Characterization of a Cryogenic Displex Refrigerator

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<u>Abstract</u>

A cryogenic system is assembled and tested for the purpose of variable temperature, optical measurements of Prussian blue analogs. The system is a retrofitted Advanced Research Systems DE-202 displex and is characterized by measuring its cooling capabilities as a function of time as well as its minimum temperature as a function of applied power. Several configurations of the refrigeration system were tested, with and without internal heat shield and exchange gas present.

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1. Introduction

The purpose of this experimental project is to assemble and to test a cryogenic system that will be used for variable temperature optical measurements of Prussian blue analogs. More specifically, a commercial closed cycle refrigerator (Advanced Research Systems DE-202), known as a displex, has been retrofitted with new thermometers, a new heat shield, and a vacuum can with optical windows. Additionally, a sample holder and an adaptor for the heat shield were designed and fabricated. Equipped with these modifications, the cooling power of the system was studied and is reported herein. Ultimately, this apparatus will be use to perform low temperature, optical measurements in a commercial Fourier-transform infrared (FT-IR) spectrometer. Ultimately, the experimental configuration will allow the C-N resonances to be studied as a function of temperature from 30 K to 300 K and energies ranging from 4000 cm^{-1} to 400 cm^{-1}. The C-N resonances of the Prussian blue analogs provide information about the analogs magnetic spin. The spin transition of the Prussian blue analog due to changes in temperature or exposure to light is a very intriguing feature and is the focus of study with the displex.

Once the displex has been modified as described in the next section, its refrigeration capabilities can be characterized by studying the temperature as a function of applied power (heat) and by observing cooling curves as a function of time, Section 3. These data have been collected for multiple configurations of the displex (using the shield, various vacuum cans, and various levels of residual exchange gas). The characterization data are then used to design a set of temperature control protocols for the system. To accomplish this task, a feedback mechanism, known as proportional-integral-differential (PID) control, is tuned to fit our system. The development of this control process is heuristic but should evolve into a recipe-book type set of protocols that are appropriate to our specific system. Finally the whole system must be made mobile to be transported to the FT-IR spectrometer and these aspects are described in the final section.

2. Retro-Fitting the ARS DE-202 Displex for Temperature Contol

The basis of our apparatus is a closed cycle helium displex, specifically an Advanced Research Systems (ARS) Model DE-202. The apparatus consists of three components: a compressor, an expander (or refrigerator), and a mechanical vacuum pump, as seen in Figure 1. The vacuum pump is used to evacuate the vacuum can. Once the can has been evacuated, the compressor and expander, which operate with gaseous helium as the refrigerant, are activated. During the cycle, the compressor receives warm, low pressure helium from the return line of the expander, this helium is then compressed, cooled, filtered, and returned to the expander by way of the supply line. The system will eventually come to a minimum temperature where the cooling power of the displex is in equilibrium with all sources of heat leaks, encompassing those intentionally from a heater and those present parasitically from the environment and electronics. The coldest part of the apparatus is referred to as a cold finger.

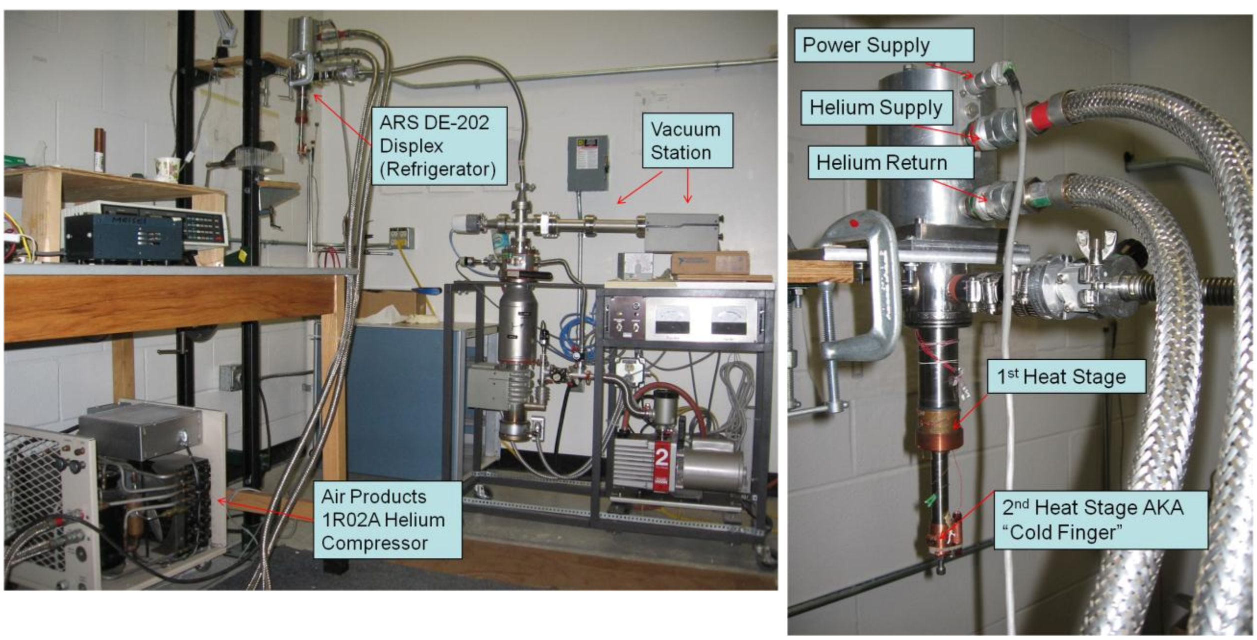

Figure 1. System Components (left panel) DE-202 Displex (right panel).

Essentially, the displex makes the system as cold as its cooling power will allow. While it is useful to simply make materials cold and to study them at low temperatures, it is often necessary to be able to control the temperature in order to explore the thermal response of the samples. A heat shield, which

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is available commercially, can be used to minimize the radiation component of the parasitic heat that reaches the low temperature environment. Although the commercial heat shield was purchased, the corresponding adaptor, which is required to install the shield, was designed and fabricated locally. The home-made adaptor, as shown in Figure 2, couples to the first heat stage of the displex and is attached with four 4-40 screws. The mount is threaded to allow it to mate with the heat shield, possesses thru-ports for wires, and has eight 2-56 tapped holes for electronics and other mounting purposes. The adaptor was fabricated in-house by the UF Physics Instrument Making Shop, and detailed drawings are in Appendix A.

