^{3}He Refrigerator
Installation and Operation

Condensed Matter NMR

2014

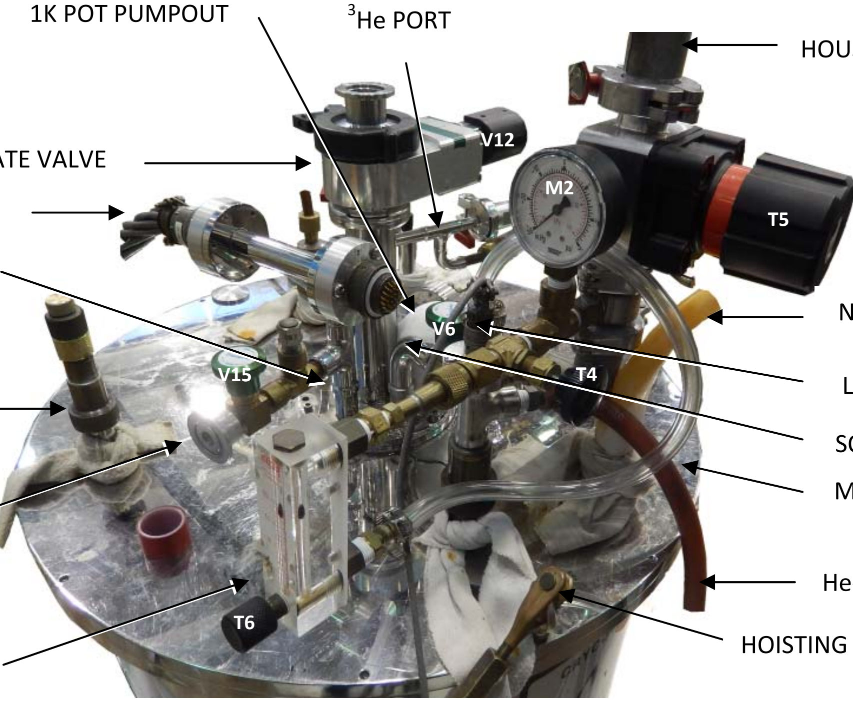


Fig. 1 The ^{3}He cryostat loaded into the dewar. See Fig. 2 for valve identification.

The Condensed Matter Nuclear Magnetic Resonance group utilizes and maintains a Janis ^{3}He refrigerator for low temperature NMR experiments. The system operates using a charcoal absorption (sorp) pump which is in direct contact with ^{3}He gas within the cryostat. The ^{3}He gas, also in thermal contact with the L^{4}He 1K Pot, is first liquefied by pumping on the pot below 3.2K while keeping the sorp at high temperatures (>40K). Thereafter, the charcoal is cooled down by circulating L^{4}He into the sorp. The large surface area of the charcoal cryo-pumps the gas resulting to lower ^{3}He vapor pressure. Through evaporative cooling, the low vapor pressure causes the liquid ^{3}He to cool below its boiling point, down to typically 0.3K.

Because of the delicate nature of this equipment, extreme care has to be taken in order to avoid possible damage to its structure or, most importantly, loss of ^{3}He gas to the atmosphere. This manual contains a lot of useful information for proper and efficient operation of the ^{3}He system. First time users of this equipment are required to read (and understand!) this manual in its entirety before attempting to operate the system.

I. Preparatory Procedures

1. Preparing the vacuum systems
   a. Leak-test the cryostat inner vaccum chamber (IVC, port V15), with particular attention on the two In o-ring seals above and below the sorp section. Test for possible internal (^{3}He space) leak as well. Make sure the inside ^{3}He space is dry.
   b. Leak-test the main dewar vacuum OVC (port V16), particularly the three o-ring seals outside and the inner joints inside. Make sure the space inside the dewar is dry.
   c. Evacuate the IVC, main dewar vacuum, and the ^{3}He space. Pump for 2-3 days.

2. Loading the main dewar into magnet
   a. Secure the main dewar onto hoist using harness. Aim the tail to the magnet.
   b. Use a torpedo level to make sure the tail is vertical and goes straight to the magnet.
   c. set the correct height so that the NMR probe reaches the bottom of the tail.
   d. in Cell2, 3 legs are adjusted to 135mm.

3. Precooling the main dewar(LN2)
   a. Secure a full LN2 storage dewar.
   b. remove the rubber stopper on the LN2 port and hook up the LN2 line to LN2 dewar.
   c. open LN2 vent port and begin transfer until full. It takes about 30 mins to complete the transfer.

