OPERATING INSTRUCTIONS
FOR THE
JANIS RESEARCH
SUPERTRAN-VP
CRYOSTAT SYSTEM

SERIAL NUMBER: 18300

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INTRODUCTION

The Janis Research Company's SuperTran-VP continuous flow cryostat combines a flexible transfer line with a cryostat to provide quick refrigeration from below 2 K to 300 K. Liquid helium is continuously transferred through a vacuum jacketed, superinsulated flexible transfer line to a vaporizer/heat exchanger at the bottom of the sample chamber. The liquid is then vaporized and heated to some specified temperature, and travels upwards to cool the sample and intercept the heat loads coming down the sample tube. A needle valve located at the bottom of the leg (which goes inside a storage dewar), controls the cryogen flow to the sample mount.

Temperatures below 4.2 K are achieved by reducing the pressure (using a mechanical vacuum pump) at the sample tube vent/pumping port. Temperatures above 4.2 K are obtained by sending an appropriate current through a bifilarly wound heater at the vaporizer. This heater, as well as any control thermometer supplied will be wired to an electrical feedthrough at the top of the cryostat vacuum jacket. An optional top loading sample positioner/sample mount assembly, containing an additional heater may be supplied with the system. The heater on this sample mount may be used for additional control.

UNPACKING AND SET-UP

After removal of the protective padding and shipping supports, the cryostat can be visually inspected for any damage incurred during shipment. Please report any visible damage to the crate or cryostat to your shipping/receiving department, and to Janis Research as soon as it is noticed.

Four tapped holes (usually ¼-20) are provided with standard systems, at the bottom of the vacuum jacket for rigid attachment to a laboratory bench. The cryostat head can also be supported by a standard laboratory stand and clamp(s) assembly, while the SuperTran leg may be supported inside the storage dewar.

PREPARATION FOR COOLDOWN

The SuperTran-VP is shipped with the transfer line vacuum space and the cryostat vacuum space evacuated. This is a result of the final testing at the factory, and it helps ensure clean vacuum spaces. As a precaution against deterioration of the vacuum, which arises sometimes during transit or a prolonged storage period, both vacuum spaces should be re-evacuated prior to use. This is best done with a good pumping station (e.g., a turbomolecular pumping station) capable of bringing the ultimate pressure down to approximately 10^{-5} Torr. Refer to the enclosed engineering drawing for the location of the evacuation (bellow sealed) valves. After evacuation, all valves should be firmly closed, but care should be exercised to avoid damaging the seat with too much pressure.

The vacuum jackets are protected against cold leaks with a pressure relief valve, which will vent any pressure that exceeds 2 to 4 psig. The transfer line pressure relief is located opposite the evacuation valve, while the shroud pressure relief is located on top of the cryostat vacuum jacket.

When evacuation of either vacuum space is initiated, always be sure that the pressure on the pump side of the evacuation valve is lower than the pressure in that vacuum space. This is done to avoid drawing oil vapor from the pump into the vacuum space. Thus, one should not pump any vacuum space while liquid helium is passing through the inner line, since the liquid helium could cryopump to a lower pressure than the pumping station in use.

The rigid leg of the transfer line has a built-in activated charcoal getter to help maintain excellent vacuum when this leg is inserted in the cryogen storage dewar. Thus, it is preferable to maintain this space under vacuum at all times and never allow helium gas or moist air inside this space. In the event moisture or helium does accidentally enter the space, a pumping station should be attached to the space for several days in order to bring the pressure down to an acceptable level.

SAMPLE MOUNTING AND INSTRUMENTATION

Most systems are supplied with a sample positioner/sample mount assembly that is top-loaded into the cryostat. This positioner generally contains two electrical feedthroughs for wiring a heater, any sample thermometer supplied and any other instrumentation desired. The positioner allows translation (±0.5") and rotation (±180°) along the axis of the cryostat.

