LIGO-T020130-00-D

Department PHYSICS

Subject CRYOSTAT – GLASSY METALS

Name HAREEM

Address LIGO 18-34

National® Brand

Computation Notebook

11 3/4" x 9 1/4", 4 x 4 Quad., 75 Sheets

43-648

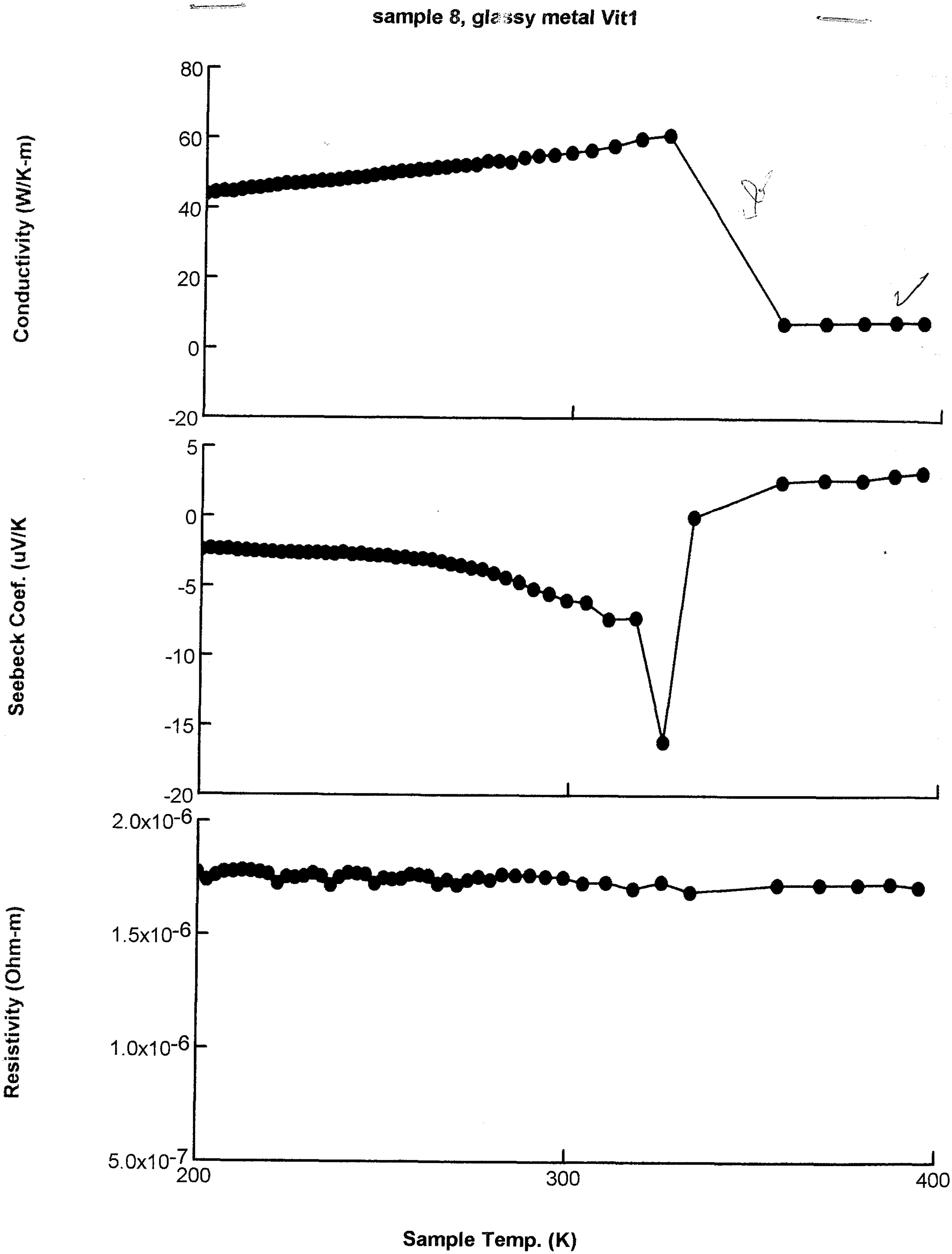
0 73333 43648 8

AVERY DENNISON

Office Products
Chicopee, MA 01022

25/2/02

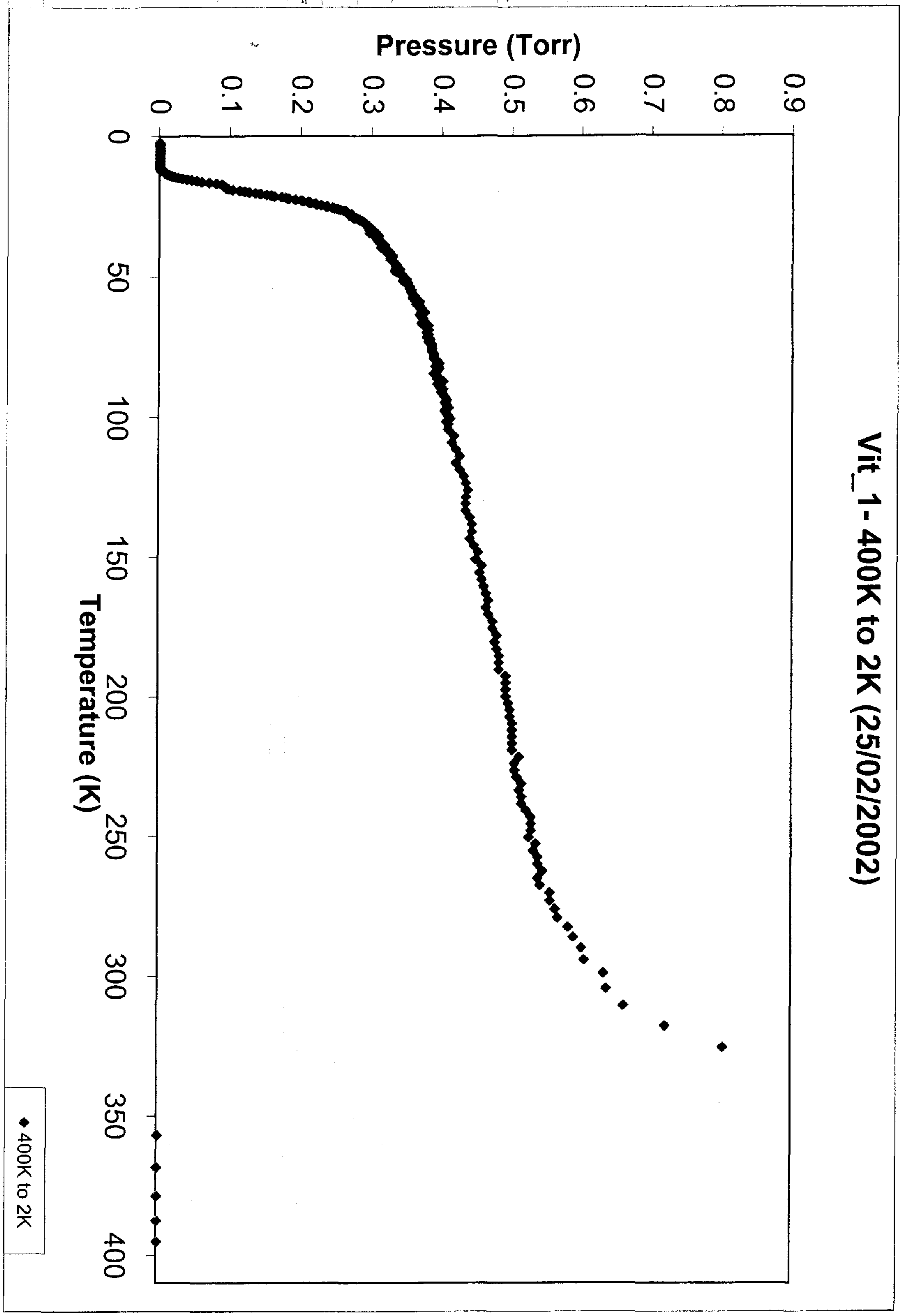
crystal measurement of VitI sample which was prepared by charlotte



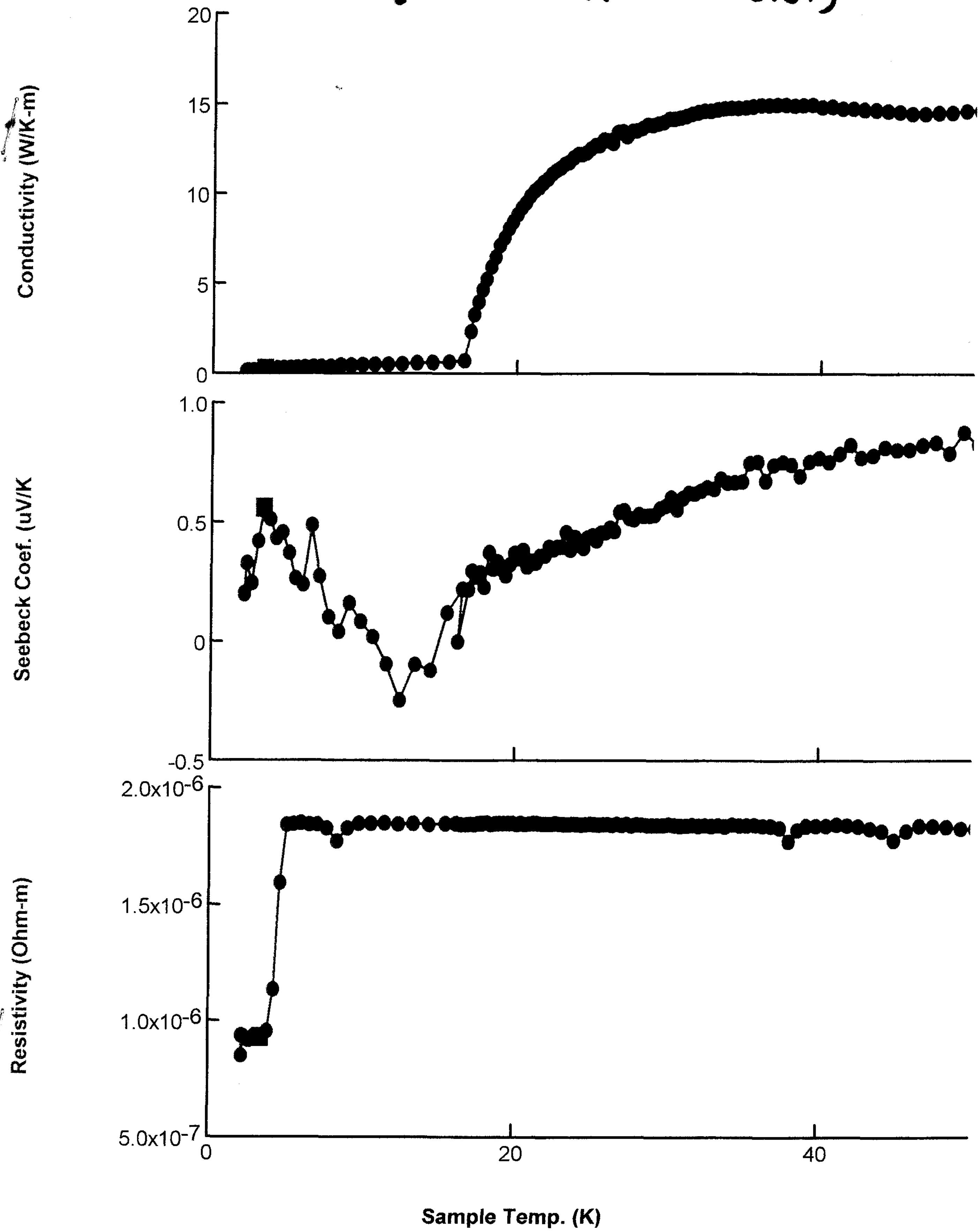
sample 8, glassy metal Vit1
Conductivity (W/K-m)
80
60
40
20
0
-20
Seebeck Coef. (uV/K
5
0
-5
-10
-15
-20
Resistivity (Ohm-m)
2.0x10-6
1.5x10-6
1.0x10-6
5.0x10-7
200
300
400
Sample Temp. (K)

26/2/2002

Experienced a strange jump in my measurement. Took another measurement to reconfirm the jump.

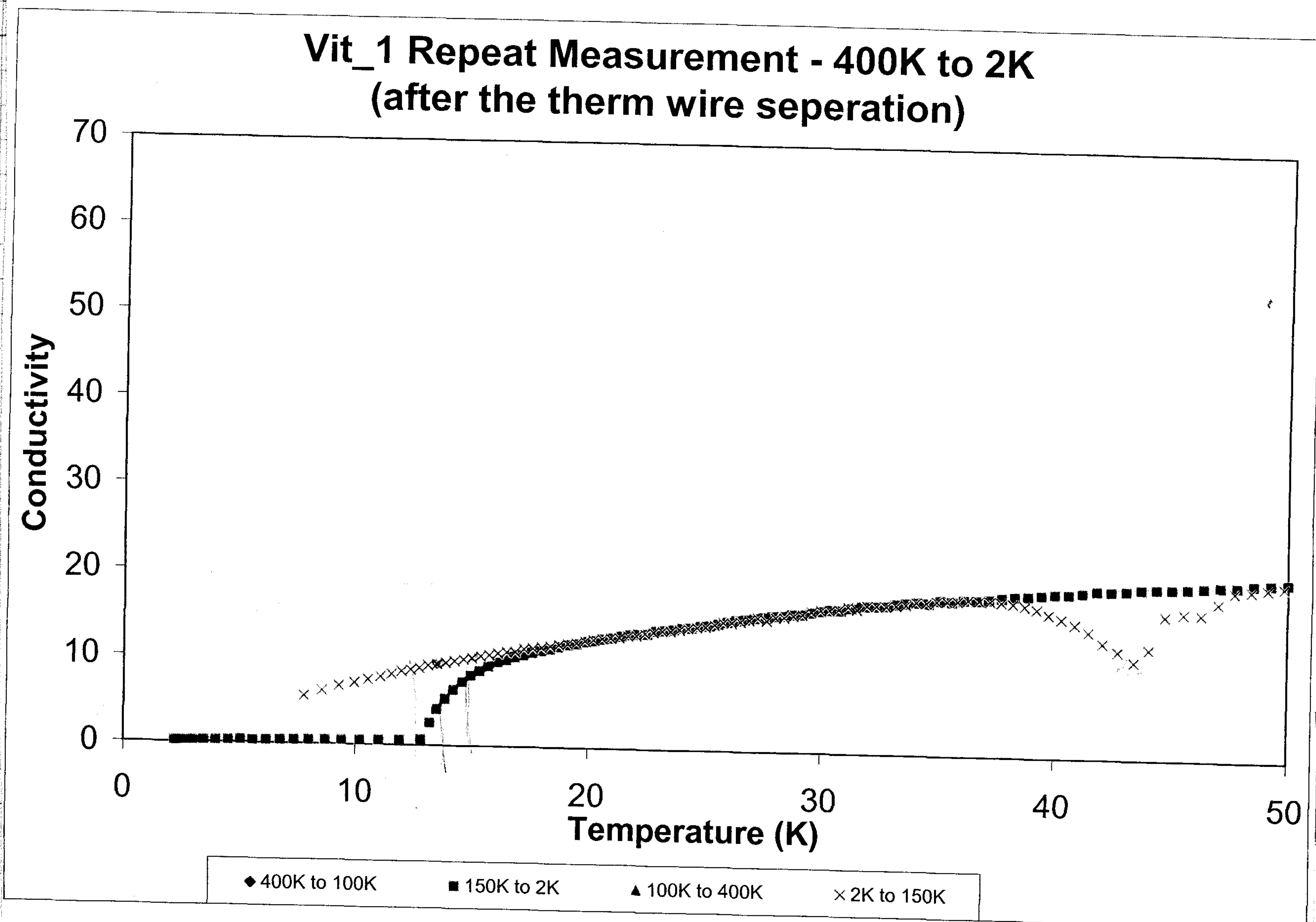


CHARLOTTE'S sample 8, glassy metal Vit1 26/2/20
(REPEAT MEASUREMENT)



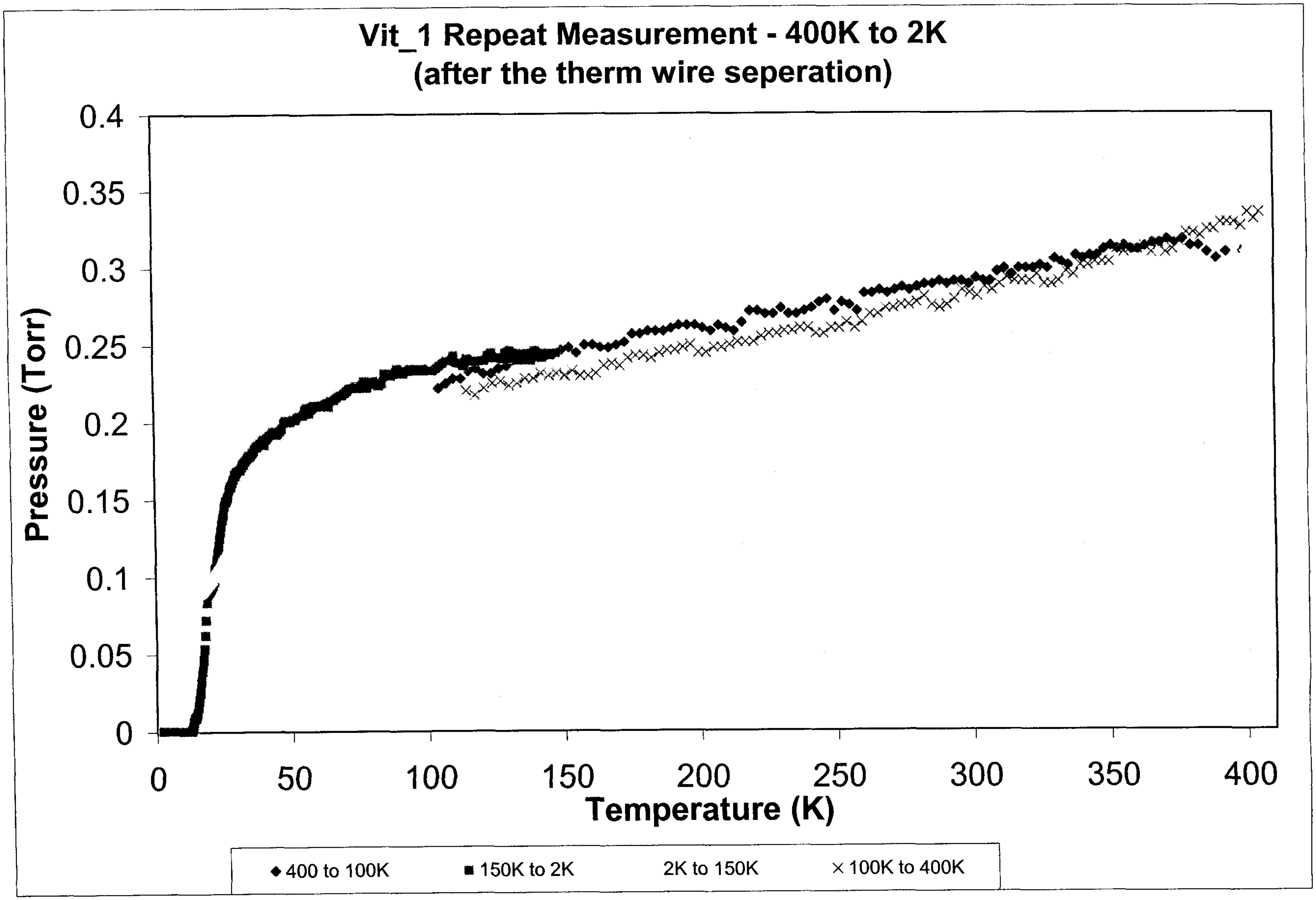
→ Checked the sample by takeng it out of cryostat → no short circuit between the sample and the puck. Seperated the wire so that none were touching the thermometer and short circuiting them.

→ Took a repeated measurement from 2K to 400K and from then 400K to 2K back again



Vit_1 Repeat Measurement - 400K to 2K
(after the therm wire seperation)
Conductivity
Temperature (K)
0
10
20
30
40
50
60
70
◆ 400K to 100K
■ 150K to 2K
▲ 100K to 400K
× 2K to 150K

8

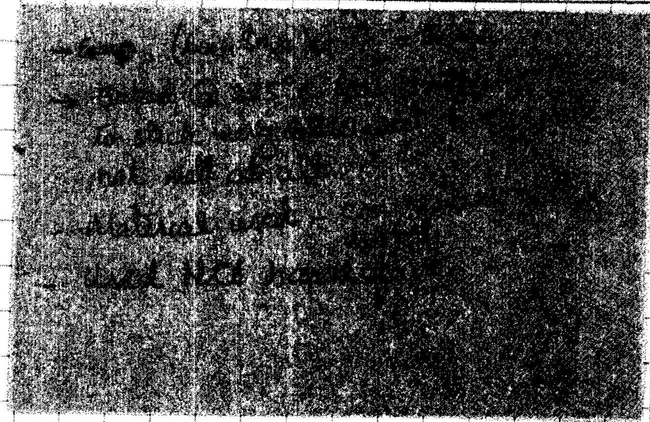


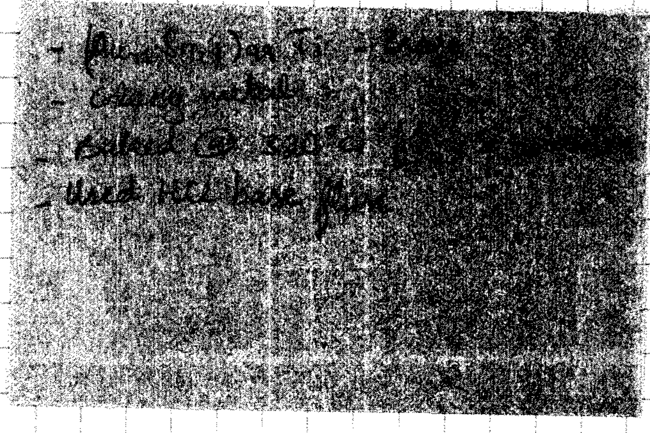
Vit_1 Repeat Measurement - 400K to 2K
(after the therm wire seperation)
Pressure (Torr)
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
50
100
150
200
250
300
350
400
Temperature (K)
400 to 100K
150K to 2K
2K to 150K
100K to 400K

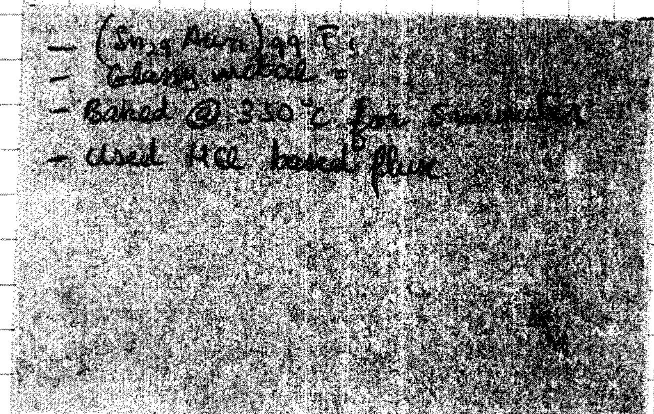
- Used HCl solu[illegible]
  machine shop [illegible]
- Baked in the [illegible]
- Flux was [illegible]
- Baking time [illegible]
  @ 350°C
- Braze = Gold [illegible]
- Commercial grade [illegible]

[illegible]

- Used HCl solution (from machine shop) as the flux & [illegible] coated with flux
- Baked in the oven while placed on top of Al square piece
- Oven temp = 350°C
- Baking time = [illegible] minutes
- Braze = Gold +
- Material = Commercial [illegible]




- Used HCl base flux


- Baked @ 330°C for
- Used HCl based flux

- Commercial glassy metal 1/4/2002
- Baked in air @ 450°C
- Matted strip used
- Baking time ≃ 20 min
- 37% HCl = flux

[illegible]

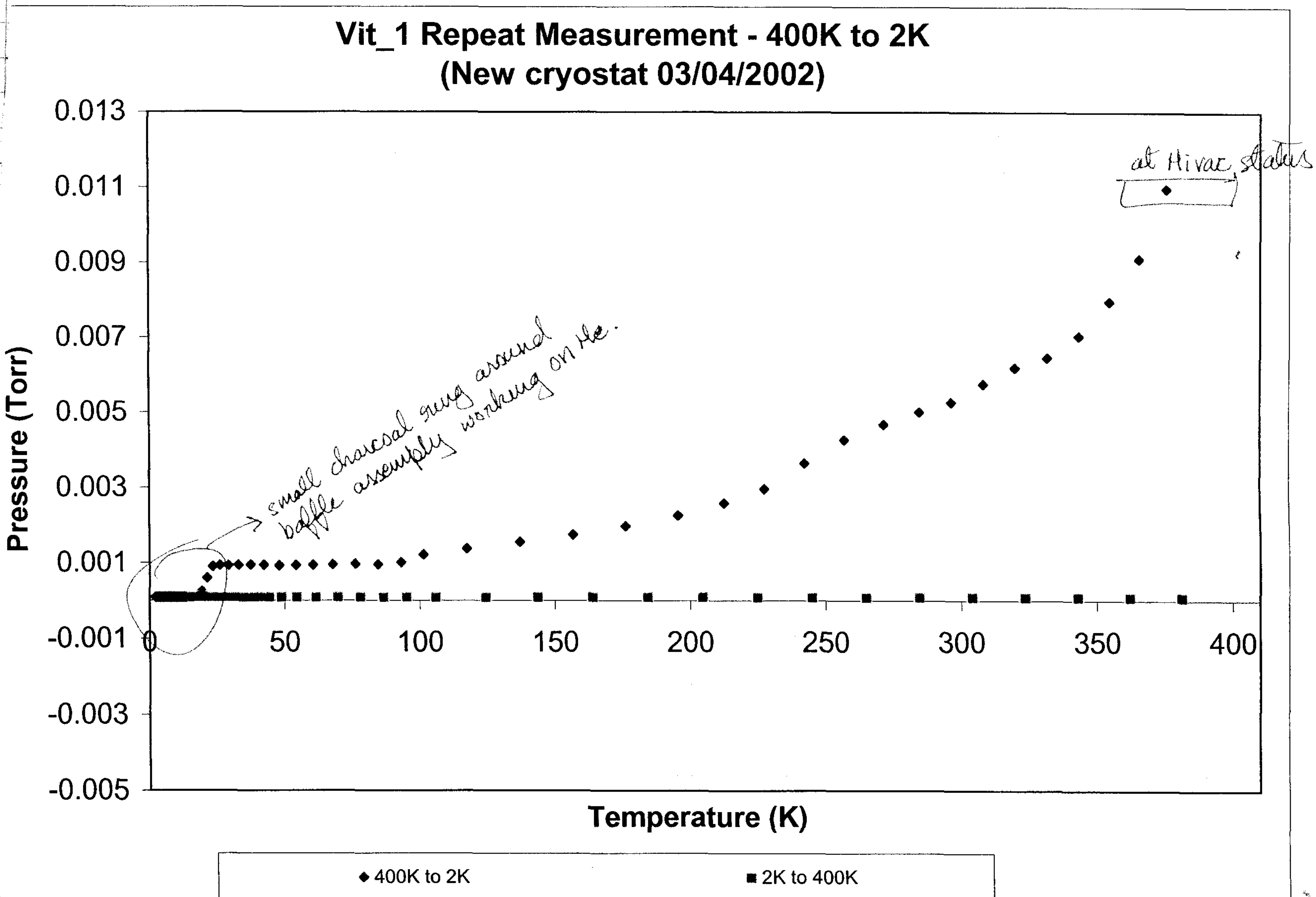
1/4/2002
- Commercial glassy metal = 2826MB
- Baked in air @ 350°C ≃ 20 min
- " " " @ 450°C ≃ 20 min
- Matted side strip used
- Flux = 37% HCl
- Braze = (Au Sn)99 Ti [made in arc melter]

[illegible]

- Commercial glassy met 2826MB 1/4/2002
- Baked in air @ 350°C ≃ 20 min
- " " " @ 450°C ≃ 20 min
- Shiny polished surface of strip
- Flux = 37% HCl
- Braze = (Au Sn)99 Ti [made in arc melter]

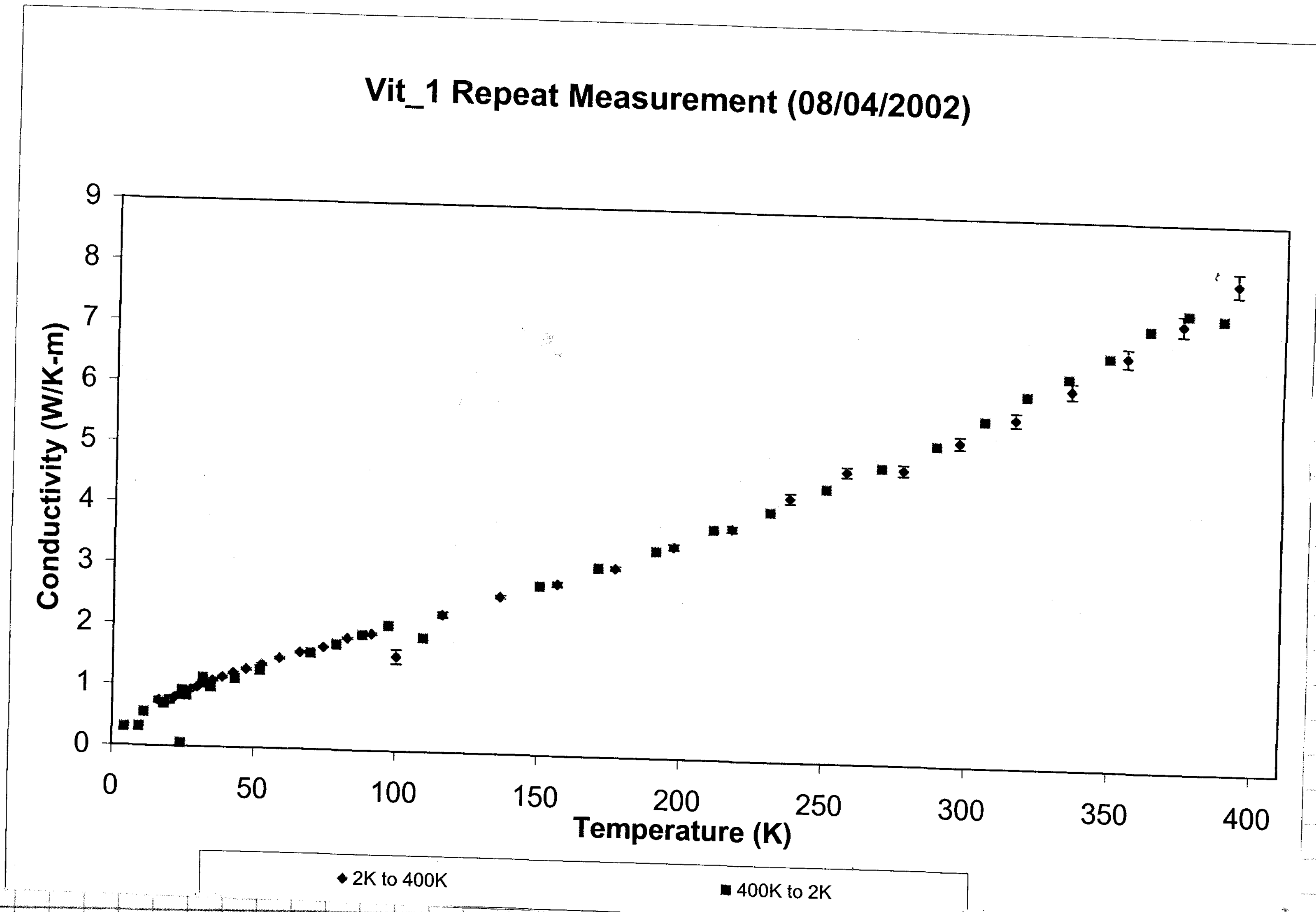
[illegible]

- Sample = 2826 MB
- Baked in air @ 450°C
- Shiny polished surface strip
- Baking Time ≃ 20 min
- Flux = 37% HCl
- Braze = (Au Sn)99 Ti (notes)



Vit_1 Repeat Measurement - 400K to 2K
(New cryostat 03/04/2002)
Pressure (Torr)
0.013
0.011
0.009
0.007
0.005
0.003
0.001
-0.001
-0.003
-0.005
0
50
100
150
200
250
300
350
400
Temperature (K)
400K to 2K
2K to 400K
small charcoal ring around baffle assembly working on He.
at Hivac status

First measurement after installation of new cryopump (cryopump #2)
-> Data on "cryostat" computer in Cryolab
-> Folder C:/cryolab/users/Hareem/~~030402~~ Vit1_030402

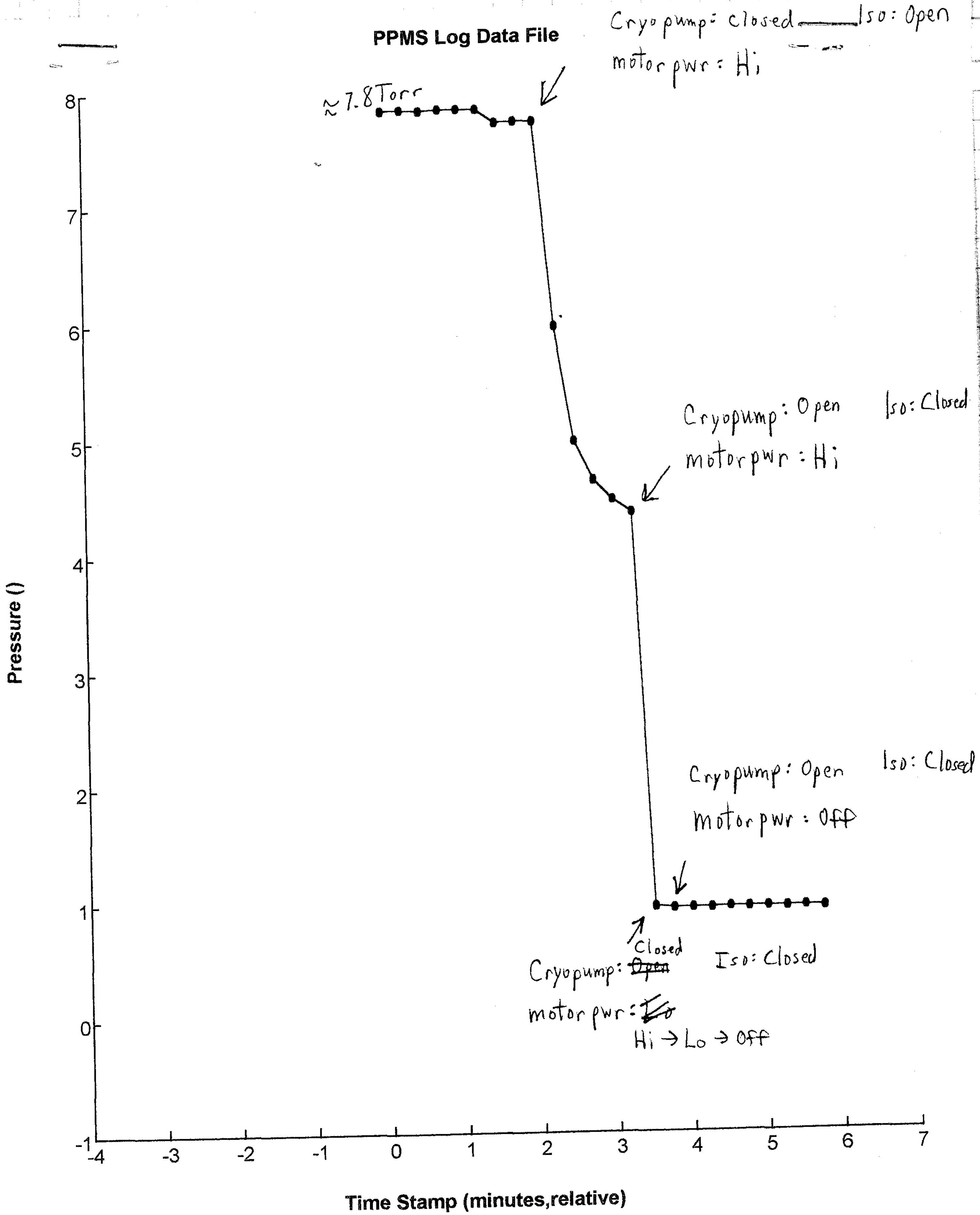

Vit_1 Repeat Measurement (08/04/2002)
Conductivity (W/K-m)
0
1
2
3
4
5
6
7
8
9
Temperature (K)
0
50
100
150
200
250
300
350
400
◆ 2K to 400K
■ 400K to 2K

→ To verify the jump in pressure at low temperatures and to verify the repeatability of the measurement another measurement was taken

→ This time the vacuum was initiated at 2K as I saw a better vacuum ($\approx 8 \times 10^{-5}$ Torr) at this lower temperature.

