Senior Design Group May 14-03

Final Report

Design and implementation of a cryogenic electrical characterization system for organic electronic devices

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1 Introduction/Motivation

1.1 Problem Statement:

Organic electronic devices are a quickly growing and active field of research, and a very promising set of technologies for energy, lighting, and other industries. One major obstacle, however, is the presence of defect-induced electronic trap states within the materials, which lower the efficiency and limit the usefulness of these devices. These defect states are still not fully understood, and our client, a research group focusing primarily on organic solar cells, would like to expand its experimental arsenal with another tool to examine these states, utilizing a method known as Thermally Stimulated Current (TSC) spectroscopy.

TSC and its variants have been around since the 1950s, but no dedicated commercial systems for conducting TSC measurements exist. Those systems built in research labs are often based on expensive cryostat systems, usually costing upwards of $30,000. Our job is to add an automated and easy-to-use TSC functionality to a small cryostat provided by our client.

TSC spectroscopy requires cryogenic temperatures--the lower the better. Our client was having difficulties getting the organic electronic samples down to the target temperature of 80 K using their cryostat, so one of our tasks is to design improvements to the current system to make 80 K consistently attainable, as well as adding a method to be directly measure the temperature on the surface of the sample.

The defect characterization measurements will be made using a nonisothermal method of monitoring thermally stimulated currents created by charge carrier detrapping and thermal escape mechanisms as the sample is heated from cryogenic temperatures. The end goal of this project is an efficient and accurate method of studying electronic defects. In addition, the test setup will be fully automated and equipped with an easy-to-use computer interface programmed in LabVIEW.

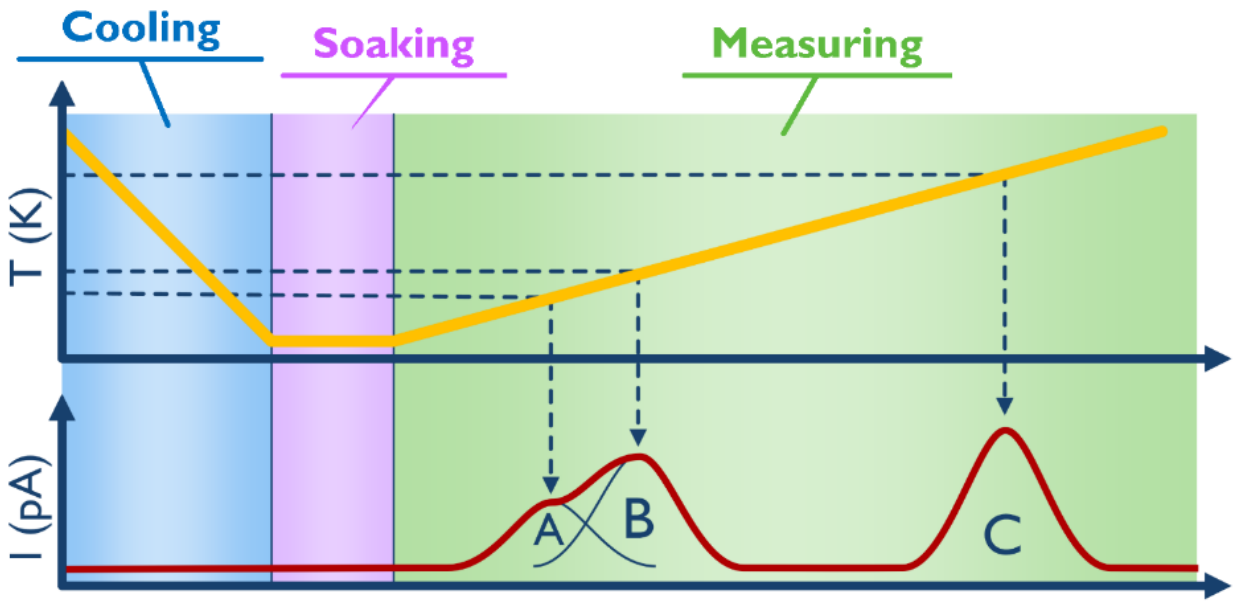


Figure 1: Schematic of a simple TSC experiment. The red line shows three different peaks, due to defects at three different energies. While conceptually simple, the implementation of a successful TSC system is notoriously tricky.

2 Background and Functional Decomposition

This design project was not intended to create an entire new system from scratch. Instead, the group was tasked with solving a technological problem by improving and extending existing equipment. The basic functionality of the required system was already in place, but did not satisfy the client's needs. The following sections describe the required functions of the system. All of the components, including the cryostat, LN_2 and atmospheric vacuum pumps, variable transformer, and temperature controller, were already owned by the client, and this setup is the starting point of this design project.

2.1.1 Samples under test

The research lab operated by our client has a fairly standardized method of fabricating organic solar cell samples for testing. The samples are built on square slides of glass which measure 1 inch on each side and are 0.7 mm thick. The glass slides are purchased pre-deposited with a layer of indium tin oxide (ITO), a commonly used, conductive, optically clear oxide for the creation of the bottom electrode. Then, between 100 and 300 nm of the active organic layer, bulk heterojunction poly-3-hexylthiopene (P3HT) is applied to the surface in an environmentally-controlled glove box, leaving a thin strip of bare ITO along one edge to allow electrical contact to the bottom electrode. The top electrodes are composed of 100-150 nm aluminum films prepared by sputtering. As both the P3HT and aluminum layers are very thin, the cells are very fragile and easy to puncture from above. Scratching through the surface of aluminum contact effectively destroys the devices themselves. Also the glass is fragile and will crack under too much applied pressure, even on the ITO tab. The fragility of these samples requires a very careful probe design to avoid scrapping a large number of samples during the testing procedures.

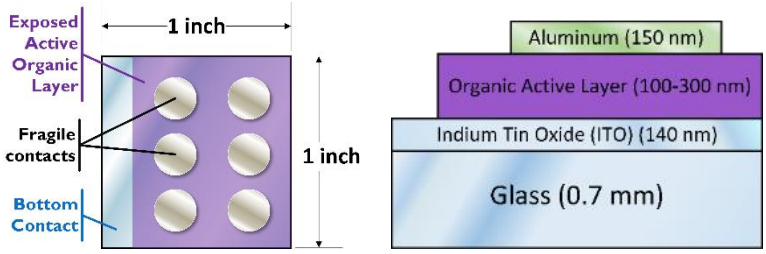


Figure 2: Schematic top and side views of the standard organic photovoltaic cells produced by our client, to be tested in our TSC system

2.1.2 Sample temperature control

In order to accurately control the heating and cooling cycles for the sample, a simple vacuum cryostat will be used with a Dewar flask filled with liquid nitrogen (LN_2). The top surface of the cryostat is a flat circular copper stage, on which the sample will be placed. A simple vacuum pump powered by a variable transformer will pull nitrogen out of the flask and up into a copper head, cooling the sample down. In order to achieve stable and repeatable temperatures, a heating coil is also included in the cryostat assembly. The temperature is monitored internally and the heater is supplied with variable power by a Model 331 Cryogenic Temperature Controller from Lakeshore Cryotronics. This system, while basically functional, had the limitation that the sample could not be brought down to the required temperature of 80 K—instead, it was limited to roughly 150 K.

2.1.3 Sample temperature monitoring

In the initial implementation of the system, there was no way to accurately determine the temperature of the photovoltaic sample itself. In order to be successful, the system must provide accurate, reliable measurement of the sample temperature in real time.

2.1.4 Electrical contact

In order to measure the thermally stimulated currents in the photovoltaic cells, reliable electrical contact must be made inside the vacuum chamber, but the contact must be soft enough to avoid damaging the cells. The contact probe must also have a low thermal mass in order to avoid altering the temperature of the cell at the contact point. The old system had a large, spring-loaded device which, while capable of making very soft contact, was proven to have far too large of a thermal mass, and it would raise the sample temperature considerably.

2.1.5 Thermal Isolation

In order to minimize convective heating of the sample, the entire cryostat head and assembly is encased in a vacuum chamber. The chamber itself, however, provides only minimal thermal insulation from the ambient temperature in the laboratory, so some amount of insulation will be required.

2.1.6 Current measurement

The system must be able to reliably and accurately measure very low currents in the sample. This functionality requires not only a high-end ammeter or source measurement unit, but also a significant amount of effort in shielding and noise management.

2.1.7 Automation Software

A software-based control system was the only system which had to be designed from scratch. The software must be able to collect and export data, as well as to control the heating and cooling cycles of the sample.

2.1.8 Operating environment

Our system is designed to be operated inside a laboratory room under standard ambient temperature and pressure conditions, and will be operated by researchers in the organic electronics field. We will therefore assume that the users will be capable of handling the fragile samples with care.

2.1.9 User interface

2.1.9.1 Hardware

As far as hardware is concerned, the user will load the sample onto the cryostat by actuating some small pins to make contact with the active aluminum area and to the ITO strip along one side, and place a sensor on the surface of the system. The user will then screw the chamber shut and make sure all the connections are properly made to the equipment. The software interface (described below) will prompt the user to turn on the vacuum pumps when the software is ready to collect data. At the end of the process (prompted again by software) the user will screw open the vacuum chamber and remove the sample.

2.1.9.2 Software

The software will have a graphical user interface which prompts for experiment parameters, shows progress information, and presents real-time temperature and current measurements. Upon starting the control program, it will automatically connect to the required devices and prompt the user to make any necessary connections or alterations to the hardware setup, as well as when to turn the cryogenic vacuum pump on. The software must also advance through the different phases of the experiment without user input, but must notify the user of these conditions at all times.