4. Precooling the main dewar (LHe)
   a. Wait 8 hours to allow the dewar tail to cool down to 77K
   b. Transfer Helium slowly to the main bath using the LHe port at <2 psi pressure. Fill halfway (30 cm) using its own level meter). Use extended tube to fill LHe.

5. Preparing the NMR probe
   a. Use the short loadlock for the long tail ^{3}He cryostat and vice versa. See Fig. 4 for length reference.
   b. By capping the bottom of the loadload on the bench, leak test the loadlock and probe.

II. Loading the cryostat into the main dewar

With LHe in the dewar, the cryostat may be loaded. The main dewar is capped by a black disk. (Note: if the cryostat is loaded before LHe4 transfer,the liquid helium may not go into the tail).

1. Evacuate ^{3}He space and close the gate valve.
2. Close the needle valve and valves V7, T5, T4, V6, and T6.
3. Install the two lifting posts and mount the cryostat onto the hoist.
4. Remove the dewar cap and lower the cryostat into the dewar very slowly (~30 min). Use lots of rags to minimize moisture intrusion. Align cryostat tail manually as it gets through the dewar tail.
5. Orient the cryostat for most efficient access to ports.
6. When loaded, secure the cryostat flange into the dewar with six bolts.
7. Remove lifting posts.
8. Optional: fill the LHe to 60cm using a short transfer tube.