- The plots to the right were taken to see if there was any difference in obtaining HiVac ~~through~~ Multi-Vu or Model 6000.

· The plots are nearly identical, which indicates ~~that there~~ is no difference. This might indicate that there is no problem with the GPIB board.

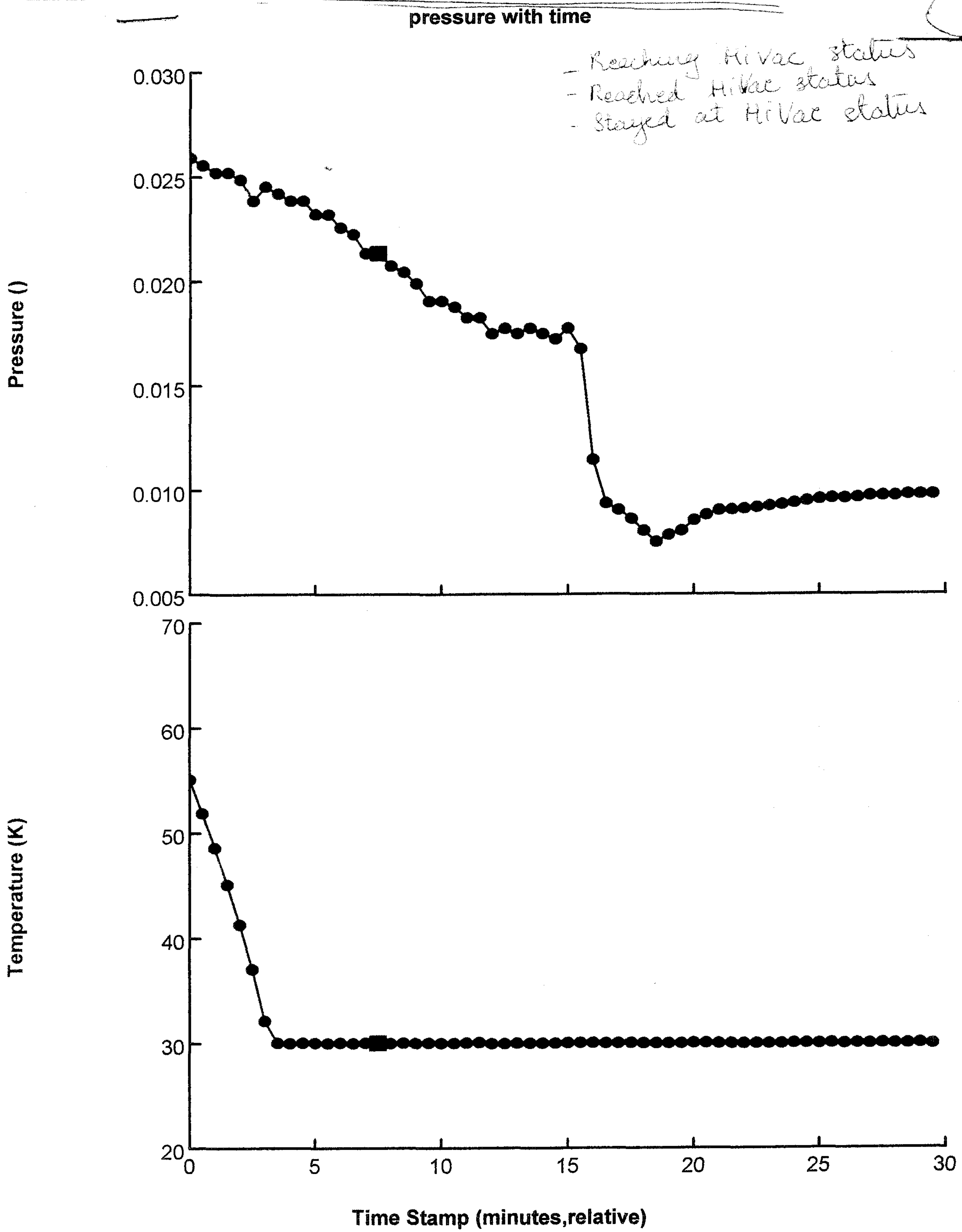


C:\cryolab\users\Mike\Diagnostic (Pressure)\model6000 pressure.04/15/02 11:52:14

Pressure monitoring while reaching HiVac

①a

- Reaching HiVac status
- Reached HiVac status
- Stayed at HiVac status



C:\WINDOWS\Desktop\LogPpmsSeq_00003.dat

04/11/02 11:22:51

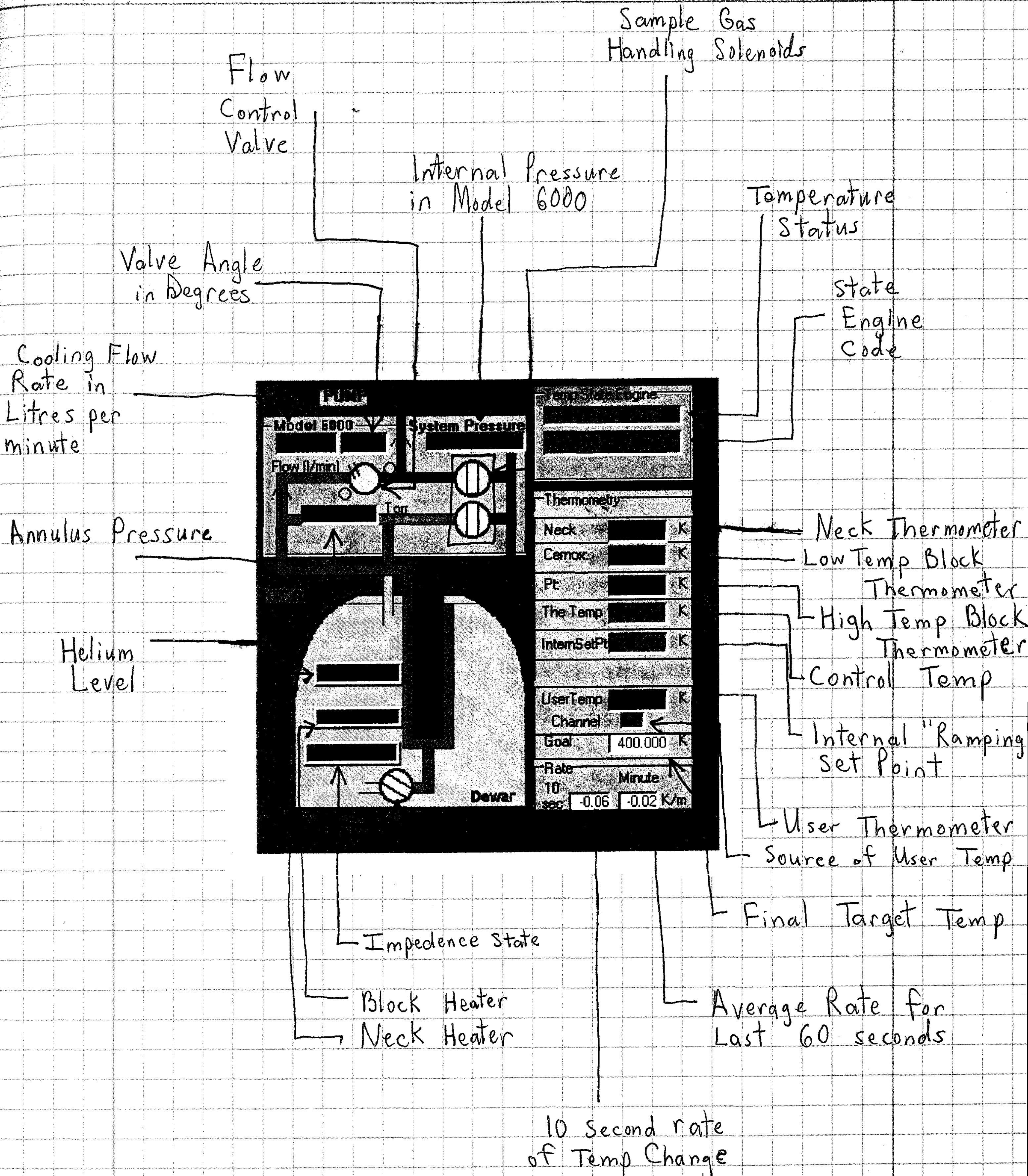


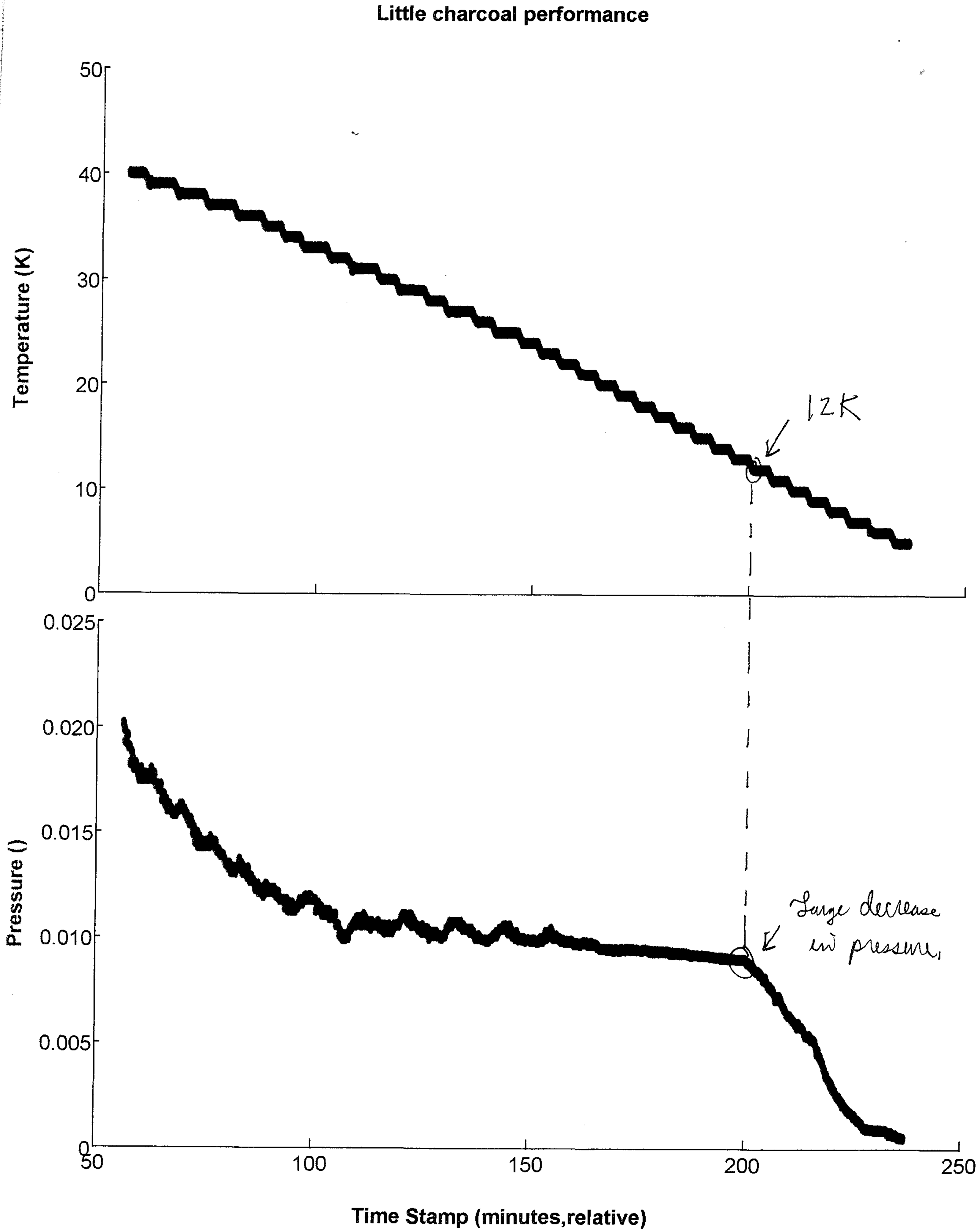
Figure → Gasmon32.exe

- The effectiveness of the charcoal located in the baffle assembly was tested before regeneration of the cryopump charcoal. At this point it appeared as if the cryopump had almost no effectiveness in pumping.

- The point of this plot was to determine when the "little charcoal" was activated, and how its pumping effectiveness varied w/ temperature.

- Online it was found that charcoal begins to absorb Helium around 10K and by 35K, it is completely released.

Little Charcoal Performance (40K - 5K)



C:\cryolab\users\Hareem\newPumpChamber\vit1_170402\LogPpmsSeq_004/18/02 14:10:54

- After charcoal regeneration, the first HiVac operation was successful, although a long time was needed to achieve 9×10^{-5} Torr. It remained stable for awhile, but the next morning the pressure spiked when no changes had been made to the system.

- Quantum Design was notified on April 19, and plots were sent to them on April 22.

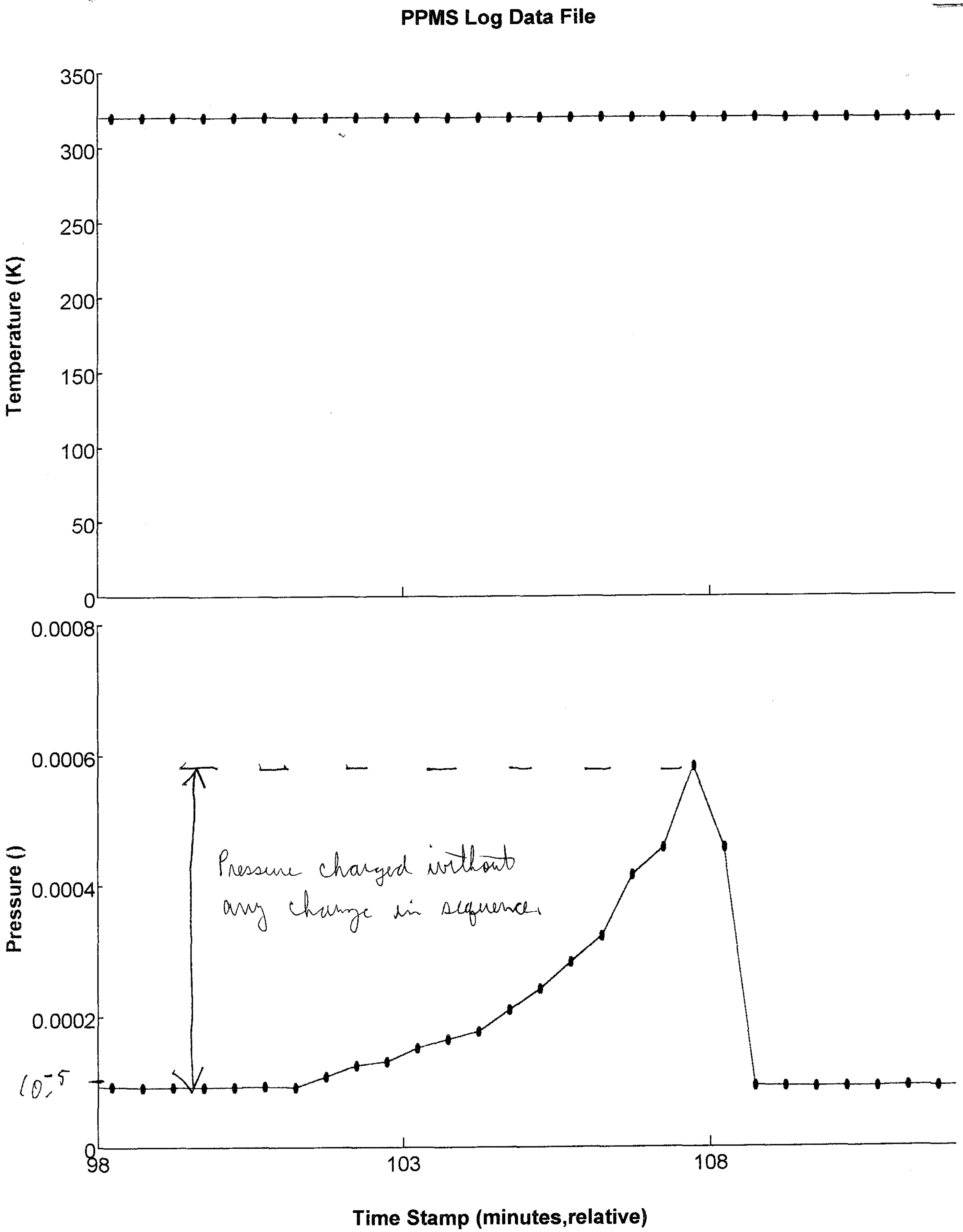
Regeneration of Charcoal

- On April 18, the charcoal was regenerated 4 times:

  1. Warm regeneration in room.
  2. Warm " in room using heat gun to warm the charcoal.
  3. In room regeneration, but "dewar" was selected to take advantage of the heater in the cryopump.
  4. Normal warm regeneration in room.

- The cryopump was then held at 400K for 30 minutes to cool down after regeneration.

Pressure "Stability" At 320K at HiVac



C:\cryolab\users\Mike\04-18-2002 - PostRegeneration\04-19-2002 04/19/02 12:40:25

Cryostat Manual Control (Valves)

Use "Cryo.exe" for manual control.

On the "Cryopump" tab, the cryopump flapper is controlled by the open/close high/low power buttons.

** Always use low power control to avoid machine damage!! **

When the red/black indicator changes state, click the "off" button IMMEDIATELY!!

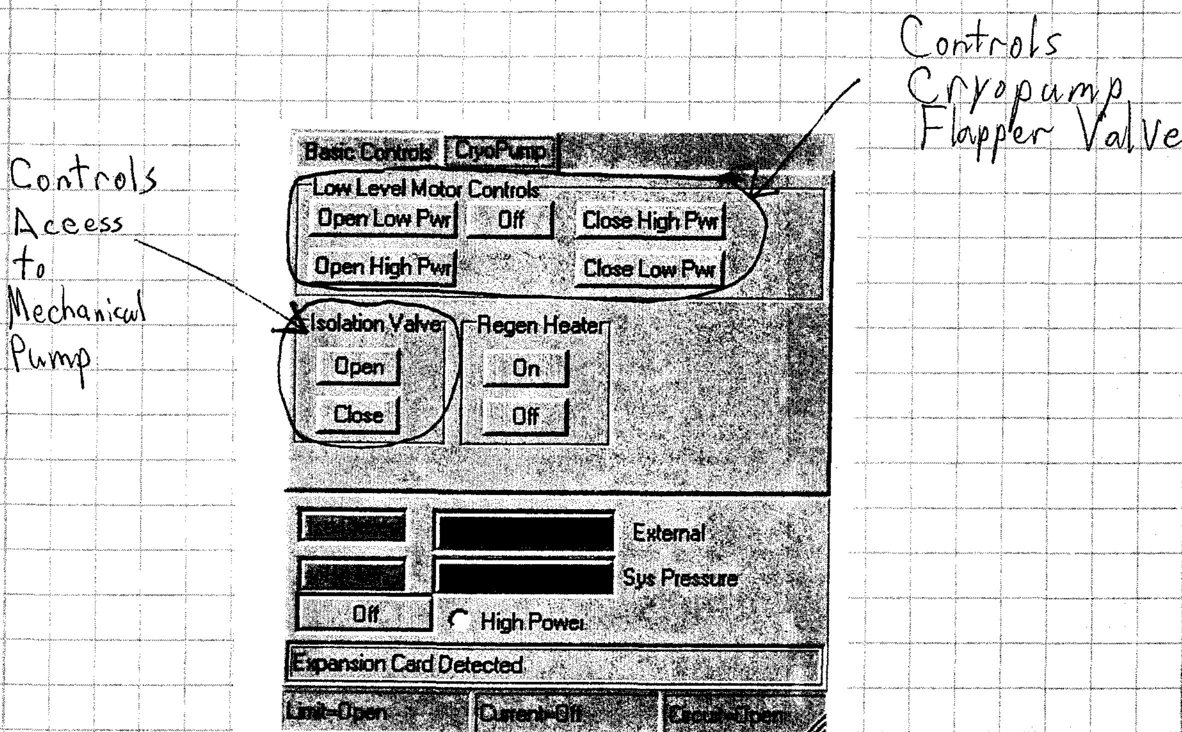


Figure – Cryopump.exe

April 22, 2002

- Thanh was notified of peculiarities found on 4-18-02 and 4-19-02, after the charcoal in the cryopump was regenerated.

- A list of these peculiarities and the e-mail sent follows on the next several pages.

耀萼BAS殖 HR殖F="虎file://C:\WINDOWS\TEMP\eud13.htm范> 萼STY蓃E> 菓TML荫{ f葯nt-葦ami葬y :荫"Times New Roman" 蔞 From: Thanh Dang <thanh.dang@qdusa.com>
To: Michael Hall <mhall@ligo.caltech.edu>
Date: Tue, 23 Apr 2002 08:34:02 -0700
Subject: Re: ATTN: Than
CC: htariq@ligo.caltech.edu, desalvo@ligo.caltech.edu
Priority: normal
X-mailer: Pegasus Mail for Windows (v2.54)

I usually keep the helium level above 60%. The best way to go is to fill your helium up to about 80-85% before running any experimet just in case your experiment runs too long.

Thanh

> Date: Mon, 22 Apr 2002 16:15:10 -0700
> To: service@qdusa.com
> From: Michael Hall <mhall@ligo.caltech.edu>
> Subject: ATTN: Than
> Cc: htariq@ligo.caltech.edu, desalvo@ligo.caltech.edu

> Than,
>
> I also had a question about how the Helium level effects the cryopump. At
> what Helium level would you expect the cryopump to lose its effectiveness
> in pumping? This question is not directly related to any of the issues
> that I have asked about in my previous email (In other words, we operated
> the above experiments with a healthy Helium level), however it was a
> question that Hareem and I had been talking about earlier today, and it's
> certainly something that we should be aware of. Thanks again for your
> help.....
>
> -Michael Hall
>
>

To: service@qdusa.com
From: Michael Hall <mhall@ligo.caltech.edu>
Subject: ATTN: Than
Cc: htariq@ligo.caltech.edu, desalvo@ligo.caltech.edu
Bcc:
Attached: C:\WINDOWS\Desktop\04-18-2002 postregen.dat; C:\WINDOWS\Desktop\04-19-2002 postregen.dat;

Than,

Attached to this e-mail is the information that we discussed on Friday over the phone. I have included all of the data that has been taken since the charcoal regeneration on Thursday, and I have pointed out the peculiarities that we have noticed and are most concerned about. Any advice and/or guidance that you can provide would be most helpful. If you have any information about why some of these peculiarities are appearing in our data, explanations would be greatly appreciated.

First, I will go over what we have noticed in the data file labeled "04-18-2002 postregen.dat".

* This plot starts off normally at 400K. We had just finished regenerating the charcoal and had waited over 30 minutes at 400K. Thereafter, we immediately entered HiVac, and although it was taking a very long time to do so, it appeared as though it would reach 9e-5, as expected. At 22 minutes, we wanted to test for a leak, so the cryopump flap was sealed. At this point, we noticed a linear increase in pressure. The pressure increase was calculated as was the corresponding leak (or out-gassing) rate:

dP/dt = 0.0627 mT/sec.

Leak Rate = dP/dt * (Volume)

Where the volume of the sample chamber is approximately:

Volume ~ (100cm) * Pi * (2.5cm) * (2.5cm) = 1962.5 cm^3

So the leak rate is:

Leak Rate = 0.123 Torr * cc/sec.

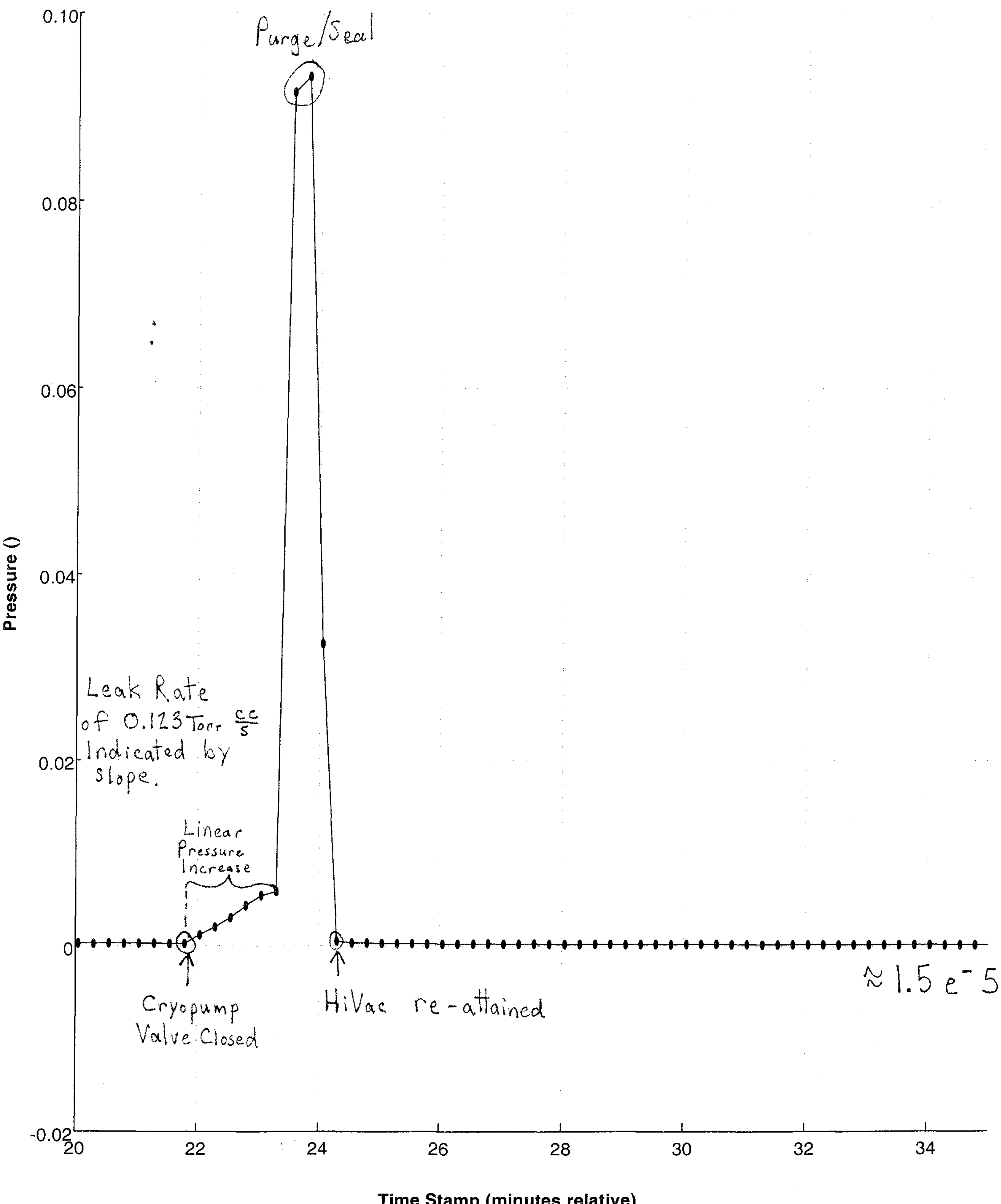
Is that small enough to be attributed to out-gassing alone, or could this indicate that a leak does exist? Please shed as much light as possible on this issue, because it is a very important one.

* HiVac was eventually attained at approximately 400K. The temperature was reduced to 10K and left running overnight. Although the pressure was very stable over this period, this run was the only one that successfully held the pressure low and stable.