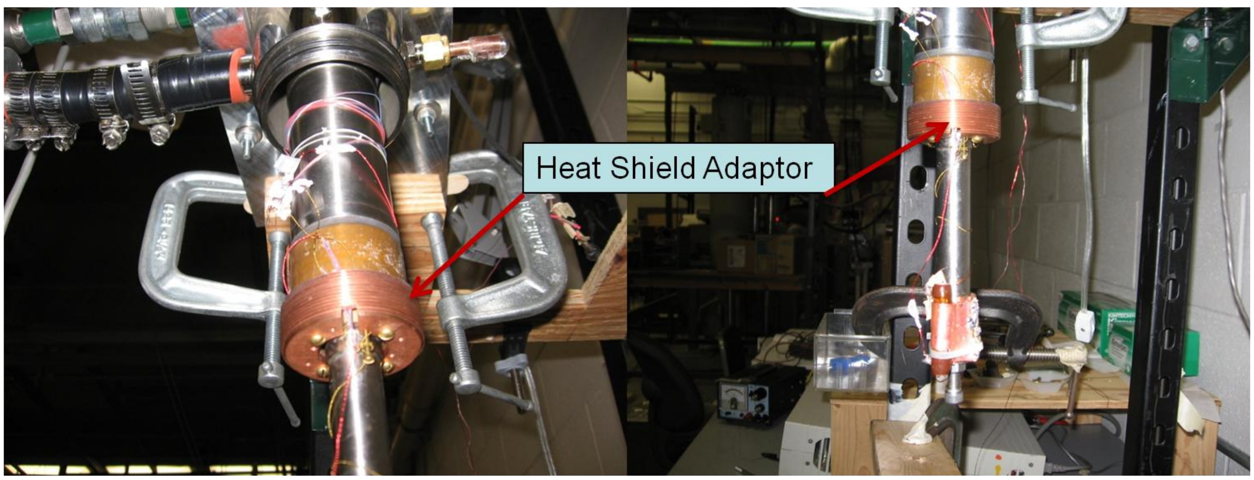

Figure 2 Heat Shield Adaptor

Thermometry is a key element for any low temperature application and for thermal control of the sample environment. For most applications, secondary thermometers, which are devices whose material properties are temperature dependent and have been calibrated either by a primary thermometer or at certain fixed points, are employed [1]. Presently, our main thermometer is a diode thermometer whose forward voltage bias, at a constant 10 μA excitation current, is temperature dependent. The thermometer is commercially produced by Lakeshore Cryotechnics, Model DT-470 and a standard calibration curve is provided. Initially, two used model DT-470 thermometers were made

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available for this work, but it was not clear whether or not they were functional. In order to ascertain their functionality, the units were biased with the appropriate excitation current and the voltage drops across the diodes were measured at known temperatures, namely room temperature and liquid nitrogen temperature, 77 K. To supply the excitation current of 10 μA, a DC constant current source was constructed, Figure 3 [2]. A digital voltage meter was used to monitor the voltage drop across the diode, and the data were subsequently compared to the calibrated curve to verify that one of the units was working properly.

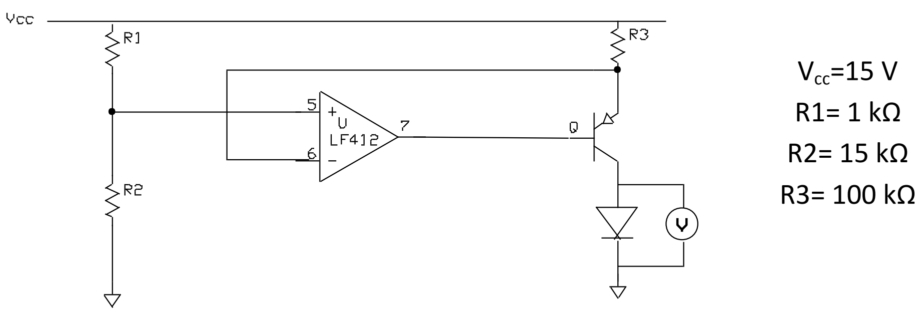

Figure 3. Circuit diagram of 10 μA constant current source.

In many applications, the primary purpose of the displex is to cool and to maintain the sample at the base temperature for long periods of time. Consequently, the displex is not equipped to operate in different modes to regulate the temperature. As a result, to control the temperature of the system, heat must be added. Our method of delivering heat is to drive an electrical current through a resistive load, thereby causing joule heating. In other words, the power output can be varied by adjusting either the current through or the voltage across the resistance. After several iterations and multiple incinerated resistors and coils of resistive wire, a 10 Ω, 10 W power resistor was selected. Finally, the heater and thermometer must be mounted in good thermal contact with the cold finger, and this requirement is achieved by mounting both units in a thin piece of copper known as a shim. The shim must be shaped in a manner that it will have intimate contact with both the device it is holding and the cold finger of the displex. To help improve thermal contact, the device and the mount are coated with a thin layer of Apiezon-N vacuum grease.

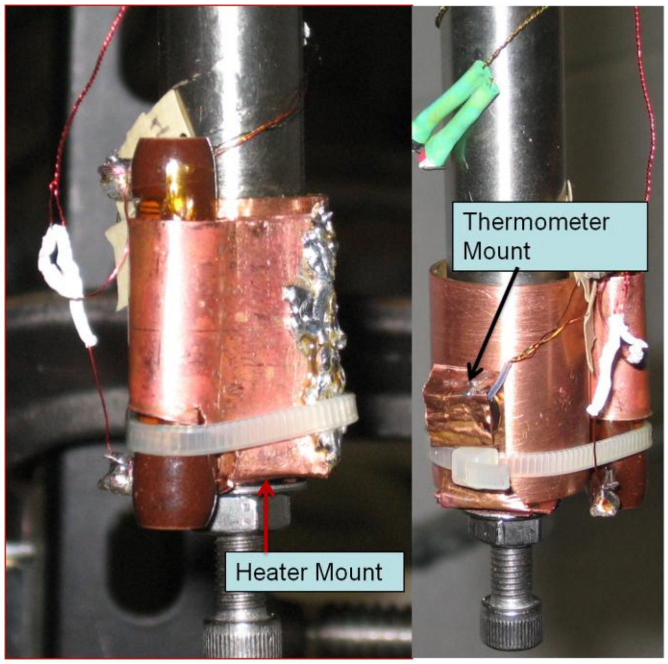

Figure 4. Heater Mount (left panel) and Thermometer Mount (right panel).

In order to utilize the electronic devices located in the cold space, the operator must have access to them electronically. Since the system is under vacuum during operation, a mechanism that will give access to the electronics via wires and maintain vacuum is required. For this purpose, a vacuum feed-through, shown in Figure 5, is utilized. The feed-through begins as a hollow copper tube. The appropriate wire sets for accessing electronics (in our case, 6 twisted copper pairs 26 AWG, and a thermocouple) are placed inside the tube. A Swage-Lok device is mounted on the exterior of the tube to mate with the vacuum port of the displex. The wires are surrounded by Emmerson and Cummins Sycast 1266 epoxy and left overnight to dry. After mounting, the internal thermometer is connected to the vacuum feed-through to a LakeShore Model DRC-93C Temperature controller, which has an input card designed specifically for the use of the DT-470 Diode thermometer. It provides the needed 10 μA excitation current and converts the voltage drop to temperature in units of Kelvin. Additionally, the DRC-93C can supply up to 1 A constant current to the heater via a different set of wires utilizing the same feed-through.