The copper sample mount has tapped holes to allow attachment of a variety of sample holders, thus locating the sample in the flowing helium vapor. The sample need not be thermally anchored to the holder or mount, since the holder provides no cooling or heating to the sample. Any thermometer placed in the vicinity of the sample should give a very good indication of the sample's temperature since both are being simultaneously cooled by the same helium vapor.

COOLDOWN

1. Close the needle valve at the bottom of the transfer line (storage dewar leg), by turning the knurled knob flow valve regulator (turn counter-clockwise when viewed from top).

2. Insert the "male" bayonet of the transfer line into the cryostat section and (hand) tighten the o-ring compression sealing knurled nut. Push the transfer line firmly into the female bayonet to ensure a good mechanical joint. After cooling the cryostat push the transfer line into the bayonet connection again, and re-tighten the o-ring compression seal to be sure that the brass tip fits snugly into the cryostat.

3. Attach a mechanical vacuum pump to the sample tube vent/pumping port and evacuate this space continuously during the first phase of cooldown. This will evacuate the sample tube and the inner line, and provide a path for the cryogen that is free from air and moisture.

4. Insert the SuperTran rigid leg (usually 0.5" OD) into a storage dewar very slowly, while allowing the cryogen vapor to vent out of the dewar. As the foot enters into the liquid cryogen, open the flow valve and close it three of four times, and listen to the "gurgle" of the pump each time the valve is opened. This ensures that the valve does not freeze shut during cooldown, and that the inner line is not blocked. After the leg bottoms out, lift it about 1 cm to avoid any frozen debris at the bottom

5. Using pressurized helium gas, pressurize the storage container to about 1 psig. Please note that it is easier (and less wasteful) to increase the pressure in the storage dewar than to decrease it.

6. Open the flow regulator about two turns, and listen to the gurgle of the pump as the cryogen starts to flow through the inner line.

7. Monitor the sample mount or vaporizer temperature and/or listen to the vacuum pump carefully. In 5-15 minutes, the inner line and cryostat head will cool down (slowly at first, then very rapidly) to helium temperature. This time can be decreased by increasing both the flow valve opening (about 4 turns) and the pressure in the storage dewar (about 4 psi). At this time the vacuum pump should be disconnected from the system, and the escaping vapor vented into the atmosphere (or a gas collecting system). Be sure to allow the pump to reach atmospheric pressure before disconnecting, to avoid introducing air and moisture into the line.

8. Check that the pressure inside the storage dewar is between 1 and 4 psi, and maintain this pressure throughout this procedure.

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 – 4 above.

1. Open the needle valve at the bottom of the transfer line (storage dewar leg) by turning the knurled knob flow valve regulator (turn clockwise when viewed from top).

2. Insert the SuperTran rigid leg (usually 0.5" O.D.) into a storage dewar very slowly and tighten the brass o-ring seal nut to form a gas tight seal. After the leg bottoms out, lift it about 1 cm to avoid any frozen debris at the bottom. Note that you will not be able to feel any gas exiting the transfer line bayonet.

3. Insert the "male" bayonet of the transfer line into the cryostat section and (hand) tighten the o-ring compression sealing knurled nut. Push the transfer line firmly into the female bayonet to ensure a good mechanical joint. After cooling the cryostat push the transfer line into the bayonet connection again, and re-tighten the o-ring compression seal to be sure that the brass tip fits snugly into the cryostat.

4. Close the needle valve and attach a mechanical vacuum pump to the sample tube vent/pumping port. This will evacuate the sample tube and the inner line, and provide a path for the cryogen that is free from air and moisture. Open the flow valve and close it three or four times, and listen to the "gurgle" of the pump each time the valve is opened. This ensures that the valve does not freeze shut during cooldown, and that the flow is not restricted.

OPERATION

For operation at 4.2 K, the flow valve should be opened just enough to maintain this temperature at the sample mount. The necessary (minimum) flow required will be a function of the heat load into the cold finger.