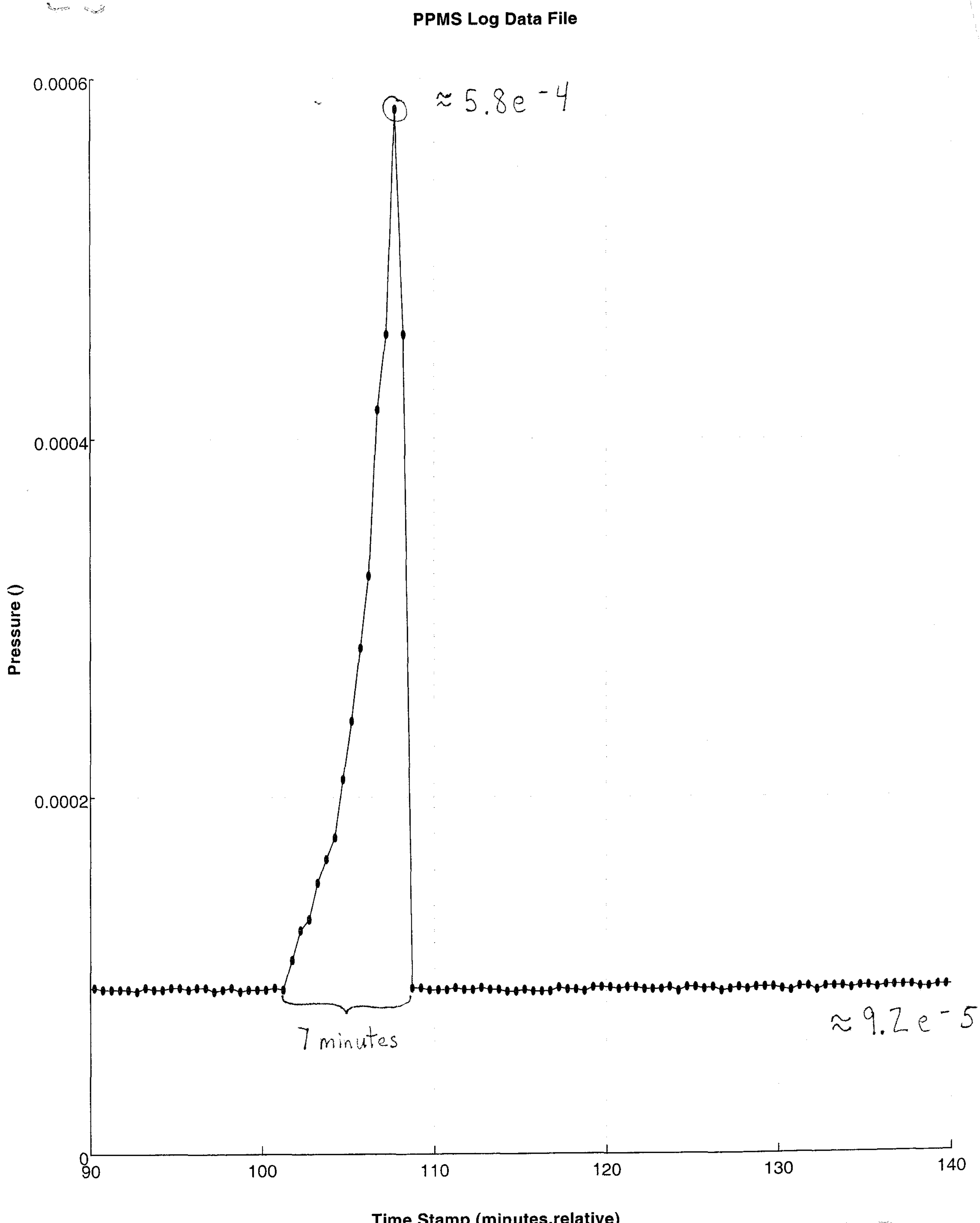
Secondly, I will go over what we have noticed in the data file labeled "04-19-2002 postregen.dat".

Seal Chamber @ 22 Minutes

PPMS Log Data File

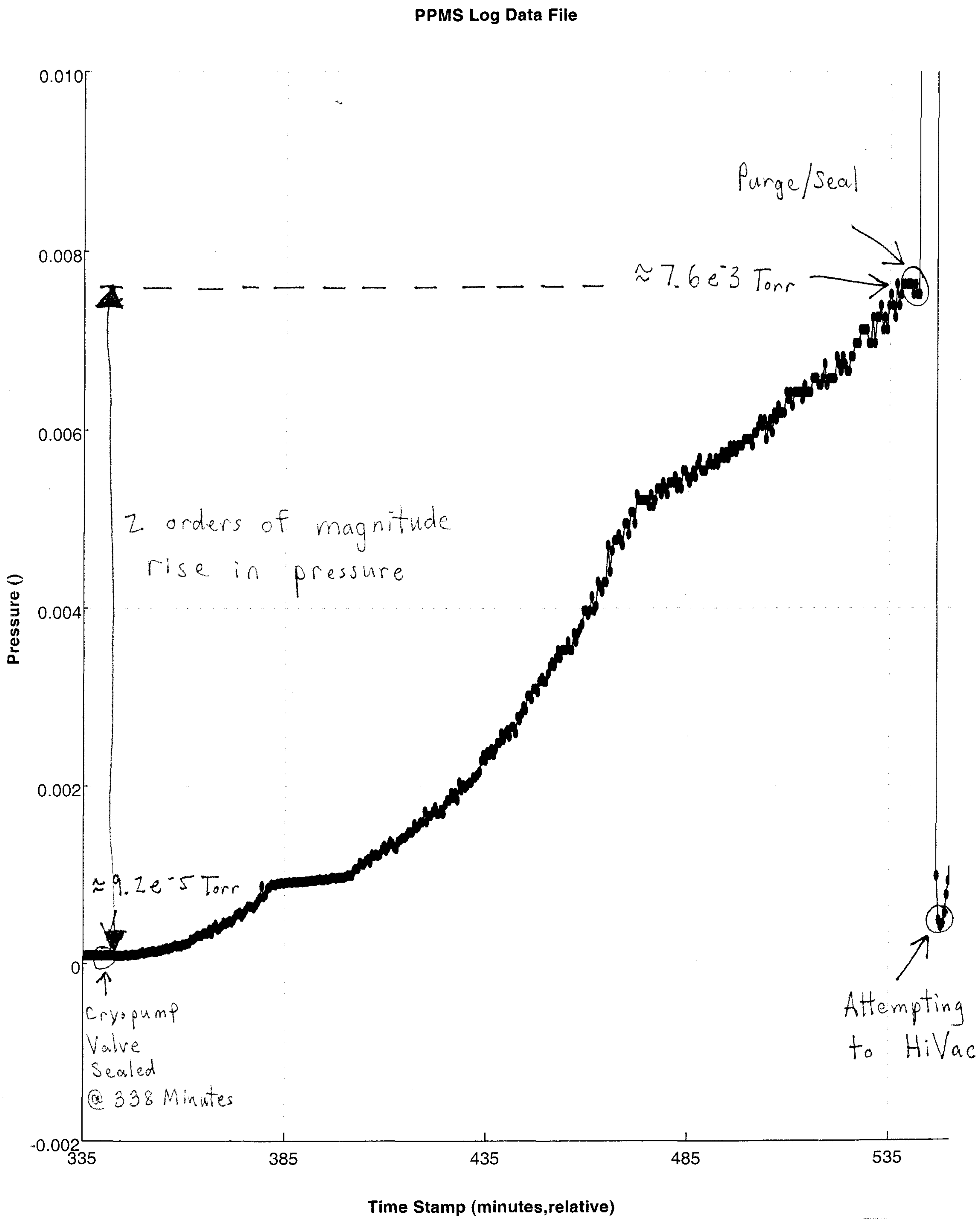


Sudden Rise in Pressure For No Apparent Reason @ 100 Minutes

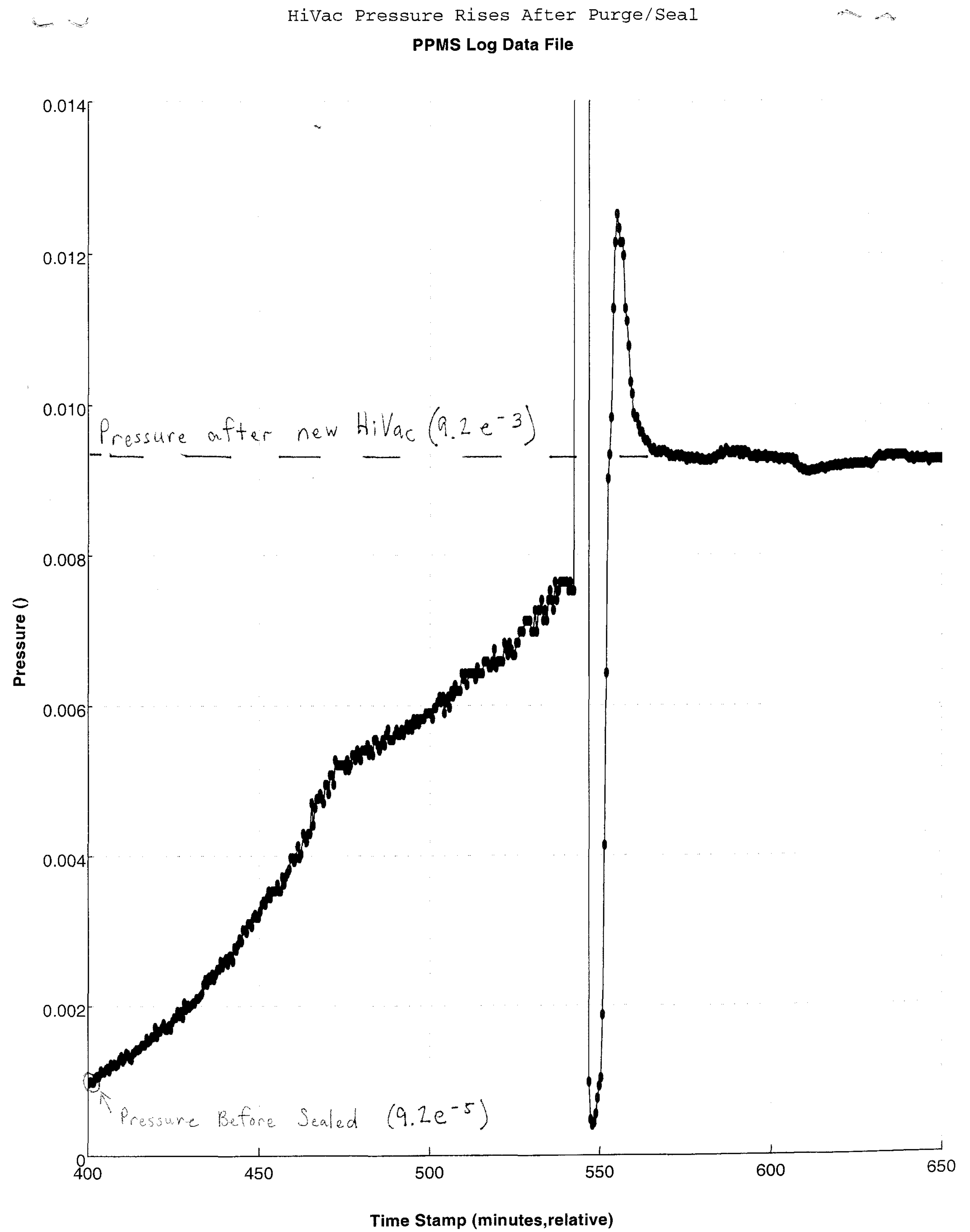


C:\WINDOWS\Desktop\04-19-2002 postregen.dat 04/23/02 11:46:58

Large Pressure Gain After Closing Cryopump Valve @ 338 Minutes



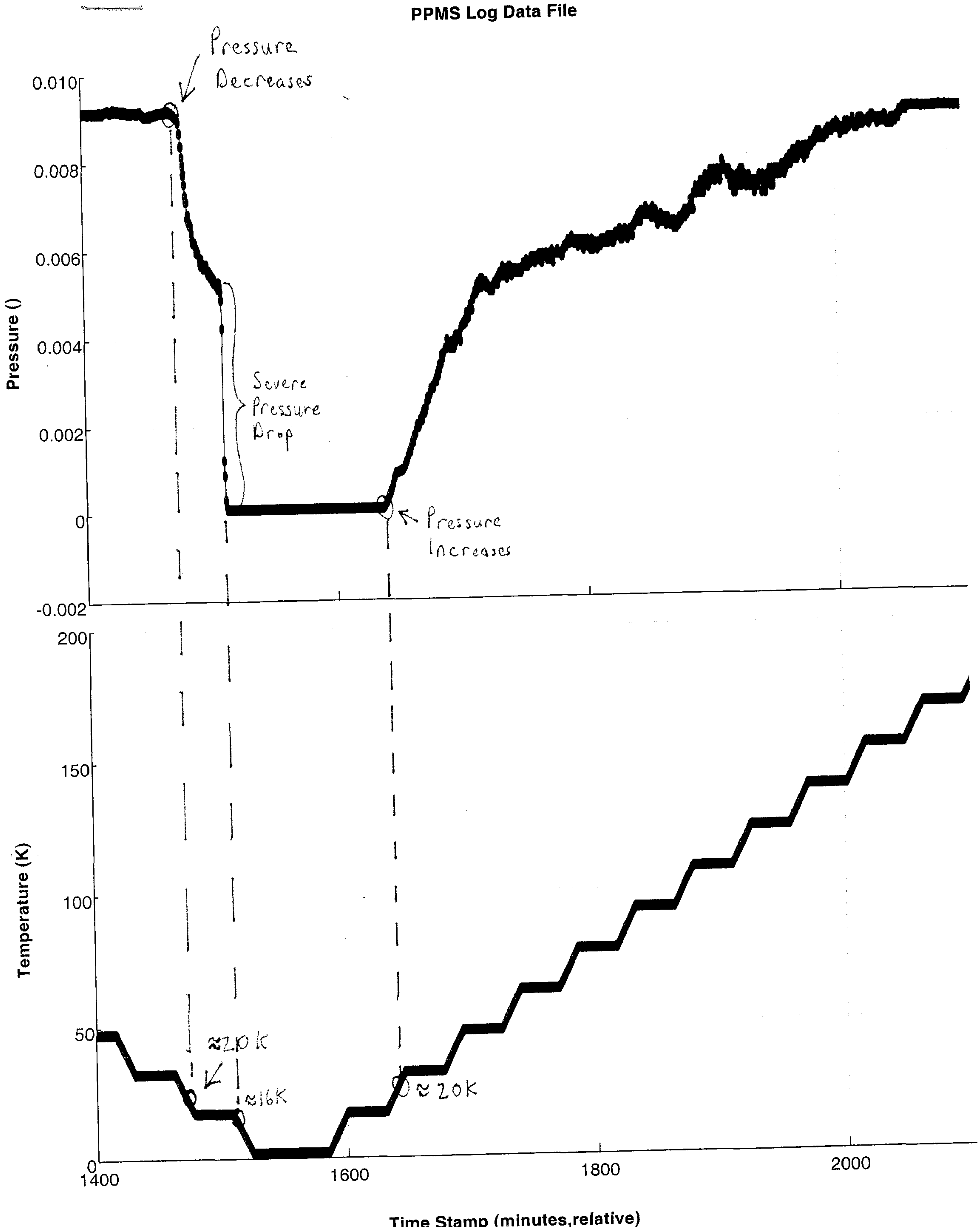
C:\WINDOWS\Desktop\04-19-2002 postregen.dat 04/23/02 11:52:29



C:\WINDOWS\Desktop\04-19-2002 postregen.dat

04/23/02 11:59:50

Pressure Decreases Below 20K Because of "Little Charcoal"

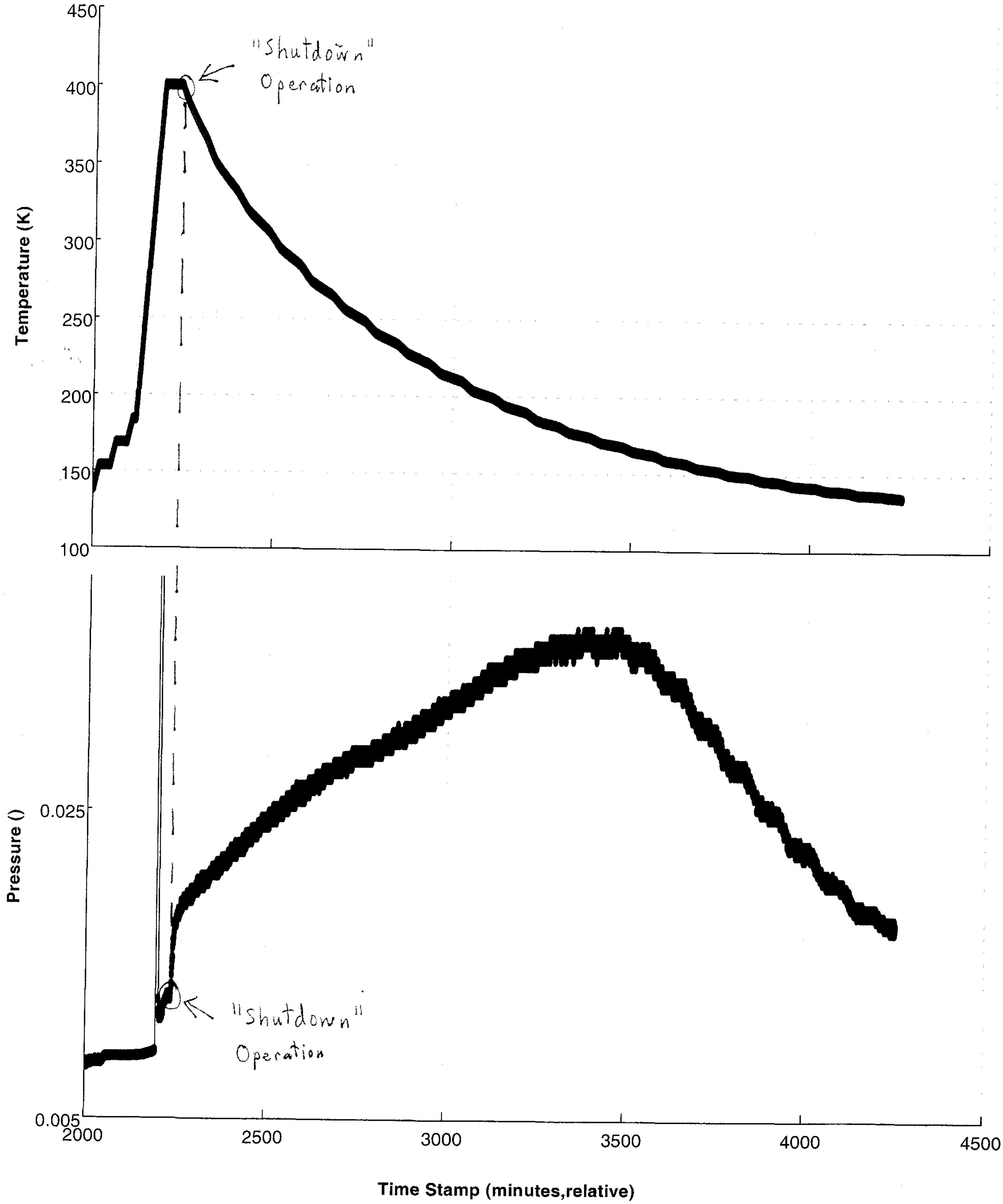


C:\WINDOWS\Desktop\04-19-2002 postregen.dat

04/23/02 12:01:47

Pressure Performance After Executing "Shutdown"

PPMS Log Data File



C:\WINDOWS\Desktop\04-19-2002 postregen.dat

04/23/02 12:03:25

Sequence File: postreg_190204

This overnight sequence tested HiVac through a range of temps.

```
 1:  LogData Start New 60.00 1048575 2097151 7 "C:\cryolab\users\Hareem\newPumpChamber\Sealed
     Cryopump Test\04-19-2002-cont.dat" "continuing the previous monitoring with changing temp
     " ""
 2:  Wait For Temperature, Delay 3600 secs, No Action
 3:  Chamber Vent then Seal
 4:  Wait For Chamber, Delay 60 secs, No Action
 5:  Chamber Purge then Seal
 6:  Wait For Chamber, Delay 60 secs, No Action
 7:  Chamber High Vacuum
 8:  Wait For Chamber, Delay 60 secs, No Action
 9:  Scan Temp from 320.0K to 2.0K at 1.0K/min, in 22 steps, Uniform, Fast
10:      Wait For Temperature, Delay 1800 secs, No Action
11:  End Scan
12:  Scan Temp from 2.0K to 320.0K at 1.0K/min, in 22 steps, Uniform, Fast
13:      Wait For Temperature, Delay 1800 secs, No Action
14:  End Scan
15:  Chamber Vent then Seal
16:  Wait For Chamber, Delay 60 secs, No Action
17:  Chamber Purge then Seal
18:  Wait For Chamber, Delay 60 secs, No Action
19:  Chamber High Vacuum
20:  Wait For Chamber, Delay 1800 secs, No Action
21:  LogData Stop ""
22:  Shutdown Temperature Controller
```

Stopped at approximately 150 K.

(Due to low Helium level.)

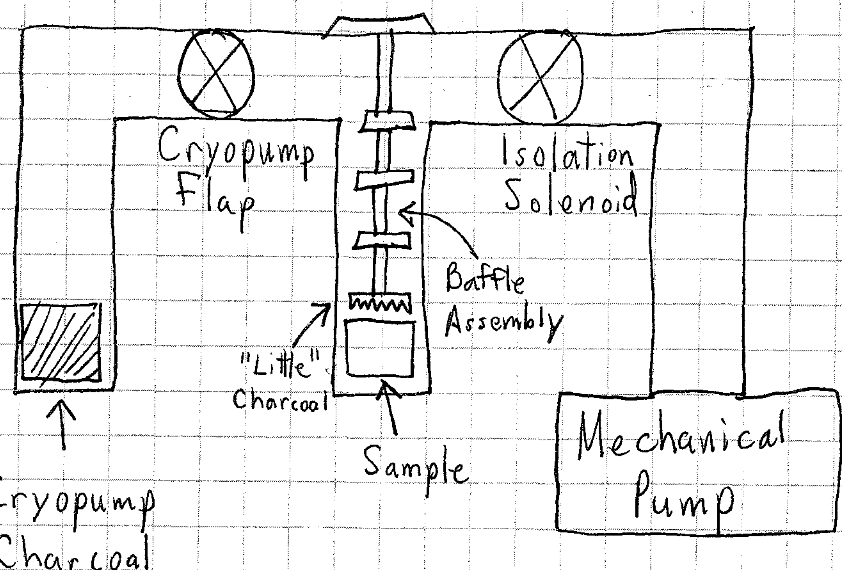
Sequence File: postreg_190204_cont200402

This was the sequence used to shutdown the system.

```
 1:  continuation of post regen measurement on the 19-04-02 as we were running out of He
 2:  LogData Start New 30.00 1048575 2094079 7 "C:\cryolab\users\Hareem\newPumpChamber\Sealed
     Cryopump Test\04-19-2002-cont04-20-02.dat" "continuing the previous monitoring with chang
     ing temp" ""
 3:  Set Temperature 400.00K at 3.00K/min. Fast Settle
 4:  Wait For Temperature, Delay 300 secs, No Action
 5:  Chamber Purge then Seal
 6:  Wait For Chamber, Delay 300 secs, No Action
 7:  Chamber High Vacuum
 8:  Wait For Chamber, Delay 1800 secs, No Action
 9:  LogData Stop ""
10:  Shutdown Temperature Controller
```

I talked to Frank on the phone today about these plots. He says he thinks it is a problem with the electronics.

After each purge / seal, the cryopump's efficiency is reduced. Frank suspects this is caused by poor timing between the two valves. (See figure):

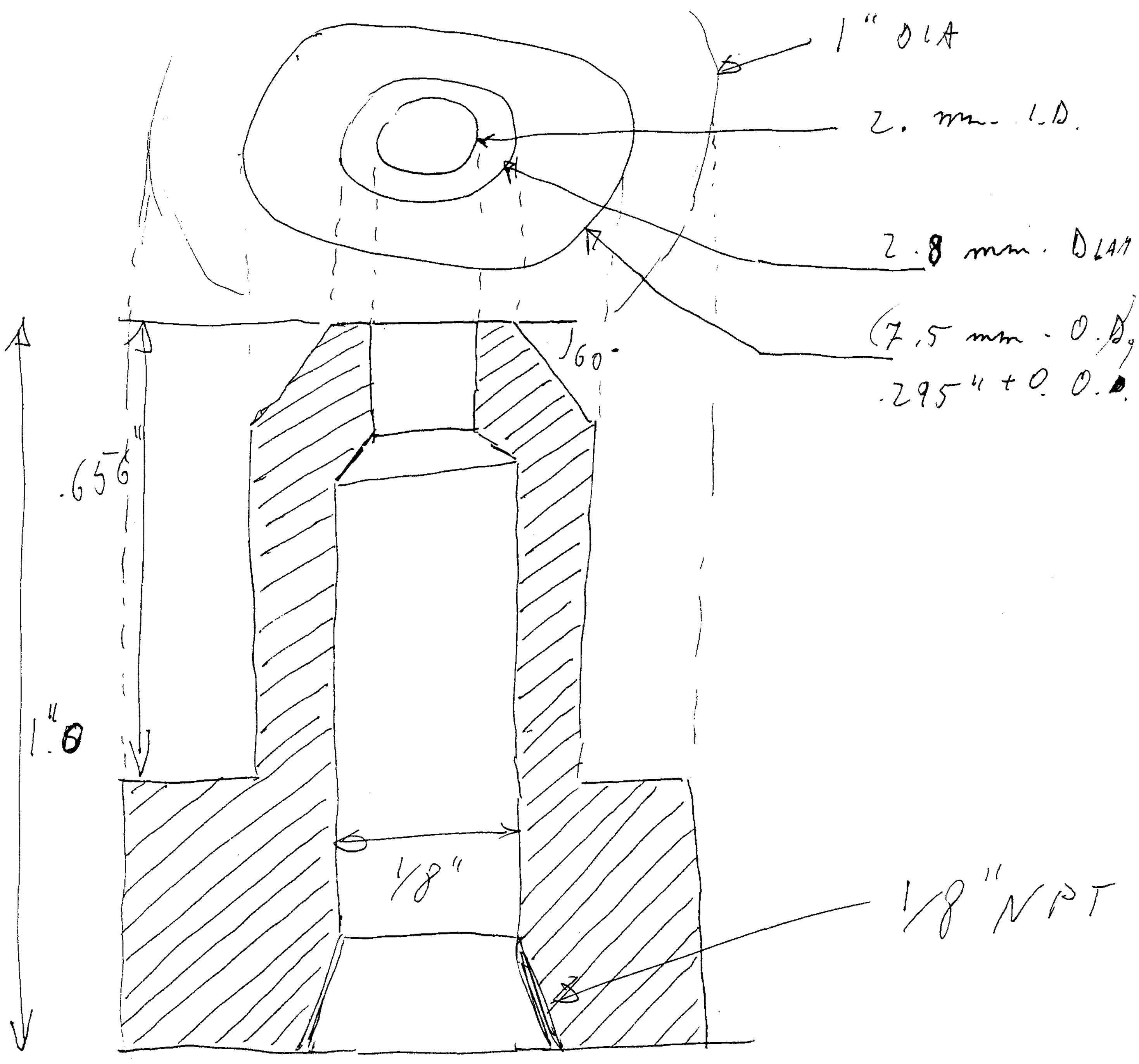


When a purge/seal takes place, it is possible that the cryopump flap does not fully close when the isolation valve opens. This would flood the cryopump charcoal with particles, eventually saturating it.

Frank says he will contact us towards the end of the week and let us know when service engineers can replace the parts.

A Heat Capacity Sample Puck Holder.

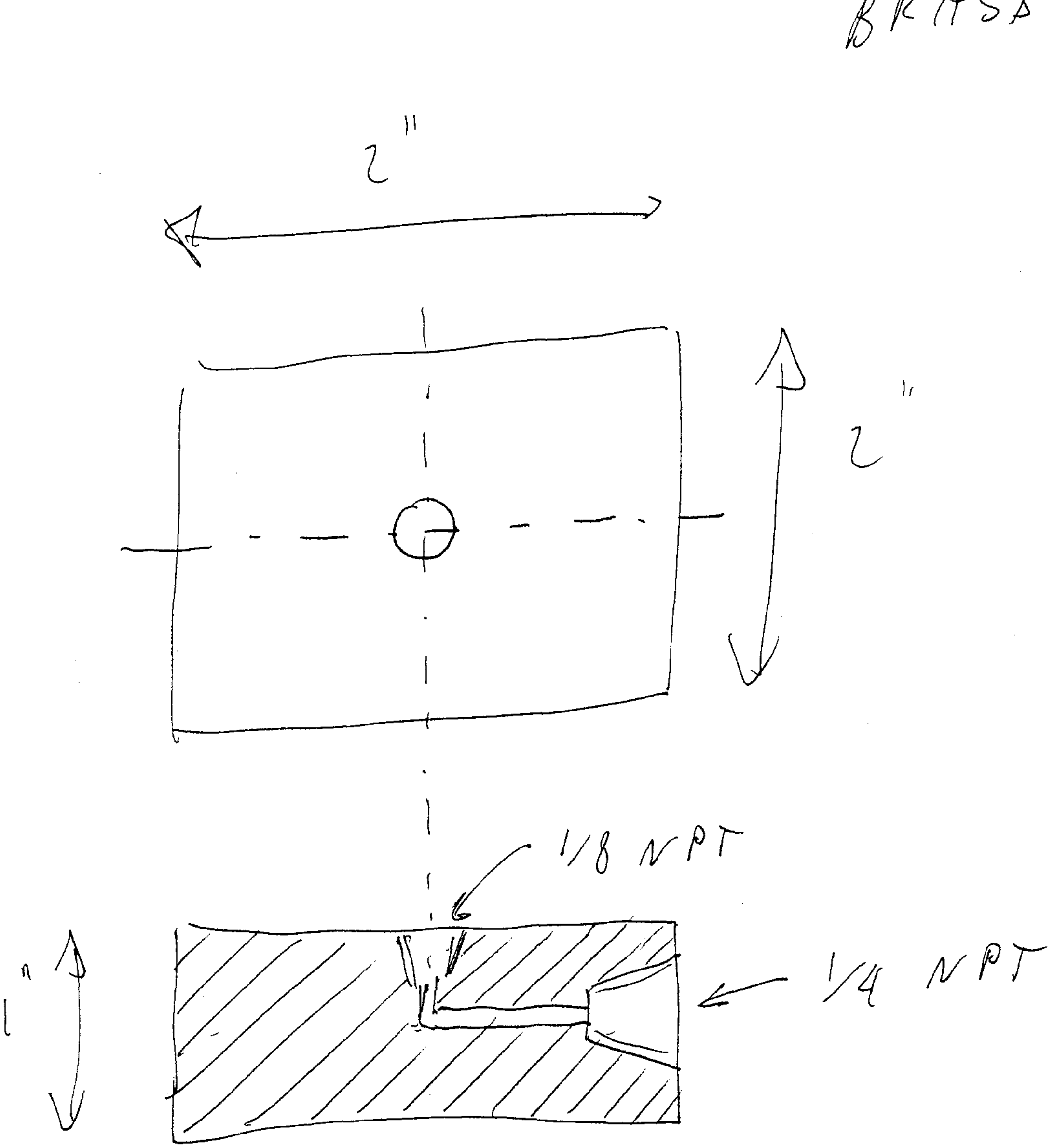
The following design was made by Riccardo as a vacuum system that will be used to keep the puck in place during the sample mounting process.



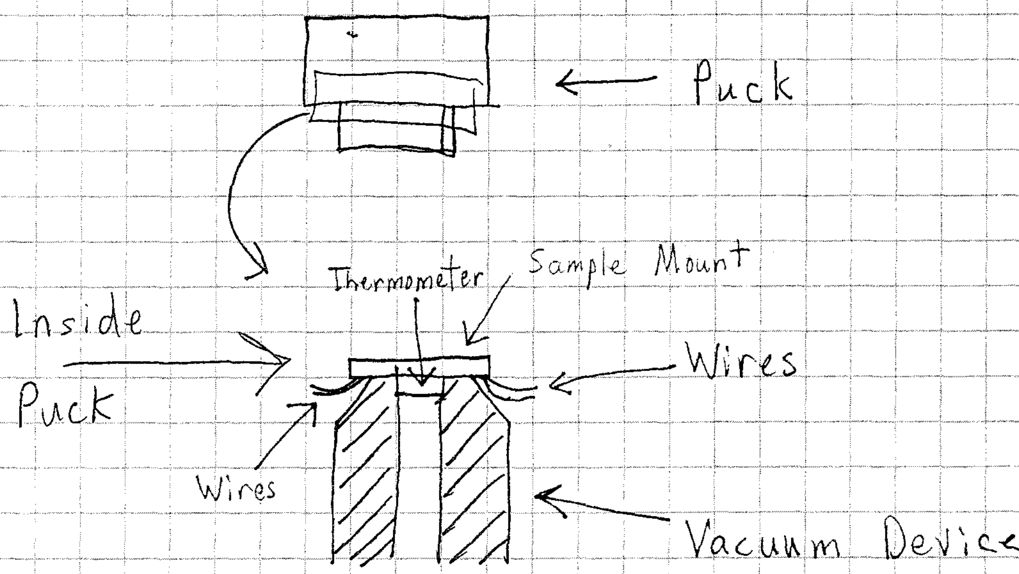
PVC 1 PIECE

The puck sits on top of the upper structure located on the previous page. That entire structure sits on top of the brass plate described below.