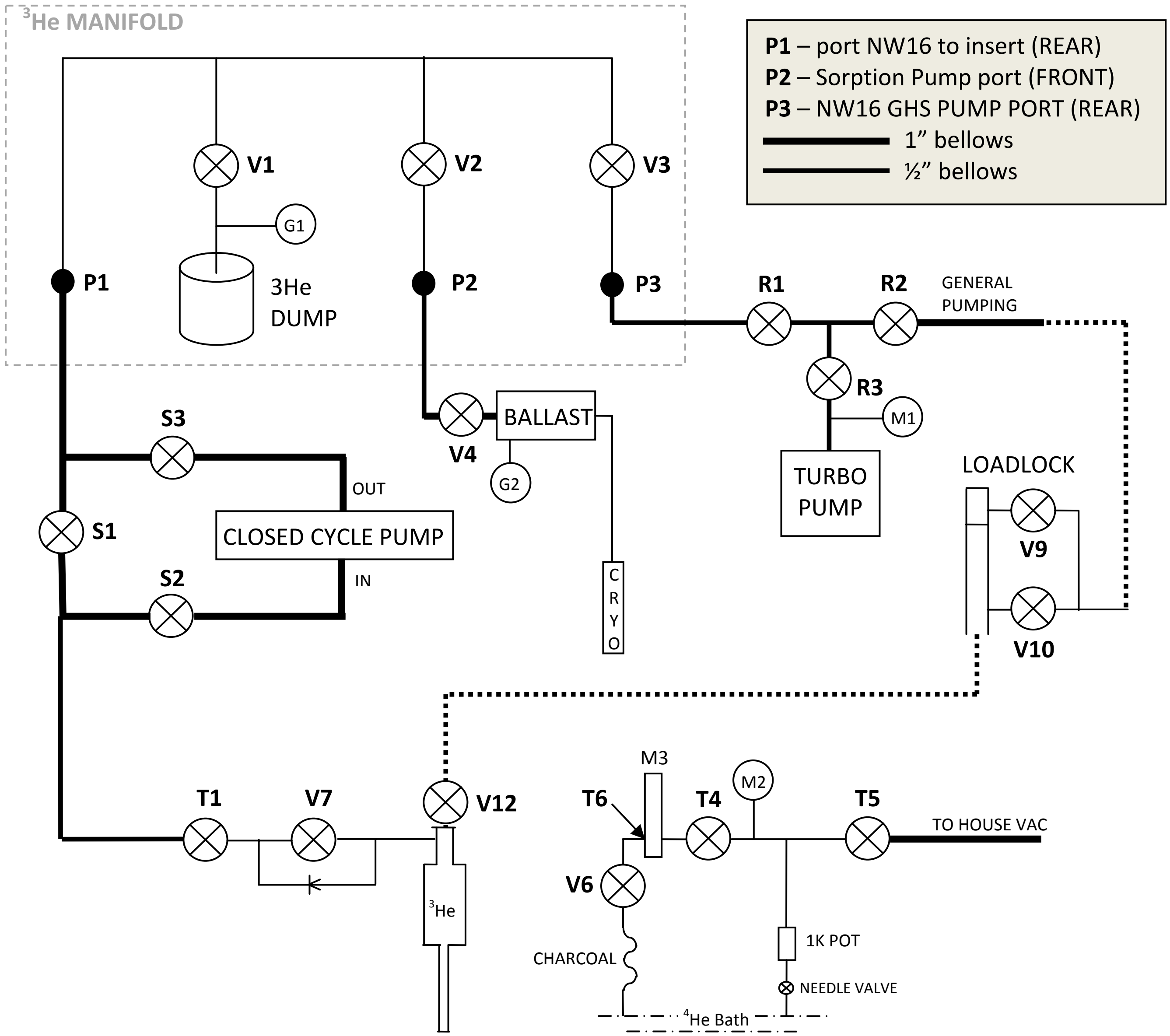


Fig. 2 ^{3}He Refrigerator System Interconnect. Dotted lines are connected only when the probe is loaded.

Sensor	Location	Type	Serial No.
A	Charcoal	Cernox	X30506
B	RuOx sample	RuOx	RO3023
C	1K-Pot	Cernox	X30302
D	Tail (Long)	Cernox	X30507
	Tail (Short)	Cernox	X30301

Table I. Sensor Connections for LS340

III. Initial Cooldown

Initial cooldown is required before introducing ^{3}He in the fridge.

1. Connect the sensors. (See Table 1). Connect the charcoal heater to the LS controller heater output.
2. Hook up the house vacuum to the 1K Pot.
3. Open house vac valve and open T5 to clear the line.
4. Check for flow on the 1K pot
   a. close needle valve, close V6 (charcoal) valve
   b. open T5, evacuate by opening house vac valve, close T5.
   c. open needle valve, pressure reading should go up. Slight boil off expected
   d. close needle valve.
5. Check for flow on the charcoal circuit
   a. open T5, evacuate by opening house vac valve, close T5
   b. open V6 and T4 and then slowly open T6, pressure reading should go up.
   c. close T4 and leave V6 open.
6. Keep T5 open and start pumping on the 1K pot. Set needle to 3-4 turns to fill the pot with LHe. Then close needle to 1.5 turns. Cooldown rate would depend on the pressure on the house vac manifold. As the temperature drops to 4K, close needle valve 3/4 turns.
7. Use T6/M3 to adjust the flow thru the charcoal. The tail temperature should slowly cool 200K/12 hrs, 0.25K/min.

IV. System plumbing

Hook up the ^{3}He storage manifold cart and turbo pump to the system as in Fig. 2. Leave the probe and the loadlock out.

V. Loading ^{3}He gas

Note: as of 4/21/2014 the ^{3}He pressure G1 in the dump is -8+ inHg. Although ^{3}He can be loaded anytime, it is recommended to load ^{3}He when the tail is already cold. Note: ^{3}He liquefies at 3.2K.

1. Hook up the plumbing system (step IV, Fig. 2). Make sure valves T1, V7, S1, S2, S3, R1, R2, R3, V1, V2, and V3 are closed.
2. Turn on the turbo pump.
3. Open T1, S1, V3, R1 and R3 to clear the lines. Open V7.
4. Pump until the turbo pressure M1 is $<10^{-5}$ mbar.
5. Close R1 and V3 and seal them to avoid accidental opening.
6. With 1K-pot at 1.5K and charcoal ~4K, introduce excess ^{3}He from the ballast. Close V2, open V4 and then slowly open V2. Pressure G2 must indicate -30 inHg. This will warm up the charcoal to ~25K, but not change the tail temperature. Rapid change in tail temperature indicates a leak. Close V2 and V4.
7. Introduce ^{3}He gas into the fridge: Open V1. Charcoal will absorb all the ^{3}He. When all is transferred, pressure G1 must register -30 inHg.
8. Set 1K pot needle valve 3/4 turns. This will run full. Pressure regulator may be used to control the flow.
9. Start to liquefy, heat up the charcoal 30-40K to push ^{3}He to the tail and liquefy. It takes about 2hrs to condense the ^{3}He gas in the tail.