2.2 Project Deliverables

2.2.1 First Semester

- Design and fabricate improvements to a cryogenic vacuum chamber system to counteract poor thermal conductivity and radiative heating effects, with the goal of consistently cooling a sample to 80 K using liquid nitrogen. This deliverable can be subdivided into several smaller steps:
    - Evaluate the feasibility of, and potentially implement, a no-grease solution for low-thermal-impedance interface between the sample and cryostat
    - Design and build a layered enclosure within the vacuum chamber to minimize radiative coupling to the chamber
    - Determine the feasibility of encapsulating solar cells in a method which allows reliable and repeatable electrical and optical measurements of the cell, is optically clear, electrically insulating, and thermally conductive
- Determine a method of heating the sample at controllable of rates of 5-10 K per minute from 80 K to 300 K.
- Design a probe contact assembly for making robust contact without damaging the fragile contacts
- Determine a method of accurately measuring low currents (resolution in the fA range) with low noise and at least 1 sample per second
- Produce a well-labeled arrangement of cables and hookups which make the system easy to connect and disconnect as other experiments require
- Run at least one full test of the assembled system

2.2.2 Second Semester

- Assess performance from first semester and make any required adjustments
- Develop control software in LabVIEW to automate the process of recording data
- Write a clear and cohesive system description and troubleshooting guide for future users of the system

3 Specifications

The new systems designed by this project as well as the improvements to the existing systems will all be expected to meet the following criteria.

3.1 Vacuum Chamber Cryostat

- Chamber must be able to reduce the sample temperature to 80 K
- Must include a temperature sensor for real-time monitoring of the sample temperature
- Any improvements or additions to the efficiency of the system must fit inside the existing enclosure
- The cold shroud enclosure must be easily and quickly removable by one person using one tool or less
- A shroud or other improvement in insulation must not interfere with electrical contact to the sample
- Requires a vacuum electrical feedthrough to enable the connection of enough wires to fulfill all measurement needs
- The final design must be flexible enough and easily enough to reconfigure to allow other users to continue other experiments using the same cryostat
- The system must remain up and running at all times during the design phase to allow other active research groups to continue their experiments.

3.2 Cell Encapsulation/Preparation

(Specialized cell preparation are not a design requirement, but if any cell encapsulation or special preparation is to be used, it must agree with the following specifications.)

3.2.1 Encapsulation chemicals

- Encapsulant must be optically clear with no visible clouding or discoloration
- Encapsulant must be electrically insulative with bulk resistivity in excess of 1 $T\Omega\ cm^{-1}$
- Encapsulant must be chemically and mechanically stable down to at least 80 K
- Encapsulant must not react with or catalyze a reaction of the active organic layer.

3.2.2 Encapsulation method

- Must significantly improve the repeatability of measurements and the endurance of cell contacts
- Must make contact to all six individual cells on each die
- Must be conducive to a contact failure rate of 33% or below (average of at least four working cells per die) after minimal training of the practitioner

3.2.3 Thermal interface material

- Must not leave a greasy residue which interferes with other types of downstream tests.
- Must have a thermal conductivity of 1 $Wm^{-1}K^{-1}$ or higher
- Must be conformal (Shore 00 Hardness of 70 or lower) to ensure good thermal contact

3.3 Measurement Setup/Control Software

- Must enable all measurements to be made through GPIB interface
- Must encompass full automation of cooling, excitation, heating, and data collection
- Must be able to detect and suggest solutions to flaws in hardware setup or set-points
- Must elegantly and safely recover from software or hardware errors
- Measurement of thermally stimulated current must have resolution measured in fA
- Must record logs of all data in an organized and readable file format
- Log files must include date, time, setpoints used, information about the samples, and other important details deemed necessary by the client

3.4 Code Functional requirements

In order to be considered functional, the system must:

- Be able to reach the target temperature of 80 K
- Smoothly and accurately change the temperature at rates of 1 K per 5-10 minutes

- Record all data in an organized and labeled, and accessible format

3.5 Experiment Design

- Hardware and software should support a customizable temperature profile for modified TSC methods such as thermal cleaning, fractional emptying, and reversibility cycles.

4 Final System Design

4.1 System block diagram

The system as a whole will be centered on a cryostat head placed in a vacuum chamber. The solar cell sample will be placed in dry, unmediated contact on top of the cryostat. The cryostat is cooled by a variable pump, which pulls liquid nitrogen from a Dewar flask through a tube in the cryostat. The temperature is monitored by a Lakeshore temperature controller, which also uses a heating coil to stabilize the system to the required temperature. Four-wire Kelvin point contacts will be made to the cell for electrical measurements, and a platinum RTD will be placed on the surface to measure the true temperature of the sample. Also shown here above the cryostat is the custom-designed shroud which will encircle the sample and provide most of the insulation. This layered structure of reflective foil and thermally insulating material will be essential in bringing the sample down to 80 K. All of the measurement and control instruments, except for the vacuum pumps, will be controlled by computer software programmed in LabVIEW, and communicated via GPIB.

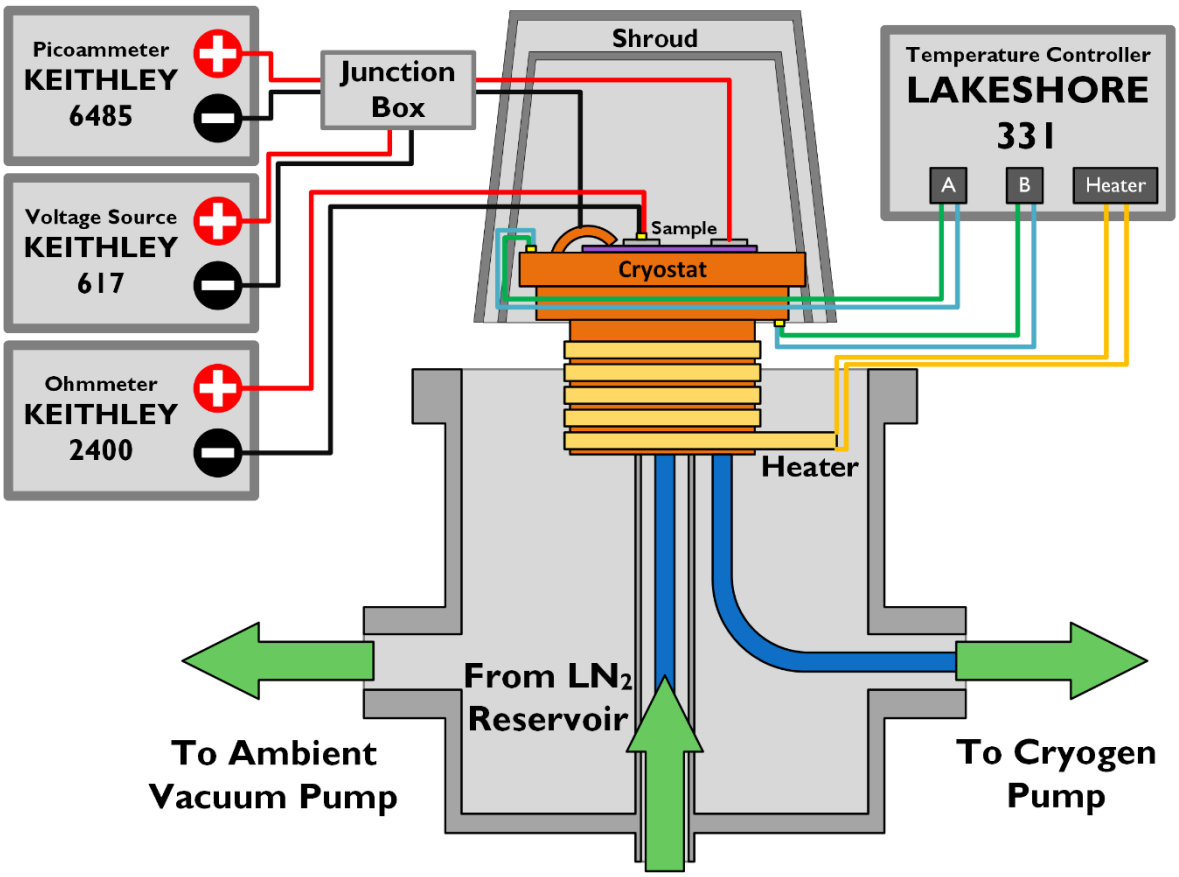


Figure 3: Schematic block diagram of all the hardware in our final system

4.2 Thermal Design

4.2.1 Temperature Control Unit

They cryostat consists of a circular copper stage about 2 inches in diameter, on which the device under test can be placed. An externally-powered vacuum pump pulls liquid nitrogen out of a Dewar flask through the copper stage. Thus cooling the sample by conduction. The Lakeshore 331 Temperature Controller monitors the temperature of the cryostat and controls a heating coil embedded in the copper assembly. The coil is used to maintain the temperature at a desired setpoint. The heating coil will also allow the user to raise the temperature of the sample at a desired rate. In order to reduce thermal loss through convection, the cryogenic chamber will be kept under vacuum of roughly 10^{-3} Torr.