For temperatures above 4.2 K, using either a manual power supply or an automatic temperature controller, send a current through the vaporizer heater (about 100 milliamps at 4.2 K) and monitor the temperature of the sample mount. After a short lag, the temperature of the sample mount will increase and then stabilize at a value determined by the heater input. IT IS IMPORTANT TO MAKE SURE THAT HELIUM IS DEFINITELY FLOWING THROUGH THE VAPORIZER PRIOR TO PASSING ANY CURRENT THROUGH THIS HEATER. IF NO FLOW EXISTS, THE VAPORIZER'S TEMPERATURE CAN INCREASE VERY RAPIDLY CAUSING HEATER BURNOUT OR DAMAGE TO THE JOINTS.

Adjustments in cryogen flow can be made at any time either to increase the cooling power (just above the cryogen boiling point), or to reduce the cryogen consumption at higher temperatures. In general, however, there is no need to change the settings of either item after the initial set-up. Control can then be entirely made through an automatic temperature controller or a manual power supply. In either case, the sample mount temperature should never exceed 300 K.

Operation below 4.2 K is achieved by reducing the pressure at the sample tube vent/pumping port with a mechanical vacuum pump. The flow valve may be partially opened (less than ¼ turn), thus developing a pressure gradient at the needle valve and continuously transferring cold helium vapor to the sample tube. This configuration provides cooling (below 4.2 K) for extended periods of time. Lower temperatures can be achieved by filling the sample tube, closing the flow valve and then reducing the pressure on top of this helium. The low temperature will be maintained until all the superfluid helium is depleted from the sample tube. A slightly higher temperature will be reached if the needle valve is throttled to continuously replenish the liquid helium in the sample tube.

In order to change samples (temperatures above 4.2 K and above), it is only necessary to remove the sample holder from the top of the sample tube and then reinsert the holder with the new sample. During this time, the helium vapor flow in the sample tube should be maintained, or even increased, in order to preclude the admission of any air to the sample zone. When operating below 4.2 K, the temperature should be brought back to 4.2 K and the pressure to 760 Torr before removing the sample holder.

SHUT DOWN

Upon the completion of the experiment, the needle valve at the bottom of the SuperTran leg should be closed, as should the sample tube vent/pumping port. This prevents any cryogen from reaching the sample mount and allows any cryogen remaining in the inner line or sample tube to vent safely outside the cryostat via the pressure relief valve at the top of the sample tube, while stopping any air or moisture from entering. The storage dewar should then be de-pressurized, and the SuperTran leg removed in order to reduce the heat input into the liquid inside the dewar.

OPERATIONAL TECHNIQUES

In order to maintain the high performance of this cryostat, it is recommended that the following suggestions and precautions be followed:

1. Always maintain the utmost cleanliness inside the vacuum shroud. Any elastomer seals should be very lightly coated with a high vacuum grease, and evacuation of this space is best done using a turbomolecular pumping station.

2. It is preferable to maintain a vacuum in the flexible line vacuum space and cryostat vacuum space at all times. Evacuate the cryostat prior to each use with a turbomolecular pumping station. The transfer line will need to be evacuated periodically, and this should be done while the inner line is at room temperature. Helium gas and moist air should never be allowed inside these spaces.

3. Do not heat the vaporizer or sample mount to a temperature above 300 K, unless the cryostat is specially designed for this purpose.

4. Do not bend the flexible transfer line to a radius of less than 12 inches (30 cm).

5. Do not over tighten the needle (flow control) valve at the bottom of the transfer leg.

6. Do not remove the brass bayonet tip from the transfer line.

7. Do not exceed a voltage of 15 volts across the heater when using a power supply for manual temperature control.

8. It is preferable to have an anti-oscillation device on your helium storage dewar, and keep the end of the transfer leg about one centimeter above the bottom of the storage dewar.