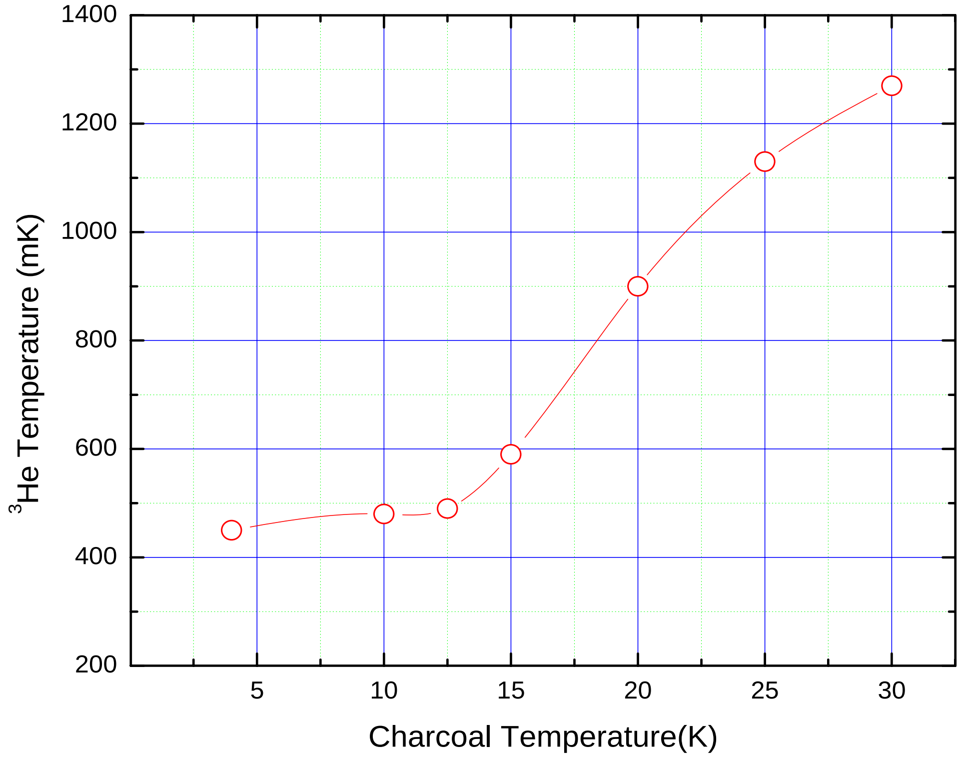


Fig. 3. Experimental asymptotic L^3He temperature as a function of charcoal temperature determined by NMR. The 1K Pot is at 1.2K. Actual temperature may vary depending on the heat load.

VI. Loading the ^{3}He NMR probe

The NMR probe is loaded when the tail is full of L^3He. NMR probe is mounted on the loadlock.

1. Measure the distance between the sample and the probe head. See Fig. 4 for reference.
2. Align the bottom of the probe with the copper anchor and the bottom of the loadlock. (Fig. 4)
3. Mark the location of the upper end of the loadlock on the loadlock shaft.
4. Mark the location of the upper end of the loadlock shaft on the probe shaft.
5. Leak-test the probe with the loadlock attached.
6. Set the loadlock on the KF flange on top of gate valve V12.
7. Connect the turbo pump to the loadlock.
8. Pump the loadlock and the loadlock seal (open R2, R3, V9, and V10))until M1 reads ~10^{-5}mbar.
9. Close V10. Keep V9 open with the pump connected to it.
10. Heat charcoal to 25K to provide small exchange gas.
11. Open the gate valve V12. ^{3}He gas should rush to the loadlock.
12. Connect the temperature controller to the probe.
13. Lower the loadlock shaft slowly 2 in. at a time until the copper thermal anchor hits the pot. Monitor the probe temperature while lowering. Use heater to control ramp to desired rate.
14. Lower the probe to the desired height.
15. Tighten the loadlock quick-connect nut.