Figure 4: Photograph of the cryostat, with a sample loaded and ready to be tested

4.2.2 Thermal interface materials

In our final design, we are using two different thermal interface materials. For semi-permanent mounting of thermal sinks, we chose to use VGE-7031 cryogenic varnish, produced by LakeShore Cryotronics. The varnish is a viscous, translucent, dark brown liquid, which can be applied to the required area with a brush and sets in 15 minutes at 400 K. If the material needs to be removed, it can be dissolved easily in ethyl alcohol. The varnish is used not only to create good thermal contact between wires and the cryostat, thus efficiently cooling the wires, but also to mechanically stabilize the whole setup.

For non-permanent thermal interfaces, we chose to use Apiezon N cryogenic vacuum grease, which is a yellow gel. This grease is used to ensure good thermal contact between the sample and the temperature sensor (described in the next section) without needing to cure or bond to the surface. Our tests have shown that this grease undergoes a solidification phase transition (described in detail in Appendix C) which causes the thermal conductivity to behave slightly nonlinearly with time, but the grease maintains most of its thermal conductivity even after the phase transition, and is therefore still useful in small quantities.

4.2.3 Temperature Sensing

The final solution we decided to use to measure the sample temperature directly was a platinum resistive temperature detector (RTD). These devices are simply a carefully specified sample of platinum, which shows a very linear temperature response over a high range. Platinum RTDs have recently been replacing thermocouples in all but the highest temperature applications. Our specific device is a PPG102A6 from US Sensor, which has an approximate linear temperature coefficient of resistance (TCR) of 3850 ppm over a temperature range of -200°C to 600°C. This device is specified to exhibit 1 kΩ (R_0) of resistance at 0°C (T_0) and the temperature can be calculated using the following formula:

$$T = 31.483 + 0.22R + 2.83 \times 10^{-5} R^2 - 6.62 \times 10^{-9} R^3$$

In order to protect the thin, fragile leads on these sensors both from shorting to each other and from fatiguing and eventually failing due to repeated contact cycles, the lead-wires themselves were encapsulated in a thin layer of cryogenic varnish for stability.

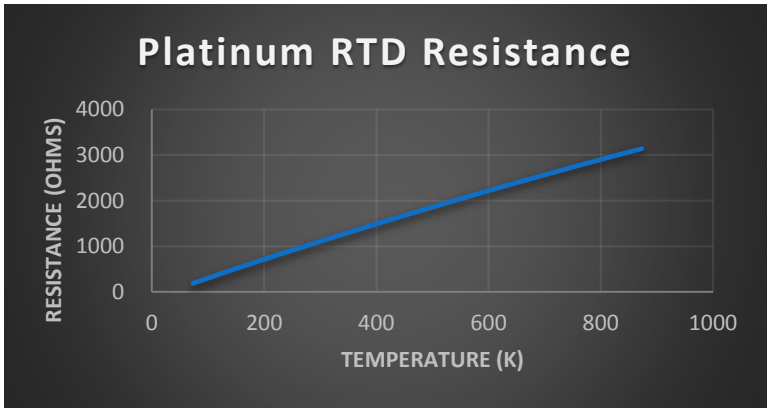


Figure 6: R-T response of the PPG102A6 RTD

Figure 5: Magnified image of the RTD sensor. The white rectangle is roughly 1 mm on a side.

4.3 Reflective Shroud

The shroud is a simple idea of adding a layer of insulation around the sample itself to cancel out any adverse radiative coupling between the external walls of the vacuum chamber and the sample itself. Our final design for the shroud consists of three parts: a Styrofoam structure; a thick, thermally conductive layer of aluminum foil on the inside; and several layers of thin, reflective aluminized Mylar.

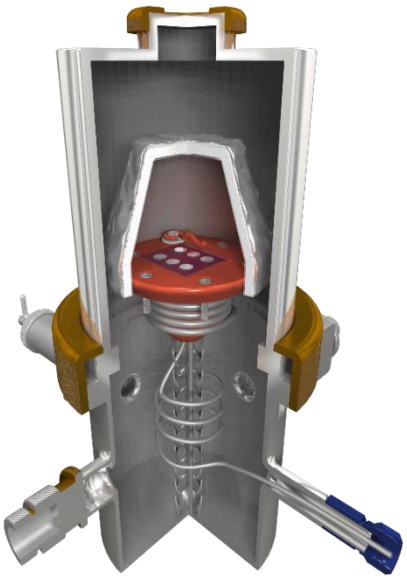


Figure 8: Cross section of the chamber, showing the placement of the shroud during an experiment

Figure 7: Exploded view of the layers involved in the shroud

The Mylar layers were repurposed from a shiny emergency blanket, such as is common for first aid, but the same material often finds uses on artificial satellites and other space-based technologies. Such Mylar films are known to reflect roughly 99% of all incident radiation, making them ideal for this purpose. In order to create maximum spacing between layers, and therefore expand even further the insulating properties of the shroud, each layer was individually crinkled, such that the layers will not lie flat against each other and thus conduct heat.

The Styrofoam was chosen initially because it was readily available, but we eventually realized that it was in fact the ideal material. Not only is it a good thermal insulator, but it has been shown to withstand the low temperatures and pressures without deforming or cracking, is flexible and thus easy to apply and remove from the cryostat, and also allows for a simple method of attaching the other layers.

The inner layer is made of heavy-duty aluminum foil, which is forced by the flexibility of the Styrofoam and the sponginess of the crinkled Mylar into good thermal contact with the cryostat. If any heat leaks in through the Mylar and Styrofoam layers, this aluminum foil layer acts as a thermal sink and conducts it down into the cryostat instead of allowing it to creep into the sample.

All of the layers are sewn together with magnet wire around the base of the shroud cone. This method was chosen to avoid adhesive materials, which traditionally delaminate at low temperatures, and to maintain the desirable flexibility of the Styrofoam base.

4.4 Other insulation

While the shroud itself does most of the work in insulating the system, we found that packing the rest of the empty space within the chamber with individual Mylar sheets improved overall performance. Insulating the top half of the material simply acted to augment the shroud and lowered the minimum achievable temperature by a few degrees. With all of this insulation in place, we managed to reach 83 K, which is only a few degrees above the boiling point of nitrogen (77 K or -321°F).

The insulation in the bottom half did not have a noticeable effect on the minimum temperature of the system, but it did significantly improve the efficiency of the heater, and therefore the ramp rate control during the measurement phase, which is an important aspect of the design.

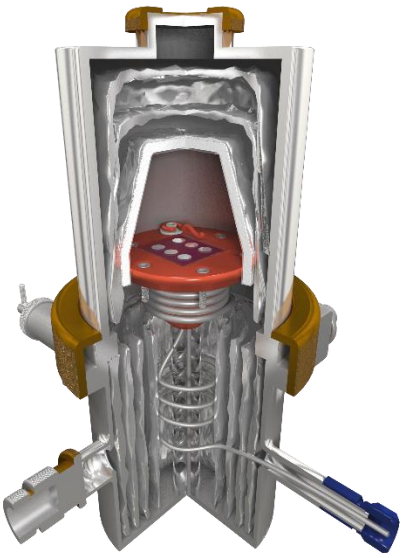


Figure 9: Cross secton showing the placement of extra Mylar insulation during an experiment

4.5 Electrical Design

4.5.1 Electrical Contact Interface

One of the main challenges of our project has been to reduce thermal coupling between the sample and the outside, while maintaining good conduction with the cryostat. This requirement motivated us to adopt a probe/clamping system as electrical contacts. However since the solar samples are very thin, we need to design the clamp and probe such that it applies enough force on the sample to enhance thermal conduction with the copper head, without creating enough stress to the device. In fact our vacuum insulation pump transmits vibrations to the sample during test runs and we had to incorporate those considerations into our design of the electrical contact interface. At first we have chosen a relatively bulky probe with a smooth pin connected to the aluminum contacts of the sample. This system provides the advantage of providing enough contact force to the sample while minimizing damage. However as we ran simulations we noticed that the thermal mass of the system was reducing the thermal efficiency of the system. The lowest thermal mass we could create was to use a thin wire as a kind of clamp, where the tension in the wire will pull the sample down onto the surface. After so many other attempts, the wire clamp method was determined to be the best option.

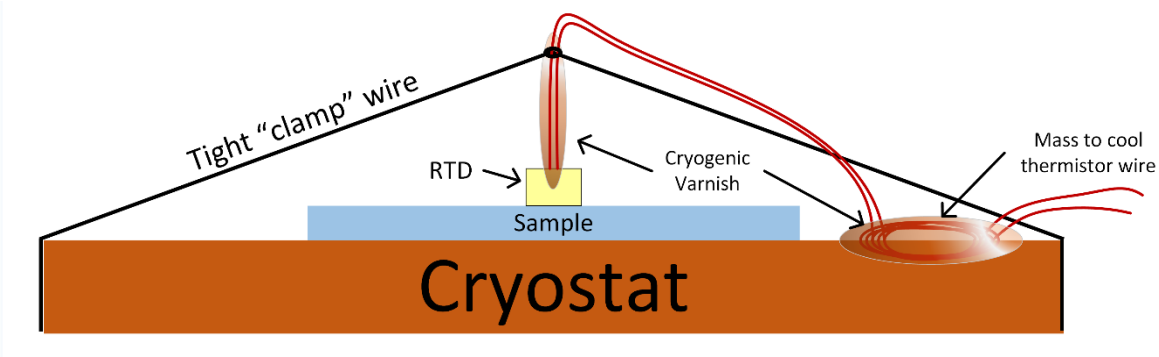


Figure 10: Schematic of the "tie-down" method of creating and maintaining contact, as well as the use of varnish to cool the sensor wire

4.5.2 Current Measurement Instrumentation

Our client provided us with a Keithley 2400 Source Measurement Unit (SMU) for current measurement purposes. However, this device had a maximum current resolution of 10 pA, which we estimated to be too rough to measure TSC signals. After much deliberation (see Appendix C for details) we decided to go with a Keithley 6485 Picoammeter. Unlike the SMU, this meter did not have the capability to source a voltage, so a secondary device was needed. Our client had a Keithley 617 electrometer on hand, and while the current measurement circuits on this device had been damaged long ago, the voltage source capabilities still worked.