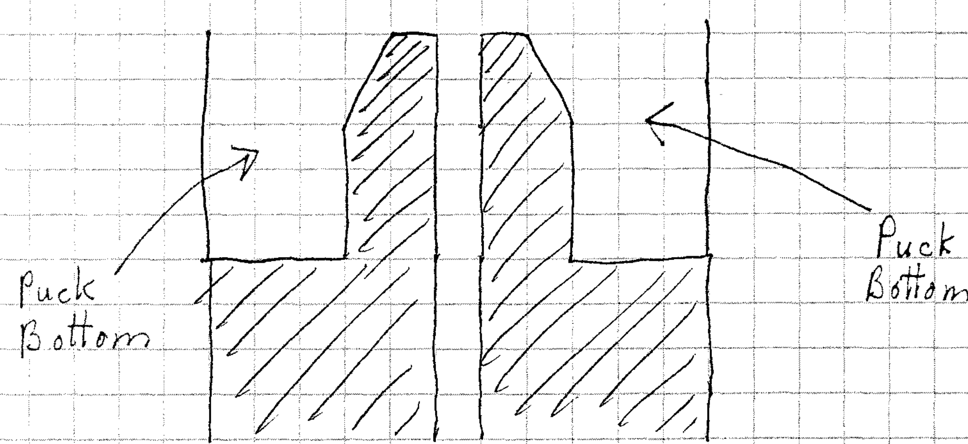
The hollowed out tube on the previous page is large enough to fit the thermometer on the underside of the puck but small enough that it does not damage the mass.



For example,



The bottom (blue) part of the puck encases the outer diameter of the vacuum device.



The picture below gives a very good, though partially incomplete, picture of the gas and pressure system of the Cryostat. The picture includes no reference to the cryopump, which is not included in the standard installation.

At the point marked ⊗, is the location of the isolation valve in the cryopump. It is for this reason that the "system pressure" does not represent the pressure in the sample chamber.

The vent valve opens when the sample chamber is purged with Helium boil-off. The flush valve opens when the sample chamber is pumped out by the mechanical pump.

The mechanical pump is always operating because it is needed to facilitate the flow of gaseous Helium through the cooling annulus, with the assistance of the flow control valve and the differential pressure sensor.

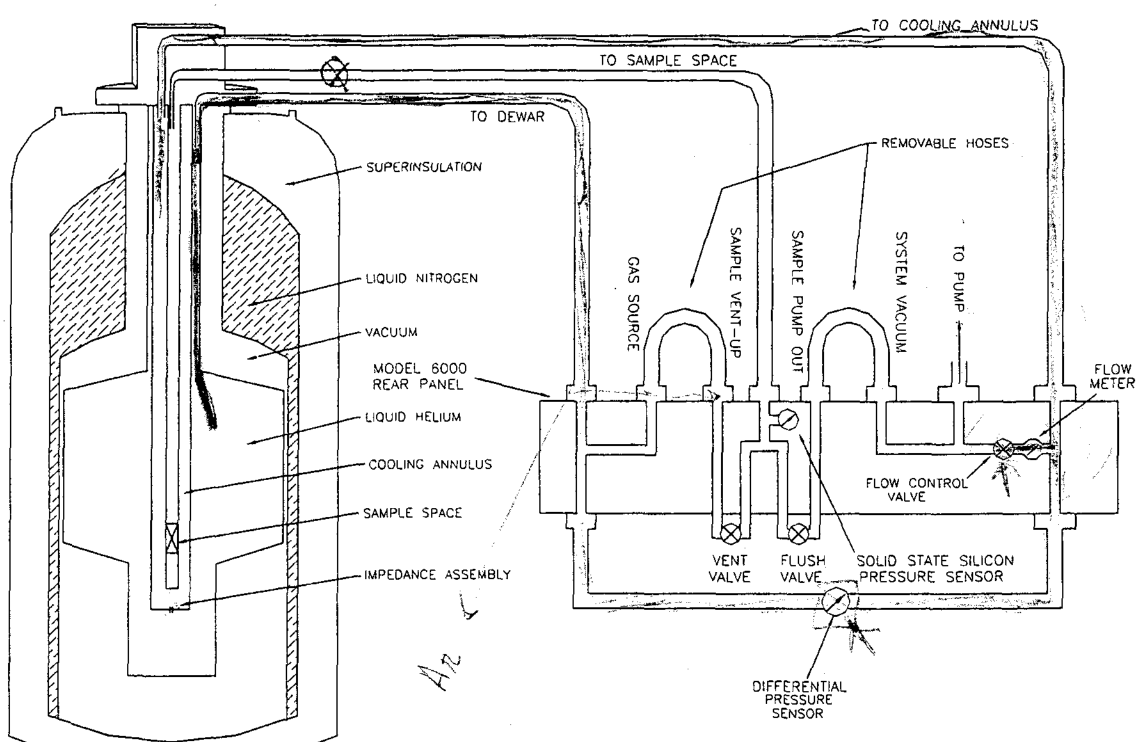
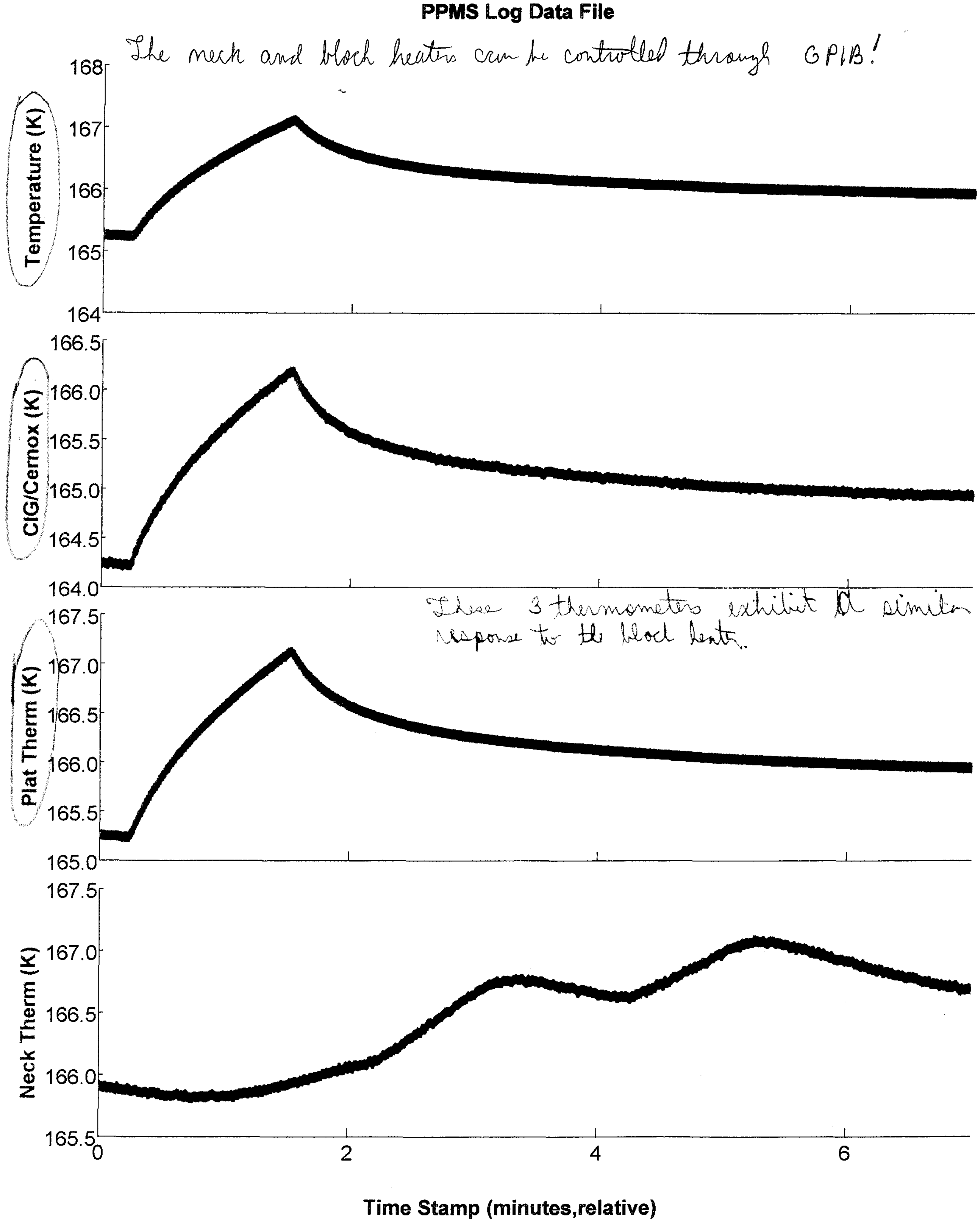


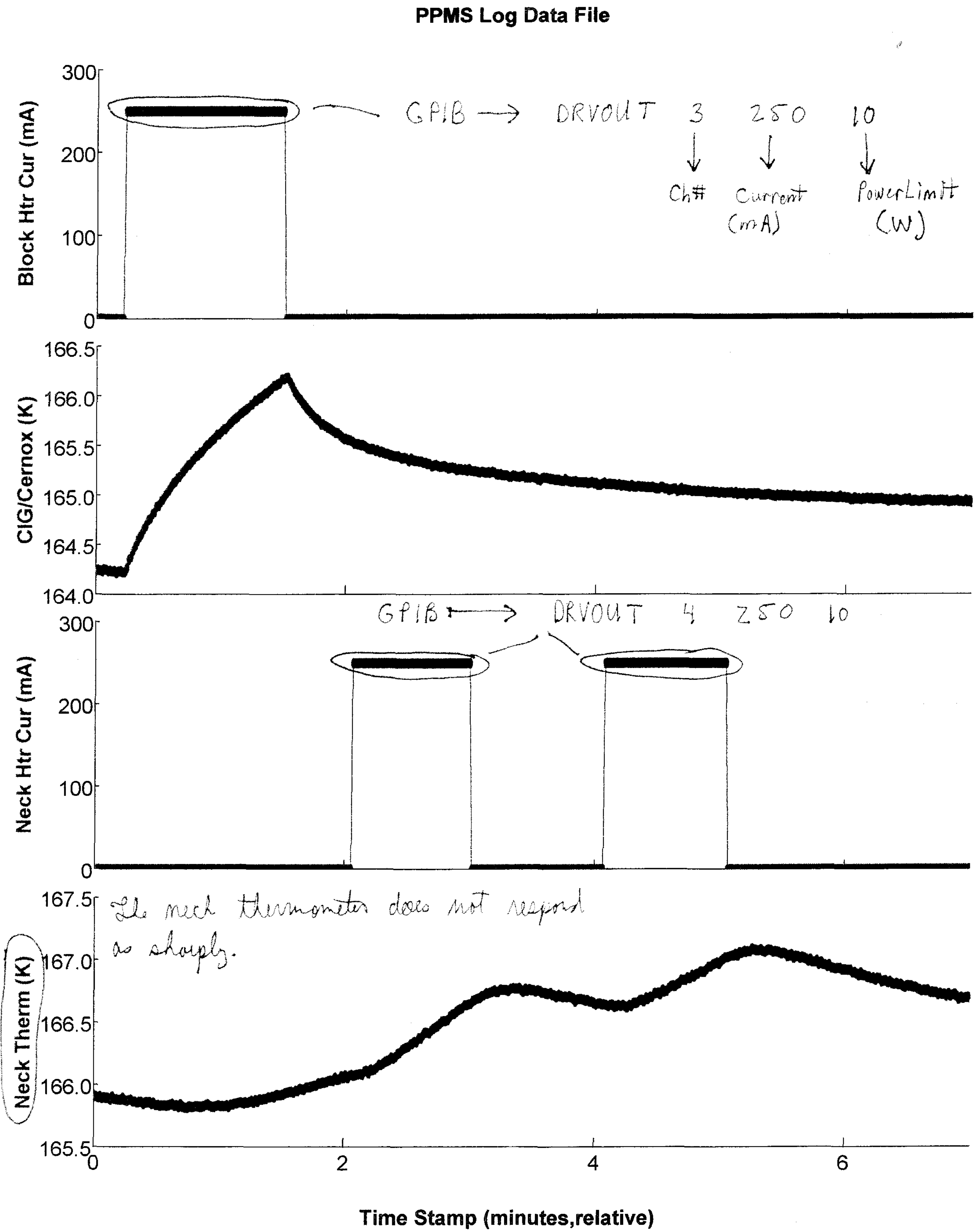
Figure - From page 3-5 in PPMS hardware Manual.

Manipulating Heater Through Driving Currents



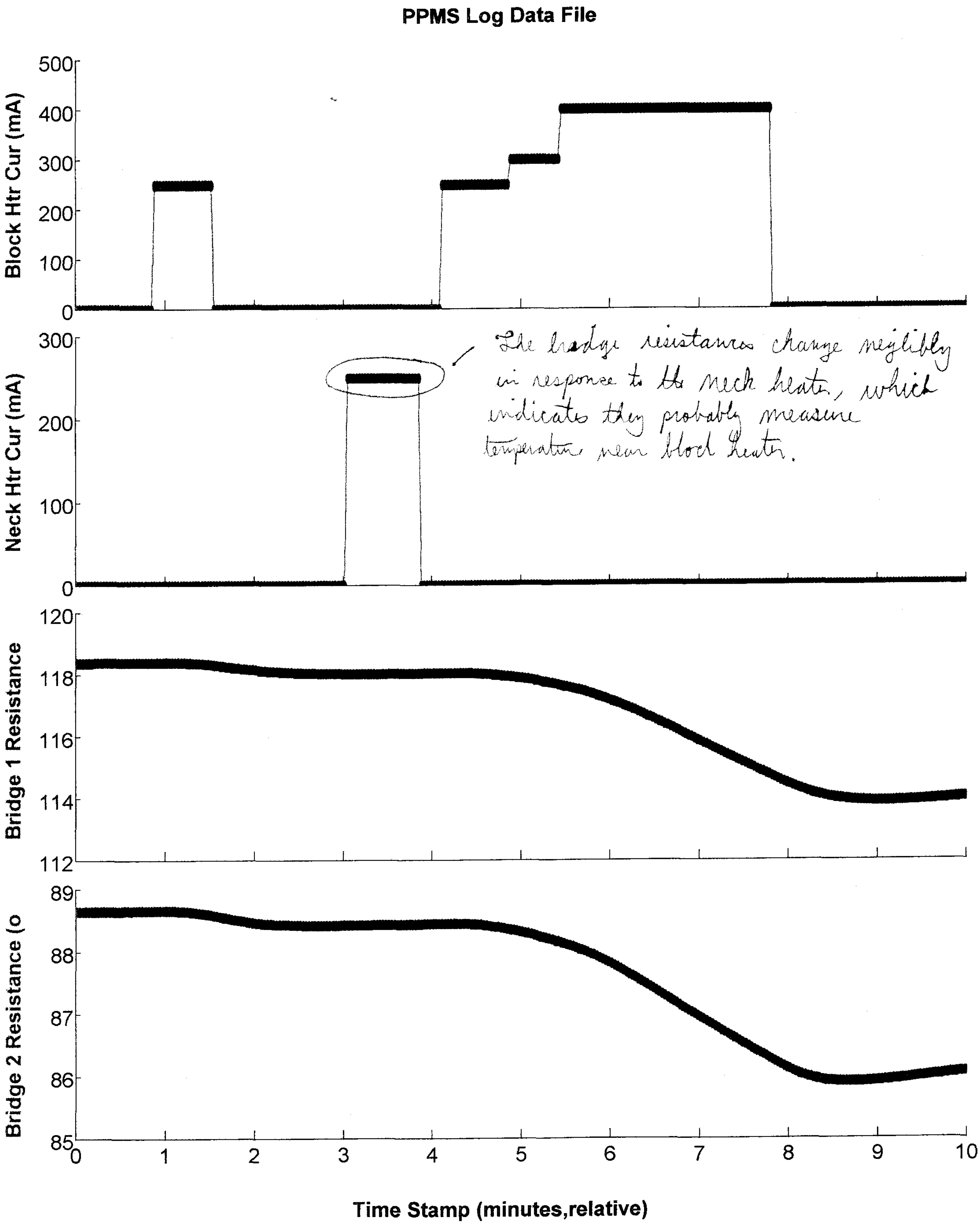
C:\WINDOWS\Desktop\testme.dat 04/25/02 14:38:27

Manipulating Heater Through Driving Currents



C:\WINDOWS\Desktop\testme.dat 04/25/02 14:40:29

Bridge Resistance vs. Drive Current



The bridge resistances change negligibly in response to the neck heater, which indicates they probably measure temperature near block heater.

C:\WINDOWS\Desktop\bridgeresistancetest.dat 04/25/02 14:55:20

April 29, 2002

A new contact at Quantum Design was made today:

Neil Dilly - Physicist - TTO Expert

Neil referred us to somebody at UC San Diego who is using the User bridge for heat capacity measurements.

Vivian - Grad. Student at UC San Diego

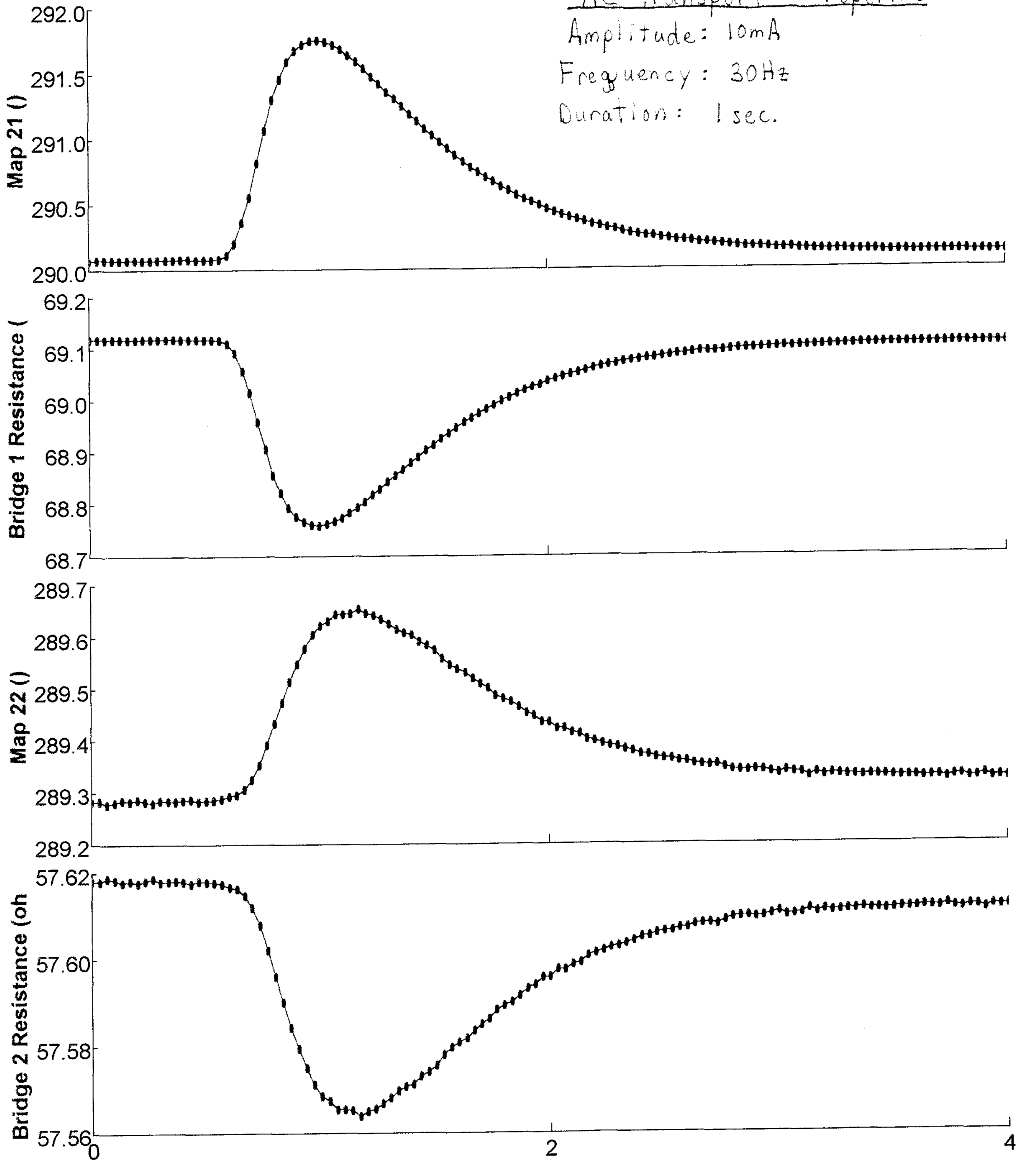
Vzapf@physics.ucsd.edu

The next 2 pages depict the response of the hot/cold thermometers (TTO) when the heater is controlled by the AC Transport option.

Warm & Cold Temperature Reaction From AC Transport Heater Control

PPMS Log Data File

AC Transport Properties.
Amplitude: 10mA
Frequency: 30Hz
Duration: 1 sec.

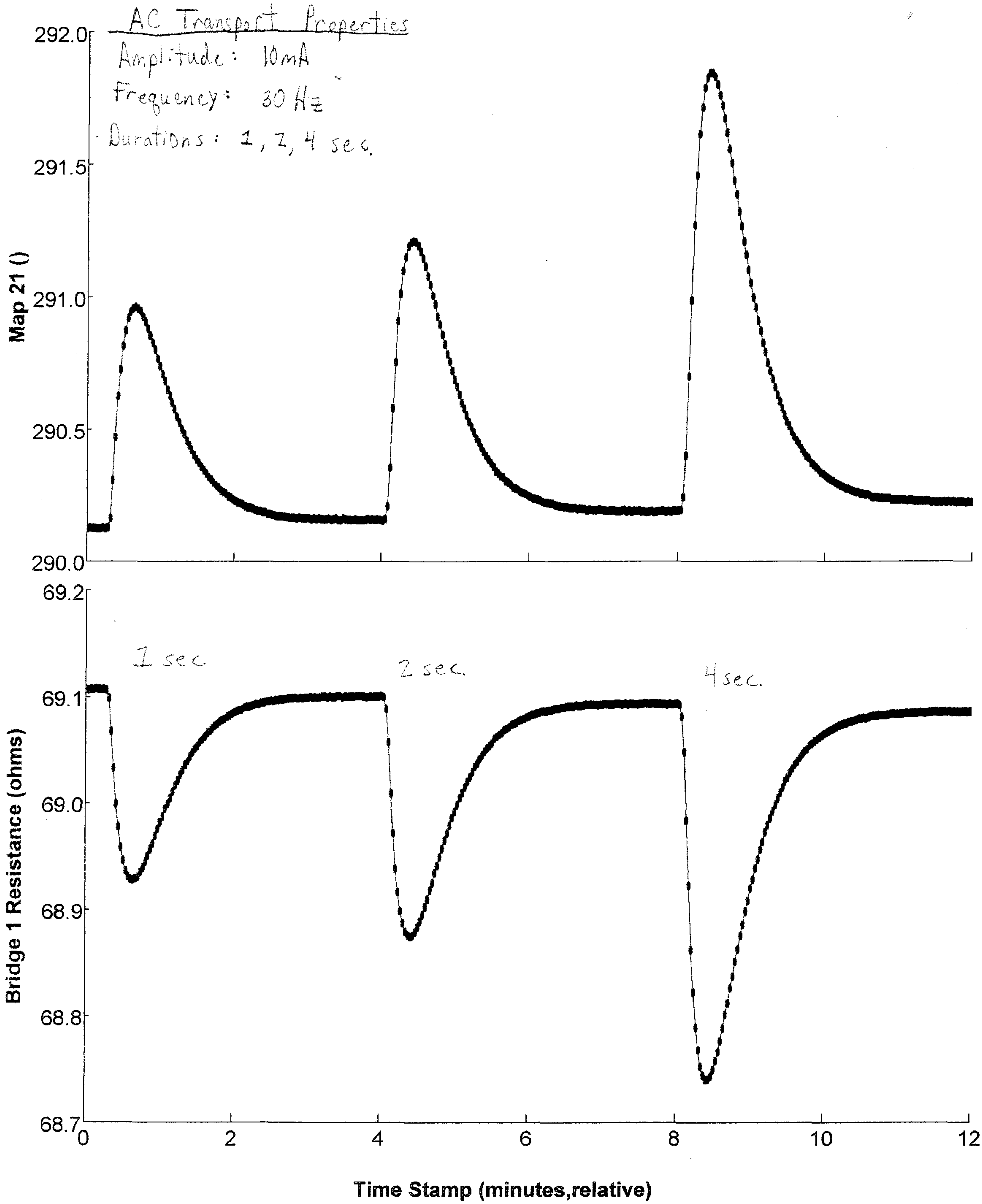


C:\WINDOWS\Desktop\delete_me.dat

04/29/02 16:09:09

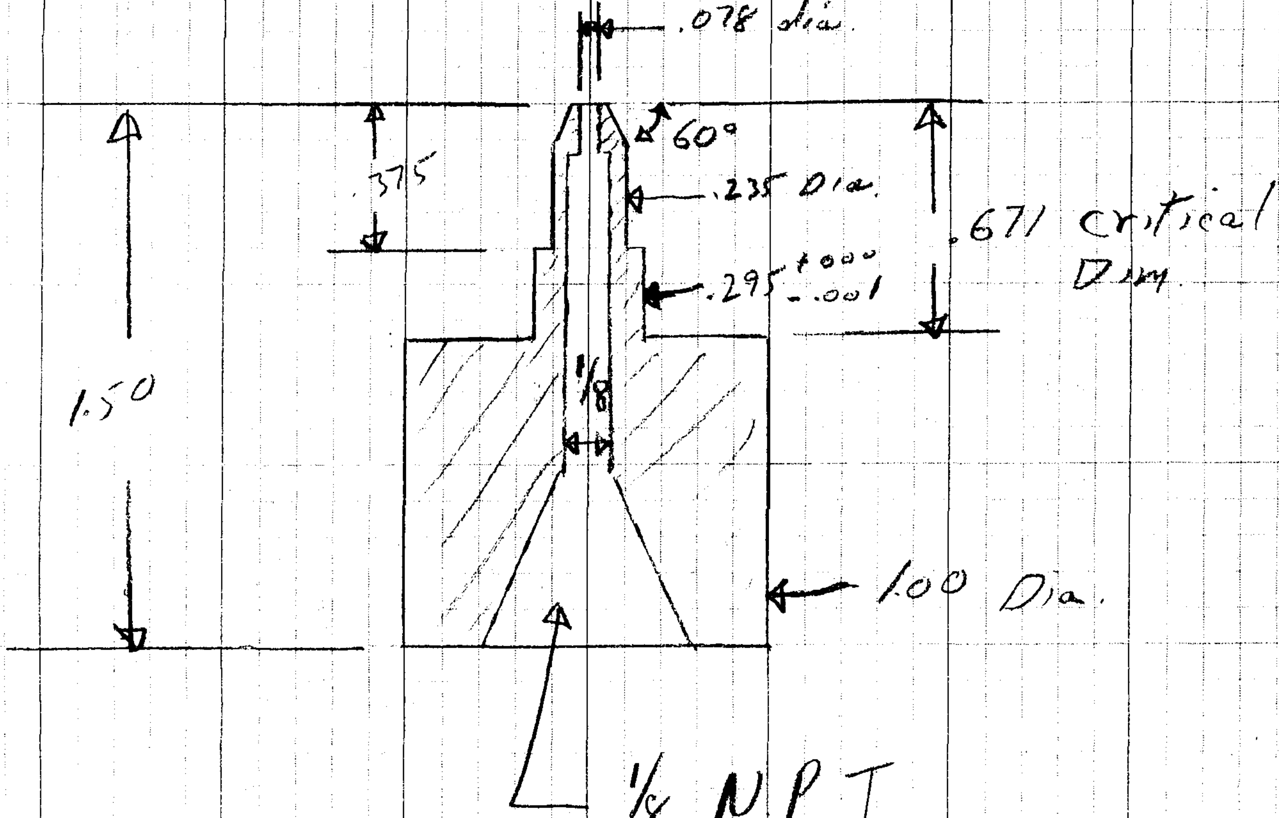
Warm & Cold Temperature Reaction From AC Transport Heater Control

PPMS Log Data File



C:\WINDOWS\Desktop\delete_me.dat 04/29/02 16:22:54

Here is the modified design for the heat capacity puck holder.



Cryopump
2

Contamination

The hair on the left was discovered on the O-ring of cryopump # 2.

(Serial No: CRYO45)

Opened top of Model 6000.

Turned Model 6000 power off.

Removed P1 ribbon cable.

Removed power cable.

Removed nut and washer.

Removed cryo expansion board.

New expansion board pressed firmly into place.

The nut & washer was tightened.

Attaching the ribbon cable (P1). } "Groove" match up properly.

Attaching the power cable.

Model 6000 cover ~~off~~ attached & screwed into place.

- Power turned ON on the model 6000 - everything on 'status' window looks normal.

- MultiVu crashed as the power shutdown was done during data logging session → Restarting the computer.

- Checking cryopump
  - Activate TIO on MultiVu
  - Set temp to 320 K (Fast settle).
  - ~~Manually open the cryopump with the 'cryoexer' programme on the PC~~
  - Open 40w power
- Doing "pump continuously" to pump the chamber down so that we can open the cryo flap.
  - The pressure is stable at 5.051 Torr. It seems higher than we expected!

- a seal operation was performed.
  - Pressure remained the same after seal.

→ Logppms started — "cryocheck1 (05-07-2002).dat"

- a purge seal operation was performed. (1.5 min.)
  - The purge peaks occur at ≈ 7.8 Torr, instead of ≈ 780 Torr as expected.

→ Regenerating the cryo-pump in room!

- The system is already at 320K.
- Removed 4 screws.
- Vented continuously.
- screws removed from side panels.
- cryopump removed from dewar.
- screws put back into side panels because it appears unnecessary to remove them.
- Waited for cryopump to reach room temperature.
- attached the cryopump pumpout fixture to bottom of cryopump.
- in room regeneration is attempted.
  - ↳ with 150 open, pressure stabilized at 5 torr.
  - ↳ cryo valve opened & pressure dropped to ≈ 4.7 Torr.

→ Following Shank's advice & performing a manual regeneration in room w/ heater on ~~thing~~ CRYO. etc.

(1.) Pump continuously (233 min.)
 - stabilize at 5.03 Torr
 - waited ≈2 minutes

(2.) opened Cryo (low power) & clicked "off"
 - Pressure 4.8 Torr

(3.) Heater on at 236 min.

(4.) Heater off at 239.7 min.

Cryo flap closed @ 4:25 PM
Venting continuously.
Blanking plate removed.
Cryopump re-installed
Purge & Seal @ 323.45.

→ Ran the calibration routine on page 2-11 of the manual.

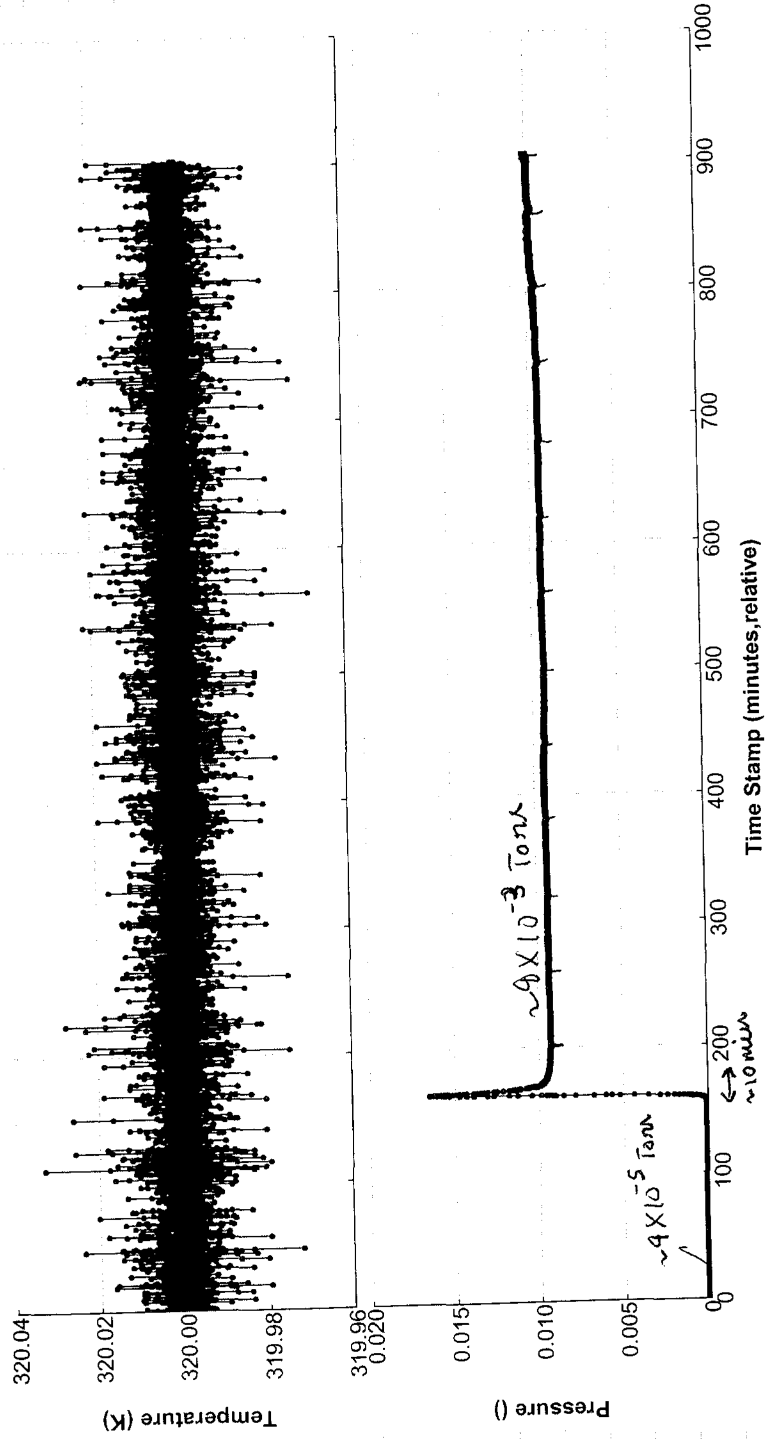
↳ This is the first time going into HiVac.