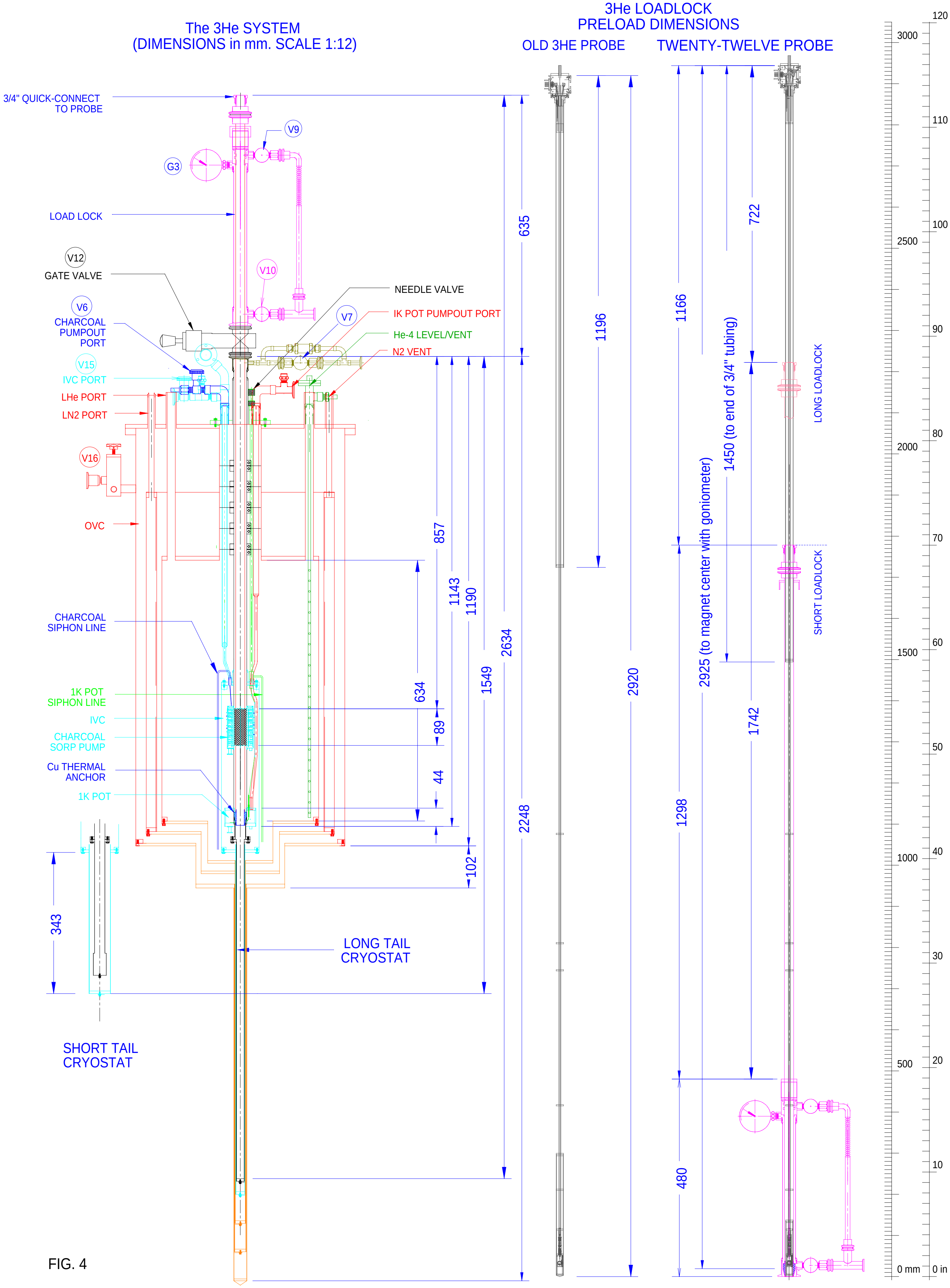

The 3He SYSTEM
(DIMENSIONS in mm. SCALE 1:12)
3He LOADLOCK
PRELOAD DIMENSIONS
OLD 3HE PROBE
TWENTY-TWELVE PROBE
3/4" QUICK-CONNECT
TO PROBE
V9
G3
LOAD LOCK
V12
GATE VALVE
V10
NEEDLE VALVE
V6
CHARCOAL
PUMPOUT
PORT
V7
IK POT PUMPOUT PORT
He-4 LEVEL/VENT
N2 VENT
V15
IVC PORT
LHe PORT
LN2 PORT
V16
OVC
CHARCOAL
SIPHON LINE
1K POT
SIPHON LINE
IVC
CHARCOAL
SORP PUMP
Cu THERMAL
ANCHOR
1K POT
LONG TAIL
CRYOSTAT
SHORT TAIL
CRYOSTAT
343
635
857
1143
1190
1549
2634
2248
634
89
44
102
1196
2920
1166
2925 (to magnet center with goniometer)
1450 (to end of 3/4" tubing)
722
1742
1298
480
LONG LOADLOCK
SHORT LOADLOCK
3000
2500
2000
1500
1000
500
0 mm
120
110
100
90
80
70
60
50
40
30
20
10
0 in

FIG. 4

VII. Operating the Fridge

Under normal operation, the ^{3}He fridge is open all the way to the dump, i.e. V1, S1, T1 and V7 are open. The L^4He comsumption is pretty good: need only to transfer every 2 days. The needle valve is set at ¾ turns during the run for max efficiency. (<1/2 is better!)