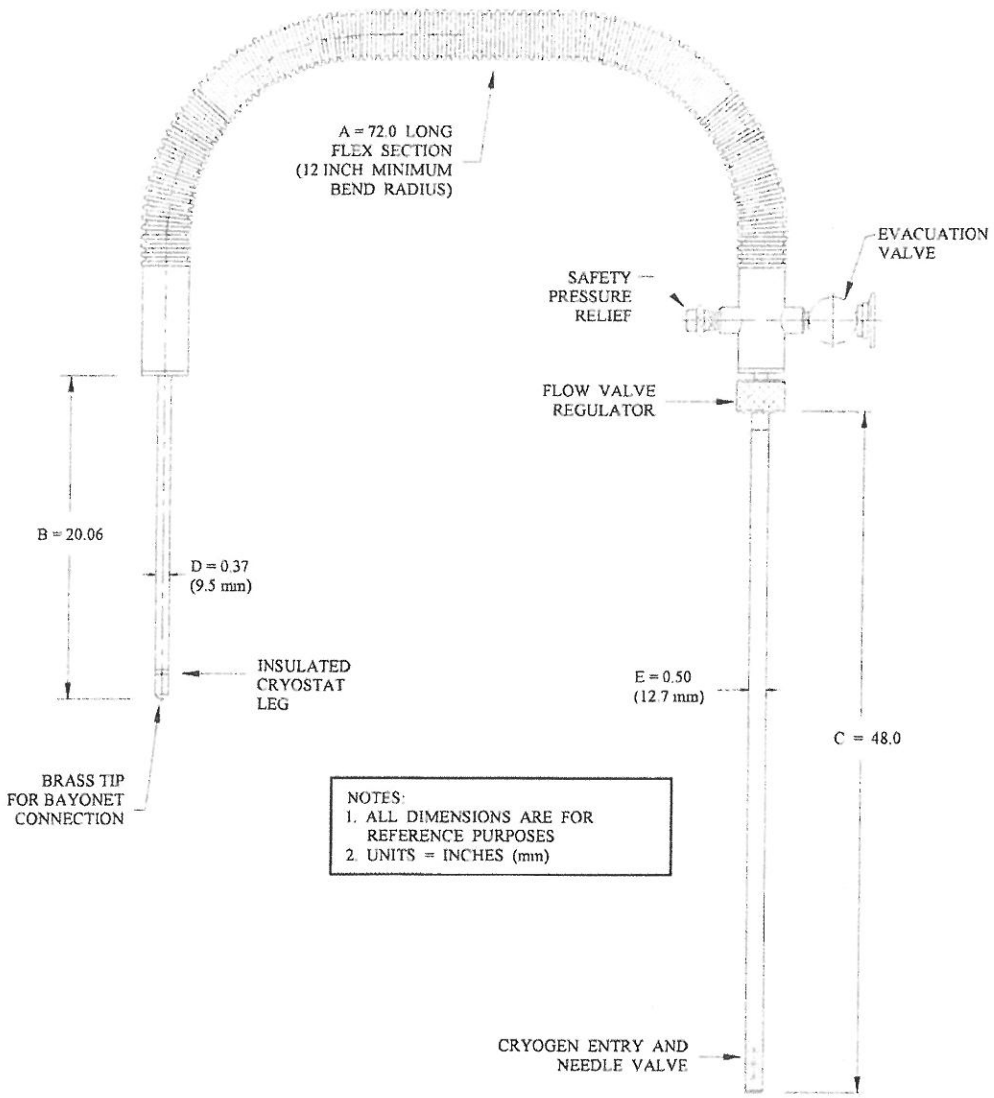

Figure 1: Model FHT-ST high efficiency flexible helium transfer line

CRYOSTAT SERIAL NUMBER: 18300

8 PIN FEEDTHROUGH

LOCATION: TOP FLANGE

PIN A - POS. CURRENT (I+) black

PIN B - POS. VOLTAGE (V+) clear **CONTROLLER INPUT "A"**
CALIBRATED CX-1050-SD-HT-1.4L CERNOX RESISTOR SN: X100775

PIN C - NEG. CURRENT (I-) red **(CONTROL SENSOR, CURVE 21)**
(ON VAPORIZER)

PIN D - NEG. VOLTAGE (V-) green

PIN E -

PIN F -

PIN G -
| 25 OHM WIRE WOUND HEATER, ON VAPORIZER
PIN H -

JANIS

SUPERTRAN VP TEST SHEET

(STVP-100, STVP-200, STVP-300, & STVP-400)