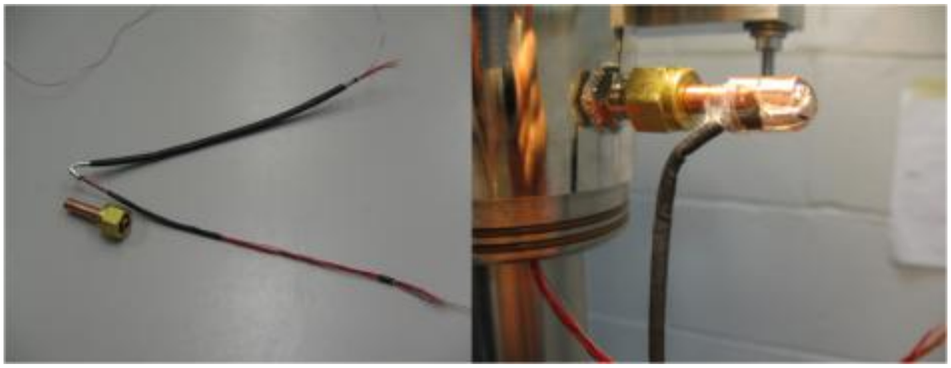

Figure 5. Feed-through components before assembly (left panel) and mounted feed through (right panel)

3. Data Acquisition and Characterization

The DRC-93C temperature controller has a GPIB port that is used to connect the unit to a computer equipped with LabVIEW. By adapting a previously written LabVIEW driver, a Virtual Instrument (VI) for data acquisition was created. The VI controls the manual heater setting, calculates heating power applied due to the current supplied and the size of the resistance in the cryostat, and writes temperatures as a function of time with applied power to a spreadsheet. The VI also has a graphical output on its front panel that is an invaluable, qualitative tool when trying to assess the approach to thermal equilibrium. Detailed code is located in Appendix B and all data analysis was performed using Origin.

By using this VI at various applied powers, the equilibrium temperatures as a function of power and Figure 6 shows the minimum temperature as a function of applied power. The response is linear, which is somewhat unexpected, as one would normally expect the response to be logarithmic. This response could be a result of non-constant cooling power from the displex. By extrapolating the linear fit it is possible to estimate the parasitic heat load on the system. Here, the parasitic heat load is 2.2 W.

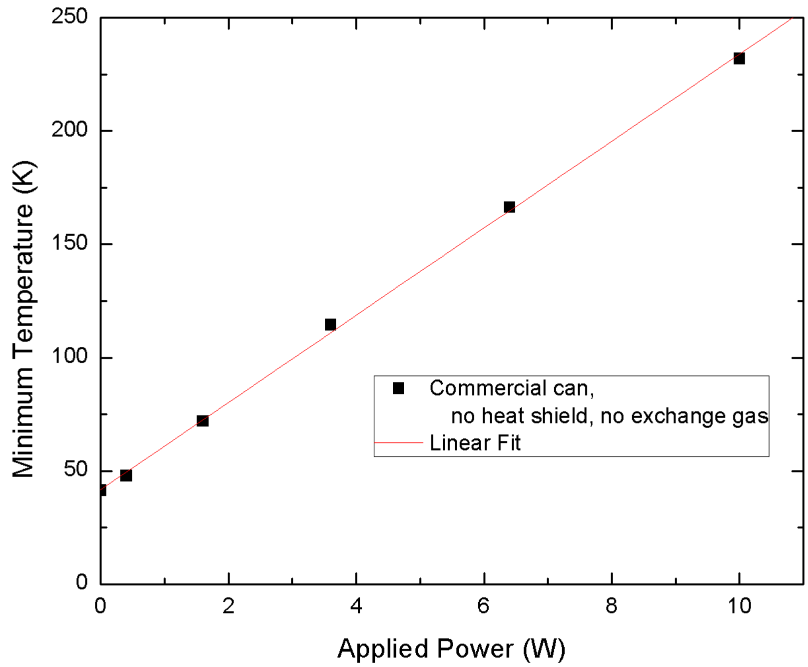

Figure 6. Minimum Temperature Vs Applied Power using a commercial vacuum can without the heat shield or exchange gas.

When using the displex in the FT-IR, trace amounts of an exchange gas may be used to improve thermal contact between the sample (an insulator) and the temperature control system. The primary drawback of using an exchange gas is that contact between the system and the external environment is also improved, thus increasing parasitic heat and raising the lowest attainable temperature. In other words, some part of the cooling power is sacrificed to ensure that the sample is in good thermal contact with the cold finger. Figure 7 shows temperature as a function of time for four configurations of the displex using the commercial vacuum can.

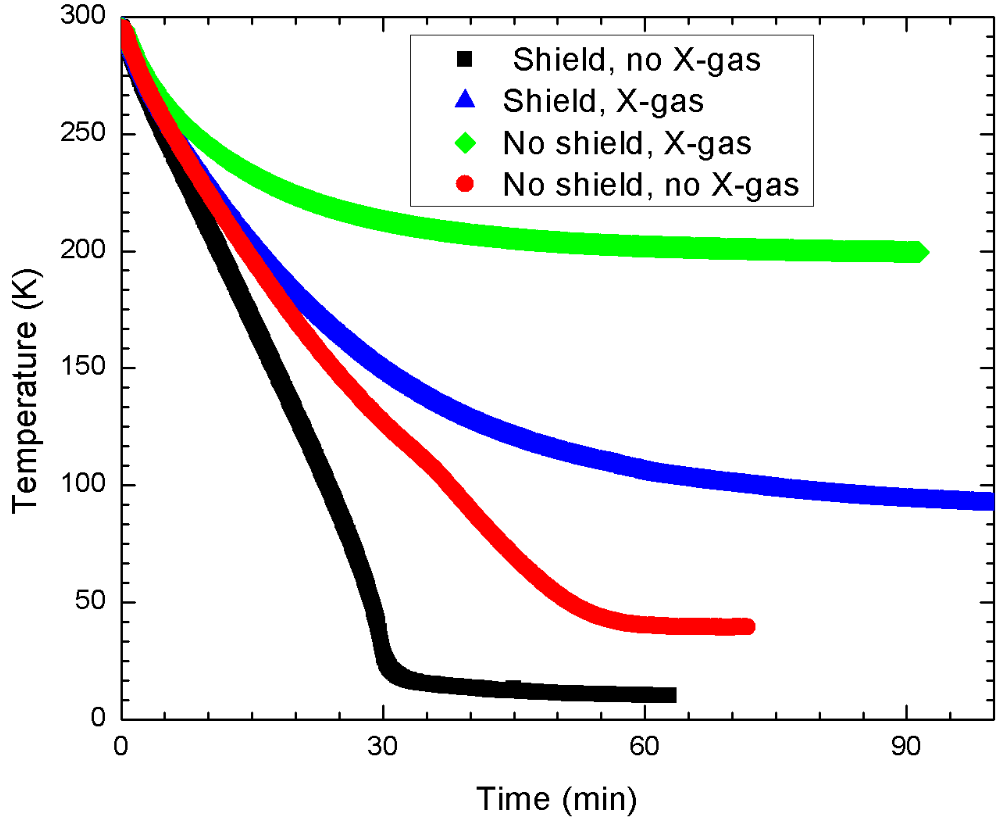

Figure 7. Minimum temperatures attained for various configurations of the displex as a function of time. Exchange gas is nominally 200 mTorr of Helium.

4. Future Research and Summary

Several logical steps are required to extend this project. For example, a sample mount and adaptor must be fabricated, and a preliminary design has been adapted from the specifications provided by Matthieu Dumont in the Talham Group (UF Chemistry), Appendix C. The temperature control of the system needs to be fine-tuned, and the DRC-93C can, in principle, provide the necessary capabilities that can be computer controlled. Finally, the system must be made transportable in order to be relocated to the Department of Chemistry for use in the IR Spectrometer.

Acknowledgements

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References

1. F. Pobell, Matter and Methods at Low Temperatures, (Springer-Verlag, Berlin Heidelberg, 1992), p 210.
2. P. Horowitz W. Hill, The Art of Electronics, (Cambridge University Press, Cambridge UK, 1989), p 181.

