control knob. The RC152 UHV vacuum and the transfer line vacuum are completely independent.

The flow vent (or vapor pumping) port is located at the top of the cryostat, just below the sample positioner assembly.

Sample access is achieved by removing the sample probe from the top of the cryostat. A 2.75 inch conflat interfaces to the sample probe and secures the sample probe assembly to the cryostat. Make sure that the sample probe design allows for thermal contraction of the center tubes when cooled down.

A kapton heater is installed on the heat exchanger (vaporizer). This heater is used to increase the temperature of the flowing vapor. For best results, the control temperature sensor should be located on this heat exchanger. Two temperature sensors are the recommended configuration for the system; one mounted on the heat exchanger for control, the other located on the sample mount for display of sample temperature.

FOLLOW STANDARD AND PROPER CRYOGENIC PROCEDURES. If you have any questions, we, at CRYO Industries, are only a phone call away and would be happy to answer any questions - TEL: (603) 621-9957 (email: cryo@cryoindustries.com).

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OPERATION

1. EVACUATE THE TRANSFER LINE.

A recommended practice is to evacuate the transfer line, with a high vacuum type pumping system, prior to EACH use. Since a high vacuum system is not always accessible, your model RC 152 transfer line contains a charcoal cryopump which allows the system to operate with a mechanical 'roughing' pump evacuation.

2. EVACUATE WORKSTATION.

Attach the vacuum pump to your vacuum chamber. Remember that the cryostat (WORKSTATION) vacuum, transfer system (line) vacuum and sample tube are completely separate volumes.

3. CHECK FLOW CONDUCTANCE & PURGE TRANSFER LINE.

Open the helium throttle valve by turning counter-clockwise the flow valve operator, approximately 5 turns. Attach a hose from the helium gas cylinder to the exit end of the transfer line. Flow helium gas through the system. Check for helium exiting at the flow control valve. The exiting gas indicates that an open flow system is present; In addition, the transfer line will be purged with helium gas which should prevent any non desirable freezing from occurring. Remove the gas hose from the transfer line. CLOSE THE VALVE CONTROL KNOB 'FINGER' TIGHT, DO NOT OVER-TIGHTEN.

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4. PURGE SAMPLE REGION.

Attach the helium gas hose to the transfer line inlet port on the WORKSTATION. Flow helium gas through the sample tube. Check for gas exiting at the vent port.

5. INSERT TRANSFER LINE LEG INTO STORAGE DEWAR.

Insert the transfer line into the neck of the storage dewar. Loosely seal the line to the storage dewar quick connect. SLOWLY lower the leg into the ullage space of the storage dewar. Open the flow control valve 3-5 turns. Continue to SLOWLY lower the transfer line leg. If liquid helium vents strongly, stop lowering the leg and hold the height position until the liquid helium settles down; then, continue SLOWLY lowering until completely inserted. At this point two conditions are possible concerning the storage dewar; follow the procedure corresponding to your status. (i). The storage dewar vent is open to atmosphere. Feel for helium gas to exit the storage dewar caused by the transfer line leg entering the liquid. Temporarily, hold the line at this position. Allow the exiting gas to cool the leg section above the liquid. Check the flow control knob to assure that it has not frozen and still rotates. When the venting settles down, continue to slowly insert the leg. Upon reaching the bottom, pull the leg up approximately 0.5" and tightly seal the leg. Attach the helium gas hose to the storage dewar and pressurize to approximately 1.0 psig. (ii). The storage dewar is pressurized and the leg was inserted through a valve, maintaining storage dewar pressure. SLOWLY lower the transfer line leg into the storage dewar. Check the flow control knob to assure that it has not frozen and still rotates. Continue to slowly insert the leg AND CHECK SYSTEM FOR FLOW. Upon reaching the bottom, pull the leg up approximately 0.5" and tightly seal the leg. Attach the helium

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gas hose to the storage dewar. A pressure between 1 to 5 psig is normal. Pressure setting is not critical. Adjust the pressure if it is outside of the normal range.