JOB # : 18300

CUSTOMER : PSI/TECO/Switzerland

DATE : 3/25/15

TECHNICIAN : LA

CRYOSTAT MODEL : STVP-NMR

STVP INVENTORY #: ______

FHT-ST INVENTORY #: ______

INSTRUMENTATION INFORMATION

TEMPERATURE CONTROLLER	Model : LCS-336	S/N : LSA-135L
VAPORIZER		
TEMPERATURE SENSOR	Model : CX-1050-SD HT 1.4	S/N : X100775
HEATER	Type: WOUND HEATER	Resistance: 25 Ohm
SAMPLE MOUNT		
TEMPERATURE SENSOR	Model :	S/N :
HEATER	Type:	Resistance:
AUX. INPUT		
LOCATION:	Model :	S/N :

AUXILIARY EQUIPMENT INFORMATION

EQUIPMENT:	Model :	S/N :
EQUIPMENT:	Model :	S/N :
EQUIPMENT:	Model :	S/N :

SUPERTRAN VP TEST

TIME		OPERATION
6:45am	→	Leak test all external and internal joints using a helium leak detector at room temperature prior to cool down.. Record room temperature sensor readings below.

DATE : 3/25/15 JOB # 18300 CUSTOMER : PSI/TECO/Switzerland

SUPERTRAN VP TEST (Continued)

ROOM TEMPERATURE DATA			
	Vaporizer	Sample	AUX
Temperature (K) :	293.93		
Resistance (ohms) :	78.05		

TIME		OPERATION
7:05am	→	Attach a mechanical pump to the NW-25 vent port. Turn on the pump with the flow regulator valve closed. Start transferring liquid helium at about 1-2 psi with the needle valve open about 2-3 turns (1 turn = 360 degrees). Pumping on the vent port helps pull the liquid helium through the transfer line and reduces the cool down time. It will take 5-10 minutes before the sample mount starts to cool.

Cool down time (300K to 10K) = 10 minutes

When the cryostat reaches ~160K, close the pump valve, wait till positive pressure builds on the pump pressure gauge, then vent through the pump manifold.

7:15am	→	Record the time the control thermometer reaches 10 K, then wait 15 minutes for the radiation shield to cool.
7:55	→	After the radiation shield is cold, record LHe sensor readings below.

LIQUID HELIUM TEMPERATURE DATA			
	Vaporizer	Sample	AUX
Temperature (K) :	4.32		
Resistance (ohms) :	3.193		

8:00	→	Fully open the flow regulator valve (~2-3 turns). Wait 1 to 2 minutes for liquid helium to collect in the sample mount. Completely close the flow regulator valve. Close the vent valve on the pump manifold and start pumping on the LHe. Record static superfluid sensor readings below.

SUPERFLUID TEMPERATURE DATA			
	Vaporizer	Sample	AUX
Temperature (K) :	1.80		
Resistance (ohms) :	12.163		

DATE : 3/25/15 JOB # 18300 CUSTOMER : PSI/TECO/Switzerland

SUPERTRAN VP TEST (Continued)

5K FLOW TEST

Open the flow regulator valve about 1 turn. Close the pump valve, wait till positive pressure builds on the inlet side of the pump, then vent through the pump manifold. Maintain the control temperature by adjusting the liquid helium flow (do not use the heater). See sales order for the expected flow rate. Note: it may be easier to control the flow by controlling the pressure in the storage dewar (1-2 psi) rather than only adjusting the flow regulator valve. Record flow data ~ every 5 minutes.

TIME	MASS FLOW (SLPM)	FLOW RATE (ML/HR) 1 SLPM (LHe) = 94 ML/HR (LHe)	CONTROL SENSOR (K)	SAMPLE SENSOR (K)
8:15am	18.3	1.720	5.0	
8:25am	16.2	1.522	5.0	
8:40am	14.6	1.372	5.0	
8:55am	13.6	1.278	5.0	

TEMPERATURE CONTROLLER OPERATION

Set the flow rate at ~10 SLPM (~800 ML/HR). Reduce to ~7 SLPM (~560 ML/HR) at 150 K and 300 K.