1. To liquefy ^{3}He gas, keep charcoal at 30K. It takes ~2 hrs to fill the tail with L^3He. (How to find out?)
2. To cool down <1.5K, cool the charcoal (see temperature chart, Fig. 3)
3. For standby operation, e.g. overnight, leave the charcoal at 4K.
4. Fig. 3 can serve as a guide in using the sorp temperature to regulate the L^3He temperature. The actual temperature may vary depending on the thermal load.
5. Fig. 5 details the P-T curves of the two helium isotopes.

VIII. Unloading the NMR probe

To unload the NMR probe, check that pot is at max 1.2K, L^3He in tail, charcoal > 32K. Sample at 1.3K. The temperature controller is connected to the probe. Turbo pump is connected to the loadlock with V9 and V10 closed.

1. Pull the probe up to the mark made in Step VI.4 (see Fig. 4 for dimensions). As the probe is pulled up, it warms to 25K as it passes through the sorp section and then back down when it goes past above IVC and then up again as it reaches the neck of the cryostat.
2. Pull loadlock shaft slowly, 1 ft at a time, up to mark made in Step VI.3. Open and close V9 for every pull to prevent air from getting in.
3. Wait until the sample is at room temperature.
4. Open V6, T6 and T4 to cool down the charcoal to 4K and cryopump the ^{3}He gas.
5. Close the gate valve V12 carefully. Make sure gate does not hit the probe or the copper thermal anchor.
6. Disconnect the turbo pump (hooked in Step VI. 7) from the loadlock
7. Open V10 to vent the loadlock.
8. Remove the loadlock and the probe fom the cryostat.

IX. Recovering ^{3}He gas

With the probe removed, the ^{3}He gas may be recovered.

1. Disconnect the turbo pump from P3.
2. Connect the charcoal heater to 14VDC variable power supply and heat slowly to get to ~180K
3. Heat the tail to 160K(set to Lakeshore to 10mW power). 1K pot warms up to 50K
4. Pressure G1 on the manifold should go up (note V2 and V4 are both open)
5. Close S1, open S2 and S3 on the close cycle pump to suck out the residual ^{3}He gas. Pressure at G1 should slowly go up to 7-8 inHg.
6. Close T1, V7. Then close T5, T4, and V6.
7. Close S2.
8. Close S3 and shut off the closed-cycle pump.