6. CHECK SYSTEM FOR FLOW.

The valve control knob should be maintained 3-5 turns open. Feel for helium gas exiting the transfer system, at the outlet end of the transfer line. Gas exiting indicates an open flow system is present, i.e., nothing has frozen in the valve or transfer line. Connect the transfer system to the RC 152 WORKSTATION. The transfer line inserts into the transfer line coupling at the top of the WORKSTATION. Install the supplied **gasket between the mating flanges** and secure with the quick connect clamp. Note: the transfer line can be connected to the WORKSTATION at any prior point, should this be preferable. If the transfer line is connected, check for exiting flow at the sample region port.

7. COOLDOWN.

MAKE SURE that the valve control knob is rotated open 3-5 turns, counter-clockwise [looking down onto the system from above]. The heat exchanger and sample will begin to cool. After sample cooldown, the flow valve knob should be rotated to approximately 1 turn open; fine adjust for minimum liquid helium flow along with sufficient cooling power.

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8. CHECK STORAGE DEWAR PRESSURE.

Maintain a check on storage dewar pressure, adjust if necessary. Exact pressure settings are not required. The nominal operating pressure is 1 psig. Check periodically during the operation and adjust if needed.

9. VARIABLE TEMPERATURE OPERATION.

Attach the temperature controller or power supply to the electrical feedthrough wired to the heat exchanger heater. Fine adjust the helium flow for the minimum cooling power required. The temperature can be varied from 5 to 300 K by adjusting only the power input to the heater. The rate of temperature increase will vary with the power supplied by the control heater; DO NOT EXCEED 50 WATTS. Lower temperatures can be obtained by turning off the electrical current supplied to the heater. If desired, more rapid temperature reduction can be obtained by increasing the flow rate. Flow settings may be returned to the previous steady state values as the new desired temperature coordinate is approached.

Users of dual heater temperature controllers (Model 32B or similar) should use the main heater on the heat exchanger and the secondary heater on the sample holder.

The most difficult region to obtain stable temperatures is between 6 and 15 K where two phase flow may be present. If difficulty is observed, turn off the heater power. Adjust the flow rate until the temperature is slightly below the desired temperature coordinate, then use the heater to obtain the required temperature. Monitor the sample temperature at the sample, a temperature differential between the heat exchanger and sample mount is normal. Attaching the liquid helium storage dewar to a helium gas cylinder with a two-stage regulator can help increase temperature stability. Adjust the regulator to the desired

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pressure setting and use it to maintain continuously the pressure in the storage dewar at a constant setting. The regulator will add helium gas to the storage dewar if the pressure drops.

10. ALTERNATE PROCEDURE, VARIABLE TEMPERATURE [10 TO 300 K].

When operating above 10 K, an alternate procedure for operation may be followed which will conserve liquid helium. Increase the temperature by reducing the flow rate using the flow control knob. Select a temperature just below but as close as easily possible to the desired set point. Use small current input(s) to the heater(s) to fine tune the temperature. The minimum liquid helium flow rate and the heat exchanger control heater will maintain the required temperature setting. The flow rate should be reduced further as the temperature is increased.

11. OPERATION BELOW 4.2 K.

Two modes of operation are possible below 4.2 K - (a). sample in vapor and (b). sample in liquid.

(a). Attach the mechanical pump to the sample tube. While pumping the sample tube, adjust the liquid helium flow to set your desired temperature. This method does limit the lowest achievable temperature of the system.

(b). Turn off any heat exchanger power. Open the flow valve control two - four turns. The sample tube will fill with liquid helium. If your system has a buffer volume, this volume will fill with liquid helium. Alternately, you can pump the liquid helium while

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filling the buffer volume. After filling sample and buffer volume, the flow valve should be set to maintain the buffer volume and sample tube full while the 'roughing' pump reduces the temperature. The flow should be fine adjusted to replenish the normal pump down losses which occur during this cooling process. Monitor the buffer volume using the liquid helium level indicator readout, if your system is equipped with this option. Obtain the lowest equilibrium temperature - while a volume replenishing flow is maintained; then, CLOSE the flow valve. At this point, your pump will begin to bring the system to the lowest terminal temperature. Switch the level meter to the sample and hold position to reduce the heat input from the probe.