The above flow rates are for a standard STVP system, and will have to be adjusted for larger or different systems.

	Control Set Point	Time Set	Time Stable	Temp. Stability (K)	Gain (P)	Reset (I)	Heater Power (Range / %)	Sample Tube Flow (SLPM)	Sample Tube Flow (ML/HR)
Vaporizer	13K	9:00am	9:15	.02	50	20	HI 6.5	6.8	639
Sample	15K								
Vaporizer	145K	9:15am	9:30	.02	50	20	HI 34	5.4	507
Sample	150K								
Vaporizer	295K	9:30am	9:45	.01	50	20	HI 45	4.4	413
Sample	300K								

After the final set-point, turn the heater power off, then close the evacuation valve. Record the amount of liquid helium used in this test below.

Total liquid helium usage for this test = 7 liters

Remarks or Comments:

CALIBRATION CERTIFICATE
Report Number: 820503

Sensor Model: CX-1050-SD-HT-1.4L	Serial Number: X100775
Sensor Type: Cernox Resistor	Sales Order: 100679
Sensor Excitation: see *Test Data* page of report	Date: February 02, 2015
Temperature Range: 1.40 K to 325 K	Due: February 02, 2016

Traceability and Calibration Method

This temperature sensor has been calibrated to the International Temperature Scale of 1990 (ITS-90) or the Provisional Low Temperature Scale (PLTS-2000) as appropriate. The calibrations are traceable to the National Institute of Standards and Technology (NIST, United States), the National Physical Laboratory (NPL, United Kingdom), the Physikalisch-Technische Bundesanstalt (PTB, Germany), or natural physical constants.

Lake Shore Cryotronics maintains ITS-90 and PLTS-2000 on standard platinum (PRT), rhodium-iron (RIRT), and germanium (GRT) resistance thermometers that have been calibrated directly by an internationally recognized national metrology institute (NIST, NPL, PTB) for T < 330 K or an ISO 17025 accredited metrology laboratory for 330 K < T < 800 K. A nuclear orientation thermometer is also used for temperatures less than 50 mK. These standards are routinely intercompared to verify consistency and accuracy of the temperature scale.

The sensor calibrations are performed by comparison to laboratory standard resistance thermometers and tested in accordance with Lake Shore Cryotronics, Inc. Quality Assurance Manual (QP-4220). The quality system of Lake Shore Cryotronics is registered to ISO 9001:2008.

Procedures used: 021-97-02, 099-00-00, 121-96-02, 029-95-02

Notes

The calibration results in this report apply only to the specific sensor specified above.

Unless stated otherwise, the uncertainties in this report are based on an approximate 95% confidence level with a coverage factor k=2.