4.5.3 Electromagnetic Interference (EMI) Shielding and the Junction Box

Given the chosen instrumentation, the path of the extremely low TSC currents must lead from the sample, to the voltage source, and to the Picoammeter. This, coupled with the requirement that our connections must not alter the connections directly to the chamber and that the voltage source did not have BNC jacks, meant that simple coaxial or triaxial cabling would not suffice to route all signals. In order to get around this issue, a shielded junction box would be required.

Figure 11: The ABS plastic box after being machined

The box consisted of a small project box made of ABS plastic, a material chosen because we had access to the tools to machine it. Three non-circular holes were drilled in the box to accommodate two regular BNC jacks and one isolated BNC jack, allowing us to route the center signal conductors to the center and shield of the isolated BNC connector as required for the Picoammeter. The box was wrapped entirely in several layers conductive aluminum shielding tape, taking special care to eliminate any gaps in the faraday cage.

Figure 12: The completed shielded junction box

The chamber had four coaxial connectors to accommodate to Kelvin point connections to the sample, but our system only required two, so we used 2-1 BNC adapters to recombine these separate wires outside the chamber. The rear panel of the voltage source only had binding posts for banana connectors, so BNC-to-banana adapters were employed. All of the shielding in the system is grounded at a single point to the "Common" port on the back of the Picoammeter, including the casing of the vacuum chamber itself, which is connected via an alligator clip.

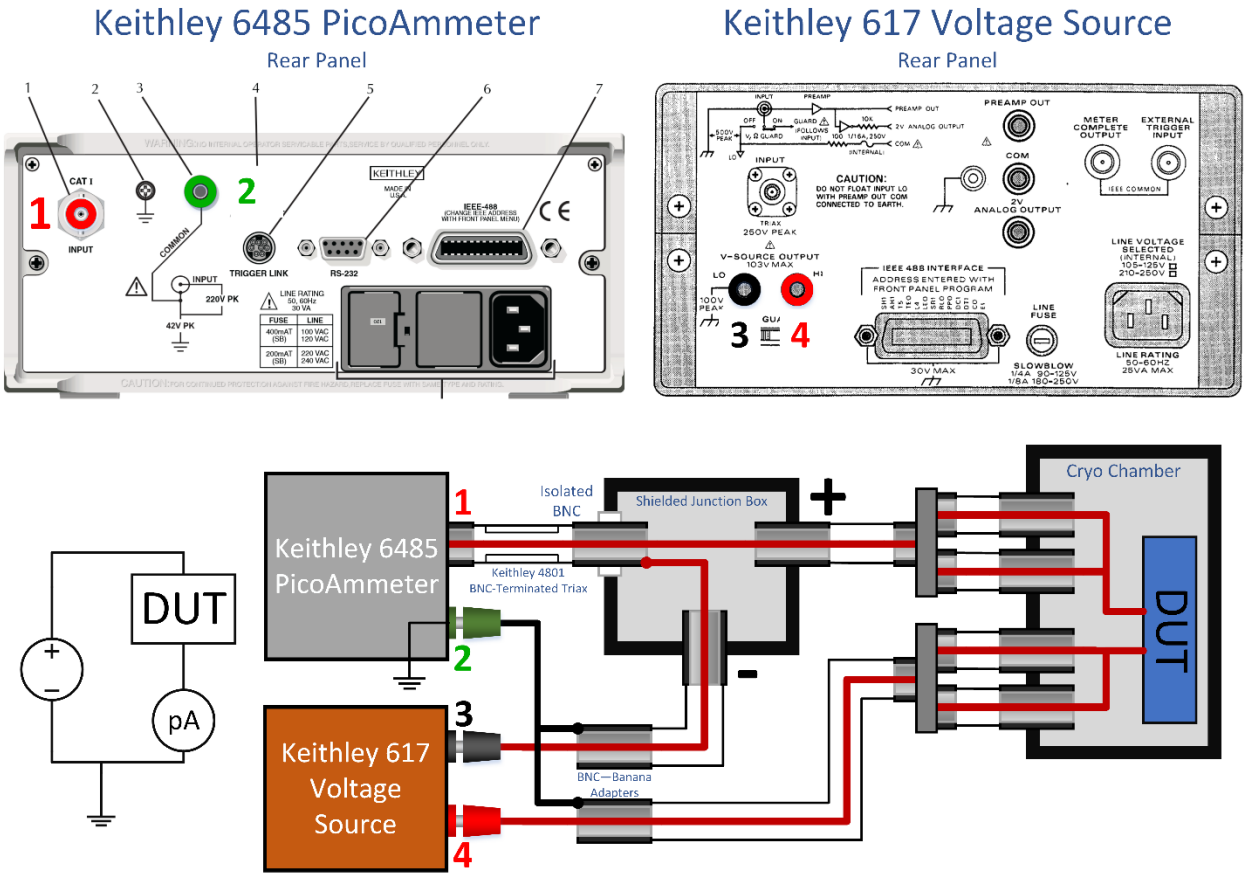


Figure 13: Connection diagram for the current sense circuit

4.6 Software Design

Our system design includes a self-contained, intuitive software platform (the "TSC Dashboard") which eases experiment design and automates experiment execution. The code was written in National Instruments LabVIEW (Student Edition 2012) and boasts an impressive list of features.

The software allows for complete customizability regarding the temperature and charging profile used in the experiment, but also allows the user to save a set of experiment parameters into profiles for easy reuse and repetition. Upon starting an experiment, the software walks the user through the sequence of turning on all of the vacuum pumps, and then the software takes over, communicating as needed with the measurement instruments. The system collects one suite of datapoints (from all four devices) in roughly one tenth of a second, allowing for great time resolution. At the conclusion of an experiment, all of the collected data is exported into an easy to read and intuitive log file, where it can be easily retrieved an analyzed in any spreadsheet manipulation program.

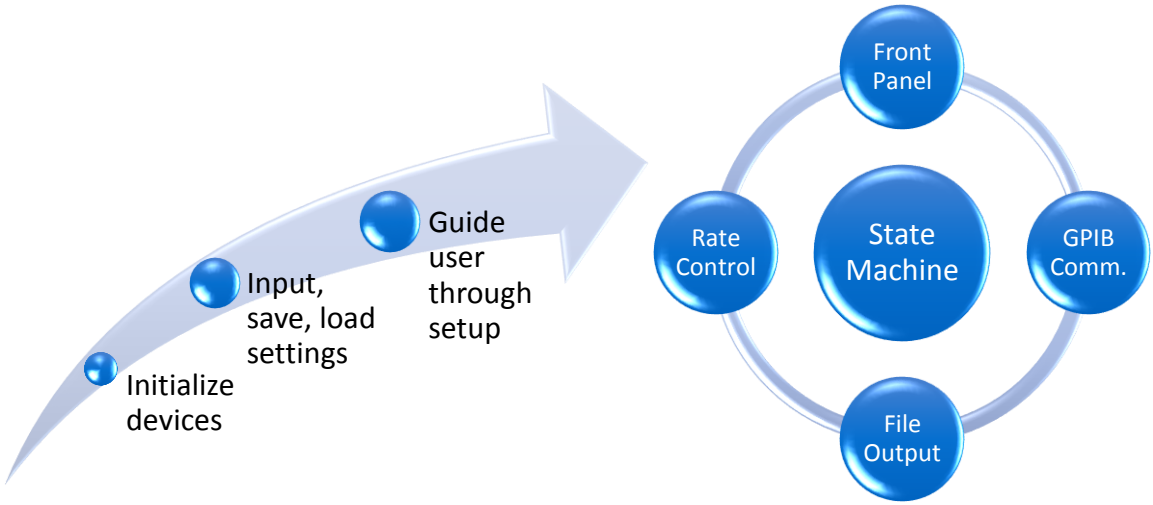


Figure 14: Flowchart describing software functionality

For more specific details on the inner workings of the TSC Dashboard program, refer to Appendix B.