The temperature of the radiation shield may be important; for operation immediately after cooldown, the radiation shield may not have had sufficient time to cool which can reduce the buffer volume hold time.

For experienced users, an optional pre-cool of the sample region with liquid nitrogen can be done by maintaining the level above the radiation shield thermal anchor position. Remove all liquid nitrogen before transferring Liquid helium; one method for removal is to slightly over-pressure the sample tube with the transfer line inlet open. The liquid nitrogen will be 'pushed' out the transfer line inlet. A simpler method of operation may be used if the time at the lowest terminal temperature remains sufficiently long when the liquid helium pump down losses are not replenished. QUICK & SIMPLE - turn off any heat exchanger power. Open the flow valve operator three-five turns. Fill the sample region with liquid helium, AFTER WHICH IMMEDIATELY CLOSE THE FLOW

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VALVED. Pump on the sample tube. The roughing pump will bring the system to the lowest possible temperature.

12. SHORT TERM SHUT DOWN. If a new experiment is to begin shortly after completion of a 'run', the transfer line insert leg can remain in the storage dewar. The helium flow valve should be closed. If preferred, the RC 152 WORKSTATION can be removed from the transfer line. The storage dewar pressure can remain. Cap off or continuously evacuate the sample tube. Restart the system by opening the helium flow valve and unsealing the vent port when atmospheric pressure is reached.

13. SHUT DOWN.

Turn off all power to the heat exchanger. Close the flow control valve. Disconnect transfer line from the WORKSTATION and cap off the inlet. If the storage dewar has a provision for removing the transfer line insert leg without de-pressurization, remove the line. Otherwise, de-pressurize the storage dewar. BE CAREFUL - during pressure reduction, helium gas will vent from the storage dewar. AVOID CONTACT WITH THE COLD GAS. After the storage dewar returns to atmospheric pressure, remove the transfer line insert leg.

Continuously evacuate the sample region of the WORKSTATION until the system returns to room temperature. If it is not possible to continue operating the mechanical pump, seal off the sample zone. The system will vent, as it warms, through the positive relief valve which protects the sample tube. Do not allow air into a COLD sample.

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GENERAL NOTES AND SAFETY PRECAUTIONS

1. It is recommended that the transfer line and cryostat be evacuated prior to EACH new use with a diffusion type pumping system. Prior overnight 'rough' pumping of the transfer line is beneficial.
2. Heater input power should not exceed 50 watts. Do not apply power to the heater without cryogen flowing.
3. The sample should not be heated to a temperature greater than 325 K.
4. Make sure that the storage dewar helium reservoir is equipped with a safety pressure relief. If needed, install a relief off the helium gas hose.
5. Never 'break' vacuum with helium gas. Remember the WORKSTATION and transfer line vacuums are completely separate.
6. Do not bend transfer line to less than a 12 inch radius.
7. Avoid contact with the cold gases.
8. The insulating vacuum spaces are protected from over pressure by safety reliefs installed at the factory. The relief has been pre-set at the factory. NEVER SET TO A HIGHER PRESSURE.
9. If pumping on the liquid helium to operate below 4.2 K, do not use, if possible, the same vacuum pump for the insulating vacuum of the dewar. Large amounts of helium may temporarily contaminate the roughing pump causing helium to backstream into the insulating vacuum space of the dewar.