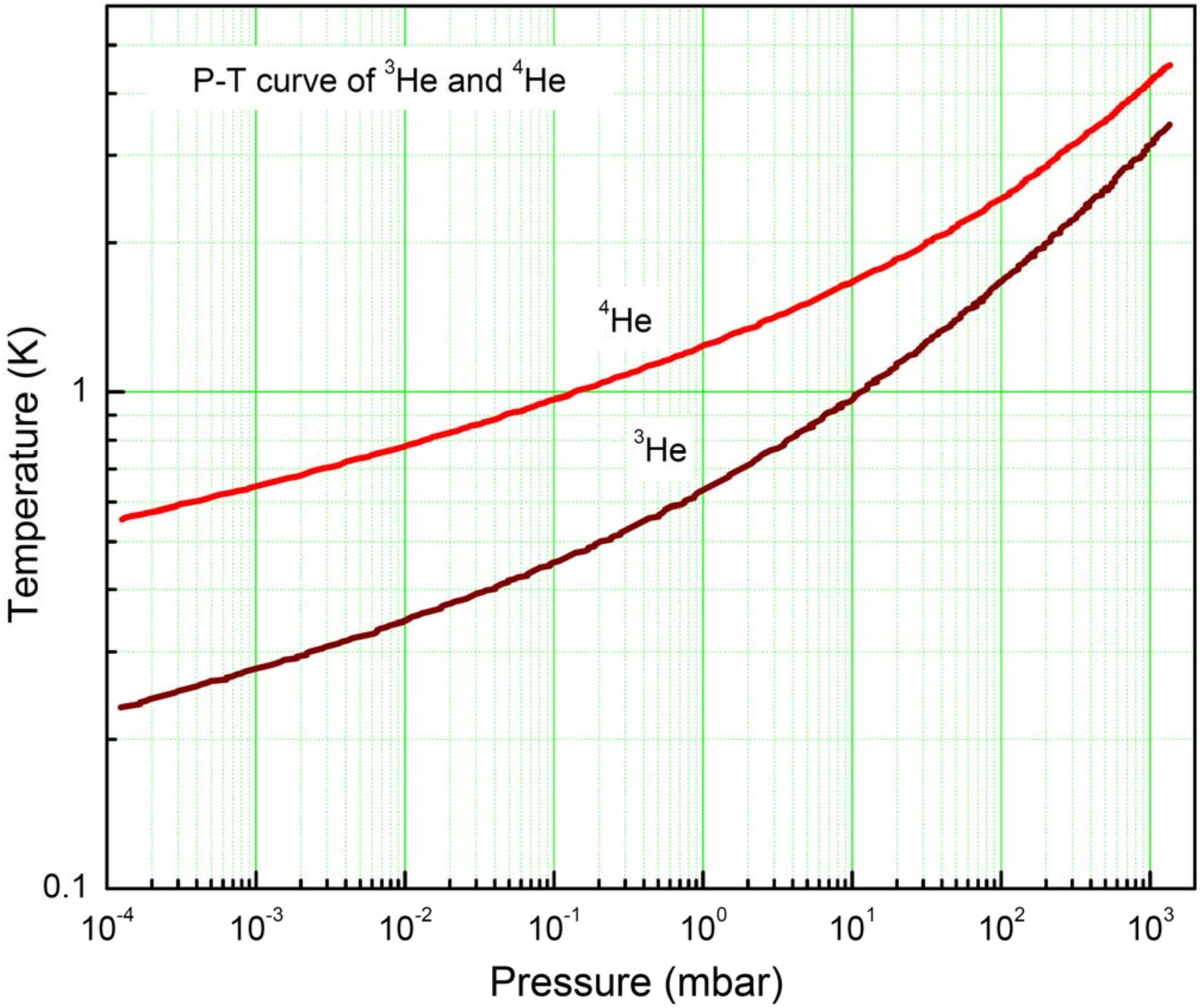


Fig. 5. P-T curve of two isotopes of Helium.

X. Clean Up procedure

This procedure to be done only if the entire ^{3}He gas has been recovered to the dump.

1. Remove the line connecting T1 and S1/S2.
2. Hook up the hoist and remove the cryostat from dewar. Cap the top of the dewar.
3. Disconnect the vent lines from the dewar.
4. Hoist up the dewar from magnet. Keep the vents open if helium is still in the system.

APPENDICES

A. EQUIPMENT DATA

Base temperature:	400mK (when 2012 probe is loaded)
Hold time:	11 hrs at base temperature
Cycle time 300mK to RT and back down:	2 hrs minimum
Dump capacity:	30 liters
STP ^{3}He gas volume:	21 liters (-7 inHg as of 5/2014)
Moles of ^{3}He:	0.94 moles
Equivalent L^{3}He volume:	58.8ml
^{3}He pot diameter:	19mm (long tail); 30 mm(short tail)
Pre-pumped liquid column height:	20.7 cm (long tail); 8.2 cm (short tail)

B. PROPERTIES OF ^{3}He and ^{4}He isotopes

Property	Symbol	^{3}He	^{4}He
Density of gas at RT (g/l = kg/m^3)	ρ_g	0.165	0.178
Density of liquid at BP (g/l)	ρ_L	58.9	125
Specific Heat (J/g-K)	c	4.61	4.48
Latent Heat (J/g)	L_v	8.49	20.90
Boiling Pt (K)	T_b	3.19	4.214
Critical Pt (K)	T_c	3.32	5.2
Thermal Conductivity (mW/mK)	K	17.1	27.2
Viscosity (uPa-s)	η	1.62	3.56
Superfluid Transition at 1 atm (K)	T_{sf}	<0.002 (^{3}He-B)	2.17

C. CALCULATIONS

Volume of L^{3}He: $V_{liq} \sim \frac{\rho_g}{\rho_L} V_{gas} = \frac{0.165}{58.9}\, 21l = 58.8\, ml$

With mass m_o at boiling point (T_b), the remaining L^{3}He due to evaporative cooling down to temperature T can be calculated as follows. Assuming no other heat losses, an infinitesimal mass dm converting into vapor will change the temperature of the liquid by an amount dT. The heat energy transfer is given by

$$-L_v dm = -\, m\, c\, dT$$

where c is the specific heat of the liquid and L_v is the latent heat of vaporization. Integrating, we get