→ Started a new data file called "First HiVac (05-02-2002)"

↳ This was started just after the completion of the calibration which put the system in its first HiVac status.

May 3, 2002

– The system failed at almost 3 hours in HiVac status.



Finished the old data taking (first HiVac test) after about 15 hours.

- Noticed the flapper valve which was open but had a very small black spot on the red flap valve. To verify whether the small black part of flapper is "normal" we run this test;

  - New sequence file - 'first HiVac01 (05-03-2002)'
  - 'Seal' chamber command to check whether the automatic command would close the flapper valve completely or not.
    → Flapper valve did close completely and we do not see the red side of the valve.

* - Manually opened flapper valve using low power on cryo.ex to see whether it is all red or mostly red w/some black like we noticed earlier.

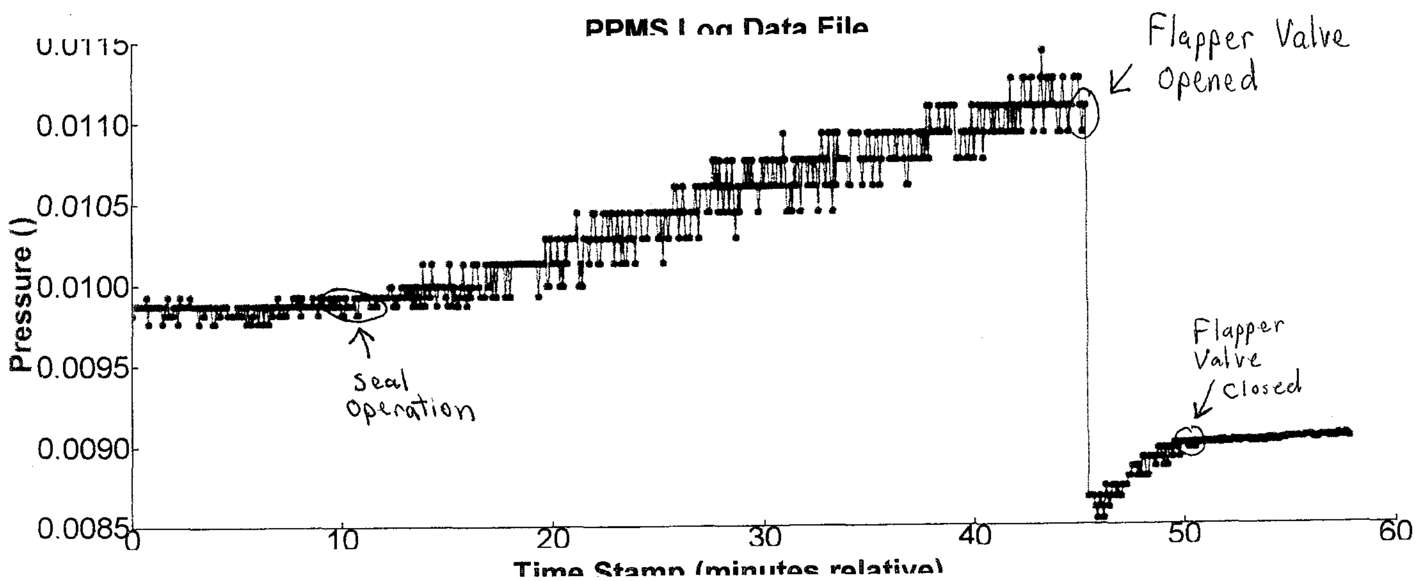
  → When fully opened it appears completely red, as opposed to red w/ black as before. (45.3 min)

~~[illegible]~~

  → The pressure went down when flapper opened. See plot.

- The cryo flapper was closed manually. (all black)

  → No significant change in pressure trend.



- Mapping the extra parameters through "Mon6000 for Win32" in the tools folder. Map by going to 'search' and then 'Map Assistant'.

→ Mapping "cryopump HiVac"

Code	Name	Map # channel
0	hiVac mode	55 (
1	Monitor state	56
2	intrupt hold (flag)	57
3	ADC reading of waitFun	58
4	Max ADC slim counts	59
5	Time out	60
6	Avg time	61
7	Start time	25
8	Flapper [mode[0:off -n:op +n: cl]]	26
9	Flapper readback (4:wire 2:limSwit lsb: current)	27
10	regin heater [flg]	28
11	iso soln(flg)	29

- We started "pumping continuously" to see if the ISO valve value changed state from 0 to 1.

  → It *did* change from 0 to 1.

- We entered into HiVac to see if it worked properly.

  → The pressure settled at ≈ 9e-3 Torr.

- Started logging a new file "first HiVac 02" to see if we can change the mapped ID.

- Monitoring the vacuum sequence to see the dependence of each mapped parameter.

  1) Vent + seal (6 min relative)

  2.) Purge + Seal (12.5 min.)

  3.) HiVac (17 min.)

thanh.dang@qdusa.com, 12:22 PM 5/3/02 -0700, Cryopump Failure Data

To: thanh.dang@qdusa.com
From: Michael Hall <mhall@ligo.caltech.edu>
Subject: Cryopump Failure Data
Cc: desalvo@ligo.caltech.edu,htariq@ligo.caltech.edu
Bcc:
Attached: C:\My Documents\Upload\firstHivacAfterRegenExpansionBoard.pdf; C:\My Documents\Upload\first hivac (05-02-2002).dat;

Thanh,

Here is the data file that was taken last night which clearly shows the cryopump failure. Just to recap what was done:

1.) The Cryopump Expansion Board was replaced (as discussed over the phone).
2.) We had thoroughly regenerated the charcoal of the cryopump out of the dewar. (As we discussed beforehand over the telephone.)
3.) The cryopump and new board were calibrated using the calibration technique (as discussed over the phone).
4.) After the cryostat reached HiVac for the first time, this data was taken. It can be seen that after 2.5 hours, the HiVac state is lost. The pressure spikes and then eventually stables out at ~9e-3, which is approximately what is achieved with the mechanical pump alone. (Again, as discussed over the telephone.)

Please take a look at this data and talk to your colleagues as required for information about what is taking place here. Myself, Hareem, and Riccardo DeSalvo will be calling you over speakerphone around 2:00pm to get your feedback on this issue. Please have the data in front of you as well as any colleagues you think may be helpful. If you would like us to call a particular phone number, please let me know through email before 2:00pm, otherwise we will contact you directly.

Thanks again for your assistance -- we hope to find a long-term solution to this problem shortly.

-Michael Hall

2:55PM Periodic dimples are observed for the second time in "HiVac" status.

- Thank has logged in remotely. (3:30PM)

(1.) Purge & Seal

(2.) Regenerating in dewar 3:35PM

(3.) Pump Continuous (few min)

(4.) Seal Chamber

(5.) Deleted data file on Model 6000.

6) Control - config - He Level change from ~62% to 70%, configured by measuring the He level with our usual "tube" method.

May 6, 2002

The following formula is used to manually calculate the amount of Helium in the dewar:

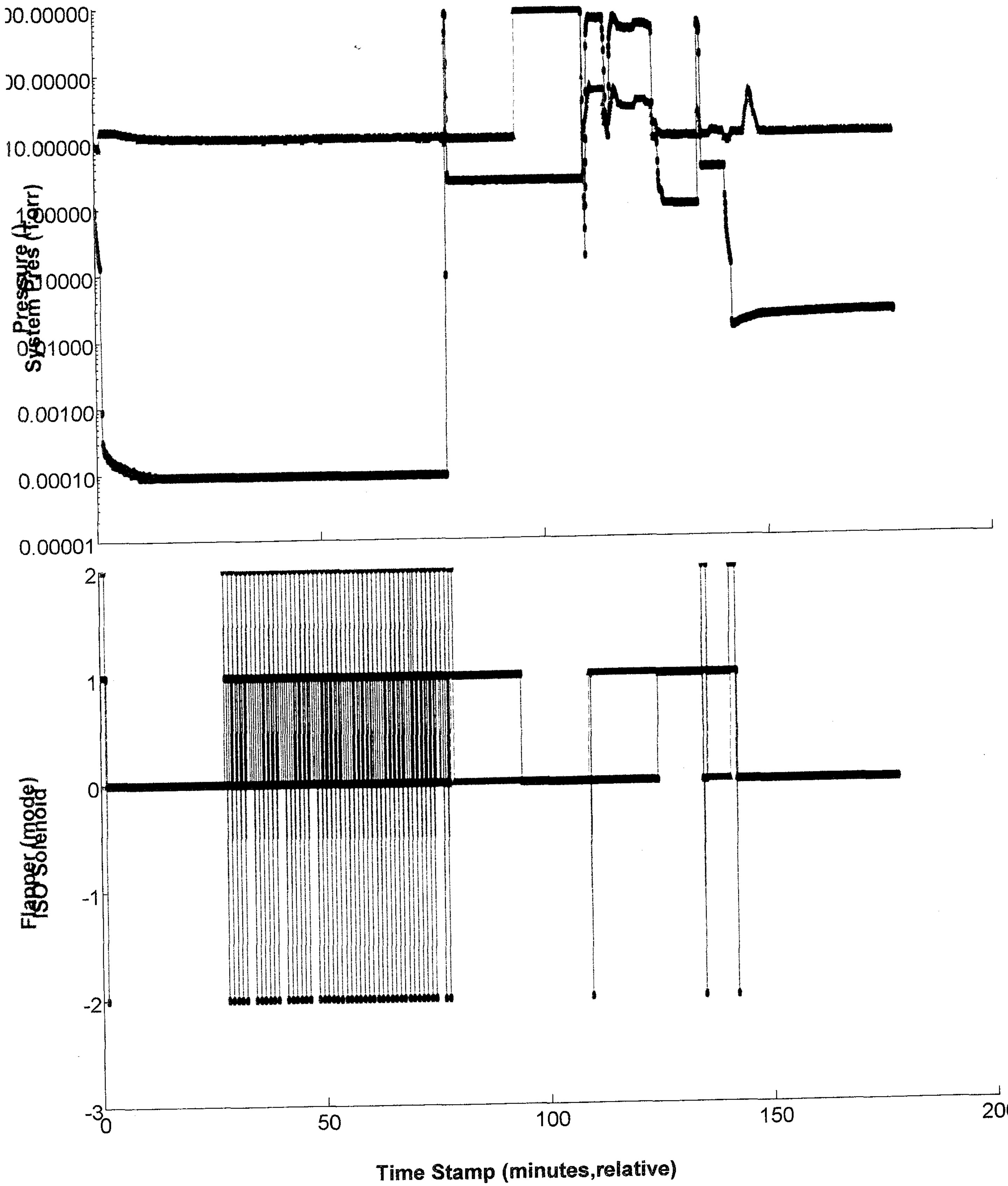
$$\frac{(\text{Inches}) - 3.6}{21} = \% \text{ of He.}$$

The plot on the next page is the result of a cryopump test performed by Frank and Brian.

Frank says these results indicate that the cryopump is operating properly.

Brian has thought the problem was a faulty motherboard all along and plans to come replace it on Thursday or Friday.

Thanh and Brian's Cryopump Test Sequence



C:\cryolab\users\ThanhTest (05-03-2002)\Cryo-Test.dat 05/06/02 16:40:06

Sequence File: thanSeq.seq

```
   CRYO VAC ISO FLAG-0 is sealed
ExecCmd PORTCMD "tblmode 20 0;"
ExecCmd PORTCMD "MAPFUN 20 12 11;"

   CRYO flapper mode 0-off
    2 is hi pwr, 1low pwr, neg values are opening?
ExecCmd PORTCMD "tblmode 21 0;"
ExecCmd PORTCMD "MAPFUN 21 12 8;"

   CRYO regen  flg- 0 is off
ExecCmd PORTCMD "tblmode 22 0;"
ExecCmd PORTCMD "MAPFUN 22 12 10;"

   CRYO Limit Switch (1=closed)
ExecCmd PORTCMD "tblmode 23 0;MAPFUN 23 11,2,4,1;"

   CRYO Motor torque (1=stalled),  0=moving freely
ExecCmd PORTCMD "tblmode 24 0;"
ExecCmd PORTCMD "MAPFUN 24,11,2,3,7;"

   CRYO Motor wireing OK->0 (only if flapper mode !=0)
   CRYO Motor wiring :  1=no current flowing,  0=OK current is flowing
ExecCmd PORTCMD "tblmode 25 0;"
ExecCmd PORTCMD "MAPFUN 25,11,2,3,6;;"

Set Temperature 300.00K at 12.00K/min. Fast Settle
Chamber High Vacuum
LogData Start New 5.00 1073741823 1073741823 7 "C:\cryolab\users\ThanhTest (05-03-2002)\Cryo-Test.dat" "" ""
Wait For Temperature, Chamber, Delay 900 secs, No Action
Scan Time 0.0 secs in 50 steps
     auto close flapper
     ExecCmd portcmd "$CRYOset 10;"
     Wait For Delay 30 secs, No Action
     auto open flapper
     ExecCmd portcmd "$CRYOset 3;"
     Wait For Delay 30 secs, No Action
End Scan

Chamber Purge then Seal
Wait For Temperature, Chamber, Delay 900 secs, No Action
ExecCmd portcmd "HIVAC_CNF 0;"
Wait For Delay 1 secs, No Action
Chamber Vent Continuous
Wait For Temperature, Chamber, Delay 900 secs, No Action

Set Temperature 200.00K at 5.00K/min. Fast Settle
Chamber Pump Continuous
iso open
ExecCmd portcmd "$CRYOset 6;"
Wait For Chamber, Delay 10 secs, No Action
auto open flapper
ExecCmd portcmd "$CRYOset 3;"
```

Sequence File: thanSeq.seq

```
54:  Set Temperature 300.00K at 10.00K/min. Fast Settle
55:  ExecCmd portcmd "HIVAC_CNF 2;"
56:  Wait For Delay 600 secs, No Action
57:  ExecCmd portcmd "CRYOREG 0;"
58:  Wait For Chamber, Delay 300 secs, No Action
59:  Chamber High Vacuum
60:  Wait For Chamber, Delay 1 secs, No Action
61:  Set Temperature 298.00K at 12.00K/min. Fast Settle
62:  Wait For Temperature, Chamber, Delay 1800 secs, No Action
63:  LogData Stop ""
64:  Shutdown Temperature Controller
```

This is the sequence that produced the plot on the previous page. It is important because this sequence contains several interesting GPIB commands for the cryopump that are normally not released to customers.

We want to reproduce the cryopump failure, but with the addition of monitoring the newly mapped cryopump parameters, in particular, the state of the 150 solenoid.

The following steps were taken @ 4:50 PM:

(1.) Cryopump regenerated in dewar.

(2.) Vent and Seal

(3.) Purge and Seal

(4.) HiVac operation

(6.) ~~Started~~ logging: "cryocheck02 (05-06-2002).dat"

(5.) Set temperature to 320K. (from ≈ 265K).

The cryopump failed, but ~~there~~ was no spike as before. More importantly, and surprisingly, there was <u>no</u> <u>correlation</u> to the 150 solenoid or the flapper valve opening.

I want to try regenerating the charcoal in the dewar & then seeing how many time I can enter HiVac before the pump fails.

9:40AM ① Pump Continuously

② Regenerate charcoal in dewar.

— Completed @ 9:43AM.

— This experiment has been delayed. New tests will be done on the isolation valve ~~[illegible]~~ tomorrow w/ Eric Black.

Swagelok Contact

Swagelok

Orange Valve and Fitting Co.

2333 S. Manchester Ave.
Anaheim, CA 92802

Contact: Brad Harrison 1-(888)-560-6764

May 8, 2002

Bryan from QD arrived @ approximately 12:30 PM.

- We tried measuring the cryo heater voltage but Model 6000 gave a fault because the LEMO was not connected
- We manually actuated the heater from cryo.exe but we measured no voltage. Afterwards we smelled something burning.
- A low, periodic chirping sound can be heard from inside the Model 6000. It sounds as if it is coming from the transformer.
- The power to the Model 6000 was restarted. An error appeared in the data file saying there was a power failure.
  - → Bryan is afraid the heater check we performed earlier damaged the power supply.
- Bryan decided to replace the Model 6000.
  - → The replacement is at least 2 years old & was used to test equipment in house. He says it worked properly before transport to Caltech.
- Bryan says the reason it burned out earlier is probably because he was touching the pin and the side of the LEMO, which shorted out his connection.
- The new Model 6000 was installed.

  (1.) Purge & Seal

  (2.) Regeneration in dewar.

  → The ISO may not have opened. (Pressure = 600 Torr)

  (3.) Manually opened ISO

  → Pressure = 5 Torr, 3 Torr

  (4.) Manually closed ISO.

(5.) Cryopump regenerated in dewar:

→ head 150 open. Pressure is low this time (3 Torr)

(6.) Manually regenerating in dewar w/ heater w/ cryo.exe.

(7.) Chose "use cryo pump" on the Model 6000.

→ pre-pump to 0.3 Torr

(8.) After reaching HiVac, manually opened 150 ~~& turned~~ on heater.

→ Pressure = 1.55 Torr.

(9) 150 closed & heater turned off manually.

→ Pressure = 7.71e-3

→ ~~##~~ Leak Check

- The cryopump installed (#3) seems to leak when compared to cryopump (#2), because the pressure is lower in #2 when the leak checker is attached.

- Bryan has concluded that both cryopumps leak, albeit differently.

→ He is taking both cryopumps back & will come tomorrow with a new one.

→ The power supply that was chirping was swapped ~~##~~ with Bryan's in-house Model 6000 power supply.

May 10, 2002

- Bryan called & said he will come Monday because:

(1.) They have not yet repaired our Model 6000.

(2.) Our old cryopump failed on their system.

(3.) They want to test a cryopump w/our Model 6000 over the weekend first.

(4.) He is bringing our original cryopump and another new one to make sure one works when he comes.

- I called QD today. Mark says the Model 6000 was repaired but Bryan is not coming today. Bryan will call back after lunch.

14th May 2002

⇒ Quantum Design Brian + Mike visit.

- Room regeneration done. New cryopump installed. Replaced Model 600
- Purge + Seal ± ≃ 14 Torr pressure
- Opened sample chamber by opening isolation value -> pressure went down + then started going up; showing drifted pressure

Recheck again the procedure above

- Purge + seal -> normal pressure change. Data logging in 'quick test.dat'

- Removed + reconnected the chamber gas line
- Opened isolation value through the cryo.ene.
- Gas line opened once more again by removing the connector at the back of chamber.
- Putting the connector back the system pressure went down + chamber pressure went up + back down again.
- Taking the gas line off from back of Model 6000 because before that quick seal in the connector was coming on
  -> system pressure went up — no changes in chamber pressure
    ∴ either pirani pressure guage broken as the chamber line was sucking on the thermo which was put in there.
      OR the flapper value is having problems the O-ring may be off?

- Checked the flapper value and the iso value by taking out the cryopump and looking physically while doing auto open + close on the cryo.ene.
- -> The flapper value did not close properly the first time but DID work the rest of the 3 or 4 times.

- Replacing everything after cleaning the O-rings.

- Regenerated in the room through Model 6000.

- Doing a manual regeneration
  - pump continously
  - Iso open - pumped for a minute
  - Cryopump open
  - Heater on

~@640 m Torr - closed flapper valve for few ~~sec~~ seconds pressure went up.

- closed the flapper valve → pressure went to 580 m Torr

- Repeated last 2 steps.

- Flooded - pressure rise to ~760 Torr.
  ↳ Continuously flooded...

- Cryopump inserted into dewar.

- After cryopump was reinstalled, the pressure dropped linearly to ≃10 Torr, then kept falling, but slower to 8 Torr
  - The pressure gauge has completely failed.

- Bryan decided to read the pressure from Iten Model 6000 to see if the pressure reading problem was the pressure gauge or our Model 6000.
  - Their Model 6000 also reads ~5.6 Torr and continued dropping. (3 Torr)

- Bryan started reading from our Model 6000 and purged & sealed the system.

~~Bryan~~

- Bryan ran a HiVac operation on our Model 6000 to analyze its reaction. The pressure attained 1×10^{-4} Torr.

15th May

Meeting with Eric Black, Ken, Mike, Haseem.

- Called Mark Seeback
- Left message on Chief Engineer Stefano Spagña
- Asked Brian to send a new "working" Pirani pressure gauge.

Will do measurements;

i) Instal an external pressure gauge
ii) Run the HiVac through model 6000 with the computer disconnected, pumping line disconned + the green Lems disconnected
iii) Systematically reconnect things one by one and find problem unit.

- Borrowed external pressure gauge (Pirani) from 40 m along with clamps + quick flange fittings; also controller for the Pirani gauge + cables were borrowed as well

- Installed external Pirani (borrowed from 40m) on the sample chamber.

- Checked + calibrated the external Pirani by venting, pumping, sealing chamber. → Cryopump Pirani seems to follow the external Pirani readings.
- Vent + seal operation through Model 6000.
- Purge + seal " " " ". 760 → venting

14 Torr → purge + seal

steadily rising with a rate ~~0.1~~ Torr/sec from 14 Torr

- Second attempt saw no rise in pressure — it was stuck @ 2 Torr.

- Upon entering HiVac, the gauge was reading -0.5 mTorr, which means it was not calibrated perfectly,

- Removed 2nd Pirani gauge.
- Activated HiVac procedure.
  ↳ stabilized @ 9e-5 after about an hour
- Removed & re-inserted green lemo
- Removed the pumping line from the back of Model 6000 but the mechanical pump was still pumping on the line.
  - Switched off the mechanical pump for few minutes but could not find an appropiate plug for pumping line
  - Switched ON the Edward pump + reconnected the pumping line because otherwise without the pump we had no control over the cooling annulus + hence the temperature.
- Disconnected green lemo @ 7:30pm.

May 16, 20

- Connected green lemo @ 9:30 AM
  - Pressure was still @ 9.1×10^{-5} Torr. The vacuum seems to have held overnight (14 hours) without the computer or Model 6000 (green lemo) w/ ~~the~~ vacuum pump connected.
- We want to repeat test by purging & sealing, and re-entering HiVac for 4 hours.
- Vent & Seal 9×10^{-5} Torr ⟶ 750 Torr
- Purge & Seal (@ 9 Torr, it flushed to 680 Torr) ⟶ Sealed @ 3.2 Torr
- Entered HiVac → pre pump started ~3.2 Torr
  ISO closed ~0.24 Torr
  Immediate jump to ~ 9×10^{-5} Torr as soon as the flapper value opened!

* All indications that the pressure gauge is not working as the jump in pressure was within few seconds of cryopump opening.

- Seal Chamber Command. (stops the mechanical pump pumping on the cryopump pumping line but it seems that under normal * HiVac sequence the pumping line is continously being pumped on → why?).

- Calibrated our Pirani gauge by reaching vacuum on leak detector & setting VAC to 0 Torr.

~~Vented & Sealed.~~

- Vented continuously after re-attaching vacuum tubes.
- attached our Pirani Gauge.
- ~~Run~~ Vent & Seal
- Purge & Seal.
  - ATM reads "Hi" on our Pirani controller.
  - After Seal, our gauge reads 6.65 Torr.
    ↳ after a minute it stabilizes at 6.80 Torr.

- Goto HiVac.
  ↳ Cryo opens @ 400 mTorr.
    ↳ Quickly decreases to 5.5 mTorr, then decreases more slowly.
  - achieved HiVac
    - Model 6000 reads 9.1×10^{-5} Torr. } 11:55 AM
    - Our gauge reads -1.3/-1.2 mTorr. }
- Seal Chamber operation.
  ↳ - Model 6000 reads 3×10^{-4} Torr and rising
    - our gauge reads 0.1 mTorr and rising slowly.
      ↳ up to 2.1 mTorr after 3 mins.
- Disconnect vacuum tubes from Model 6000.
  ↳ Our gauge reads 3.0 mTorr

- The pressure on our gauge appears to rise at approximately 0.3 $\frac{mTorr}{min}$ as observed for 3 minutes.

↳ @ 12:06PM, P = 5.5 mTorr

@ 12:44PM, P = 15.1 mTorr

@ 2:51PM, P = 22.2 mTorr

@ 3:23PM, P = 28.6 mTorr

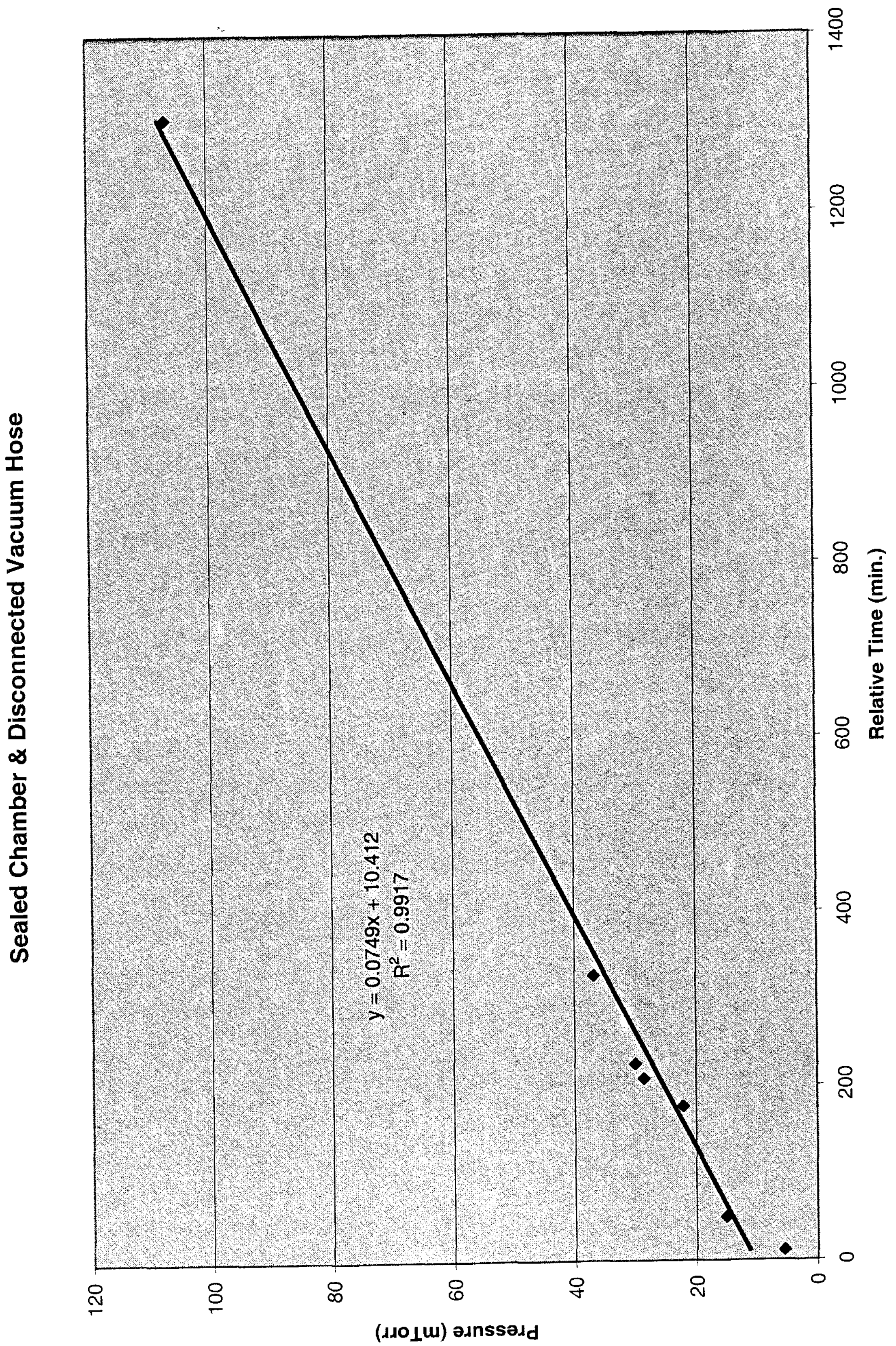
@ 3:40PM, P = 30.0 mTorr

@ 5:22PM, P = 36.8 mTorr

May 17, 200

@ 9:41AM, P = 107 mTorr

Plotted Results from QD requested experiment.



- To see if the linear rise in pressure is normal or if it is the fault of the Model 6000, the original experiment will be repeated w/ the Pirani gauge attached.

- Reconnected vacuum hose.

- Vent & Seal (Gauge Reads "Hi")

- Purge & Seal. (Gauge Reads 6.25 Torr after Sealed.)
  - ↳ Increases to 6.35 Torr & stabilizes,

- Goto HiVac. (Mechanical pump pumps to 486 mTorr)
  - when cryo opens, pressure jumps to ~2.5 mTorr, then decreases more slowly.
  - HiVac reached @ ~1.5 mTorr on our gauge (9.1×10⁻⁵ Torr on Model 6000) → @ 11:06 AM

- Pressure remained steady until 11:17 AM.
- Seal Chamber. (Gauge reads ~1.2 mTorr & increasing)
- Remove green lemo. (Pressure reads ~0.1 mTorr & increasing)

@ 11:19 AM, P = 0.1 mTorr
@ 11:25 AM, P = 3.2 mTorr
@ 11:33 AM, P = 5.5 mTorr
@ 11:50 AM, P = 8.8 mTorr
@ 12:06 PM, P = 11.0 mTorr
@ 1:30 PM, P = 18.8 mTorr
@ 1:42 PM, P = 19.6 mTorr

@ 4:08 PM, P = 30.4

Here is a plot of the data w/ a best fit curve, as described on the previous page.

```
> restart:  with(stats):  with(plots):  with(stats[statplots]):
  with(fit):
> TIME := [2,8,16,33,49,133,145];
```

$$TIME := [2, 8, 16, 33, 49, 133, 145]$$

```
> PRESSURE := [0.1,3.2,5.5,8.8,11,18.8,19.6];
```

$$PRESSURE := [.1, 3.2, 5.5, 8.8, 11, 18.8, 19.6]$$

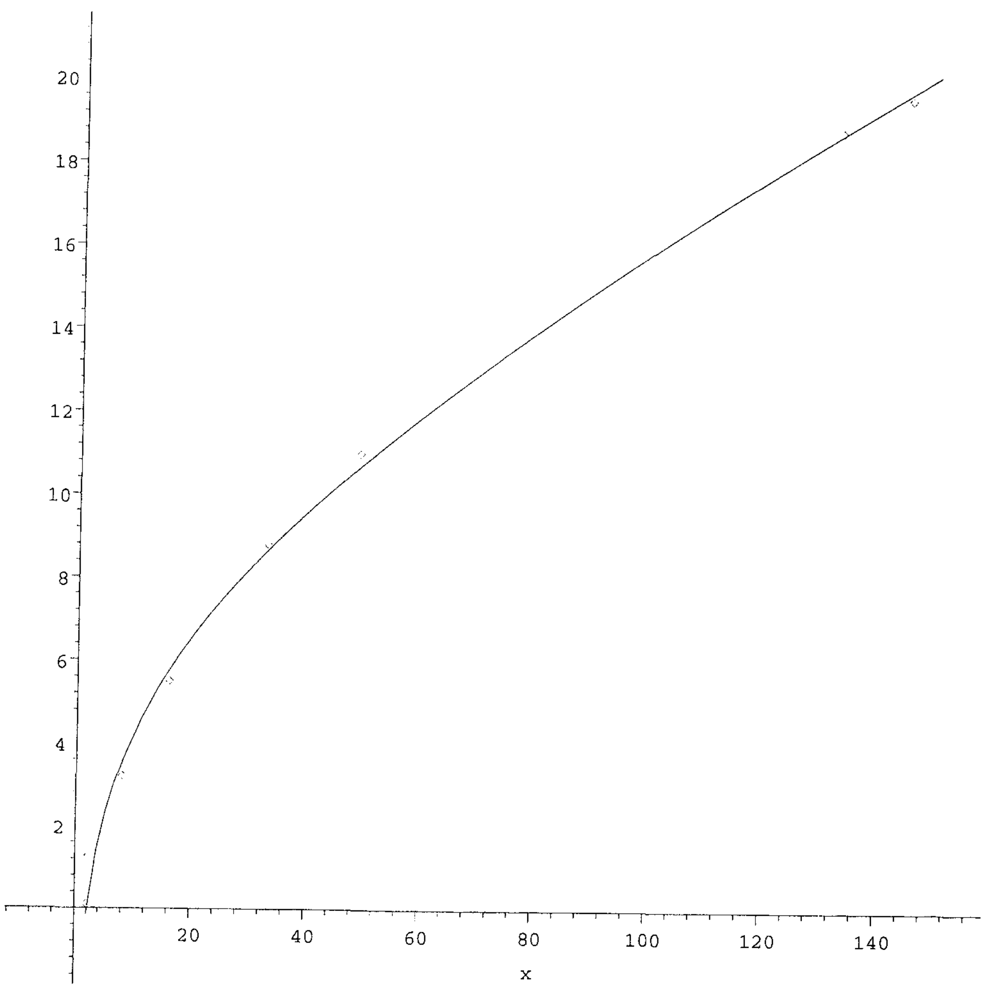
```
> SCATPLOT := scatterplot(TIME,PRESSURE,color=red):
> FIT := op(fit[leastsquare[[x,y], y=a*ln(x)+b*x+c,{a,b,c}]]([TIME,
  PRESSURE]))[2];
```

$$FIT := 2.390651512 \ln(x) + .06759057337\, x - 1.942603132$$

```
> FIT;
```

$$2.390651512 \ln(x) + .06759057337\, x - 1.942603132$$

```
> FITPLOT := plot(FIT,x=2..150,color=black):
> display(FITPLOT,SCATPLOT);
```



4:43 PM

- Green lemo re-connected.
- Vent & Seal. (P = "Hi")
- Purge & Seal. (P = 6.00 Torr after seal)
- HiVac. @ 4:11PM

NOTE: very quickly went to 9×10^{-5} Torr! (2nd time it did this)

- System reached HiVac @ 4:17PM. (P = -1.3 mTorr)
- 7:15pm - external pressure gauge = -1.6 mTorr
  Model 6000 = 9×10^{-5} Torr.