Curve DT-670



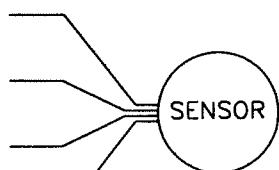
Standard Curve DT-670: Measurement Current = 10 $\mu\text{A} \pm 0.05\%$

| T (K) | Voltage (V) | dV/dT (mV/K) | T (K) | Voltage (V) | dV/dT (mV/K) | T (K) | Voltage (V) | dV/dT (mV/K) |
|-------|-------------|--------------|--------|-------------|--------------|--------|-------------|--------------|
| 1.20 | 1.646540 | -9.87 | 18.00 | 1.228300 | -15.25 | 125.00 | 0.939242 | -1.96 |
| 1.40 | 1.644290 | -12.49 | 18.50 | 1.220700 | -15.18 | 130.00 | 0.929390 | -1.98 |
| 1.60 | 1.641570 | -14.79 | 19.00 | 1.213110 | -15.20 | 135.00 | 0.919446 | -2.00 |
| 1.80 | 1.638370 | -17.15 | 19.50 | 1.205480 | -15.34 | 140.00 | 0.909416 | -2.01 |
| 2.00 | 1.634720 | -19.30 | 20.00 | 1.197748 | -15.63 | 145.00 | 0.899304 | -2.03 |
| 2.20 | 1.630670 | -21.14 | 21.00 | 1.181548 | -16.98 | 150.00 | 0.889114 | -2.05 |
| 2.40 | 1.626290 | -22.61 | 22.00 | 1.162797 | -21.11 | 155.00 | 0.878851 | -2.06 |
| 2.60 | 1.621660 | -23.63 | 23.00 | 1.140817 | -20.77 | 160.00 | 0.868518 | -2.07 |
| 2.80 | 1.616870 | -24.16 | 24.00 | 1.125923 | -9.42 | 165.00 | 0.858120 | -2.09 |
| 3.00 | 1.612000 | -24.67 | 25.00 | 1.119448 | -4.60 | 170.00 | 0.847659 | -2.10 |
| 3.20 | 1.606970 | -25.63 | 26.00 | 1.115658 | -3.19 | 175.00 | 0.837138 | -2.11 |
| 3.40 | 1.601730 | -26.80 | 27.00 | 1.112810 | -2.58 | 180.00 | 0.826560 | -2.12 |
| 3.60 | 1.596260 | -27.91 | 28.00 | 1.110421 | -2.25 | 185.00 | 0.815928 | -2.13 |
| 3.80 | 1.590570 | -28.99 | 29.00 | 1.108261 | -2.08 | 190.00 | 0.805242 | -2.14 |
| 4.00 | 1.584650 | -30.21 | 30.00 | 1.106244 | -1.96 | 195.00 | 0.794505 | -2.15 |
| 4.20 | 1.578480 | -31.59 | 31.00 | 1.104324 | -1.88 | 200.00 | 0.783720 | -2.16 |
| 4.40 | 1.572020 | -32.91 | 32.00 | 1.102476 | -1.82 | 210.00 | 0.762007 | -2.18 |
| 4.60 | 1.565330 | -33.97 | 33.00 | 1.100681 | -1.77 | 220.00 | 0.740115 | -2.20 |
| 4.80 | 1.558450 | -34.74 | 34.00 | 1.098930 | -1.73 | 230.00 | 0.718054 | -2.21 |
| 5.00 | 1.551450 | -35.25 | 35.00 | 1.097216 | -1.70 | 240.00 | 0.695834 | -2.23 |
| 5.20 | 1.544360 | -35.60 | 36.00 | 1.095534 | -1.67 | 250.00 | 0.673462 | -2.24 |
| 5.40 | 1.537210 | -35.92 | 37.00 | 1.093878 | -1.64 | 260.00 | 0.650949 | -2.26 |