Reported by: Romerero Prince
Calibration Engineer/Technician

Approved by: John Krause
Metrology

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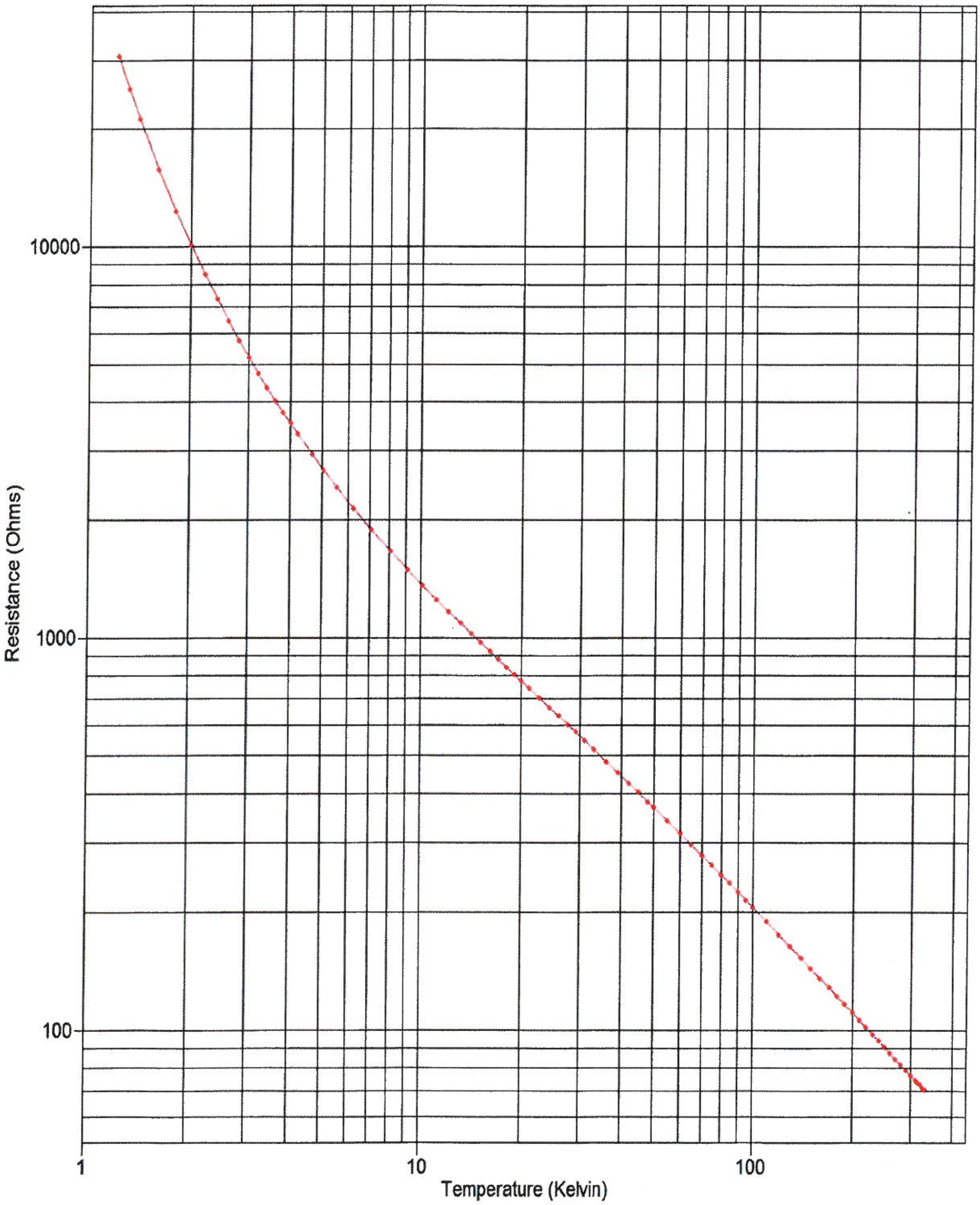
DATA PLOT

Calibration Report: 820503
Sensor Model: CX-1050-SD-HT-1.4L
Sensor Type: Cernox Resistor