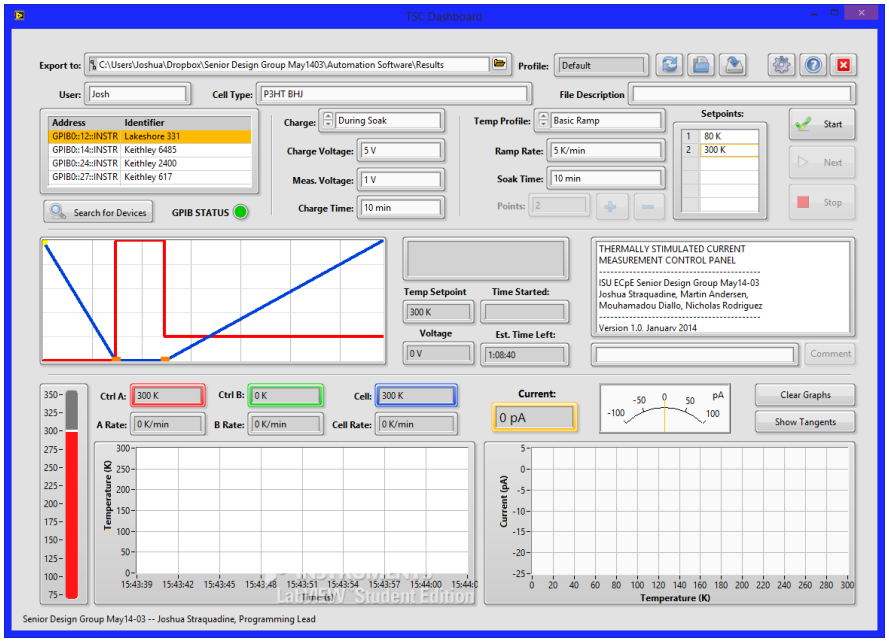


Figure 15: Dashboard screen seen on startup

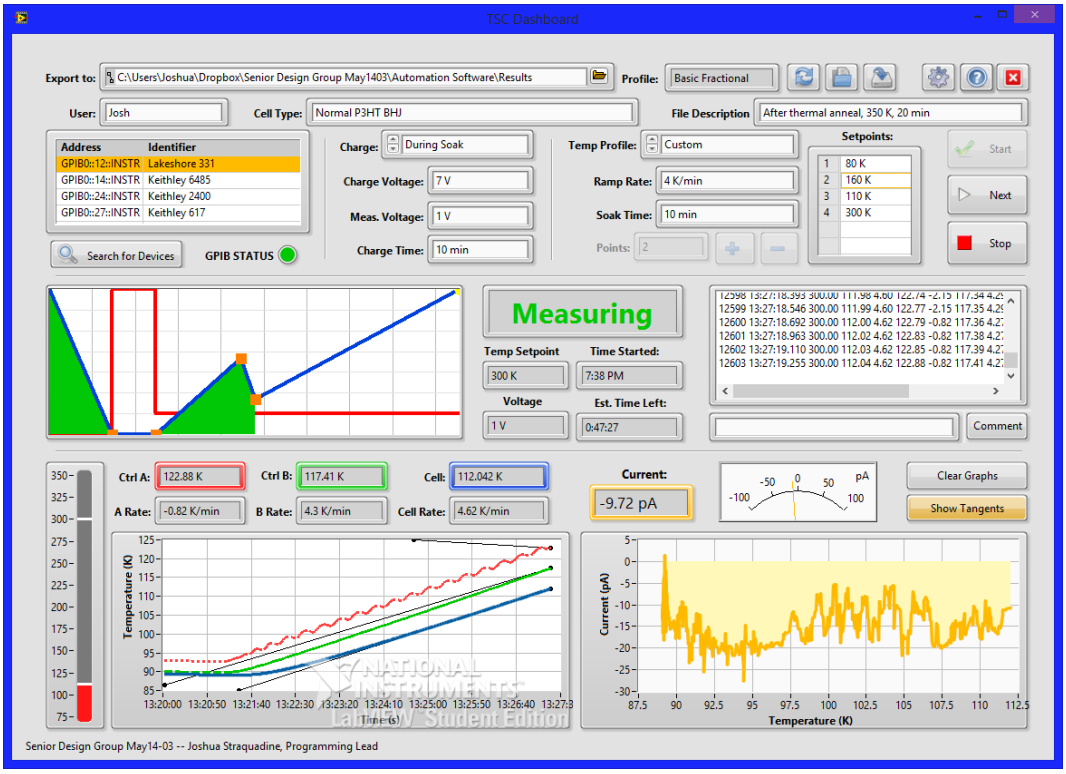


Figure 16: Dashboard screen seen during an experiment

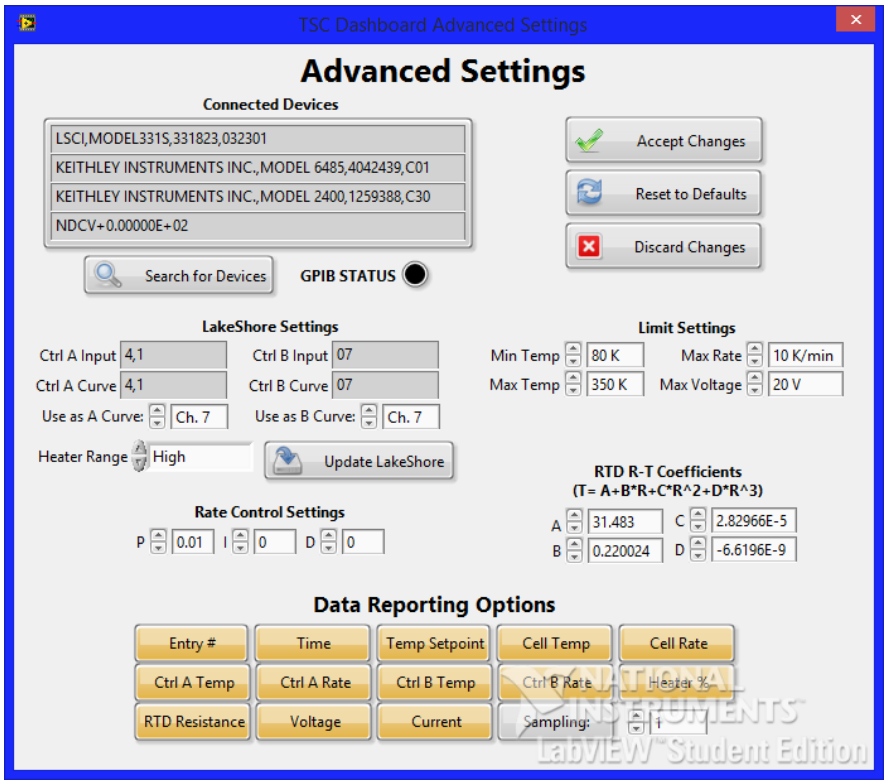


Figure 17: Advanced settings pane

4.7 Relevant Standards

In our project, we are working with a highly customized vacuum cryostat for low temperature exploration of defect states in custom-build organic solar cells. With that in mind, most of our project will be built for the first time ever, meaning that there are hardly any standards regarding our project. The only standard which truly affects the way we will design our project is the interface we will be using to achieve computer control of our equipment.

The IEEE-488 General Purpose Interface Bus (GPIB) is a short range digital 8-bit parallel communications interface protocol for communicating between a computer-based control system and up to 15 other laboratory apparatuses. All reputable modern laboratory power, measurement, or signal generation hardware has a GPIB port, so it is generally fairly simple to control several devices simultaneously or to integrate new devices into an existing system. GPIB hardware is recognizable by the unique stacking connectors, which make rewiring a lab bench simple. All of our test and measurement hardware with the exception of our vacuum pumps (which don't have processors, just "ON" switches) will eventually be controlled using this interface.

GPIB was developed by Hewlett-Packard (under the name HPIB) in the late 1960s for exactly this purpose. The standard interface became a huge selling point for HP, because all of their equipment was easy to integrate with other HP devices. In 1975, IEEE adopted this protocol as a standard across the industry, and many companies were quick to join in. Since then, the standard has undergone a few revisions regarding specific commands, signal handshaking, and other things to speed up and ensure data transmission, but the core of the standard has remained largely the same.

Thanks to the GPIB standard, we will not need to learn all of the small details of this standard to complete our task. We will be using National Instrument's LabVIEW software, which, given NI's impressive line of test and measurement equipment, already supports GPIB interfaces quite well through built-in libraries. We're fortunate that this standard exists, however, because we'll be trying to integrate equipment produced by several different companies. If no industry standard protocol such as existed, we would have to write much more code to speak to all of those devices in their own languages.

5 Implementation Details

5.1 Resource requirements

The majority of the most expensive components involved in our system, including the cryostat, SMU, vacuum grease, and assorted cabling was found by looking and asking around the Microelectronics Research Center (MRC), which has an extensive inventory of scientific equipment similar to our needs. This helped us to keep our project-specific costs down. The materials we used and their prices are all listed below.

Resource	How will we get it?	Cost
Cryostat	Provided by client	---- N/A ----
Dewar flask	Provided by client	---- N/A ----
Liquid nitrogen	Provided by client	---- N/A ----
Aluminum Foil	Provided by client	---- N/A ----
Source measurement unit	Provided by client	---- N/A ----
Temperature controller	Provided by client	---- N/A ----
Computer with LabVIEW installed	Provided by client	---- N/A ----
GPIB interface cables	Provided by client	---- N/A ----
Thermally conductive grease	Provided by client	---- N/A ----
Cryogenic Varnish	Provided by client	---- N/A ----
New Platinum RTDs	PPG101A6, US Sensor (Digikey)	$ 58.44
Thermally conductive epoxy	Stycast 1266 Cryogenic Epoxy (CMR Direct)	$ 197.75
Thermal interface pad	5519 or 5591S Pads from 3M	$ 61.06
Connectors and wires	Digikey	$ 109.62
Junction Box Materials	Digikey	$ 50.48
Mylar Insulation	Edmund Scientifics	$ 31.90
Keithley 6485 Picoammeter	Keithley/Tektronix	$ 1,660.00
	TOTAL:	$ 2,169.25

5.2 Project schedule

Within the first few weeks of the project, we set a schedule for the rest of the year. For the most part, we were able to follow this schedule fairly accurately, with only some small slippage in February, when we needed to purchase some new parts to finish assembling the unforeseen junction box. The largest obstacle to our schedule was working around the schedule of the MRC, where we were not permitted to work without our advisor there to supervise. Also, other research groups were actively using the system, so we often would have to delay and reschedule our work because someone's experiment was running long.