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Appendix A

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PHYSICS RESEARCH SHOP JOB REQUISITION

Job Title and Number Displex Shield adaptor

Brief Job Description copper unit that mates with existing Shield for mounting.

Submitted By Ann Ziegler (Summer REU) Yitzi Calm Authorized By Meisel

Date Submitted 3 June 2010

Phone No. and Email 605-645-3381, Ziegler@phys.ufl.edu

USE SPACE BELOW FOR SKETCH:

Departments of Physics and Astronomy
University of Florida
Phone: (352) 392-0480 Email: mshop@phys.ufl.edu

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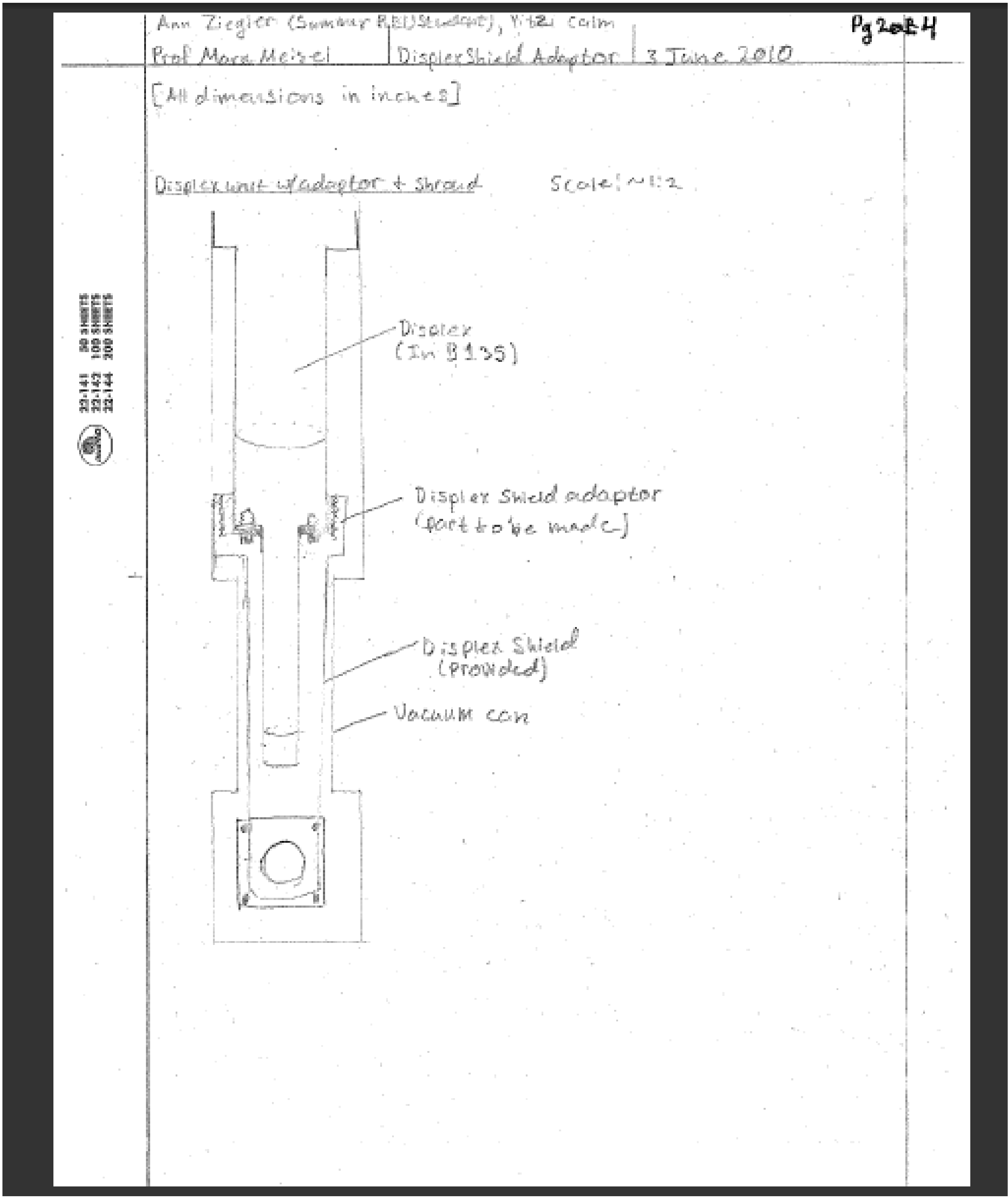
Ann Ziegler (Summer RET Student)
Pg 2 of 4
Displex Shield Adaptor
3 June 2010
[All dimensions in inches]
Displex unit w/adaptor + shroud
Scale: ~1:2
Displex
(In B135)
Displex Shield adaptor
(part to be made)
Displex Shield
(provided)
Vacuum can

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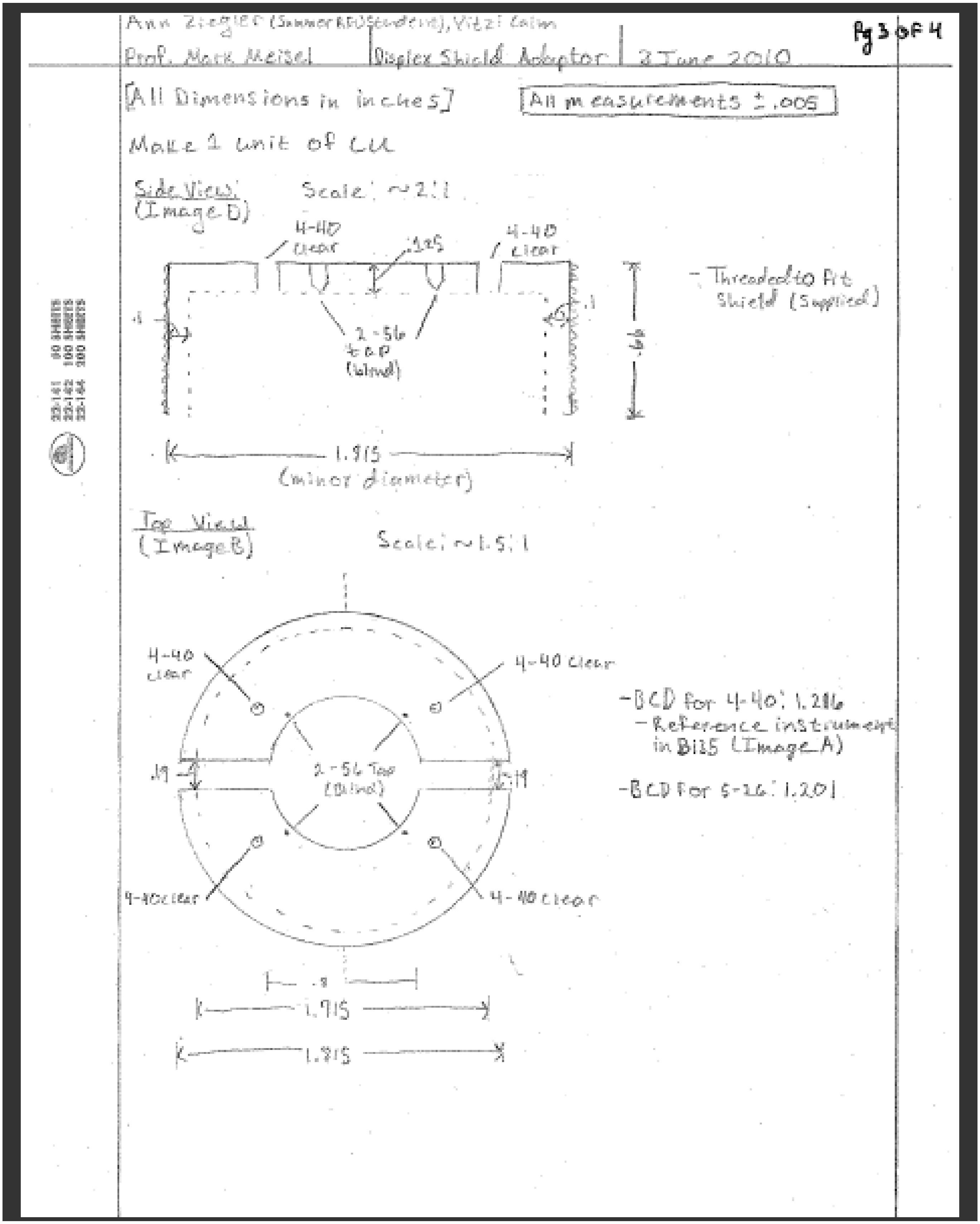
Ann Ziegler (Summer REU Student), Vitzi Calm
Prof. Mark Meisel
Displex Shield Adaptor
2 June 2010
Pg 3 of 4
[All Dimensions in inches]
All measurements ± .005
Make 1 unit of Cu
Side View: (Image D)
Scale: ~2:1
4-40 clear
.125
4-40 clear
Threaded to fit Shield (Supplied)
.1
.1
2-56 tap (blind)
.96
1.915
(minor diameter)
Top View (Image B)
Scale: ~1.5:1
4-40 clear
4-40 clear
-BCD for 4-40: 1.286
-Reference instrument in B135 (Image A)
.19
2-56 Tap (Blind)
.19
-BCD for 5-26: 1.201
4-40 clear
4-40 clear
.8
1.915
1.815