| 5.60 | 1.530000 | -36.22 | 38.00 | 1.092244 | -1.62 | 270.00 | 0.628302 | -2.27 |
| 5.80 | 1.522730 | -36.48 | 39.00 | 1.090627 | -1.61 | 273.15 | 0.621141 | -2.28 |
| 6.00 | 1.515410 | -36.71 | 40.00 | 1.089024 | -1.60 | 280.00 | 0.605528 | -2.28 |
| 6.50 | 1.496980 | -36.86 | 42.00 | 1.085842 | -1.59 | 290.00 | 0.582637 | -2.29 |
| 7.00 | 1.478680 | -36.21 | 44.00 | 1.082669 | -1.59 | 300.00 | 0.559639 | -2.30 |
| 7.50 | 1.460860 | -35.00 | 46.00 | 1.079492 | -1.59 | 305.00 | 0.548102 | -2.31 |
| 8.00 | 1.443740 | -33.42 | 48.00 | 1.076303 | -1.60 | 310.00 | 0.536542 | -2.31 |
| 8.50 | 1.427470 | -31.67 | 50.00 | 1.073099 | -1.61 | 320.00 | 0.513361 | -2.32 |
| 9.00 | 1.412070 | -29.95 | 52.00 | 1.069881 | -1.61 | 330.00 | 0.490106 | -2.33 |
| 9.50 | 1.397510 | -28.31 | 54.00 | 1.066650 | -1.62 | 340.00 | 0.466760 | -2.34 |
| 10.00 | 1.383730 | -26.84 | 56.00 | 1.063403 | -1.63 | 350.00 | 0.443371 | -2.34 |
| 10.50 | 1.370650 | -25.51 | 58.00 | 1.060141 | -1.64 | 360.00 | 0.419960 | -2.34 |
| 11.00 | 1.358200 | -24.31 | 60.00 | 1.056862 | -1.64 | 370.00 | 0.396503 | -2.35 |
| 11.50 | 1.346320 | -23.20 | 65.00 | 1.048584 | -1.67 | 380.00 | 0.373002 | -2.35 |
| 12.00 | 1.334990 | -22.15 | 70.00 | 1.040183 | -1.69 | 390.00 | 0.349453 | -2.36 |
| 12.50 | 1.324160 | -21.17 | 75.00 | 1.031651 | -1.72 | 400.00 | 0.325839 | -2.36 |
| 13.00 | 1.313810 | -20.25 | 77.35 | 1.027594 | -1.73 | 410.00 | 0.302161 | -2.37 |
| 13.50 | 1.303900 | -19.41 | 80.00 | 1.022984 | -1.75 | 420.00 | 0.278416 | -2.38 |
| 14.00 | 1.294390 | -18.63 | 85.00 | 1.014181 | -1.77 | 430.00 | 0.254592 | -2.39 |
| 14.50 | 1.285260 | -17.92 | 90.00 | 1.005244 | -1.80 | 440.00 | 0.230697 | -2.39 |
| 15.00 | 1.276450 | -17.31 | 95.00 | 0.996174 | -1.83 | 450.00 | 0.206758 | -2.39 |
| 15.50 | 1.267940 | -16.77 | 100.00 | 0.986974 | -1.85 | 460.00 | 0.182832 | -2.39 |
| 16.00 | 1.259670 | -16.31 | 105.00 | 0.977650 | -1.88 | 470.00 | 0.159010 | -2.37 |
| 16.50 | 1.251610 | -15.94 | 110.00 | 0.968209 | -1.90 | 480.00 | 0.135480 | -2.33 |
| 17.00 | 1.243720 | -15.64 | 115.00 | 0.958657 | -1.92 | 490.00 | 0.112553 | -2.25 |
| 17.50 | 1.235960 | -15.41 | 120.00 | 0.949000 | -1.94 | 500.00 | 0.090681 | -2.12 |