May 20, 20

@ 9:20AM, P = -1.9 mTorr
Model 6000 = 9×10^{-5} Torr

May 21st,

- System is idle since yesterday after the chamber was sealed + the HiVac status released.
- Received new Pirani gauge from QD. (No serial or ID #)
- Pressure reading @ external Pirani = 76.3 mTorr } @ 2:50pm
  Cryostat Pirani = 0.054 Torr }

⇒ Changing the pressure gauge.
- Vent & Seal → then vent continously.
- Taking the green lemo off to rem
- Two screws removed from current sensor.
- Old pressure sensor removed.
- O-ring removed & cleaned.
- O-ring re-inserted.
- Pirani power cable re-attached.
- Two screws re-inserted.

[illegible] May 22, 2002

- Helium fill & waited 40 min to cool.
- Connected computer.
- Calibration wizard
- Disconnected computer.
- Vent & Seal
- Purge & Seal.
- HiVac (@ 5:18 PM)
  → M. Pump to ~ 209 mTorr
- Reached HiVac @ 5:33 PM
- We want to hold @ HiVac overnight to see if it is successful. Tomorrow the same test will be run w/ the computer on & data logging.

@ 5:34 PM
$P_{M6000} = 8.72 \times 10^{-5}$ Torr
$P_{PIR} = -1.4$ mTorr

May 23, 2002

@ 9:31 AM
$P_{M6000} = 8.72 \times 10^{-5}$ Torr
$P_{PIR} = -1.8$ mTorr

- Next, we want to try the same thing w/ the computer attached & logging data.
- Vent & Seal
- attach computer.
- Open Multi-Vu.
- Start logging data. (Log PPMS)

<u>Note:</u> I will continue to send commands from Model 6000.

- Purge & Seal. ($P_{M6000} = 2.9$ Torr, $P_{PIR} = 6.00$ Torr)
- HiVac (@ 9:39 AM)
  → M. Pump to ~ 143 mTorr

- Reached HiVac @ 10:06 AM

$P_{M6000} = 8.82 \times 10^{-5}$ Torr
$P_{PIR} = -1.6$ mTorr

@ 2:07 PM $P_{M6000} = 8.72 \times 10^{-5}$ Torr
$P_{PIR} = -1.8$ mTorr

- Next, we want to install the sample & baffle assembly & see if the results are the same.
- Vent + Seal.
- Stop logging.
- Start logging new files
- Purge & Seal. ($P_{M6000} = 3.04$ Torr, $P_{PIR} = 6.10$ Torr)
- HiVac (@ 2:14PM)
  - ↳ M. Pump to ~ 135 mTorr
- Sealed Chamber.
- Vent + Seal.

} Forgot to remove Pirani gauge & insert sample!!!

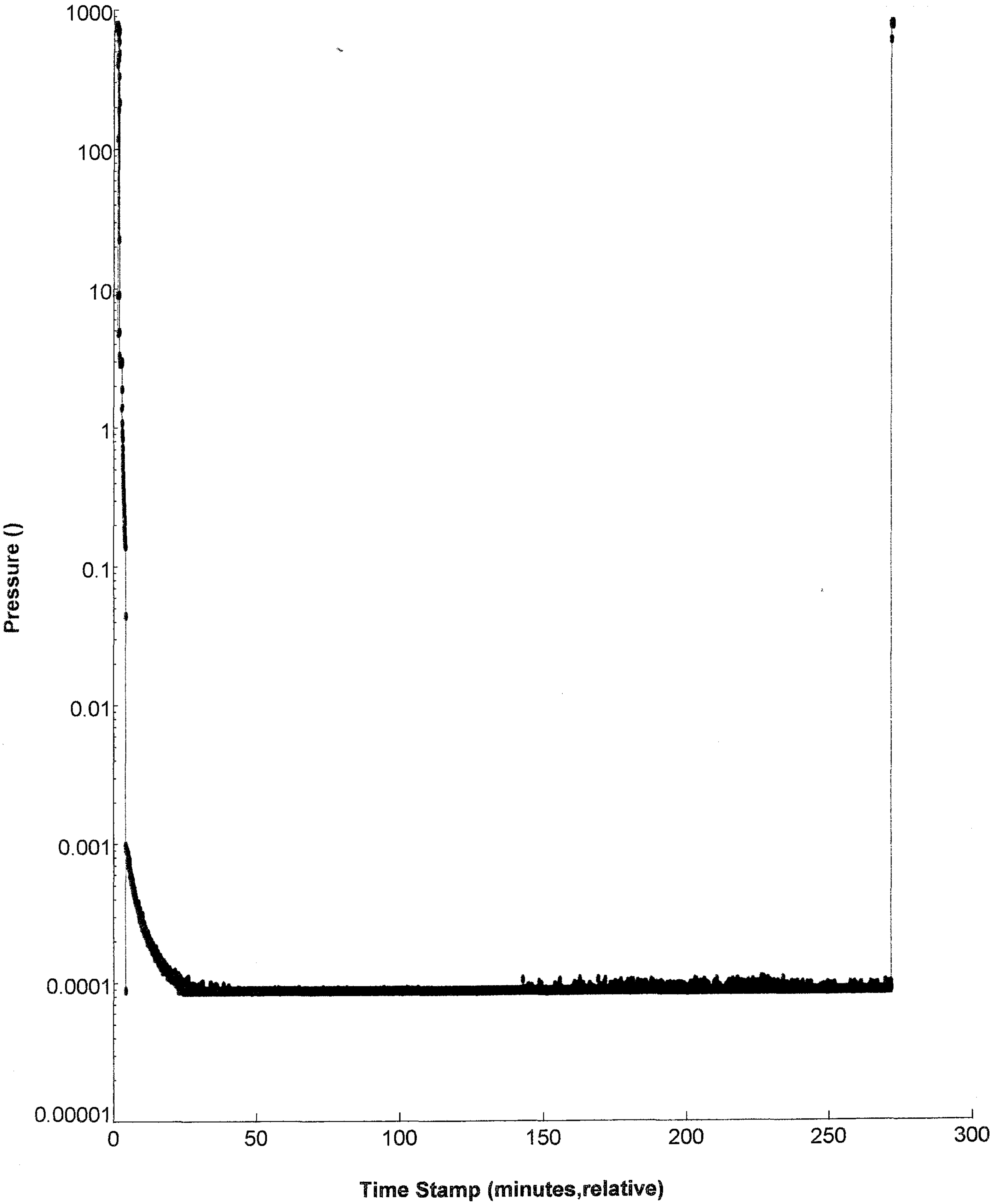
- Stopped logging.
- Vent Continuously.
- Removed Pirani Gauge.
- Inserted Sample.
- Inserted Baffle assembly.
- Vent + Seal. ⟶ The pressure goes to ATM, + then falls quickly!!!
  - ↳ as before w/ Dunn + Mike
- ~~Started logging data.~~

- Vent continuously.
- Remove baffles & sample.
- Reattach Pirani gauge.
- Vent & Seal.
  - ↳ Pressure rises to ≈ 640 Torr & drops again!
- Disconnect computer.
  - ↳ Pressure still drops.
- Reconnect computer.
  - ↳ Pressure still drops.
- Used Romcfg32.exe to upload config file.
  - ↳ no effect on pressure reading.
- Called QD & reported the problem to Robert.
  - ↳ He is shipping out another pressure gauge.
- Purge & Seal
- HiVac
  - ↳ M. Pump to ~ 320 mTorr
  - ↳ Cryopump doesn't appear to be as effective as before the pressure gauge failed.
- The system eventually reached HiVac.

$P_{MG000} = 8.82 \times 10^{-5}$ Torr
$P_{PIR} = -1.8$ mTorr

rst HiVac Test Of New Pressure Gauge With Computer Attached (No Sample or Baffle

PPMS Log Data File



C:\cryolab\users\New Pressure Gauge\05-23-2002\firstcomputerlog05/23/02 14:11:37

- Vent & Seal.
  - ↳ Pressure responds normally & is stable.
- Purge & Seal.
  - ↳ Pressure responds normally.
- Vent & Seal.
- Vent continuous...
- Removed Pirani gauge & installed sample & Baffle Assembly.
  - ↳ Pressure started dropping after assembly inserted & chamber closed.
- Removed baffle assembly.
  - ↳ Pressure started rising.
  - ↳ stabilized @ ≈ 763 Torr
- Inserted baffle assembly.
  - ↳ Pressure decreases.
- I found that the pressure decreases as soon as the baffle assembly is pressed firmly against the sample. It increases as soon as it is released from the sample (even very slightly).
- Removed baffles.
- Removed sample.
  - ↳ Pressure increased to ≈ 807 Torr & stable
- Installed baffle assembly.
  - ↳ Pressure reached ≈ 9.1 Torr & seemed to continue to drop, but <u>very</u> slowly.

- Vent & Seal.
- Seal.
  - ↳ No apparent change in pressure.
- Purge & Seal.
  - ↳ Pressure stabilizes ~~[illegible]~~ @ ≈ 3.1 Torr & increases slowly.
- Vent continuously...
  - ↳ Pressure reaches ≈ 760 Torr & appears stable.
- Baffles ~~[illegible]~~ removed.
- Sample & baffles replaced.
  - ↳ Pressure drops.
- Purge & Seal.
  - ↳ Pressure @ ≈ 2.3 Torr after seal.
- Vent & Seal.
  - ↳ P = ≈ 796 Torr after seal.
- Purge & Seal.
  - ↳ Pressure @ ≈ 2.3 Torr after seal.
- HiVac @ 5:38 PM.
  - ↳ M. Pump to ≈ 400 mTorr & struggles to reach lower pressures.
- Vent continuous...
- Remove ~~[illegible]~~ baffles & sample.
- Baffles reinserted.
  - ↳ Pressure drops.

- Purge & Seal.
  - ↳ P = 2.3 Torr after seal.
- HiVac @ 5:44PM.
  - ↳ M. Pump to ≈ 396 mTorr.
- Sealed!
  - ↳ Pressure rises.
- Vent continuous.
- Baffles removed.
- Sample inserted.
- Our Pirani gauge attached.
  - ↳ Pressure drops.
- Purge & Seal.
  - ↳ ($P_{M6000} \approx 2.5$ Torr after seal, $P_{PIR} \approx 6.60$ Torr)
- HiVac @ 5:50PM
  - ↳ M. Pump to ≈ 350 mTorr
- Sealed when cryopump reached $\approx 5 \times 10^{-3}$ Torr.

<u>NOTE</u>: Is it a problem w/ ~~outgassing~~ saturating the cryopump?

- Vent continues.
- Old sample removed.
- Empty sample holder (#2) installed.
- Our Pirani gauge attached.
  - ↳ Pressure drops.
- Purge & Seal.
  - ↳ after seal → $P_{M6000} = 2.8$ Torr, $P_{PIR} = 6.40$ Torr
- HiVac @ 5:58PM
  - ↳ M. Pump to ≈ 342 mTorr.
- Sealed when Cryo reached ≈ 3.8×10^{-3} Torr.
- Vent continues.
- empty holder (#2) removed. Original sample installed w/ baffles.
  - ↳ Pressure drops.
- Purge & Seal.
- ~~HiVac @~~
- ~~Started~~ logging data
- HiVac @ 6:05 PM.
  - ↳ M. Pump to ≈ 356 mTorr
- Reached HiVac @ 6:37PM
  - ↳ $P_{M6000} = 1.721 \times 10^{-4}$ Torr.

May 24, 2002

- The Model 6000 held HiVac overnight.
  ↳ (See plot on next page.)

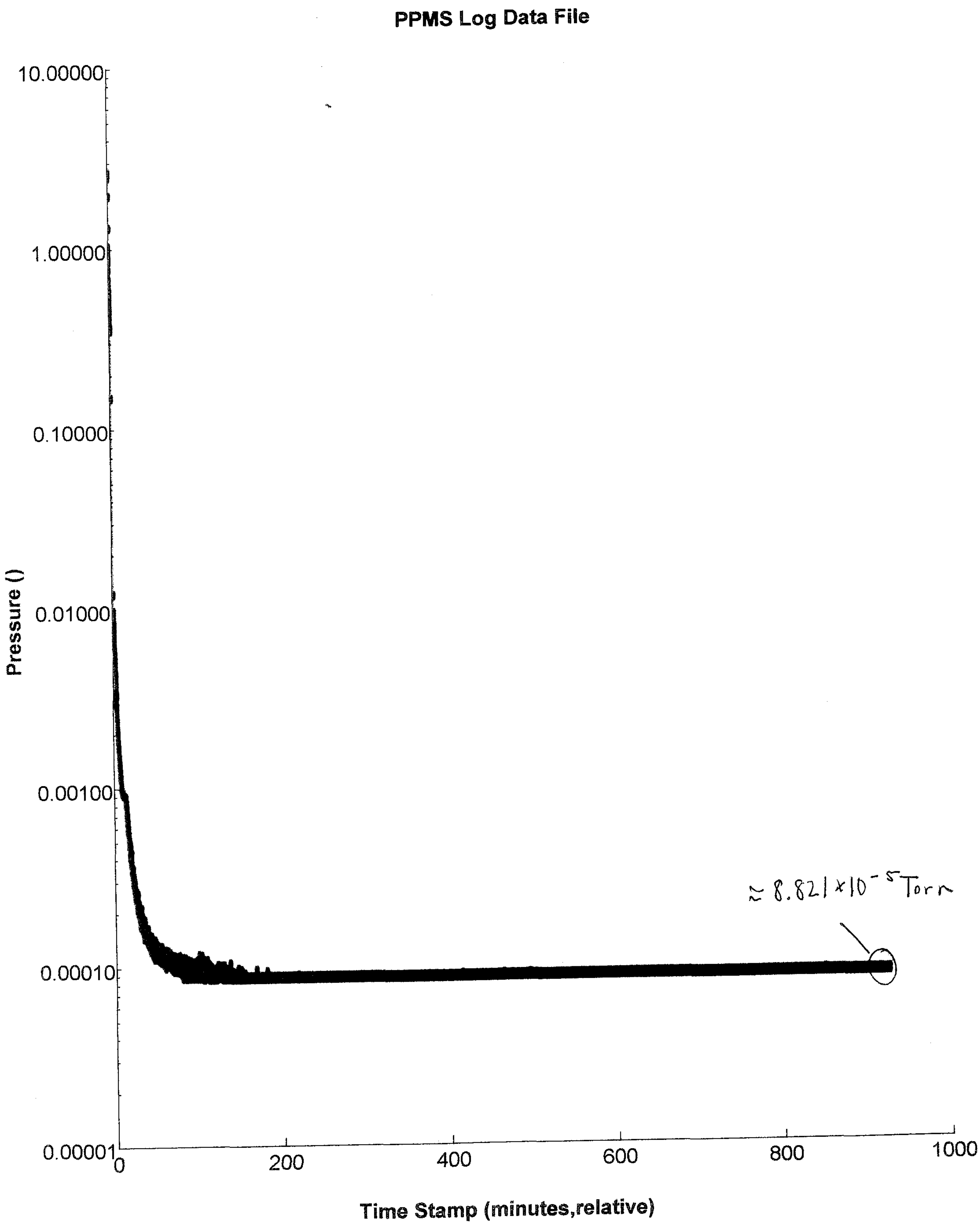
- I decided to run a TTO measurement on the Nickel sample currently installed to see if it works properly.
  ↳ The run started at approximately 9:35 AM.
  - As temperature approached 400K, the pressure started rising from 8.82×10^{-5} Torr to $\approx 1.5 \times 10^{-4}$ Torr.
  - After ~~pressure~~ temperature decreased from 400K, the pressure appeared to start decreasing as well. (1.2×10^{-4} Torr @ 392K).
  - By the time the temperature reached ≈ 385K, the pressure returned to 8.821×10^{-5} Torr.

NOTE: Robert & Ron from QD just called. They are sending me the expected Nickel sample TTO results.

— The measurement was stopped after 3 measurements, because the warm/cold thermometers did not appear to be reading.

- Sample checked & re-mounted.
- Purge & Seal.
- Temp. Set to 325K.
- Vent & Seal.
- Purge & Seal.
- HiVac @ 11:31PM
  ↳ M. Pump to 221 mTorr
- Noticed physics students had unplugged the TTO cord & did not plug it back in!
- ~~Stopped~~ logging data.
- Plugged TTO cable back into User Bridge Port.

Overnight HiVac Performance With Sample AND Baffles Installed



C:\cryolab\users\New Pressure Gauge\05-23-2002\sample&bafflesin05/24/02 09:32:35

- After plugging cable lead in, following values:

  negative resistance & 999 μA

  changed to:

  ≈ 50 Ω & ≈ 150 μA.

- Now, we are going to try running the same sequence again.

- Reached "HiVac" @ 12:03PM. ; $P = 1.4 \times 10^{-4}$ Torr

  ↳ Will wait for pressure to attain $\approx 8.8 \times 10^{-5}$ Torr.

- Set temp to 300K.
- Pressure does not attain $\sim 8.8 \times 10^{-5}$ Torr.

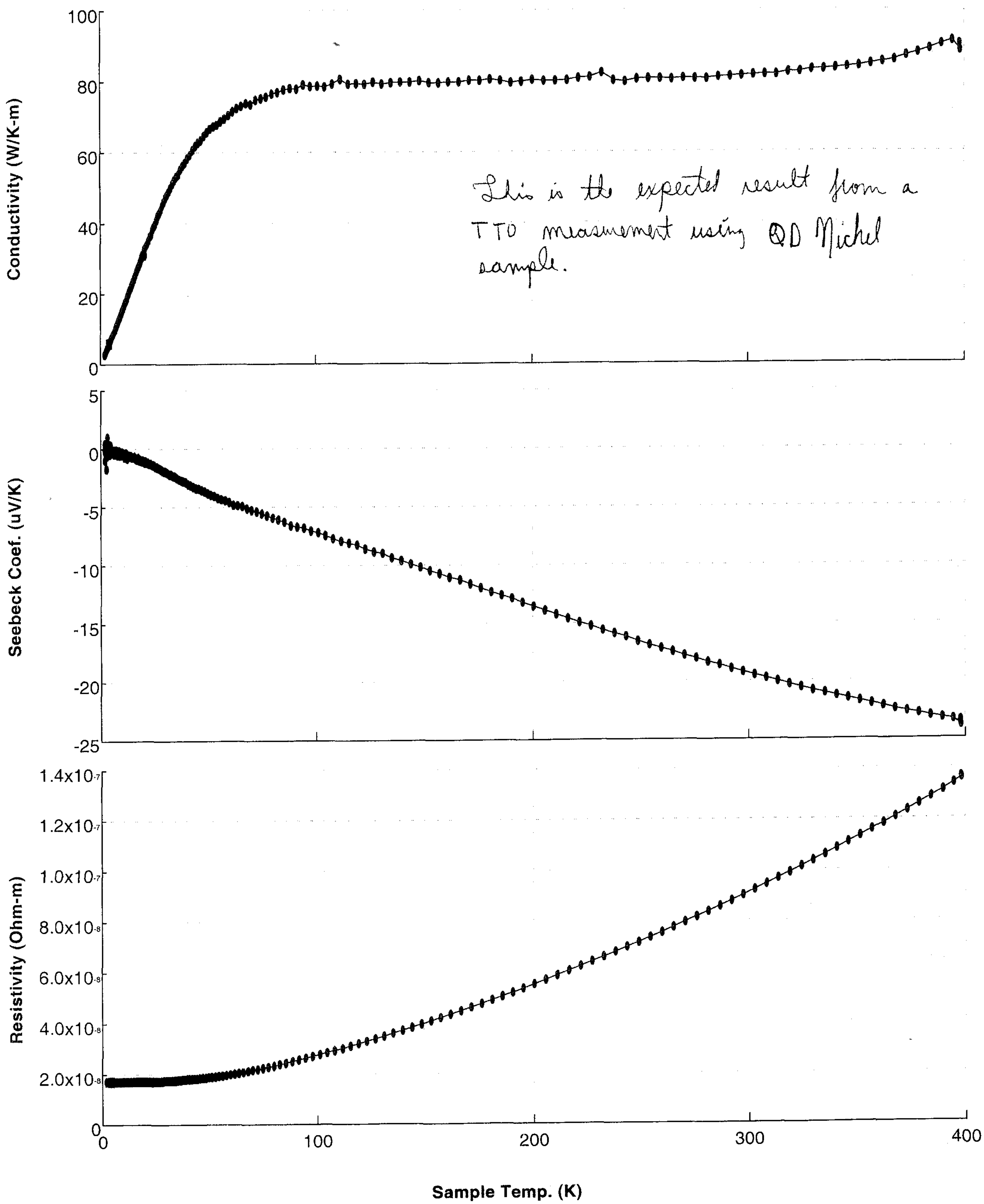
- Run TTO measurement sequence.

- The results were "noisy", so I stopped the sequence @ 5:43PM.

- ~~Set temp to~~ 300K
- ~~[illegible]~~. Purge & Seal.
- Shutdown mode.

Quantum Design Factory Measurement On Nickel Sample

Factory Calibration



Here is the manual for the pressure gauge we've been using w/ the Cryostat.

Model 906 Convection Gauge Controller

for use with Granville-Phillips CONVECTRON® gauges

1/8 DIN Vacuum Gauge Controller

Measures from 0.1 mtorr to 1000 torr

- *low cost*
- *highest performance available at any price*
- *measures to 0.1 mtorr*
- *1/8 DIN - smallest controller for CONVECTRON® gauges*
- *two process control set points with 2 amp relays*
- *RS-232 input/output*
- *calibrated logarithmic analog output*

The Granville-Phillips CONVECTRON® gauge is the most popular convection-Pirani gauge tube in use. Terranova's Model 906 Gauge Controllers offer significant advances in performance and cost savings over other gauge controllers that are compatible with CONVECTRON® gauges.

Our Model 906 is the most advanced controller for CONVECTRON® gauges available. Through use of highly integrated electronics and sophisticated software algorithms, we were able to reduce the size and cost of the controller, and at the same time dramatically improve performance. The 906 is housed in a 1/8 DIN enclosure — half the size of 1/4 DIN enclosures, the industry norm. This size reduction allows the user greater latitude in system design.

The Model 906 measures to lower pressure than other controllers — to 0.1 mtorr. A precise logarithmic analog output makes data collection easy; other CONVECTRON® gauge controllers have highly non-linear outputs requiring a cumbersome look-up table.

An easy-to-use RS-232 serial port is provided for digital data accumulation; also included are two heavy-duty process-control relays for control of small motors and actuators. All of these are standard features, provided at no additional cost.

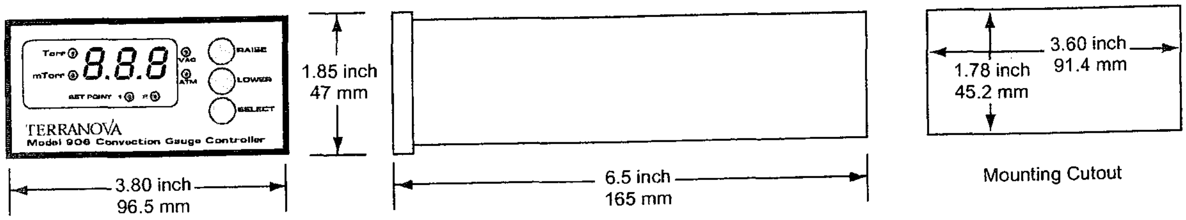
The Terranova 906 is the best controller you can get for any CONVECTRON® gauge application — and at a cost that's about the same as a basic analog controller.

TERRANOVA®

Vacuum Instruments for Science and Industry

Specifications

Dimensions – standard 1/8 DIN enclosure



Mounting Cutout

Measuring Range
0.1 mtorr to 995 torr, for air or nitrogen; (10^{-4} torr to 10^{3} torr); pressure range selection is automatic

Display Range
-29 mtorr to 995 torr; pressures lower than -29 mtorr display ***LO***; pressures higher than 995 torr display ***HI***; if cable is not plugged in display shows ***OFF***

Accuracy of Gauge Interface
Pressure calculation algorithm is accurate to ±1% of published Granville-Phillips data for the CONVECTRON®* gauge (this is for the 906 controller only, and does not include uncertainty of the CONVECTRON® gauge).

Units of Display
torr or mbar; specify at time of ordering

Vacuum Gauge Sensor
uses Granville-Phillips CONVECTRON® gauge; uses standard cable connections

Operating Temperature Range
+2 to +50 deg Celsius

Pressure Display
3-digit bright red LED, 10 mm high

Display Indicators
bright red LED for UNITS, VAC, ATM, SET PT 1, SET PT 2

Display Resolution
varies; from 0.1 mtorr below 10 mtorr, 5 torr above 100 torr

VAC and ATM Adjust
set by front panel pushbuttons

Process Control Set Points
two, set by front panel pushbuttons

Process Control Relays
two relays, contacts rated 2 amp/240 VAC, 30 VDC; +5 volts is provided for TTL applications; D15 accessory connector

Nonvolatile Memory
for VAC, ATM and SET POINTS

Analog Output
calibrated logarithmic, 0.50 volts/decade: 1 mtorr or less = 0 volts; 10 mtorr = 0.50 volts; 100 mtorr= 1.00 volts; 1 torr=1.50 volts,etc.; D15 accessory connector

RS-232 Input/Output
9600 baud, 8-N-1; D15 accessory connector; e.g. send "p" (ASCII value 112), pressure is returned; e.g. 54e-3 for 54 mtorr

Operating Voltage
universal input, 85 - 265 VAC, 30 VA; industry standard IEC 320 instrument power input receptacle on rear panel

Weight
1 lb. / 0.5 kg

Mounting
Side clips are provided for panel mounting in standard 1/8 DIN cutout (see above).

Environmental Considerations
not for use with explosive or corrosive gases

Warranty
Terranova products are warranted to be free from defects in material and workmanship for one year from the date of shipment Warranty details available on request.

Shipping
F.O.B. Mountain View, California; other terms quoted on request.

Ordering Information

Model 906 Convection Gauge Controller for Granville-Phillips CONVECTRON® Gauges
Model 906 Gauge Controller; specify:
units of measure: torr or mbar

includes: AC power cable, 7 feet / 2.0 meters CONVECTRON® gauge cable, 10 feet / 3.0 meters; panel mounting brackets, accessory D15 connector

Specifications subject to change without notice.

TERRANOVA®

Vacuum Instruments for Science and Industry

9702M20

DUNIWAY STOCKROOM CORP.
1305 Space Park Way
Mountain View, CA 94043
Toll Free: 800-446-8811
Phone: 650-969-8811
Fax: 650-965-0764

May 28, 2002

The results of TTO measurements are very noisy, so we are checking the puck to make sure it is mounted properly.

- Set temperature to 300K.
- Vent container.
- The sample was removed & checked out ok.
- Re-inserted sample.
- Purge & Dew.
- TTO measurement was "noisy".

- Mike called Stefano and the following was discussed.

1. Come to QD on Friday w/ samples, TTO puck, and possibly the cryopump.

2. Call him ~~tomorrow~~ to confirm.

3. Send him Raw data of "noisy" TTO measurement.

4. They have been correcting the problem w/ charcoal attachment to cryopump & are not ready to send out another cryopump.

5. They have not found the problem w/ our second cryopump.

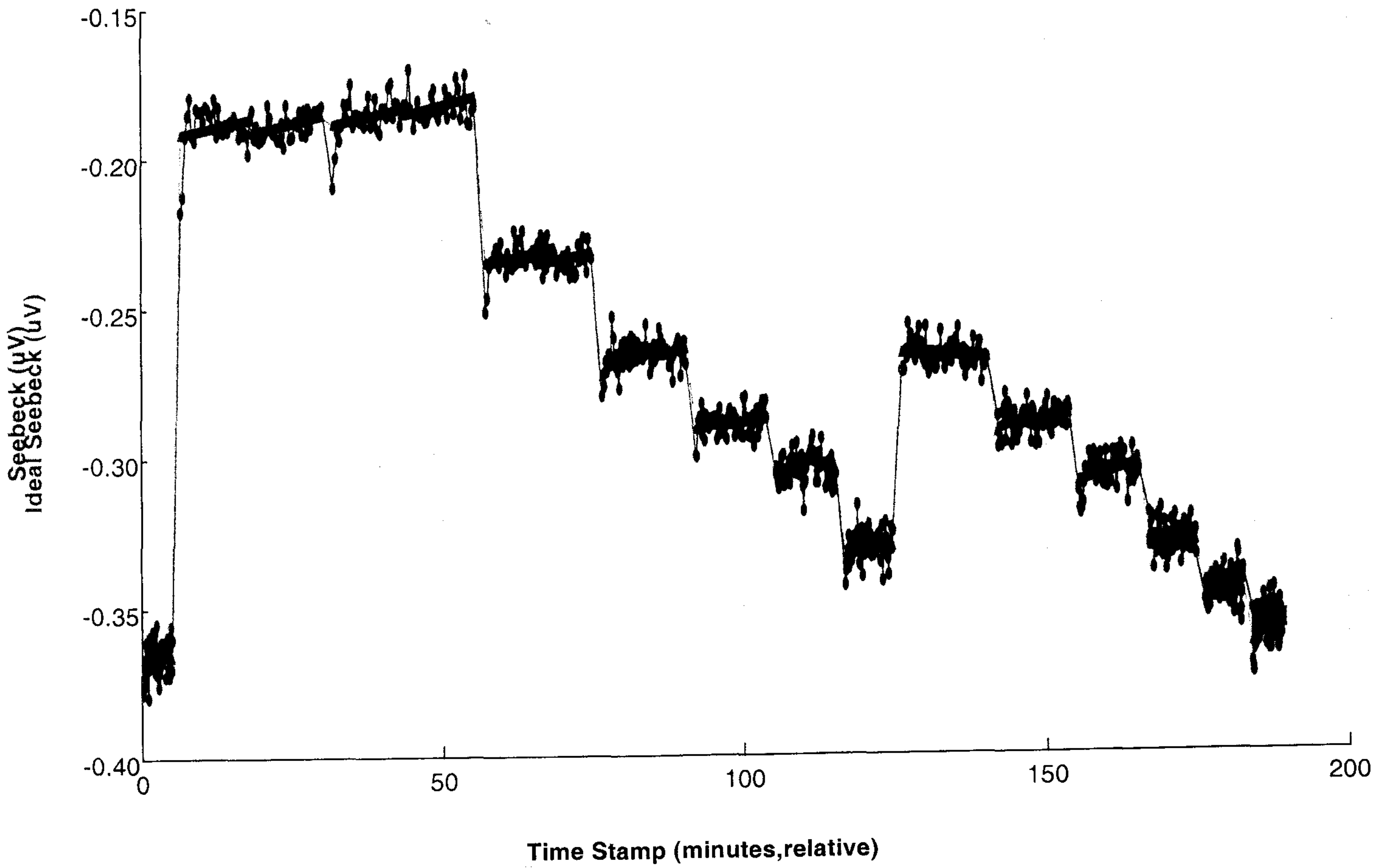
6. We discussed Bryan burning out the power supply. He says the "noise" is probably not due to the Model 6000 motherboard but conceded a possibility of it being a damaged bridge board!