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Ann Ziegler (summer RET student) Displex Shield Adaptor 4 June 2010 Pg 4 of 4
Yitzi Calm
Prof Mark Meisel

Image A

Image B

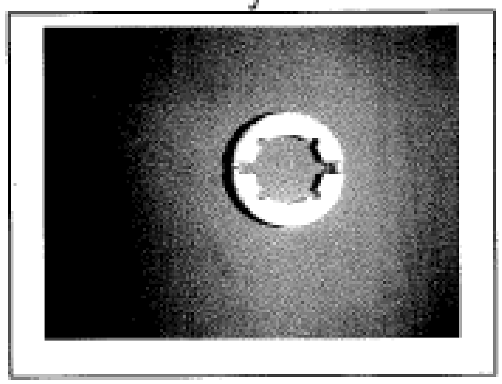


Image C

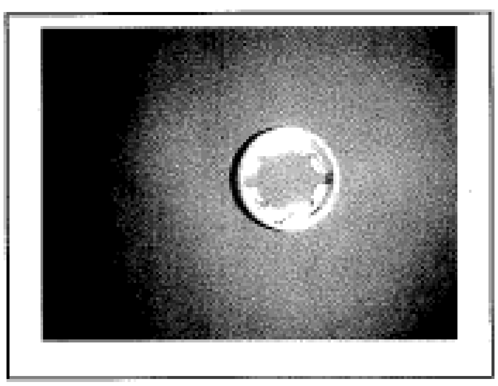
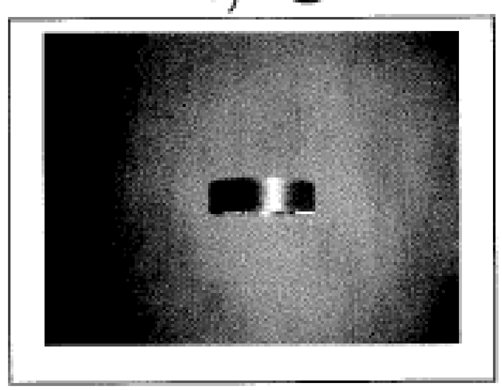
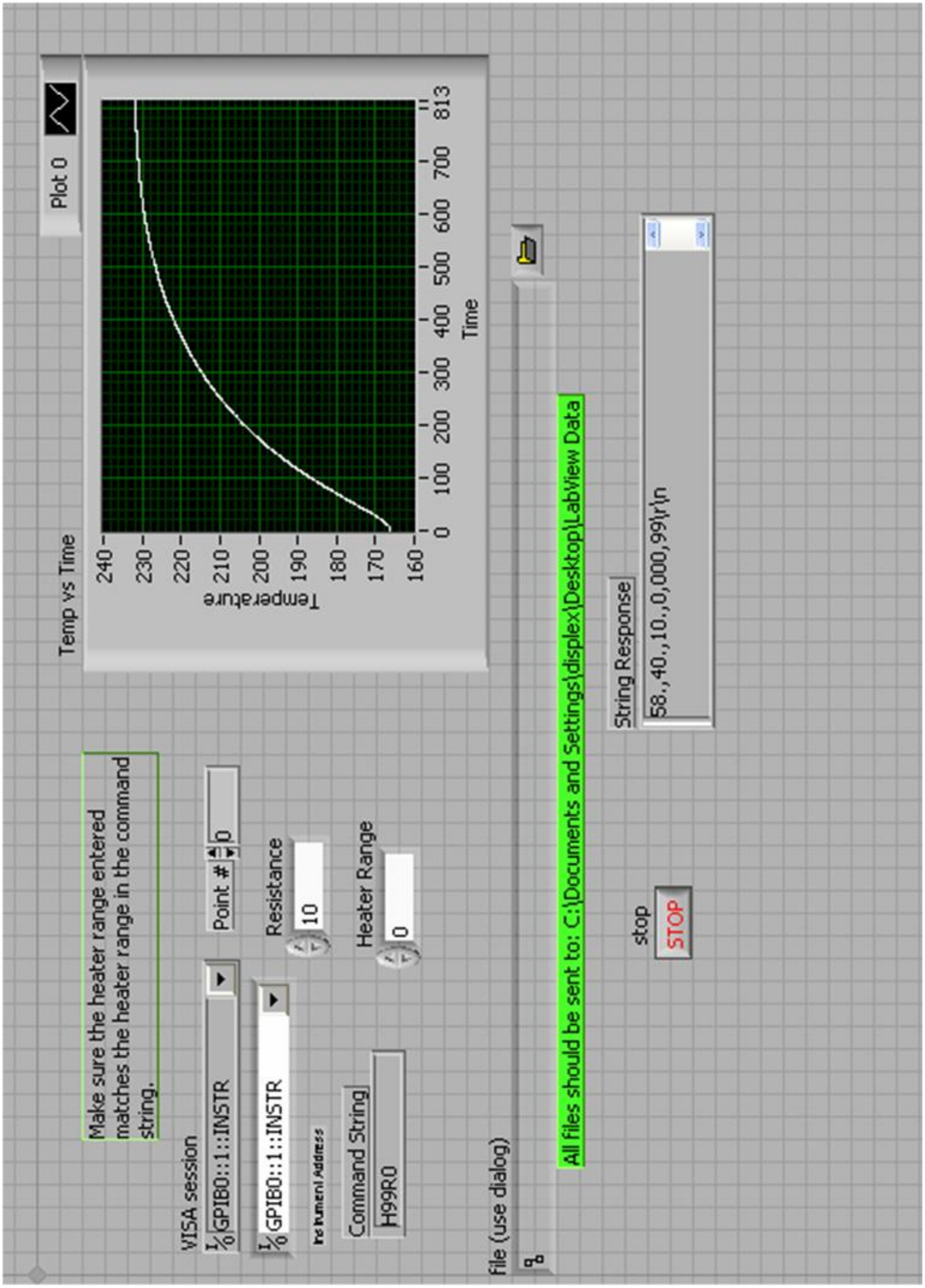