10 PIN FEEDTHROUGH INTERNAL WIRING FOR TWO SENSORS AND ONE HEATER

DATE: ----- JUNE 27, 2003
 LAB TECH. NAME --- MARC POIRIER

JOB NO: ----- 3664
 CUSTOMER: ----- UNIVERSITY OF ALBERTA (WITH TRIUMF)
 SYSTEM DESCRIPTION: --- 152-1797-SV (CONT. FLOW UHV CRYOSTAT)
 FEEDTHROUGH LOCATION: --- MINI 1.33" CONFLAT

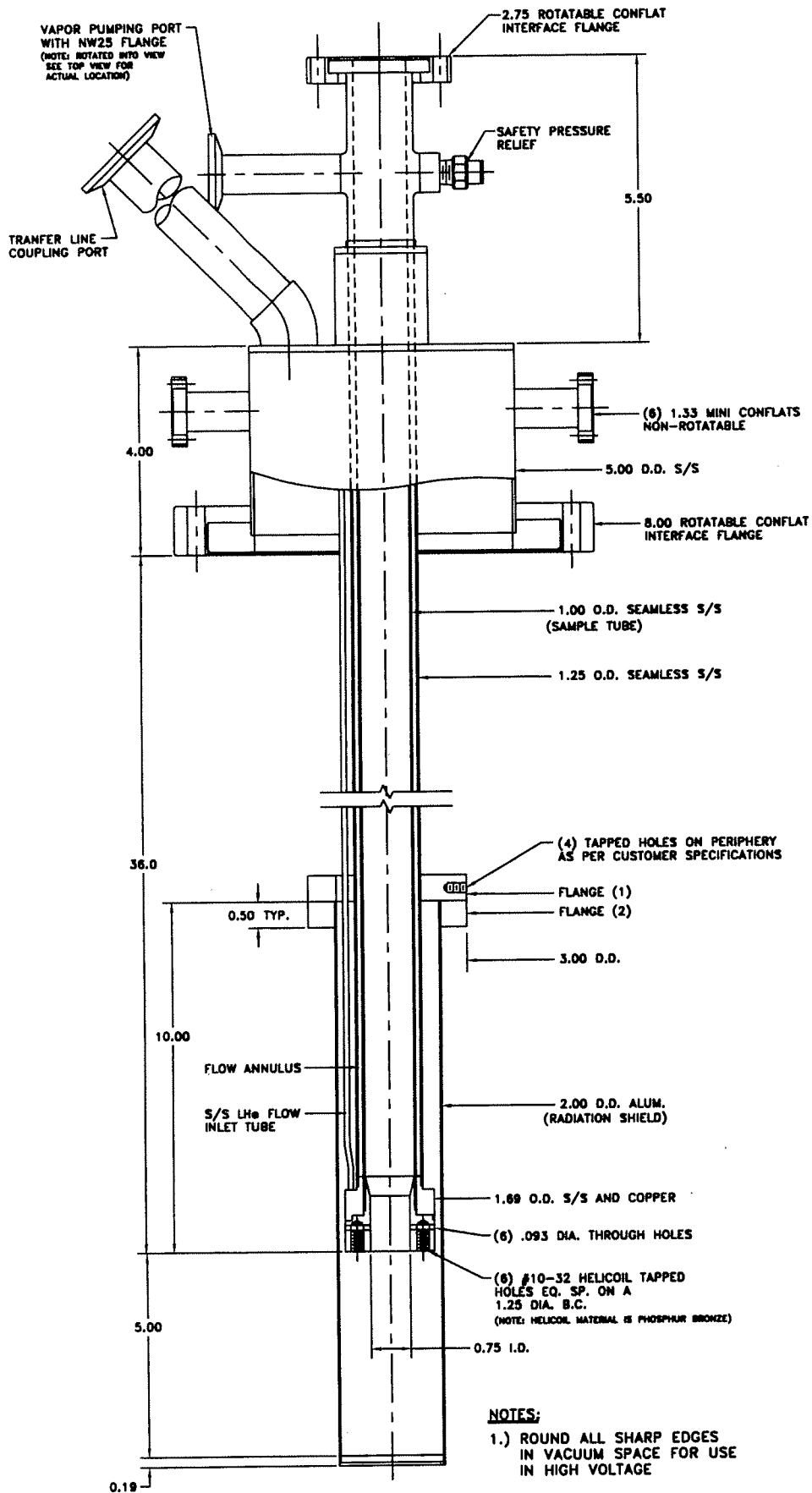
| PINS | INTERNAL WIRES | ITEM | SENSOR INFORMATION |
|------|----------------|---|---|
| A | I+ BLUE |  | TYPE: --- SILICON DIODE |
| B | V+ CLEAR | | MODEL NO.: DT-670B-SD |
| C | V- GREEN | | SERIAL NO.: D56240 |
| D | I- RED | | CURVE NO.: LOCATION: VAPORIZER ADAPTER |