$$\ln m = \frac{c}{L_v} \int_{T_b}^{T} dT$$

Thus, the relative amount of liquid remaining at temperature T is

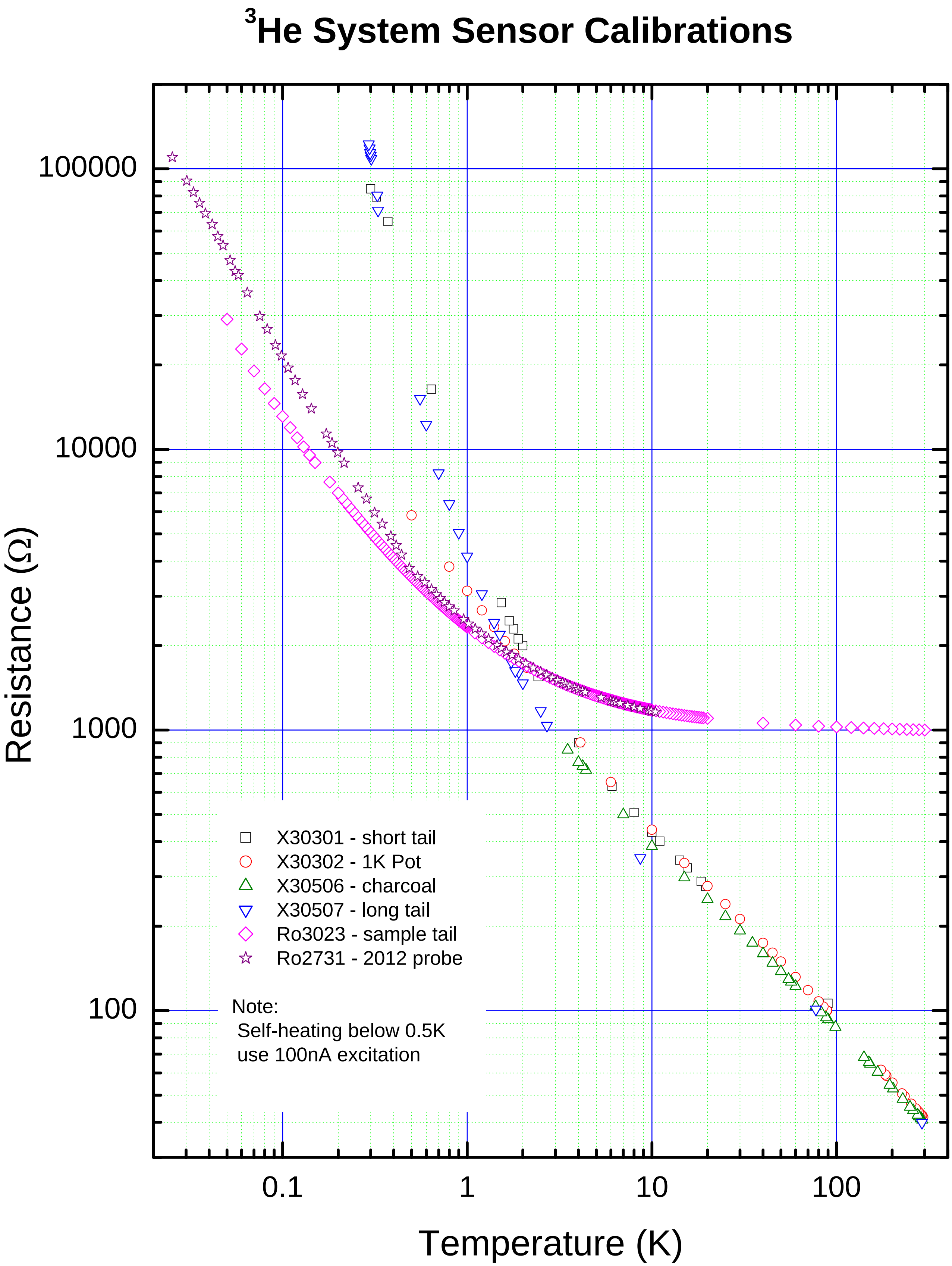
$$\frac{m}{m_o}(T) = e^{-\frac{c}{L_v}(T_b - T)}$$

At 300 mK and using the values in the table, we get $\frac{m}{m_o}(0.3K) = 20\%$!

Note: At 50% volume, the temperature of the liquid would be

$$T_{\frac{1}{2}} = T_b - \frac{L_v}{c}\ln 2$$

which for L^{3}He is about $T_{\frac{1}{2}}$ =1.92K. For L^{4}He, $T_{\frac{1}{2}} = 0.9$K, and $\frac{m}{m_o}(1.2K)$ =53%.



³He System Sensor Calibrations
100000
10000
1000
100
Resistance (Ω)
0.1
1
10
100
Temperature (K)
X30301 - short tail
X30302 - 1K Pot
X30506 - charcoal
X30507 - long tail
Ro3023 - sample tail
Ro2731 - 2012 probe
Note:
Self-heating below 0.5K
use 100nA excitation