Image D

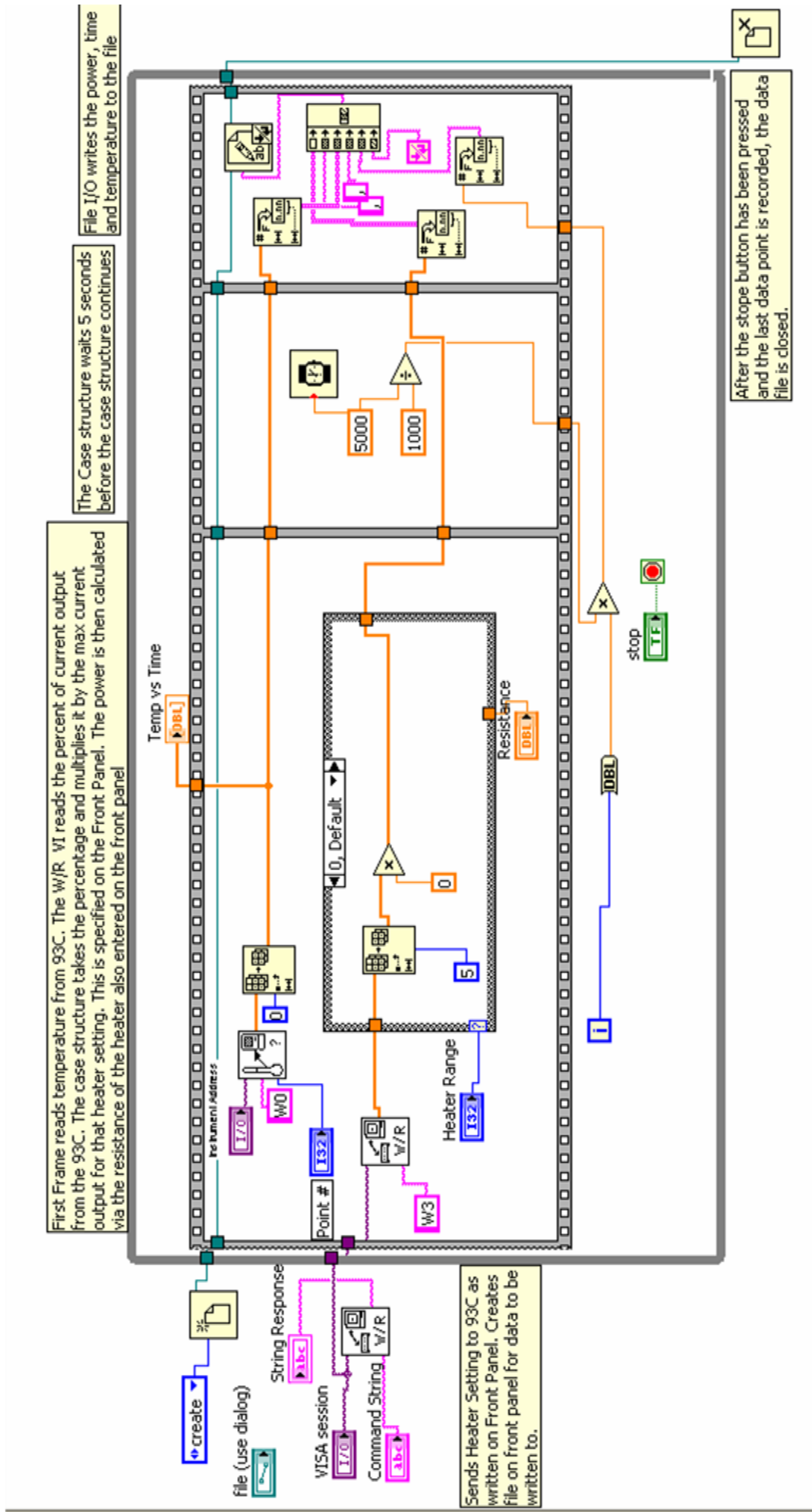


Appendix B

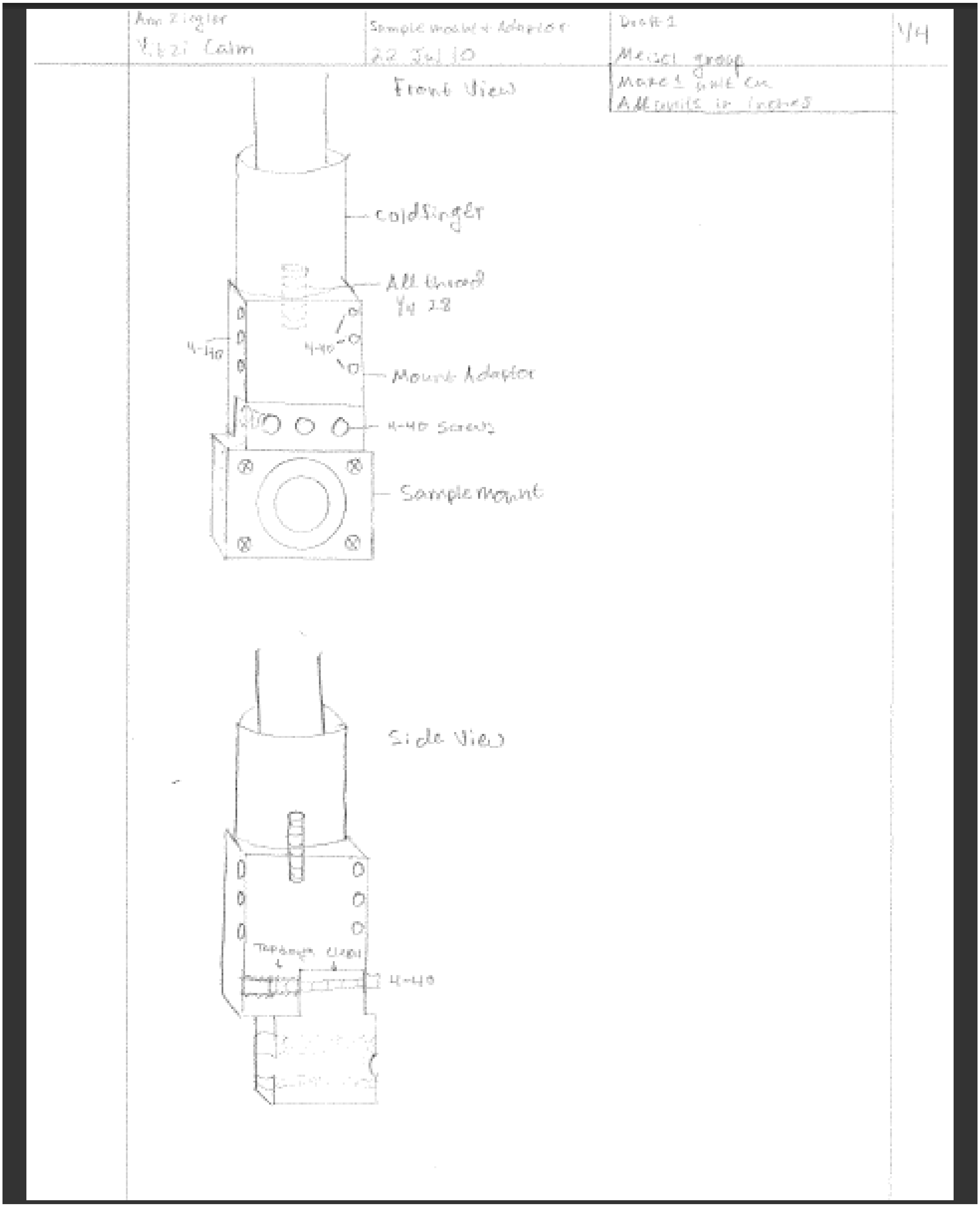


Temp vs Time
Plot 0
Temperature
240
230
220
210
200
190
180
170
160
0
100
200
300
400
500
600
700
813
Time
Make sure the heater range entered matches the heater range in the command string.
VISA session