| PINS | INTERNAL WIRES | ITEM | SENSOR INFORMATION |
|------|----------------|---|---|
| E | I+ BLUE | | TYPE: --- SILICON DIODE (SPARE) |
| F | V+ CLEAR | | MODEL NO.: DT-670B-SD |
| J | V- GREEN | | SERIAL NO.: D56248 |
| K | I- RED | | CURVE NO.: LOCATION: VAPORIZER ADAPTER |

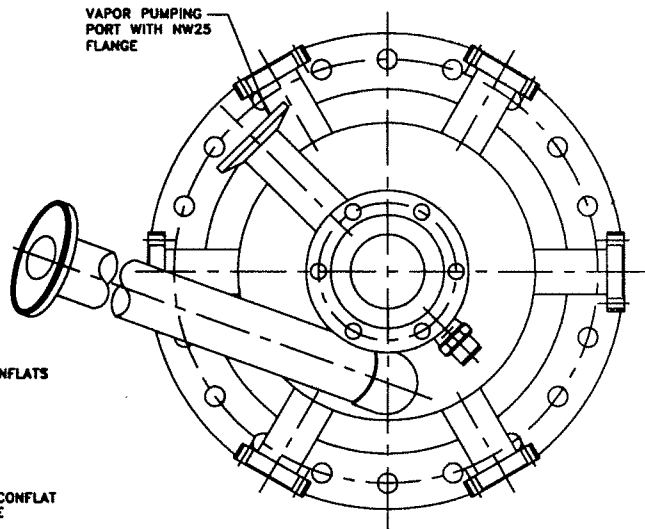
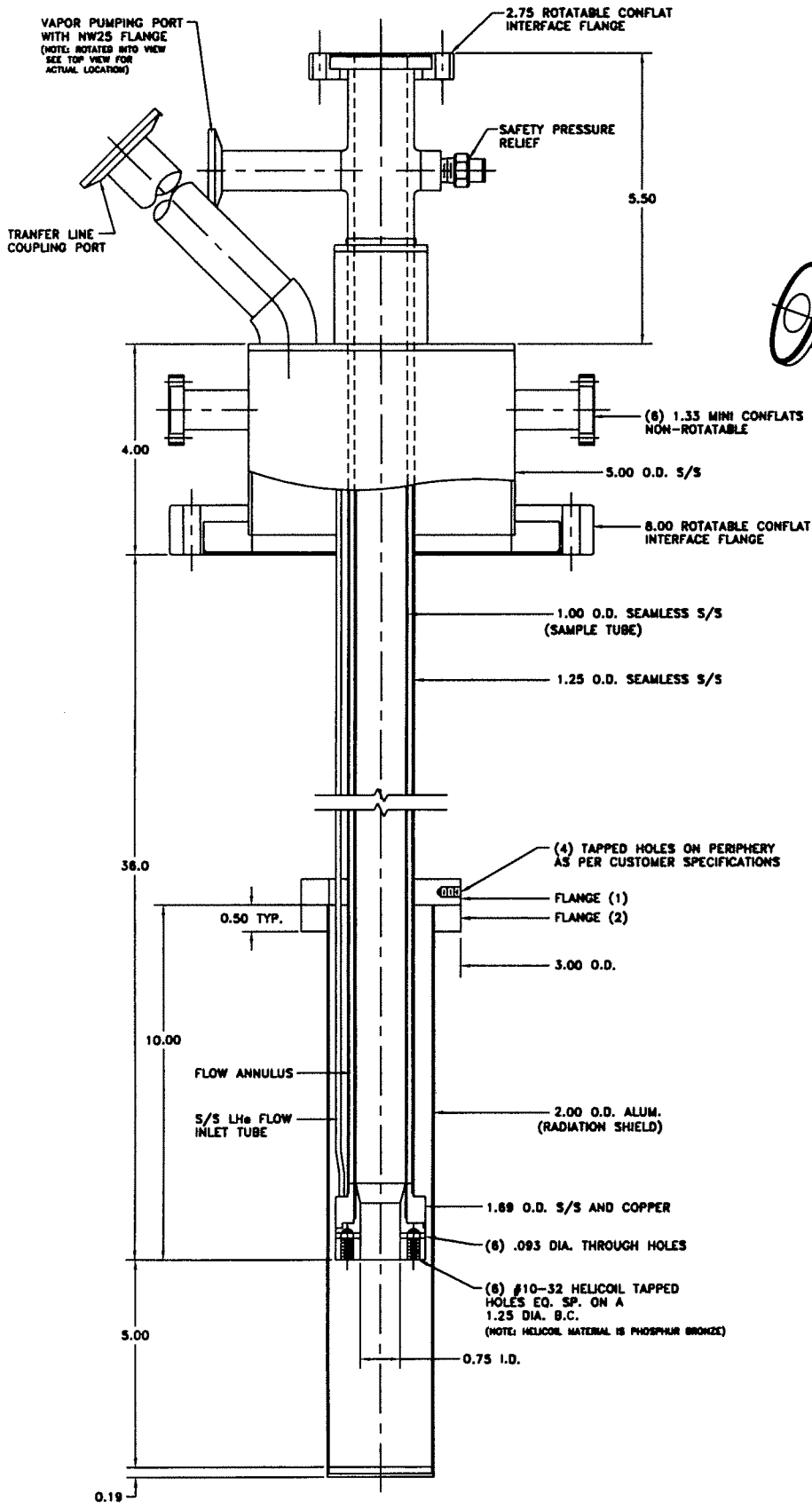
| PINS | INTERNAL WIRES | ITEM | HEATER INFORMATION |
|------|--|--------|---------------------------------|
| G | | HEATER | TYPE: --- RIBBON HEATERS |
| H | | | RESISTANCE: (2) @ 25 OHMS EACH |
| | | | LOCATION: --- VAPORIZER ADAPTER |