- The sequence file, TTO measurements, raw data, & LogPPMS files were sent to:

service@qdusa.com

to the attention of Stefano Spagna @ 12:30PM.

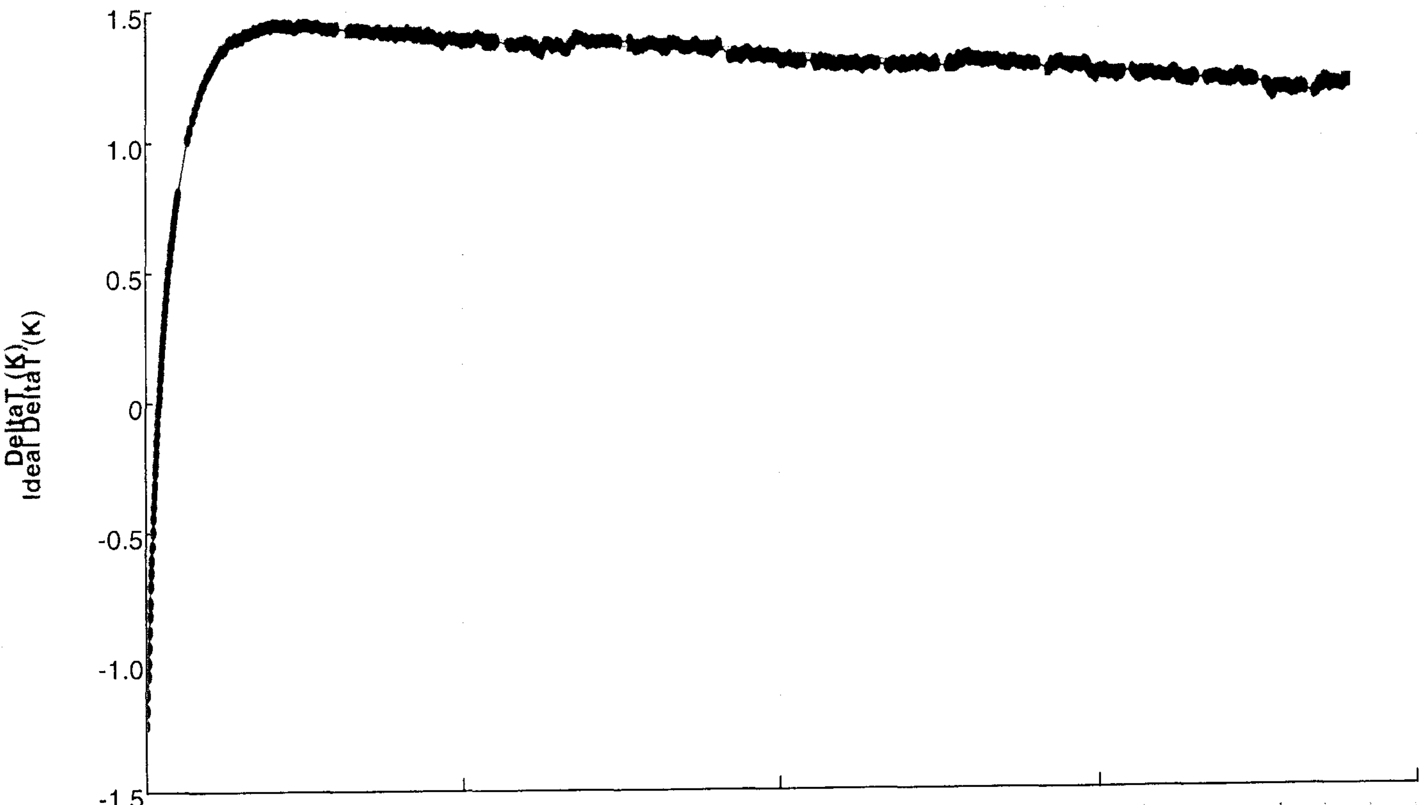
- The plots on the following two pages show the poor TTO measurement behavior.



C:\WINDOWS\Desktop\FirstTTOMeasurement(Nickel)_00001.raw 05/29/02 12:15:34

TTO Measurement Raw Data

First TTO Measurement With New Pressure Gauge (Nickel Test Sample)



- The expected TTO measurement should appear as follows:

May 30, 2002

Stefano & Denish believe the data shows the heater isn't working. I checked the heater resistance & it is 2KΩ. Denish says this is normal.

It turns out that when trying to measure the resistivity, the following error appeared:

"Error: Voltage or Compliance Erorr"

I finally noticed that the J3 cables were disconnected. This prevented the Model 6000 & Model 7100 from communicating

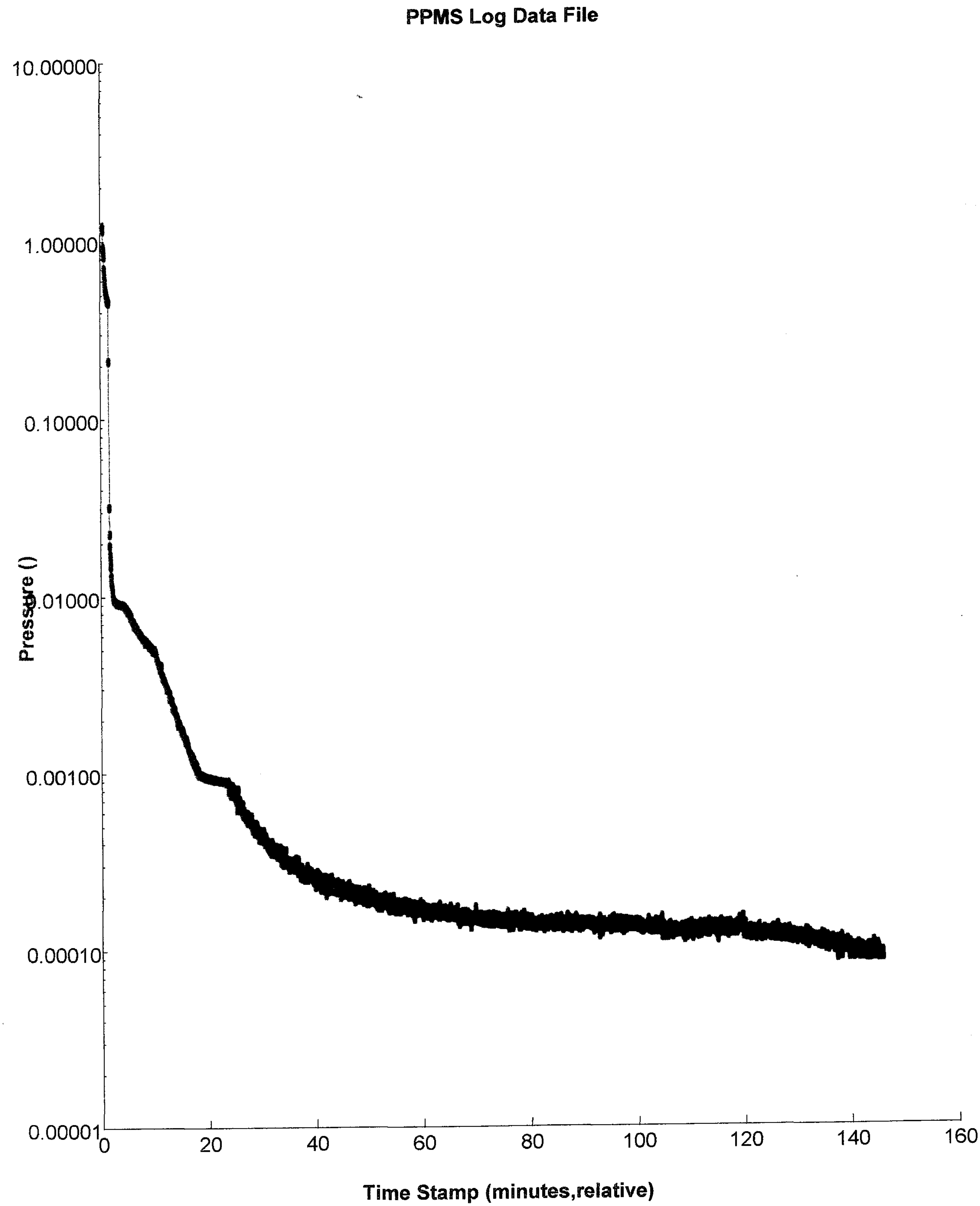
The plugs were reconnected & the TTO measurement look much improved.

- HiVac @ 11:57 AM
  ↳ M. Pump to 458 mTorr
- It took >140 minutes to reach HiVac as can be seen on the plot on the next page.
- This plot was sent to Stefano Spagna after talking w/ him on the phone. We also told him our plans were cancelled to come down tomorrow.
- Most sites on the internet indicate that at 273 K:

For Nickel @ 273K

Thermal conductivity $\approx 90.7 \frac{W}{mK}$

HiVac Attempt Previous To First TTO Measurement



C:\cryolab\users\New Pressure Gauge\05-30-2002\hivacoperation.d05/30/02 14:23:40

~~May 30, 2002~~

~~Stefano & Derek called & said the data appears to clearly indicate the heater is not turning on.~~

~~I will check this [illegible].~~

~~[illegible] continued~~

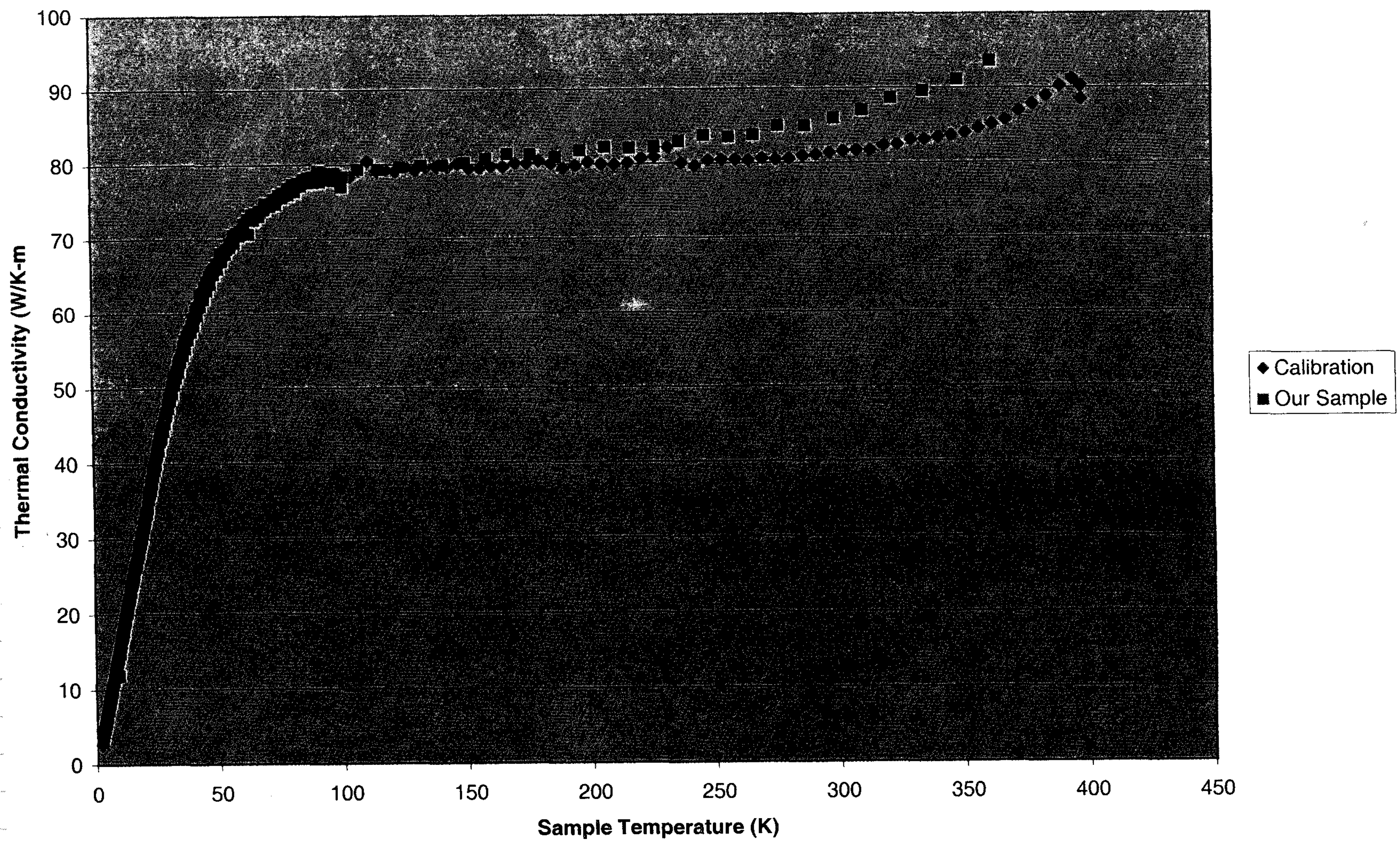
May 31, 2002

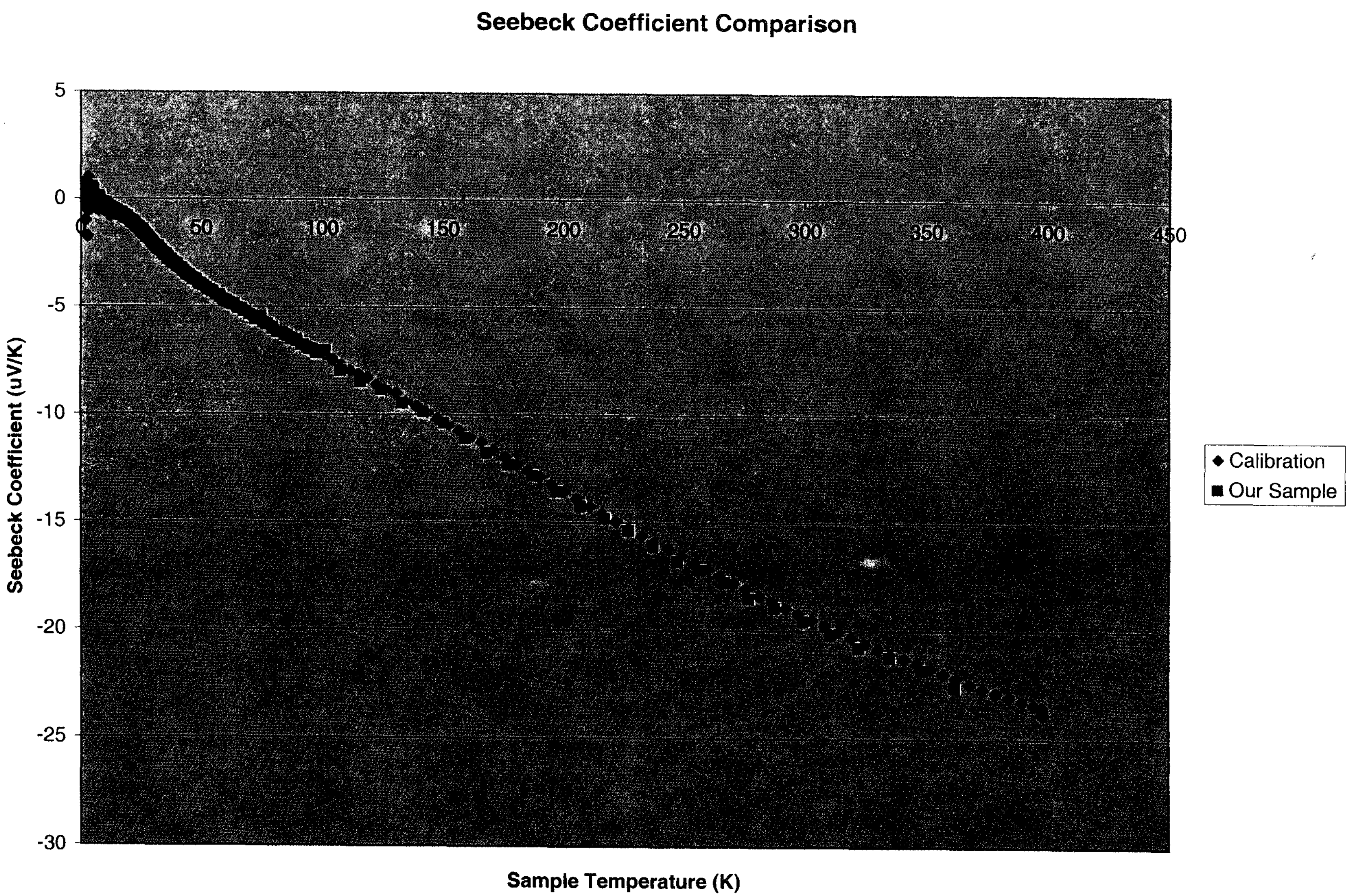
The TTO measurements are encouraging, yet still show some pressure problems above $\approx 150K$.

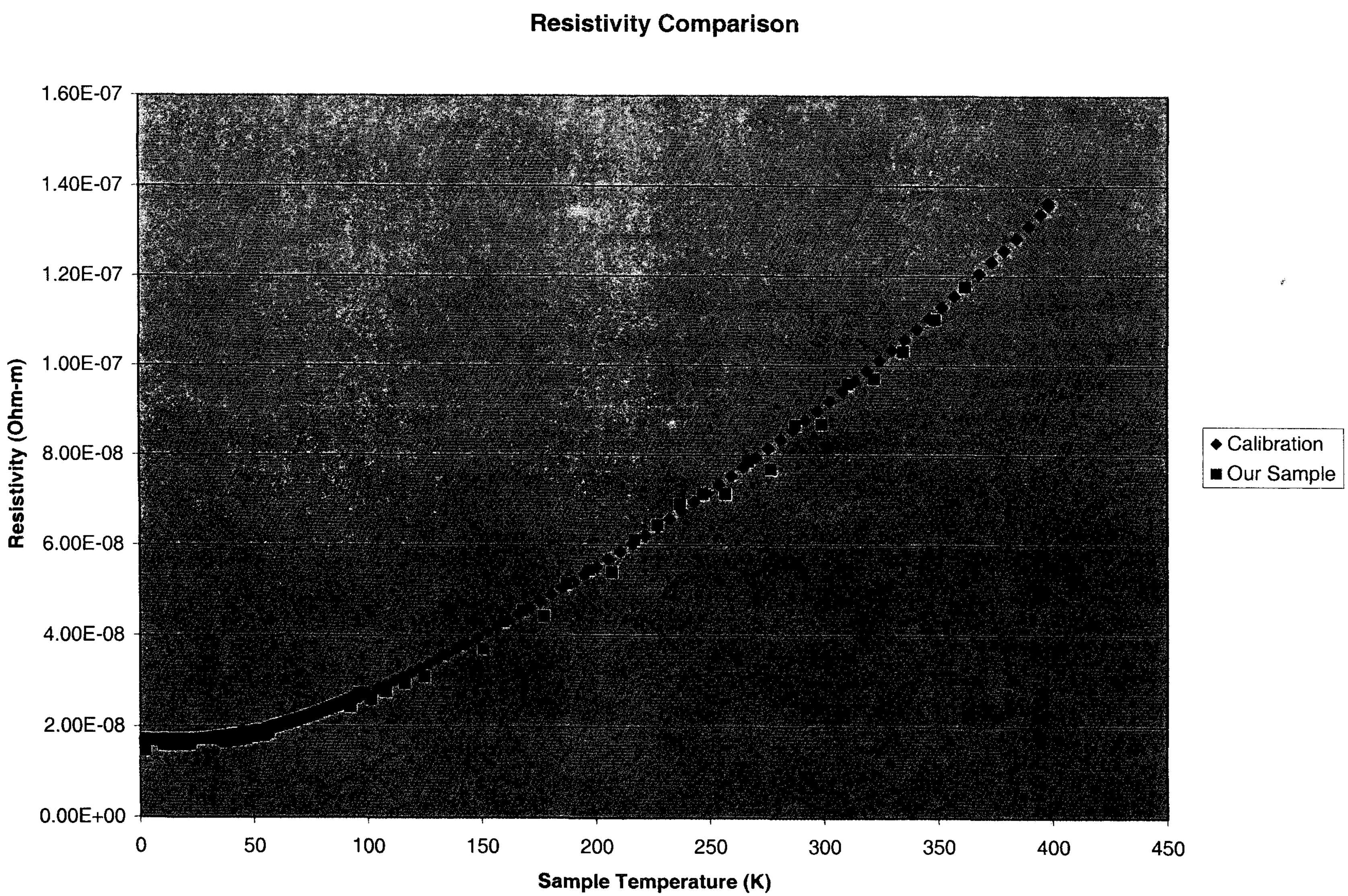
The next 3 plots show the comparison to Ron's Calibration file.

- Notice the deviation in conductivity above 150K. This is due to pressure instability at this point.
- Derek has been notified, & the data will be e-mailed as soon as the measurement completes.
- Although the data was completely repeatable ~~the~~ pressure fluctuated as can be seen by the first plot in this series.

Thermal Conductivity Comparison




Seebeck Coefficient Comparison
5
0
-5
-10
-15
-20
-25
-30
0
50
100
150
200
250
300
350
400
450
Seebeck Coefficient (uV/K)
Sample Temperature (K)
Calibration
Our Sample


Resistivity Comparison
1.60E-07
1.40E-07
1.20E-07
1.00E-07
8.00E-08
6.00E-08
4.00E-08
2.00E-08
0.00E+00
Resistivity (Ohm-m)
0
50
100
150
200
250
300
350
400
450
Sample Temperature (K)
Calibration
Our Sample

Here is the thermal paste we're testing on Vitaloy 1:

High Temperature and High Thermally Conductive Paste

MADE IN USA

B

OMEGATHERM® thermal conducting paste is a high temperature and high thermally conductive paste product. They are specially formulated for permanent and temporary bonding of thermocouples, thin film RTDs, thermistors and other temperature sensors, to most surfaces–metals, ceramics, glass, plastics, paper products.

OMEGATHERM® products is compounded and packaged for convenient, easy mixing and application. Each formulation exhibits important characteristics necessary for accurate, fast, reliable temperature measurement. These are: good adhesion and strength, high temperature rating, high thermal conduction, high electric insulation, thixotropic consistency, fast cure, and easy application.

OMEGATHERM® 201

OMEGATHERM® 201 — is a very high thermally conductive filled silicone paste, ideally suited for many temperature measurement applications. This thick, grey, smooth paste wets most surfaces and will not harden on long exposure to elevated temperatures. It is rated for continuous use between -40 and 200°C (–40 and 392°F).

OMEGATHERM® 201 provides an excellent means of conducting heat and expanding the heat-path area from a surface to a temperature measurement sensor, thus increasing the speed of response and improving accuracy. Some applications are:

a) **Surface Measurement Probes —** dab a small amount on the surface and push the sensor into this area.

b) **Temporary bonding and encapsulating of temperature sensors —** simply dab OMEGATHERM® 201 onto the surface or in the cavity, plant the sensor in the paste, and tape to hold in place.

This highly versatile paste is supplied in ½- and 2-ounce jars, as well as in one- and two-pound containers.

To Order *(Specify Model Number)*

Kit size vs Material	Model No. & Price per kit
	OT-201-□
"A"	OT-201-1/2 $5 one ½ oz. Jar
"B"	OT-201-2 $7.50 one 2 oz. Jar
1 lb. Two-Can Kit	OT-201-16 $55
Two 1 lb. Kits	OT-201-32 $110

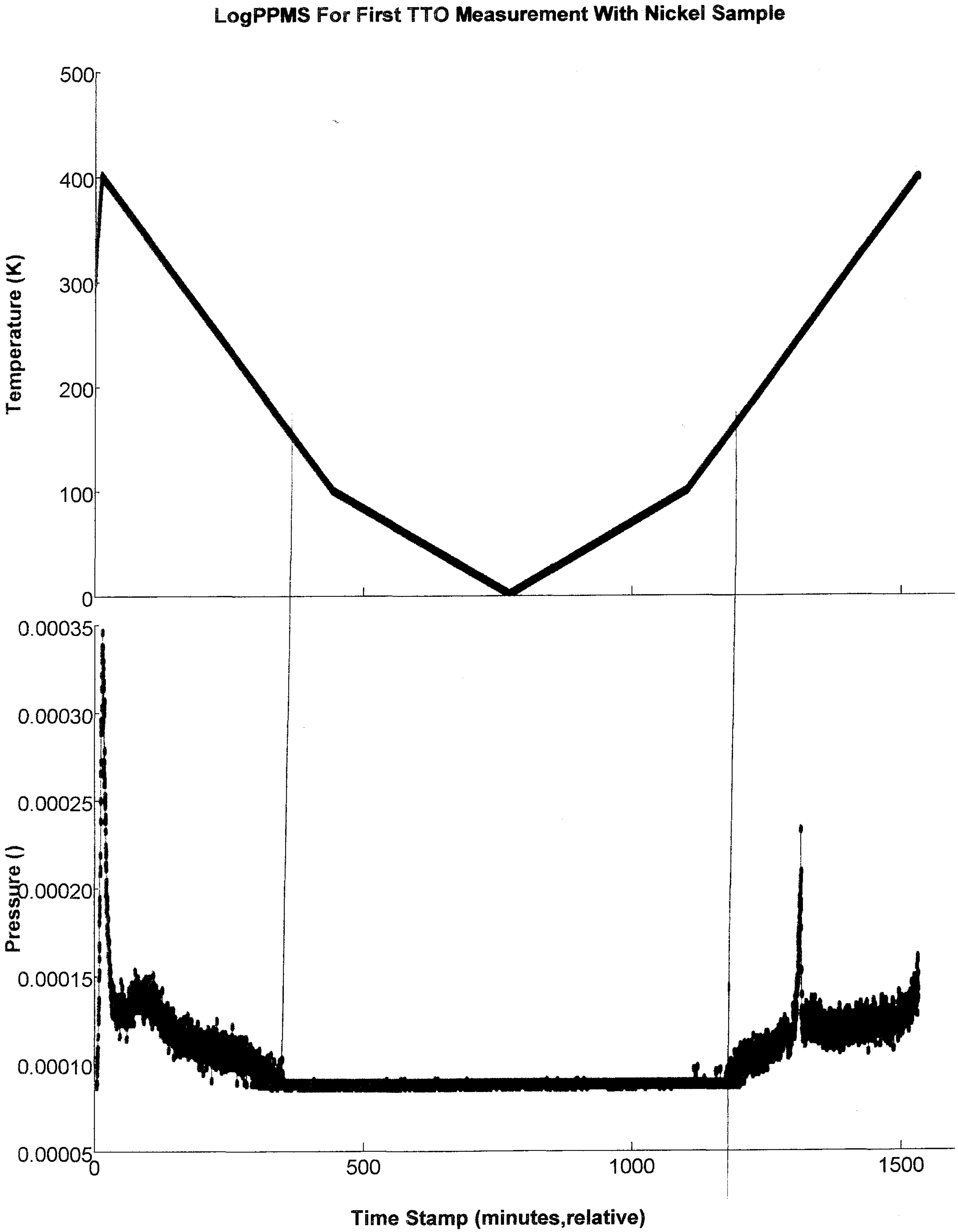
Typical Properties

Model No.	OB-100
Material	Silicone Grease
Continuous Temperature	200°C (392°F)
Cure	Not required
Adheres to Most*	Wets most Surfaces
Thermal conductivity (k) (BTU) (in)/(hr) (ft^2) (°F)	Extremely High 16
Electrical Insulation Volume Resistivity ohm–cm	Very High 10^{14}

*M=Metal
C=Ceramic
PL=Plastic
PA=Paper Products
W=Wood

The above information, while determined by tests and evaluation, is offered only as a general guide. Actual suitability for a particular purpose must be determined by material user. This information is not to be taken as a warranty for which we assume legal responsibility.

First TTO Measurement Using Nickel



C:\cryolab\users\New Pressure Gauge\05-30-2002\FirstTTOMeasurem05/31/02 16:33:03

- I want to try pumping continuously to see if outgassing is the problem.
- Set temp to 300K. → MISTAKE!!!
- Set temp to 400K.
  - The pressure is increasing, because the chamber was sealed @ "shut down".
- Pump continuously... @ 4:42PM...
- Purge & Seal. @ 4:46PM...
- Pump continuously... @ 4:47PM...
- Set T = 375 K.

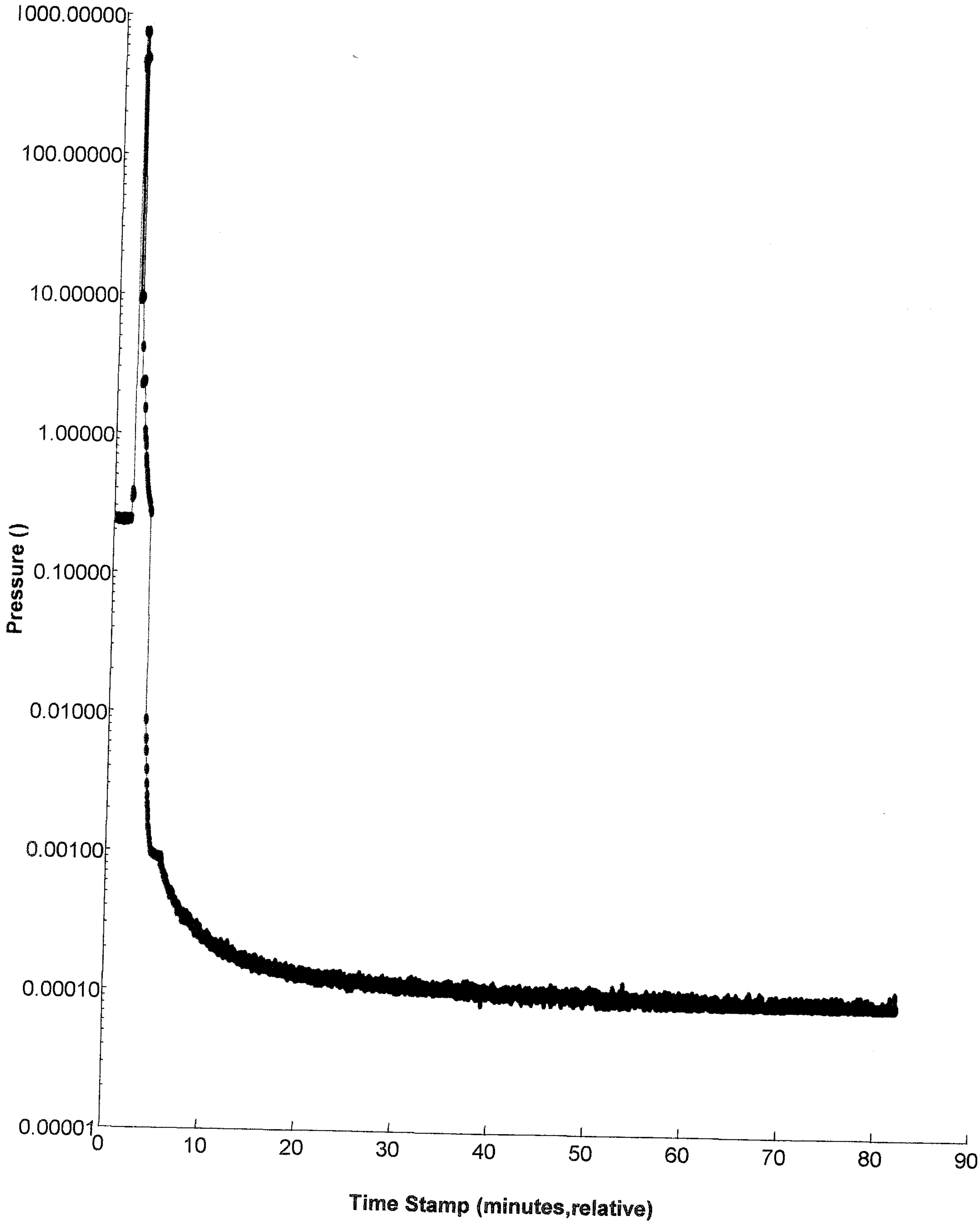
I want to see if it again takes 2 hours to reach HiVac.

~~[illegible]~~

- Set T = 300K.
- Purge / Seal.
- Goto HiVac (@ 3:37 PM)
  - ↳ M. Pump went to ≈ 400 mTorr.
- Eventually came close to 8.8×10^{-5} Torr. (~~See attached~~ plot.)