GPIB0::1::INSTR
GPIB0::1::INSTR
Instrument Address
Point #
0
Resistance
10
Heater Range
0
Command String
H99R0
file (use dialog)
All files should be sent to: C:\Documents and Settings\displex\Desktop\LabView Data
String Response
58.,40.,10.,0,000,99\r\n
stop
STOP

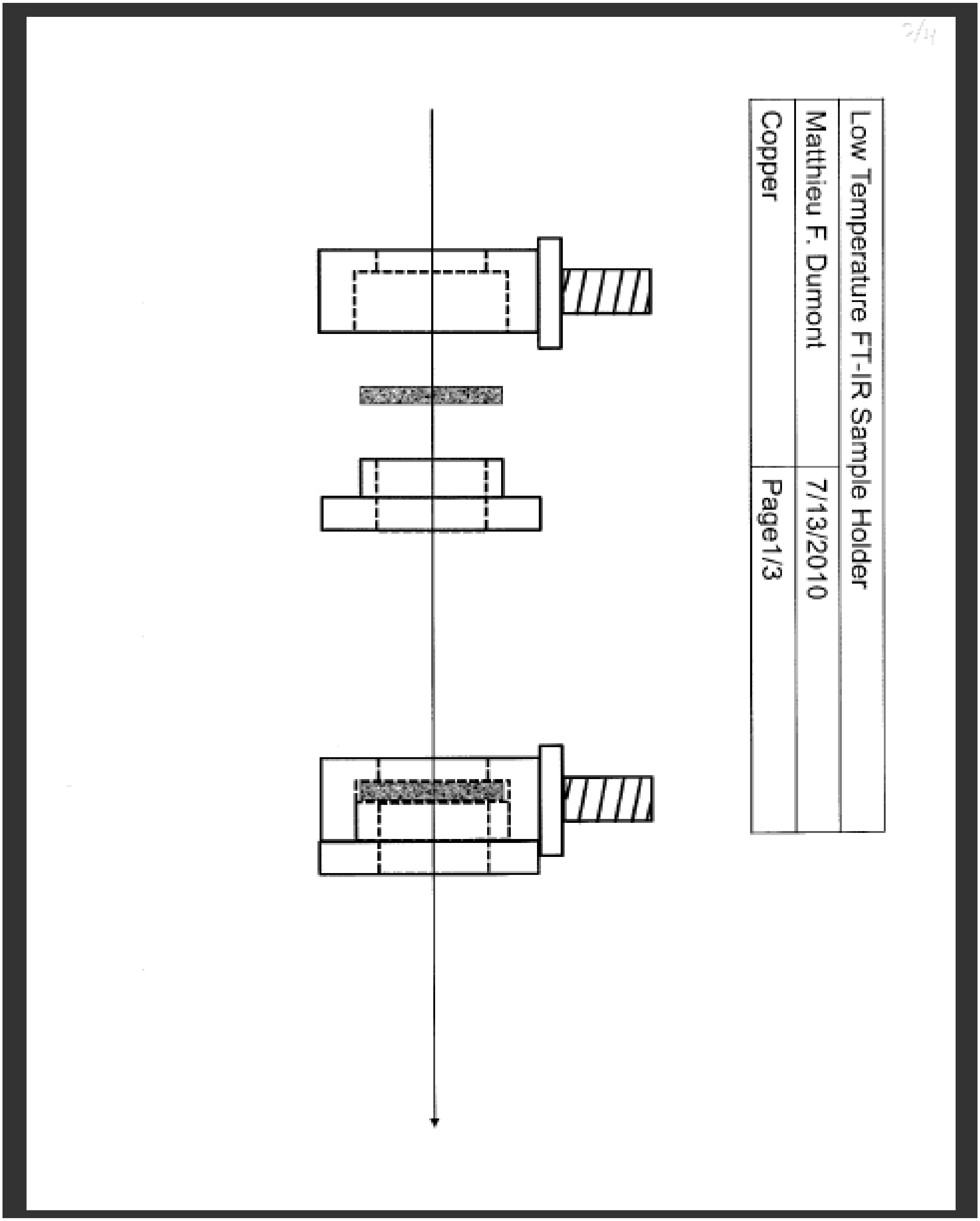
.