NOTES: THE RIBBON HEATERS WERE WIRED IN SERIES TO EQUAL 50 OHMS

DWG#: 3664 (WR-403)
 (FORMERLY DWG: 10-PIN)



| | | | | |
|--|---------------------|-------------|---------|-----|
| CRYO Industries of America Inc. 11124 So. Willow St., Manchester N.H. 03103 | | DWG. NO. | | |
| | | 152-1797-SV | | |
| TITLE: SPECIAL MODEL RC152 CRYOGENIC WORKSTATION | | REV. | DATE | BY |
| | | 1 | 1-28-03 | GJS |
| | | 2 | 2-07-03 | OC |
| | | 3 | 3-13-03 | GJS |
| DWN. BY: GJS | SCALE: NTS | 4 | 3-18-03 | GJS |
| CHKD. BY: OC | DATE: JAN. 17, 2003 | | | |



TOP VIEW

NOTES:
 1.) ROUND ALL SHARP EDGES IN VACUUM SPACE FOR USE IN HIGH VOLTAGE

| | | | |
|--|--|---------------------|-------------|
| CRYO | | DWG. NO. | |
| Industries of America Inc. | | 152-1797-SV | |
| 11124 So. Willow St., Manchester N.H. 03103 | | REV. | DATE BY |
| TITLE: SPECIAL MODEL RC152 CRYOGENIC WORKSTATION | | 1 | 1-28-03 GJS |
| | | 2 | 2-07-03 OC |
| | | 3 | 3-13-03 GJS |
| DWN. BY: GJS | | 4 | 3-18-03 GJS |
| SCALE: NTS | | DATE: JAN. 17, 2003 | |
| CHKD. BY: OC | | | |