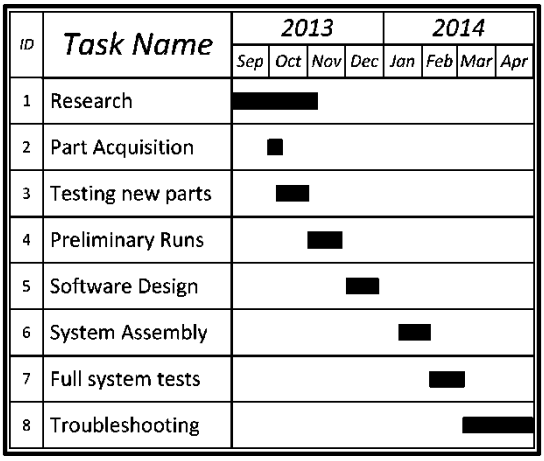


Figure 18: Schedule for the project

6 Testing Procedure and Testing Results

By the nature of our system, testing individual components was a very time-consuming task. Depending on the state of the insulation, reaching low temperatures would take anywhere from 20 to 40 minutes, and ramping back up to simulate an experiment could take several hours. Also, the performance of each component couldn't be adequately In order to maximize the useful data we could extract from a single run, we designed a coherent succession of experiments to evaluate our materials.

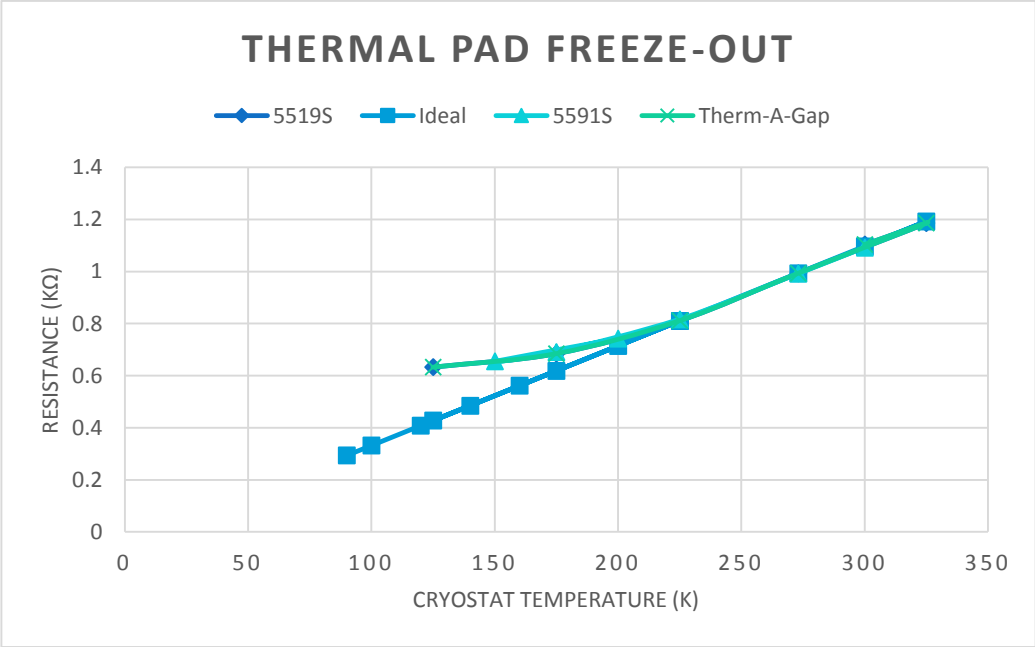
6.1 Probe development

In order to prove the efficacy of one probe setup over another, we used two different standard setups: in one, the temperature sensor was placed directly on the copper stage with a small bead of thermal grease, and in the other the sample was placed on a glass slide resting on the copper stage, with thermal grease at both interfaces. Under both testing setups, experiments were run under vacuum and without any insulation. Through these tests, every solution which had a large solid mass failed miserably. Only the setups consisting only of thin wires managed to produce reasonable values for temperature. The rest of the deviation could be handled through the insulation.

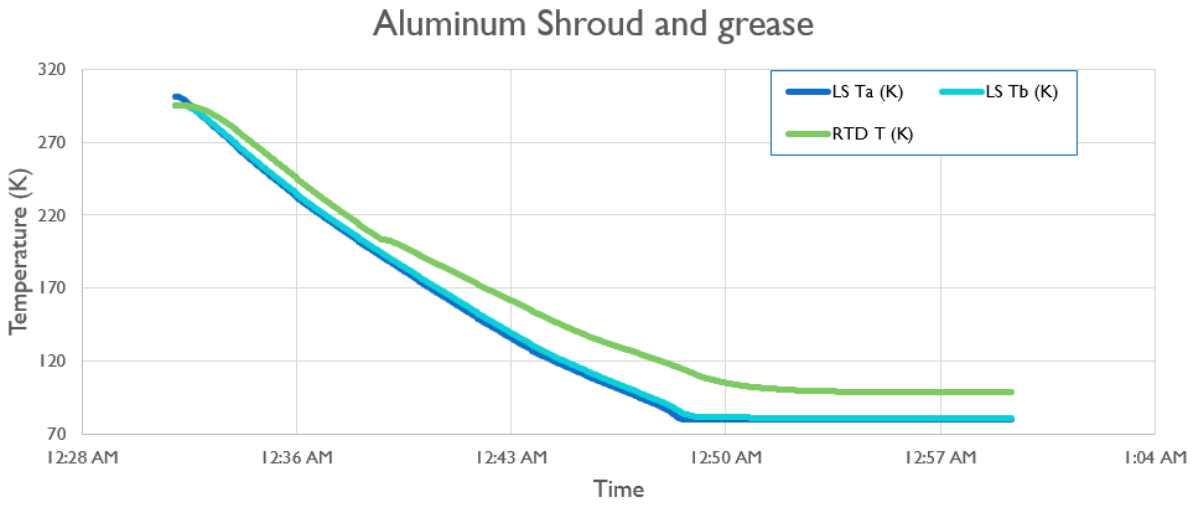
6.2 Thermal interface selections

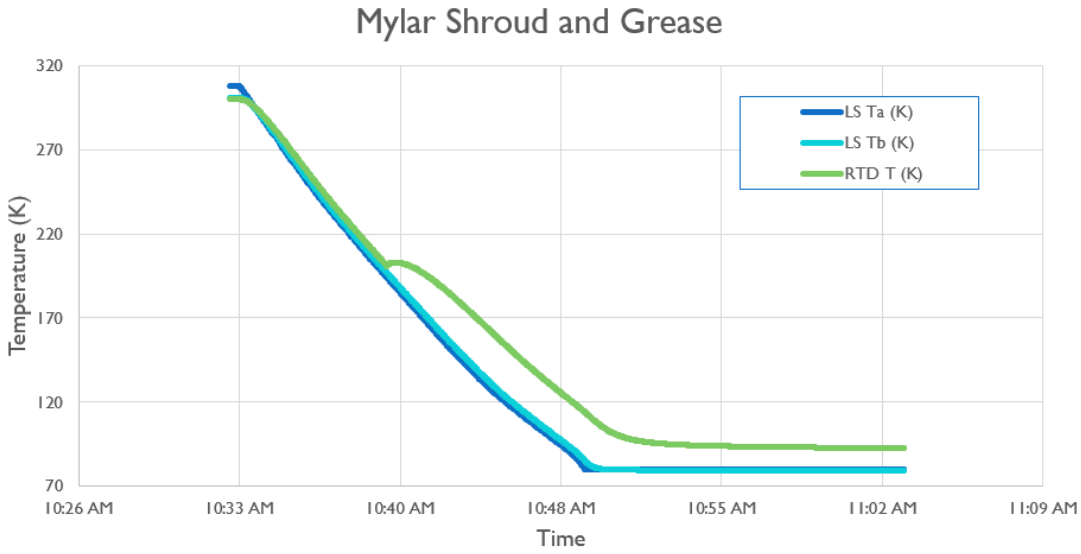
Once we had a reliable probe to place on the sample surface, we spent a significant amount of effort to find a suitable thermal interface material to place between the copper cryostat and the glass sample. For each material candidate, we ran a test using no insulation and no electrical contact on a bare glass slide identical to those on which the samples were fabricated, and placed the RTD probe on the surface.

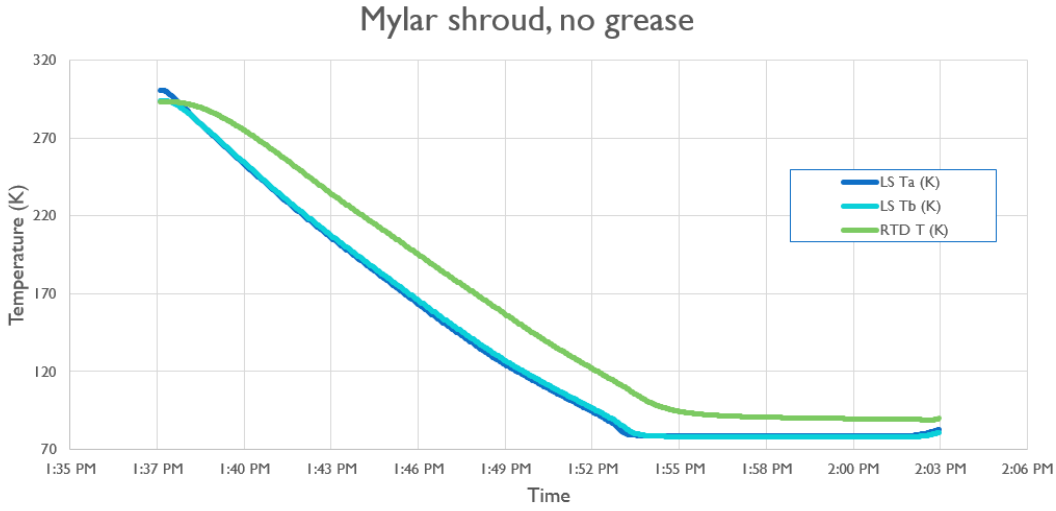
We first examined three different conformal pads (details in Appendix C) and quickly realized that none of those options would function beneath 200 K. The pads all seemed to freeze and lose their thermal properties around that temperature.