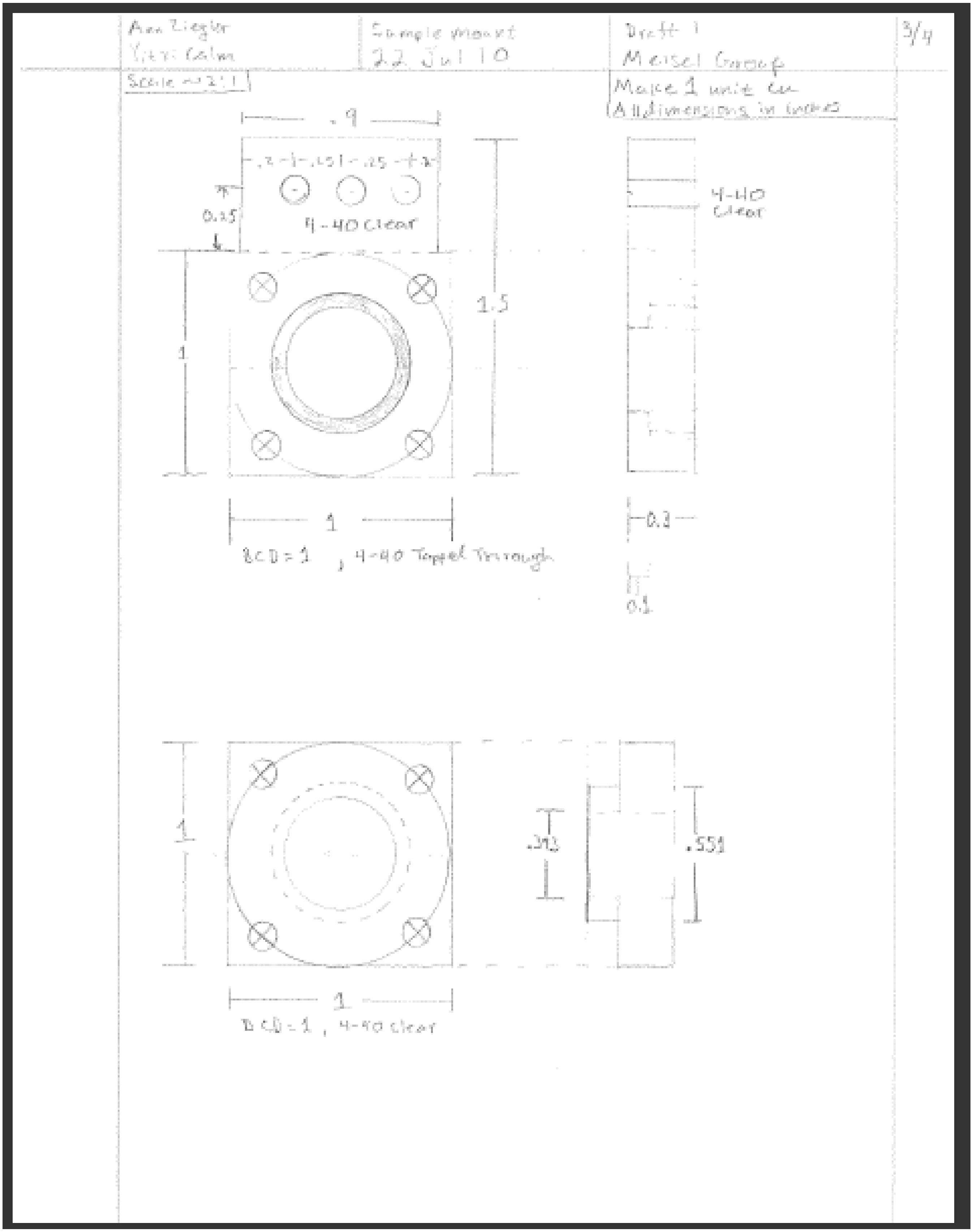

File I/O writes the power, time and temperature to the file
The Case structure waits 5 seconds before the case structure continues
First Frame reads temperature from 93C. The W/R VI reads the percent of current output from the 93C. The case structure takes the percentage and multiplies it by the max current output for that heater setting. This is specified on the Front Panel. The power is then calculated via the resistance of the heater also entered on the front panel
Temp vs Time
Resistance
5000
1000
0, Default
0
5
Heater Range
Point #
Instrument Address
W0
W3
stop
After the stope button has been pressed and the last data point is recorded, the data file is closed.
create
file (use dialog)
String Response
VISA session
Command String
Sends Heater Setting to 93C as written on Front Panel. Creates file on front panel for data to be written to.

Appendix C

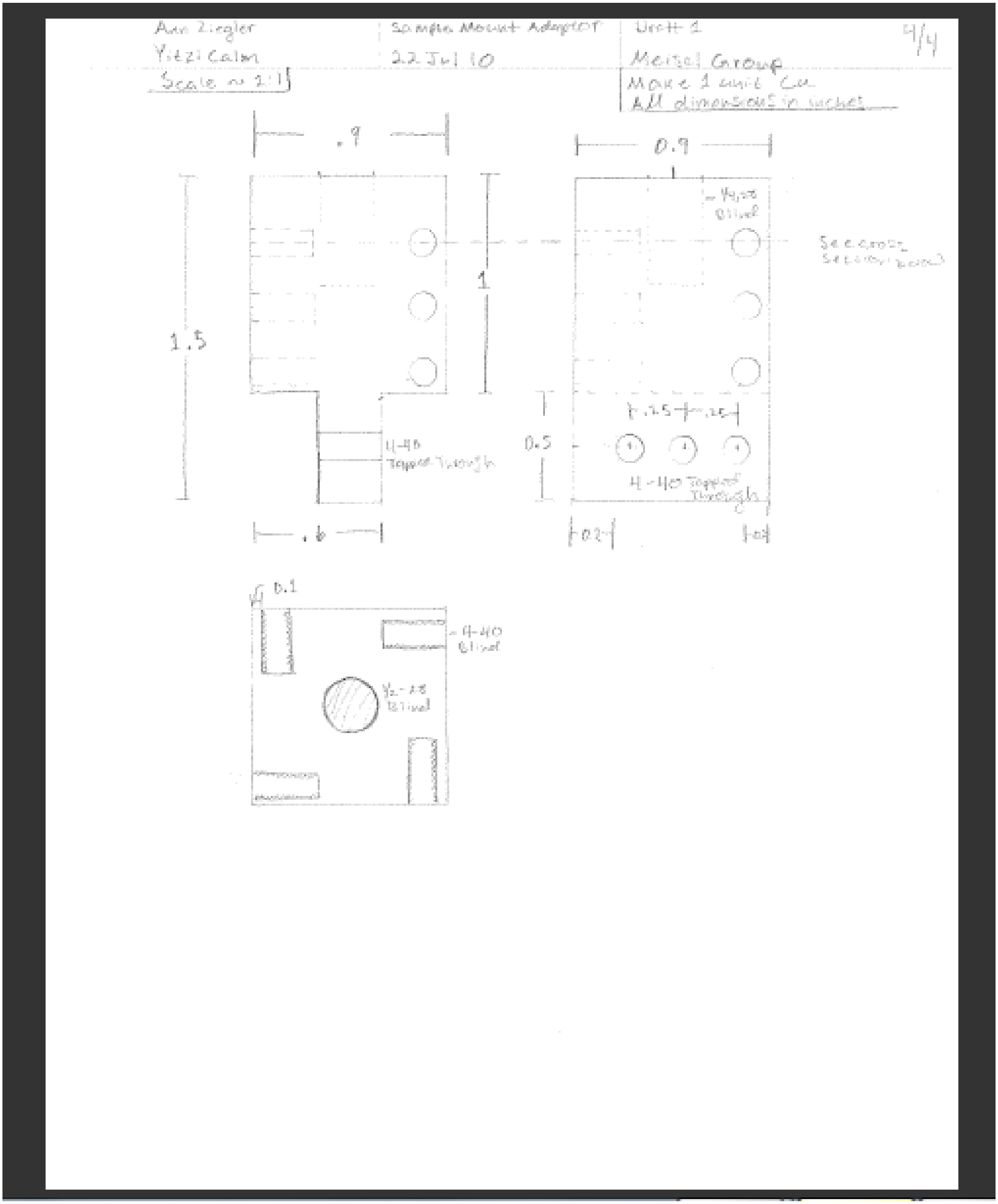

Sample mount + Adaptor
22 Jul 10
Draft 1
1/4
All units in inches
Front View
coldfinger
All thread
1/4 28
4-40
4-40
Mount Adaptor
4-40 Screws
Sample mount
Side View
4-40

.


Low Temperature FT-IR Sample Holder
Matthieu F. Dumont
7/13/2010
Copper
Page1/3


Scale ~2:1
Sample mount
22 Jul 10
Draft 1
Meisel Group
3/4
Make 1 unit
All dimensions in inches
.9
.2
.25
0.25
4-40 Clear
1.5
1
4-40
Clear
0.3
0.1
BCD = 1 , 4-40 Tapped Through
.551
BCD = 1 , 4-40 clear

.


Ann Ziegler
Yitzi Calm
Scale ~ 2:1
Sample Mount Adaptor
22 Jul 10
4/4
Make 1 unit Cu
All dimensions in inches
.9
0.9
1.5
1
4-40 Tapped Through
0.5
.25
4-40 Tapped Through
.6
0.1
4-40 Blind
1/2-28 Blind

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