HiVac Time Trial

PPMS Log Data File



C:\cryolab\06-03-2002\hivactimingtest.dat

06/03/02 16:56:29

Dinesh recommends checking to see if the little charcoal is emitting water vapor.

- Remove "little charcoal" from baffle assembly.
- Insert sample & baffles.
- Purge & Seal. (x 3)
- HiVac @ 10:26 AM.
  ↳ M. Pump to 407 mTorr.
- @ 11:28 AM, the pressure reads ≃ 1.2×10^{-4} Torr.
  I am going to try a regeneration.

- Vent continuously.
- Re-attach charcoal to baffle assembly.
- Close sample chamber.
- Purge & Seal.
- Regenerate in dewar. (X 2) @ 11:37 PM
- Waited ≃ 45 min.
- HiVac...
  ↳ M. Pump to ≃ 220 mTorr.
- HiVac seemed to stabilize at ≃ 1×10^{-4} Torr after ≃ 90 min
- Decrease temp to see pressure reaction.
- As the temperature decreased it appears as though the pressure trends towards greater stability.

- When T passed 230K, the pressure ~~stabilized~~
  ≈ 8.8×10^{-5} Torr. (w/ small fluctuations)
- Stopped data collection @ 2:15 PM.
  (Due the Physics lab students)

Here is a full price list from Epoxy Technology.
They say H20E is either impregnated & works reliably down to 4K.

PRICE LIST
ELECTRICALLY CONDUCTIVE ADHESIVES

PRICES ARE SUBJECT TO CHANGE AT ANYTIME WITHOUT PRIOR NOTICE

OCTOBER 1999
Revised 1/1/2000

EPO-TEK PRODUCT	EVALUATION KIT SIZE	EVALUATION KIT PRICE	1 - 24 LBS	25 - 99 LBS	100 - 499 LBS
E2001,E3001	1 oz	73.00			
	4 oz	292.00	584.00	545.00	
E2101	1 oz	96.00			
	8 oz	465.00	795.00	746.00	734.00
E3084	1 oz	153.00			
	8 oz	660.00	1132.00	1068.00	1056.00
E4110	2 oz	112.00	566.00	531.00	519.00
H20E, H20F,	1 oz	90.00			
& H20S,H20S-MC	8 oz	429.00	736.00	656.00	645.00
H20E-175	1 oz	103.00			
	8 oz	507.00	847.00	758.00	746.00
H20E-PFC	1 oz	109.00			
	8 oz	519.00	889.00	832.00	817.00
H22, H24	1 oz	74.00	577.00	513.00	501.00
H21D, H31D	1 oz	86.00	613.00	545.00	534.00
H27D	1 oz	90.00	736.00	656.00	645.00
H31, H31LV	1 oz	74.00	577.00	513.00	501.00
H35-175MP, H35-175MPLV H35-175MPT	1 oz	118.00	996.00	936.00	922.00
H37-MP, H37-MPT	1 oz	118.00	996.00	936.00	922.00
N20E & N30	8 oz	72.00	121.00	105.00	98.00
P-1011,	1 oz	100.00			
P-1011S	8 oz	489.00	845.00	755.00	744.00

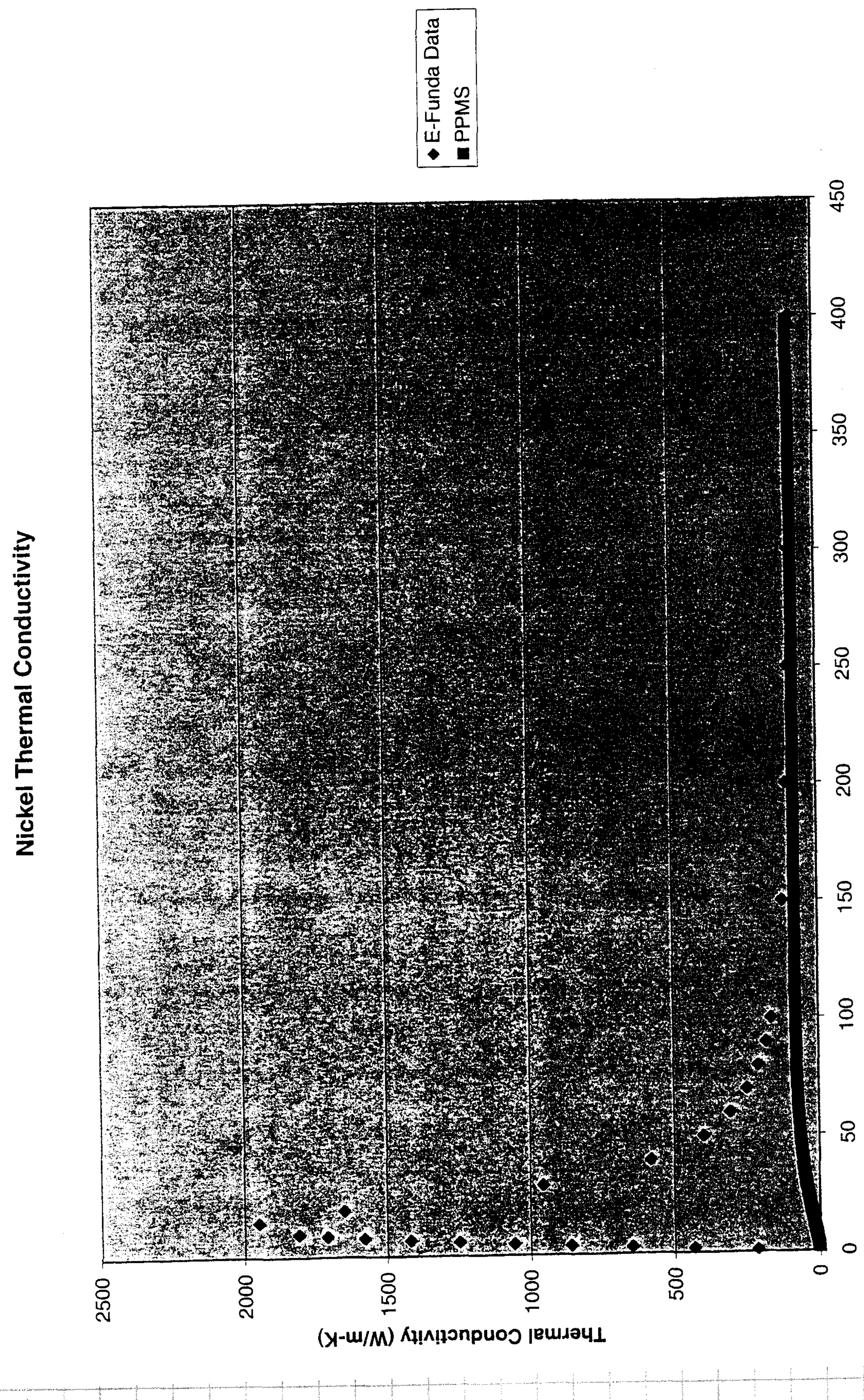
AVAILABILITY: Shipment within 10 days ARO. Payment terms are NET 30 days, FOB Billerica MA. Production prices are on bulk packaging. A handling charge is imposed on two component systems when specified lot sizes (lb. increments only) are requested: i.e. 20 lbs. in 4 x 5 lb. lots. Trial evaluation kit size and prices are firm - no quantity discounts.

EPOXY TECHNOLOGY, INC.
14 FORTUNE DRIVE BILLERICA MA 01821-3972
Phone: 978-667-3805 Fax: 978-663-9782
Web Site: www.epotek.com

EPO-TEK™

June 6, 2002

Here is a comparison of our collected data vs. data taken from the CRC handbook of materials.



Here is the raw data from CRC:

THERMAL CONDUCTIVITY OF METALS AND SEMICONDUCTORS AS A FUNCTION OF TEMPERATURE

This table gives the temperature dependence of the thermal conductivity of several metals and of carbon, germanium, and silicon separate entries are given for the thermal conductivity parallel (||) and perpendicular (⊥) to the layer planes. The thermal conductivi materials is very sensitive to impurities at low temperatures, especially below 100 K. Therefore, the values given here should be reg values for a highly purified specimen; the thermal conductivity of different specimens can vary by more than an order of magnitu temperature range. See Reference 2 for details.

REFERENCES

1. Ho, C. Y., Powell, R. W., and Liley, P. E., *J. Phys. Chem. Ref. Data*, 1, 279, 1972.
2. White, G. K., and Minges, M. L., *Thermophysical Properties of Some Key Solids*, CODATA Bulletin No. 59, 1985.

Thermal Conductivity in W/cm K

				Carbon (C)						
				Diamond (type)			Pyrolytic graphite			
T/K	Ag	Al	Au	I	IIa	IIb	‖	⊥	Cr	Cu
1	39.4	41.1	5.46						0.402*	422
2	78.3	81.8	10.9	0.0138*	0.033*	0.0200*			0.803	84.0
3	115	121	16.1	0.0461	0.111	0.0676			1.20	125
4	147	157	20.9	0.108	0.261	0.160			1.60	162
5	172	188	25.2	0.206	0.494	0.307			2.00	195
6	187	213	28.5	0.344	0.820	0.510			2.39	222
7	193	229	30.9	0.523	1.24	0.778			2.27	239
8	190	237	32.3	0.762	1.77	1.12			3.14	248
9	181	239	32.7	1.05	2.41	1.53			3.50	249
10	168	235	32.4	1.40	3.17	2.03	0.811	0.0116	3.85	243
15	96.0	176	24.6	3.96	8.65	5.66			5.24	171
20	51.0	117	15.8	7.87	16.8	11.2	4.20	0.0397	5.93	108
30	19.3	49.5	7.55	18.8	38.9	26.5	9.86	0.0786	5.49	44.5
40	10.5	24.0	5.15	29.4	65.9	44.0	16.4	0.120	4.25	21.7
50	7.0	13.5	4.21	35.3	92.1	59.1	23.1	0.152	3.17	12.5
60	5.5	8.5	3.74	37.4	112	67.5	29.8	0.173	2.48	8.29
70	4.97	5.85	3.48	36.9	119	69.1	36.6	0.181	2.07	6.47
80	4.71	4.32	3.32	35.1	117	65.7	42.8	0.181	1.84	5.57
90	4.60	3.42	3.28	32.7	109	60.0	47.5	0.176	1.69	5.08
100	4.50	3.02	3.27	30.0	100	54.2	49.7	0.168	1.59	4.82
150	4.32	2.48	3.25	19.5	60.2	32.5	45.1	0.125	1.29	4.29
200	4.30	2.37	3.23	14.1	40.3	22.6	32.3	0.0923	1.11	4.13
250	4.29	2.35	3.21	11.0	29.7	17.0	24.4	0.0711	1.00	4.06
300	4.29	2.37	3.17	8.95	23.0	13.5	19.5	0.0570	0.937	4.01
350	4.27	2.40	3.14	7.55*	18.5*	11.1*	16.2	0.0477	0.929	3.96
400	4.25	2.40	3.11	6.5*	15.4*	9.32*	13.9	0.0409	0.909	3.93
500	4.19	2.36	3.04				10.8	0.0322	0.860	3.86
600	4.12	2.31	2.98				8.92	0.0268	0.807	3.79
800	3.96	2.18	2.84				6.67	0.0201	0.713	3.66
1000	3.79		2.70				5.34	0.0160	0.654	3.52
1200	3.61*		2.55				4.48	0.0134	0.619	3.39
1400							3.84	0.0116	0.588	
1600							3.33	0.0100	0.556	
1800							2.93	0.00895	0.526*	
2000							2.62	0.00807	0.494*	

THERMAL CONDUCTIVITY OF METALS AND SEMICONDUCTORS AS A FUNCTION OF TEMPERATURE (continued)

T/K	Fe	Ge[a]	Mg	Ni	Pb	Pt	Si[a]	Sn	Ti	W
1	1.71	0.274	9.86	2.17	27.9	2.31	0.0693*	183	0.0144*	14.4
2	3.42	2.06	19.6	4.34	44.6	4.60	0.454	323	0.0288*	28.7
3	5.11	5.35	29.0	6.49	35.8	6.79	1.38	297	0.0432	42.8
4	6.77	8.77	37.6	8.59	22.2	8.8	2.97	181	0.0575	56.3
5	8.39	11.6	45.0	10.6	13.8	10.5	5.27	117	0.0719	68.7
6	9.93	13.9	50.8	12.5	8.10	11.8	8.23	76	0.0863	79.5
7	11.4	15.5	54.7	14.2	4.86	12.6	11.7	52	0.101	88.0
8	12.7	16.6	56.7	15.8	3.20	12.9	15.5	36	0.115	93.8
9	13.9	17.3	57.0	17.1	2.30	12.8	19.5	26	0.129	96.8
10	14.8	17.7	55.8	18.1	1.78	12.3	23.3	19.3	0.143	97.1
15	17.0	17.3	41.1	19.5	0.845	8.41	41.6	6.3	0.212	72.0
20	15.4	14.9	27.2	16.5	0.591	4.95	49.8	3.2	0.275	40.5
30	10.0	10.8	12.9	9.56	0.477	2.15	48.1	1.79	0.365	14.4
40	6.23	7.98	7.19	5.82	0.451	1.39	35.3	1.33	0.390	6.92
50	4.05	6.15	4.65	4.00	0.436	1.09	26.8	1.15	0.374	4.27
60	2.85	4.87	3.27	3.08	0.425	0.947	21.1	1.04	0.355	3.14
70	2.16	3.93	2.49	2.50	0.416	0.862	16.8	0.96	0.340	2.58
80	1.75	3.25	2.02	2.10	0.409	0.815	13.4	0.915	0.326	2.29
90	1.50	2.70	1.78	1.83	0.403	0.789	10.8	0.880	0.315	2.17
100	1.34	2.32	1.69	1.64	0.397	0.775	8.84	0.853	0.305	2.08
150	1.04	1.32	1.61	1.22	0.379	0.740	4.09	0.779	0.270	1.92
200	0.94	0.968	1.59	1.07	0.367	0.726	2.64	0.733	0.245	1.85
250	0.865	0.749	1.57	0.975	0.360	0.718	1.91	0.696	0.229	1.80
300	0.802	0.599	1.56	0.907	0.353	0.716	1.48	0.666	0.219	1.74
350	0.744	0.495	1.55	0.850	0.347	0.717	1.19	0.642	0.210	1.67
400	0.695	0.432	1.53	0.802	0.340	0.718	0.989	0.622	0.204	1.59
500	0.613	0.338	1.51	0.722	0.328	0.723	0.762	0.596	0.197	1.46
600	0.547	0.273	1.49	0.656	0.314	0.732	0.619		0.194	1.37
800	0.433	0.198	1.46*	0.676		0.756	0.422		0.197	1.25
1000	0.323	0.174		0.718		0.787	0.312		0.207	1.18
1200	0.283	0.174		0.762		0.826	0.257		0.220	1.12
1400	0.312			0.804		0.871	0.235		0.236	1.08
1600	0.330					0.919	0.221		0.253	1.04
1800	0.345*					0.961			0.270*	1.01
2000						0.994*				0.98

[a] Values below 300 K are typical values.
* Extrapolated.

Here is the datasheet for our epoxy.

DATA SHEET

EPO-TEK H20E

Electrically Conductive, Silver Epoxy

Rev. III
4/00

TYPICAL PROPERTIES

(To be used as a guideline only)

NUMBER OF COMPONENTS .... Two
MIXING RATIO .... PARTS BY WEIGHT
- Part "A" (epoxy resin and silver powder) 1
- Part "B" (hardener and silver powder) 1
- NOTE: Mix contents of each container (A and B) thoroughly before mixing the two together.

CURE SCHEDULE (minimum)
- 175°C .... 45 seconds
- 150°C .... 5 minutes
- 120°C .... 15 minutes
- 80°C .... 90 minutes

PHYSICAL PROPERTIES
- Color .... *Bright Silver*
- Consistency .... Smooth, thixotropic paste
- Specific Gravity
  - Part "A" 2.03
  - Part "B" 3.07
- Viscosity (@ 100 rpm/23°C) .... 2,200 - 3,200 cPs
- Glass Transition Temp. (Tg) .... > 80°C
  - (cured 150°C/1 hour .... typically 100°C
- Coefficient of Thermal Expansion (CTE)
  - Below Tg .... 31 x 10^{-6} in/in/°C
  - Above Tg .... 120 x 10^{-6} in/in/°C
- Lap Shear Strength .... 1,500 psi
- Die Shear Strength .... > 10 Kg/3,400 psi
- Degradation Temperature .... 410°C
- Weight Loss @ 200°C (TGA) 0.16%
- Operating Temperature
  - Continuous .... 200°C
- Storage Modulus .... 750,000 psi

ELECTRICAL - THERMAL PROPERTIES
- Volume Resistivity .... < 0.0004 ohm-cm
- Thermal Conductivity .... 2.0 W/m°K
- Thermal Resistance: (Junction to Case)
  - TO-18 package with nickel-gold metallized 20 x 20 mil chips and bonded with EPO-TEK H20E (2 mils thick)
  - Eutectic Die attach .... 4.8 to 5.3°C/watt
  - EPO-TEK H20E .... 6.7 to 7.0°C/watt
  - Pass Thermal Shock - Gold backed silicon chips bonded to a gold metallized ceramic substrate will pass: 5 cycles from -62°C to +125°C
  - Bonded Silicon Chips (100 x 100 mils) when placed on a 300 - 340°C heat column will resist a shear force of 16 oz.

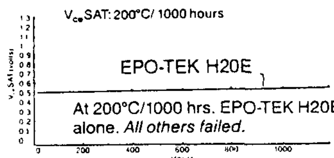
SCHOTTKY DIODE

INITIAL	2 WEEKS @ 200°C
C_j 1pF (typical)	
$V_b \geq 5V$ @ 10 µa	4.8V @ 10 µa
$V_f \leq 0.4V$ @ 1 ma	0.32V @ 1 ma

POT LIFE .... 4 days

SHELF LIFE
- One year when stored at room temperature. REFRIGERATION NOT REQUIRED

TO-3 package, 2N3055 chips, medium power transistor - 4 amp pulse



H20E EXHIBITS SUPERIOR V_{ce}SAT PERFORMANCE.

EPO-TEK H20E is a 100% solids, two component silver filled epoxy with a soft, smooth, thixotropic consistency designed specifically for chip bonding in microelectronic and optoelectronic applications.

The excellent handling characteristics and the extremely long pot life at room temperature for this unique Electrically Conductive Adhesive (ECA) are obtained without the use of solvents. In addition to the high electrical conductivity, the short curing cycles, the proven reliability and the convenient mix ratio, EPO-TEK H20E is extremely simple to use and make it an ideal material for use in electronic applications. The pure silver powder is dispersed in both the resin and the hardener so that it can be used in a convenient 1:1 mixing ratio. In fact the EPO-TEK H20E is the easiest-to-use two component silver epoxy that has ever been developed for the microelectronic industry.

EPO-TEK H20E is especially recommended for use in high speed epoxy chip bonding systems where very fast cures are highly desirable. This cannot be obtained with single component systems. Because EPO-TEK H20E can be cured very rapidly, it is an excellent material for making fast circuit repairs. EPO-TEK H20E can be screen printed, machine dispensed or stamped and can withstand wire bonding temperatures in the range of 300 – 400°C.

EPO-TEK H20E has proven itself to be extremely reliable over the many years of service and is still the conductive adhesive of choice for new applications.

- NASA APPROVED
- NON TOXIC – complies with USP Class VI Biocompatability standards

When placing an order, please specify whether EPO-TEK H20E is to be used by volume or weight.

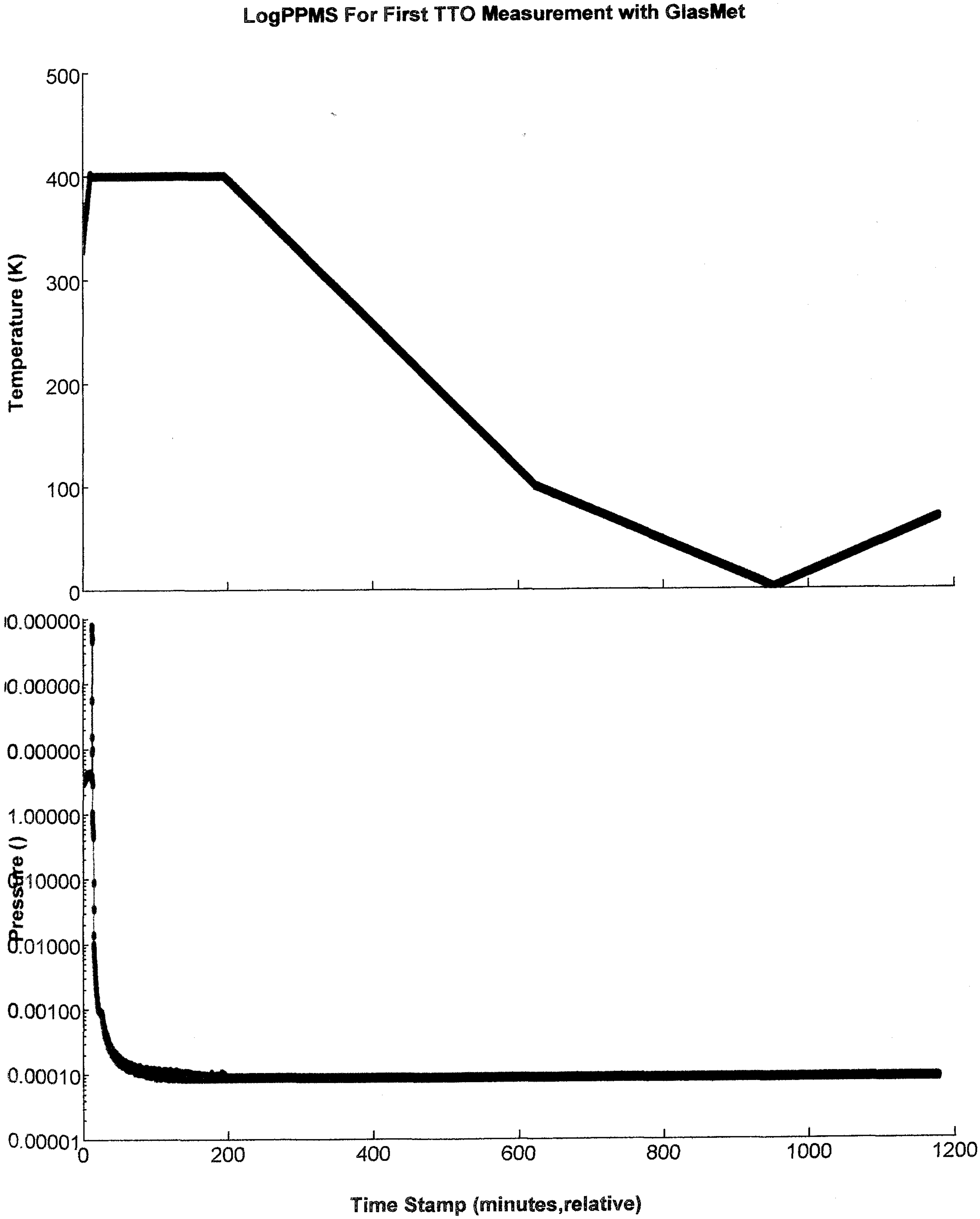
EPOXY TECHNOLOGY, INC. 14 Fortune Drive Billerica, MA 01821-3972 USA
PHONE: 978.667.3805 1.800.227.2201 **FAX:** 978.663.9782

June 10, 2002

— The following page is the pressure for the TTO measurement of Glasnet on June 7. It was ~~stopped~~ over the weekend when Hareem noticed the Resistivity cable was left plugged in, instead of the TTO cable.

— The measurement has been restarted.

Pressure Stays Stable From 400K and Below



C:\cryolab\06-07-2002\GlasMet(Smaller)_logPPMS.dat 06/10/02 09:16:38

June 11, 2002

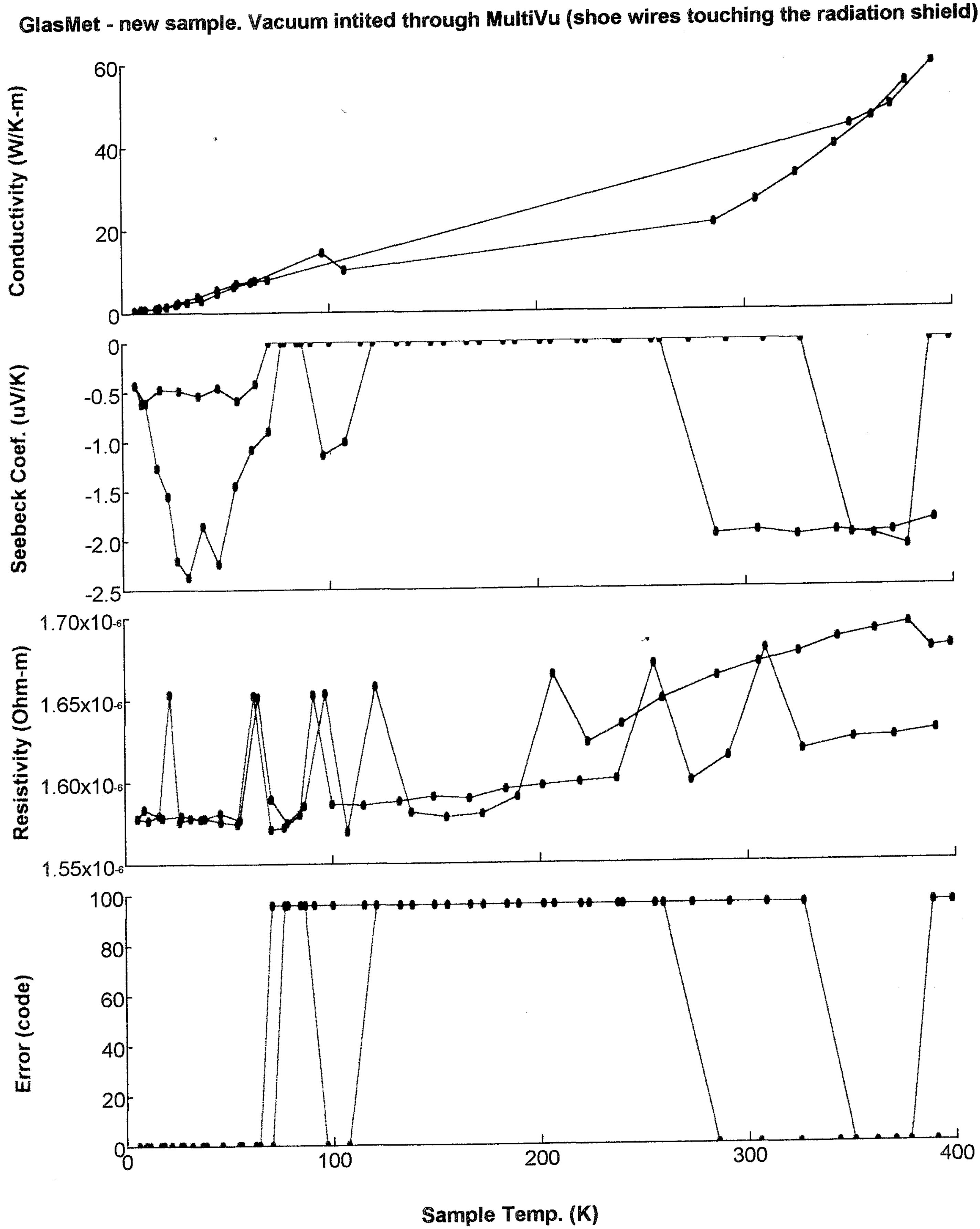
The TTO for Glasnet is shown on the right.

The thermal conductivity loops repeatedly at the ends, but could not be measured for T = 100 → 300 K.

The next page shows how peculiarities correspond to measurement errors.

I checked the puck & I think there was a short circuit somewhere. It seemed okay before I reinserted it.

Glasmet (Smaller) Data

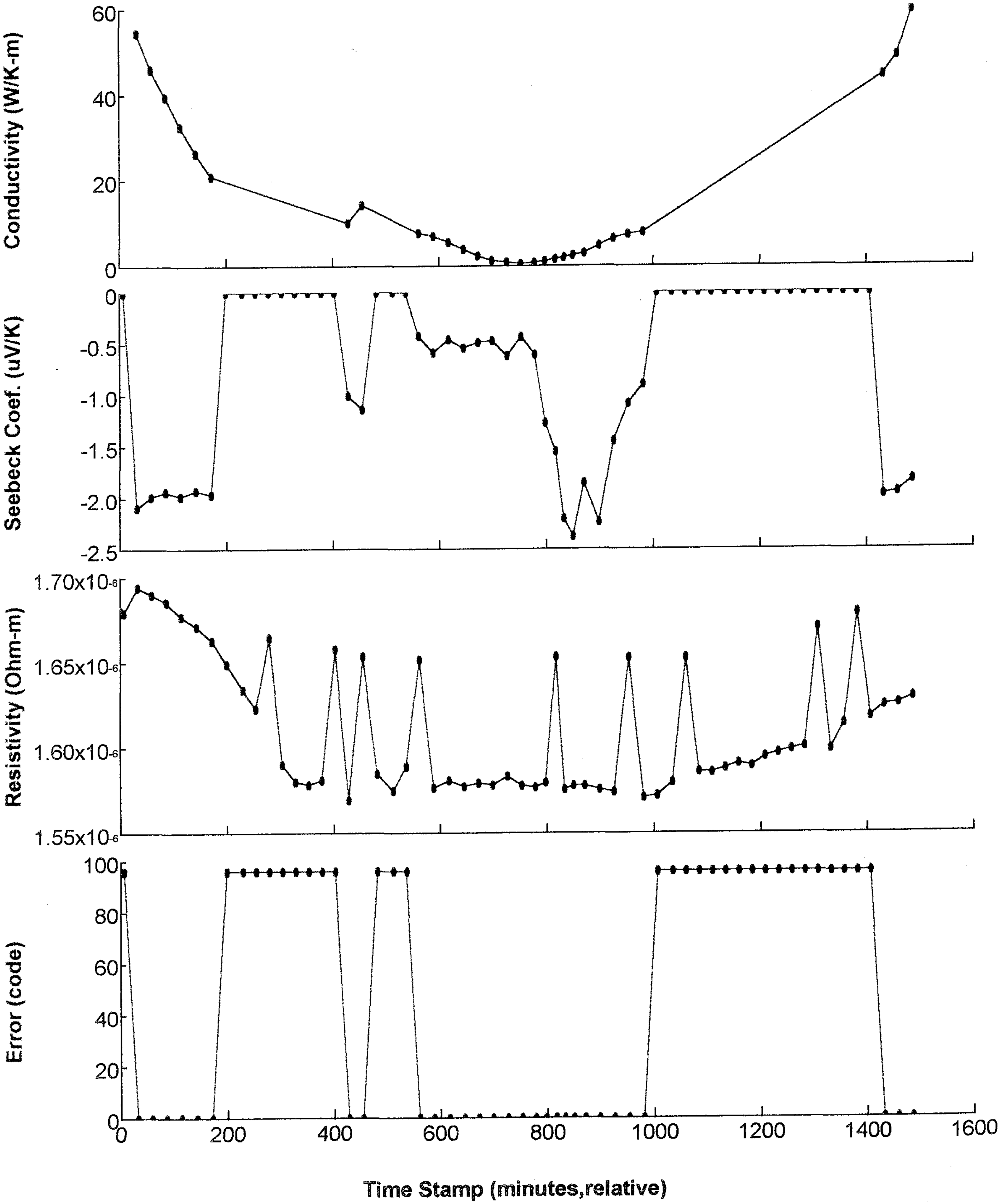


C:\cryolab\06-10-2002\GlasMet(Smaller)_00001.dat

06/11/02 15:29:28

TTO Data Correlation to Errors

GlasMet - new sample. Vacuum intited through MultiVu (shoe wires touching the radiation shield)



C:\cryolab\06-10-2002\GlasMet(Smaller)_00001.dat

06/11/02 16:04:36

6/12/2002

Mark ✓ GPIB Card   external measurment
back of Model 6000   RS232 Connector

— Talk to Application Scientist

(Should not drill some hole)

Steve Application scientist. (Don't recommand it.)

steve@qdusa.com   Send the picture
x226

Some document on GPIB.
www.qdusa.com
→ Go to "Tech Resource"
→ Go to "Technical Document"
→ Under "PPM", go to "Application Note"
→ Go to "Interfacing Third Party Instrument to the PPMS Software Environment - 1070-202"
(which is Acrbat format)

June 20, 2002

I e-mailed Mark Seebach the data collected from Mike's sequence. I will call tomorrow.

I wanted to try a 3rd Glasmet measurement but the sample's 3rd lead broke. I must remount it tomorrow.

July 10, 2002

The Hi-Vac system has been functioning properly for several weeks now w/out error. Several TTO measurements have been taken and the only problem so far have been setting the correct TTO parameters.

Michael Hall's logbook carries the details of the most recent Cryostat runs.