The next candidate was the Apiezon grease, so we applied a consistent amount of grease to the cryostat and pressed the glass slide down, rubbing it around in circles in an effort to create a thin layer beneath the slide. Early tests using the prototype aluminum foil showed a small nonlinearity around 200 K as well, but the grease still maintained most of its thermal conductivity after this transition. Once the Mylar shroud was developed, which had a significantly lower thermal mass than the aluminum one, this transition was even more pronounced, and became a large obstacle to a linear ramp during the measurement phase. With this advanced shroud, we found that removing the thermal interface material entirely created a larger time lag between the cryostat and the sample surface, but that the sample temperature still achieved the same end temperature. This was a result we could not have expected given our initial understanding of the system.


Aluminum Shroud and grease
320
270
220
170
120
70
Temperature (K)
LS Ta (K)
LS Tb (K)
RTD T (K)
12:28 AM
12:36 AM
12:43 AM
12:50 AM
12:57 AM
1:04 AM
Time


Mylar Shroud and Grease
320
270
220
170
120
70
Temperature (K)
LS Ta (K)
LS Tb (K)
RTD T (K)
10:26 AM
10:33 AM
10:40 AM
10:48 AM
10:55 AM
11:02 AM
11:09 AM
Time


Mylar shroud, no grease
320
270
220
170
120
70
Temperature (K)
LS Ta (K)
LS Tb (K)
RTD T (K)
1:35 PM
1:37 PM
1:40 PM
1:43 PM
1:46 PM
1:49 PM
1:52 PM
1:55 PM
1:58 PM
2:00 PM
2:03 PM
2:06 PM
Time

6.3 Insulation Development

Similar to the thermal interface analysis, we also conducted an extensive comparison between various shroud designs and Mylar packing schemes. All of these tests were run using the 40 V setting on the liquid nitrogen pump, without the contact wire, and with the vacuum pump on. The effectiveness of the shroud was rather definitively demonstrated through these tests.

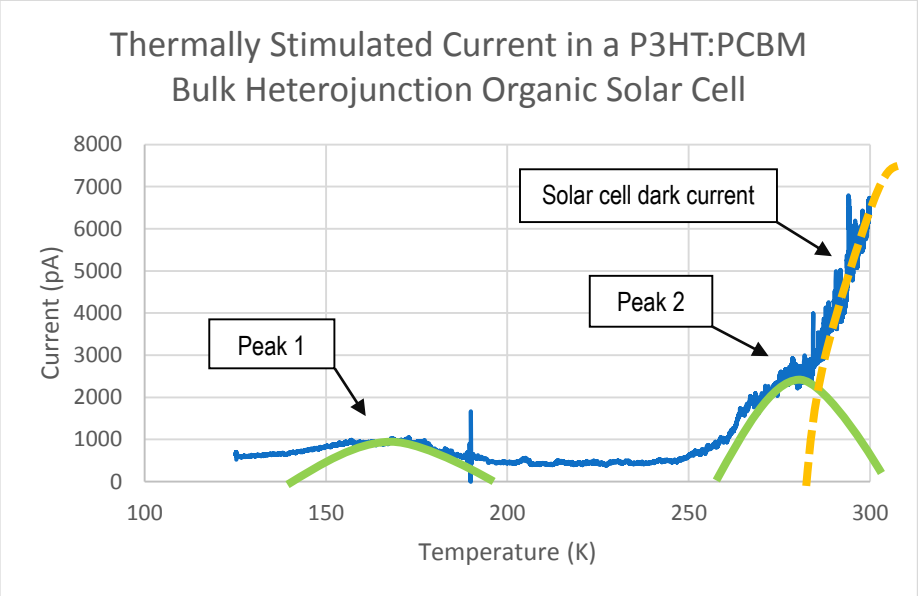
Setup	Minimum Temperature (K)	Time to stable (min)
Bare cell	136.64	21
Aluminum shroud, top grease only	92.20	22
Grease alone, both sides	135.01	20
Grease and aluminum shroud	98.55	24
Mylar shroud, no grease top or bottom	95.80	26
Mylar shroud and grease, top only	90.54	25
Mylar shroud and grease, both sides	92.75	29
Mylar Shroud and grease, top only, with extra insulation	88.65	20
Final Shroud, grease top only, all extra insulation	83.73	20

6.4 Noise characterization and shielding

Upon connecting the Picoammeter to the system for a test run, we noticed that the noise levels were significantly higher than we expected. In order to determine where and how noise was coupling into the system, we conducted tests of parts of the system and connected one part at a time, noting the extent to which the noise changed. We found that the noise was not coming from any of the instrumentation in the current path, but rather from the LakeShore temperature controller. By ensuring that the ITO clamp was fully electrically isolated from the cryostat (which is electrically connected to the heater we were able to minimize this noise. Also, as the noise was periodic and predictable, we were able to apply digital filtering to remove it from the final signal.

6.5 Full system assembly

Once we had fleshed out the details of each component, we ran a few full TSC tests on real organic solar cells. Data analysis was not within the scope of our project, but our preliminary results detected two potential defect peaks, illustrating the successful operation of our completed system.



APPENDIX A: Hardware Setup Walkthrough

A.1 Sample Prep

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. Before placing the sample into the chamber, make sure that the bottom glass surface is free of any cryogenic grease or other substance. If any such material is present, use a lint-free wiper and some isopropyl alcohol to gently clean the surface. Do NOT apply the alcohol directly to the sample, but rather apply it to the wiper first, and then rub the wiper on the surface.

A.2 Chamber Prep

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. If the lower chamber insulation is not already in place, carefully insert aluminized Mylar sheets into the chamber beneath the cryostat one at a time, taking care to pack them all the way to the bottom, but avoiding the wiring within the chamber. The thin cryogenic wires are fragile and will not withstand any considerable strain. The packing does not need to be too dense: five or so Mylar sheets (roughly one square foot each) should suffice. Finally, take care not to allow the insulation to make contact with the suspended heater wire, as contact can cause the insulation to melt.

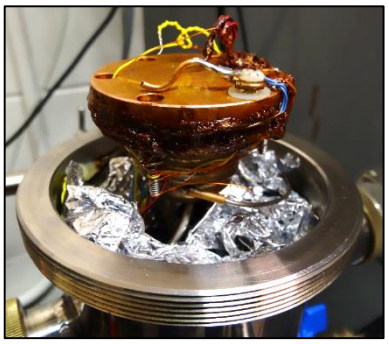


Figure 19: Aluminized Mylar packed into the bottom half of the chamber

2. Mylar sheets should similarly be added to the chamber cover as well. Loosely pack aluminized Mylar along the inside walls of the cover, making sure to leave enough space to accommodate the shroud when fully assembled. About three sheets of Mylar should be enough. The blue cap for the liquid nitrogen Dewar can be used to compact the Mylar enough to make room for the shroud.

3. Make sure that no screw is placed in the marked screw hold in figure 3. The tie-downs for the RTD wire and the electrical contact wire should be threaded through this hole.

Figure 20: Aluminized mylar packed into the top cover of the vacuum chamber, leaving enouh space for the

4. Before placing the sample in the chamber, make sure that the cryostat surface is clear of any cryogenic grease or other substances. If any such material is present, clean the surface by applying isopropyl alcohol to a lint-free wiper and rubbing the surface, taking care not to apply any alcohol to the brown varnish. Isopropyl alcohol slowly dissolves the varnish and will degrade it over time. With the same alcohol and wiper, gently clean the tip of the yellow, electrical contact wire, to ensure that it will make good contact to the sample.

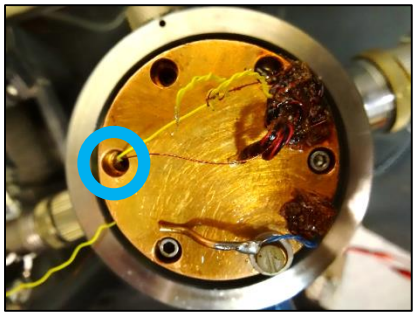


Figure 21: The marked screw hole should be left open, and the tie-down wires should be threaded through this hole.

5. Using a sheet of fine grit (>400) sandpaper, gently sand the underside of the copper ITO clamp. Copper oxide slowly forms on the clamp at ambient temperature and humidity, and if not removed it can lower the quality of the measurement.

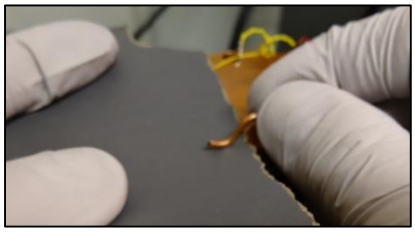


Figure 22: Sanding the underside of the

A.3 Loading a Sample

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. Pull up on the contact and RTD wires to create a large enough space to accommodate a sample on the surface of the cryostat head. Loosen the nylon screw on the copper ITO clamp and rotate the clamp away from the center of the cryostat.