Sales Order: 100679
Serial Number: X100775
Temperature Range: 1.40 K to 325 K



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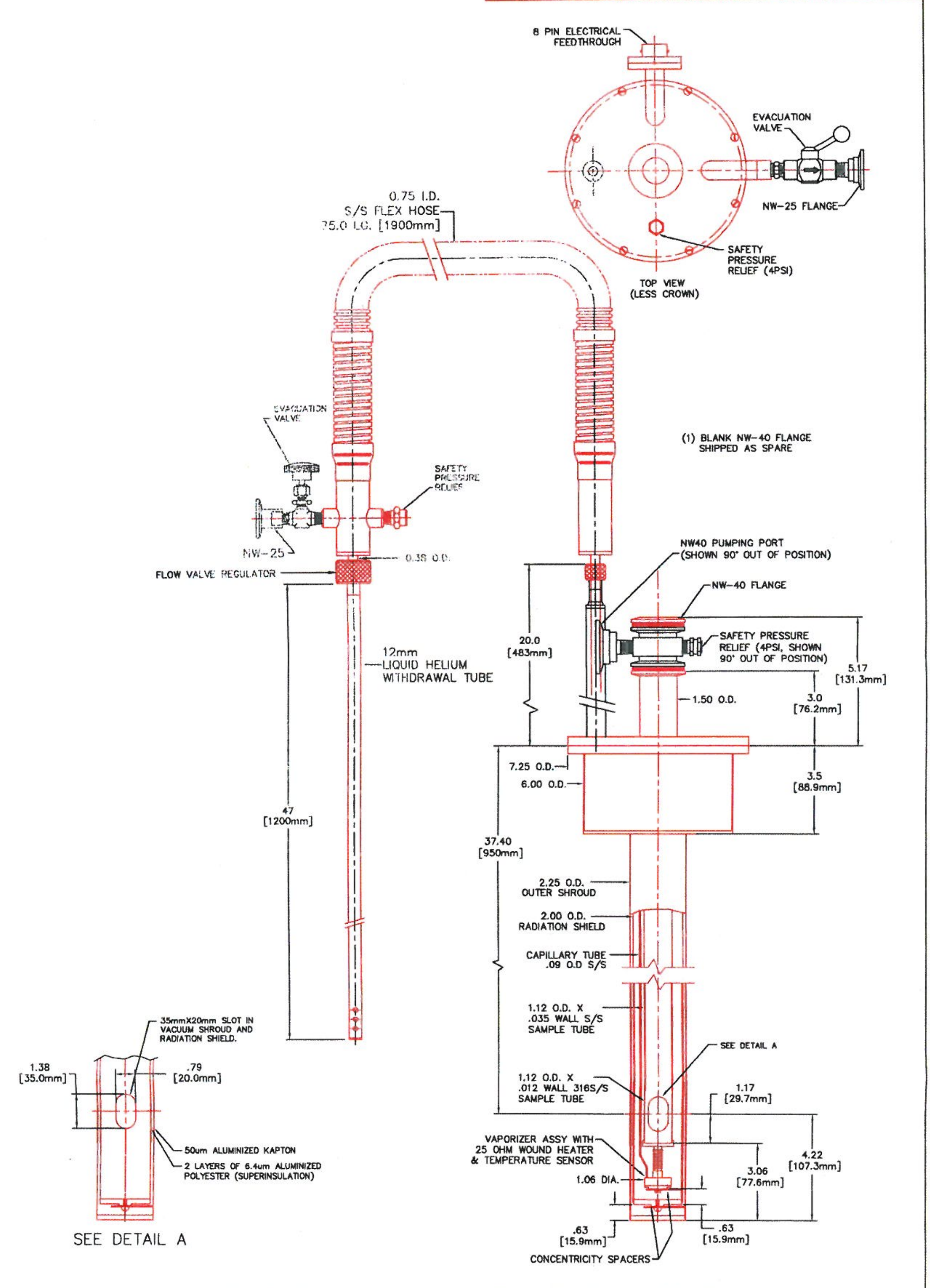
REV.	ECO #	REVISION DESCRIPTION	DATE	INITIALS	APP'D
A		RELEASE TO PRODUCTION	12/16/14	MG	



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UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES DO NOT SCALE DRAWING REMOVE ALL SHARP EDGES AND CORNERS MACHINED SURFACES 32

THIRD ANGLE PROJ. DRAWING ANSI Y14.5

TOLERANCE: .X ± .1, .XX ± .02, .XXX ± .005, .XXXX ± .0005, FRAC. ± .1, ANGLE ± 0°30'

MAT'L:		DRAWN / DATE	MG 12/1/14
ASS'Y:		APP'D / DATE	
	STOCK	JOB NO.	18300
	WELDING	FINISH:	
	MODIFY	QTY REQ'D	

TITLE: STVP-NMR, CONT. FLOW NON-OPTICAL SYSTEM

SIZE	DRAWING NO:	REV.
D	D11-26-14B	A

SCALE – | ITEM NO. | SHEET 1 OF 1