2. Carefully, and without scratching the top surface, slide the sample onto the copper stage. Center it as well as possible, and orient it such that the ITO clamp can make solid contact.

3. Rotate the ITO clamp back over the ITO region, carefully lower it to the surface, and press down on the clamp. While holding it down with one hand, use your other hand to tighten the screw and lock the clamp in place.

4. Designate one contact on the surface to act as the thermal reference. Using the tip of a needle, wire, or other thin metallic object, carefully apply a small dot of cryogenic vacuum grease to that contact.

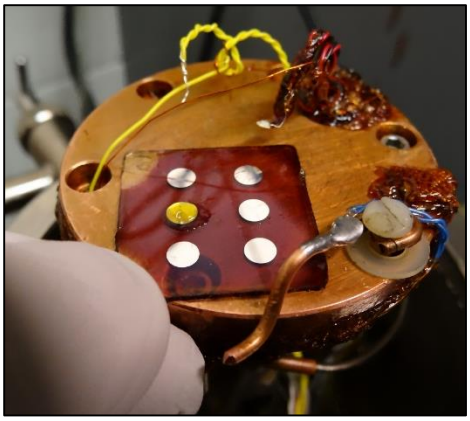


Figure 23: Carefully sliding the OPV sample onto the cryostat. This sample had already been used for a few tests, so the yellow grease had already been deposited on one of the contacts.

5. Using the tie-down wire connected to the RTD, lower the RTD onto the greased contact. For best results, bend the RTD wire such that the RTD naturally comes into contact with the closer edge of the contact, and then pulling the tie-down tight causes

the head to slide into the center of the contact. The head should be flat on the sample surface. Wrap the tie-down wire around the screws on the underside, as close to the copper as possible.

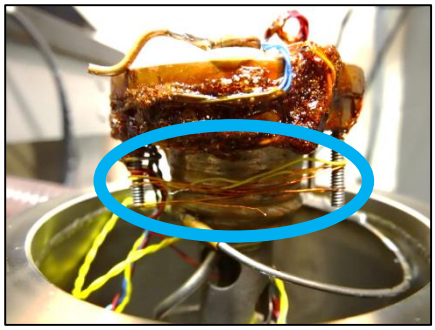


Figure 24: Stabilizing the tie-down wires by wrapping them around the screws on the

6. Repeat the same procedure with the electrical contact wire. Because this wire is smaller, care must be taken to ensure that the wire makes perpendicular contact to the surface, or else it will buckle and slide under the tie-down force. Wrap the tie-down wire on the same screws, taking care not to disturb the RTD tie down.

7. Carefully lower the shroud onto the cryohead, and gently push down until the shroud is snug and secure. Make sure the shroud remains upright, and does not become canted off to one side, as this can disrupt the contacts to the sample.

Figure 25: The shroud on the

8. Carefully lower the vacuum cover onto the chamber, making sure that no Mylar or tie-down wires get caught between the vacuum chamber halves. Tighten the brass collar to complete this process.

A.4 Connecting the Instructions

1. First, ensure that all four instruments (Keithley 2400, Keithley 617, Keithley 6485, and Lakeshore 331) are interconnected by stacking GPIB cables. The order of these cables does not matter as long as all devices are connected to the same bus.

2. In order to protect the sensitive amplifiers in the Keithley 6485 Picoammeter, make sure that the “Zero Check” function is activated. When it is enabled, either “ZZ” or “ZC” will appear after the current measurement. If this is not the case, turn this function on by pressing the “ZCHK” button on the front panel. Do not attempt to connect or disconnect any cables until this is done.

Figure 26: The stack of measurement instruments

3. Two of the female connectors on the chamber are labeled with ITO. These two should be connected together using the 2-1 BNC adapter, and then connected directly to the positive terminal (marked with a plus sign) on the shielded box.

4. Connect a BNC cable to the negative terminal on the box (marked with a minus sign) and then, using a BNC-Banana adapter, plug the center terminal (the red connector) into the black binding post on the rear panel of the Keithley 617. Connect the black connector to the "Common" terminal on the back of the Keithley 6485.

5. With a second BNC Banana adapter, connect the red terminal to the red binding post on the back of the Keithley 617, and the black connector also to the "Common" terminal on the back of the Keithley 6485. Then connect the other end of the BNC cable to the two remaining connectors on the chamber by using a 2-1 BNC adapter.

6. Finally, use the Keithley BNC-terminated-triax cable to connect the isolated jack on the shielded box (the center connector with the white plastic collar) to the rear panel of the Keithley 6485.

7. Ensure that the KUSB-488 GPIB to USB adapter is connected to a GPIB cable on the rear panel on any one of the instruments. Connect the USB terminal to the computer which will be running the automation software.

Figure 27: The 6485 Picoammeter front panel, with the position of the ZCHK button marked.

Figure 28: The shielded junction box with the connectors and adapters

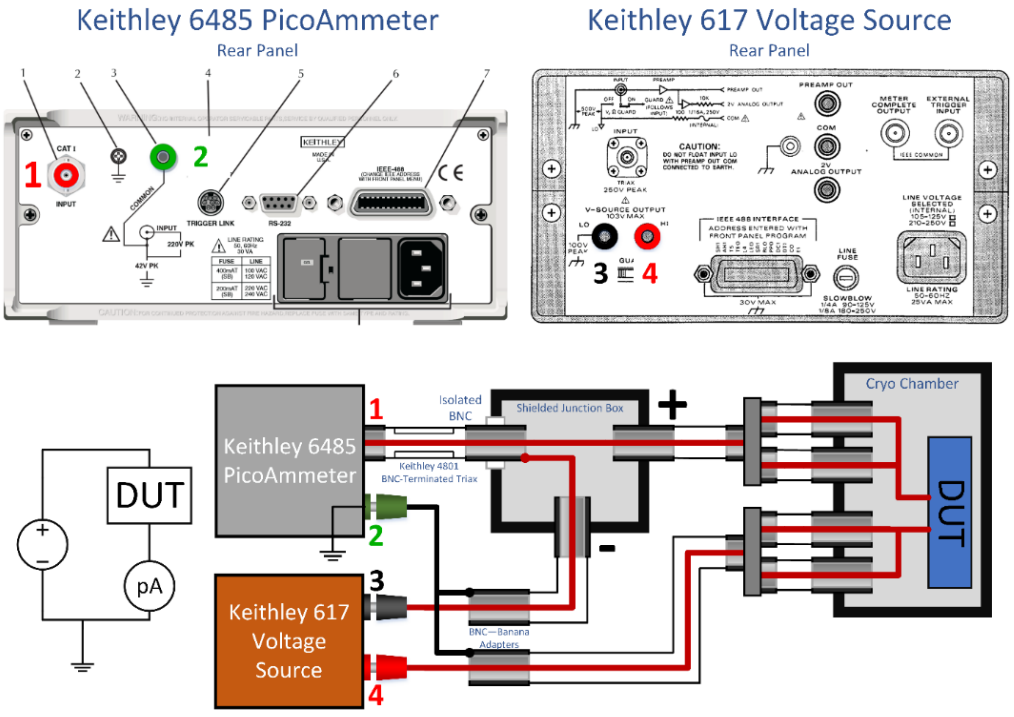


Figure 29: Schematic diagram of the necessary connections for running TSC experiments

A.5 Running the Software

1. To run the software, open Labview 2012 or later. Use the file browser to navigate to the VI titled "TSC Control.vi". If the computer in the lab is being used, a desktop shortcut exists with this name.

2. Open program, and Labview runs the program automatically. Input your setpoints, and click on the "Start" button when ready. For more details, see the TSC Software Manual, located on our website.

A.6 Starting the Vacuum Pump

1. Once you press the "Start" button on the software, the software will prompt you to turn on the vacuum pump. First, check to make sure each feedthrough into the chamber is snug, then close the chamber valve. Flip the small switch near the back of the pump to turn it on, and allow it to equilibrate to a low pressure. Turn the vacuum off, and observe the pressure for five seconds. If the pressure gauge changes noticeably with the vacuum off, there is a leak somewhere between the pump and the chamber valve. Check each joint and try again.

2. Once the hose and pump are leak free, turn the vacuum back on, and open the valve to the chamber. The pressure gauge will rise momentarily as the chamber evacuates, but will eventually settle down. Turn the pump off one more time and check for leaks. If the pressure changes noticeably, there is a leak in the chamber, which most likely is caused by getting some material stuck between the top and bottom halves of the vacuum chamber.

3. If such a chamber leak is suspected, vent the chamber by turning off the vacuum pump and unscrewing the valve connector from the chamber. Once the pressure is equilibrated back to ambient, open the chamber, readjust, and try again.

Figure 30: The vacuum pump. Ensure that the oil levels (visible on the bottom left) is within the recommended limits.

A.7 Starting the Cryogen Pump

1. The cryogen pump is a variable-strength pump controlled by a variac. Tests have shown that the optimal voltage setting is 40 V. To minimize sample vibrations, turn the dial down to zero while the variac is off, turn it on, and then turn the voltage up smoothly to 40 V.

A.8 Unloading the Sample

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. The software will automatically activate the "Zero-Check" function on the picoammeter. This will be denoted by the letters "ZC" after the current reading. Take care NOT to disturb this setting, as turning the zero-check off leaves the sensitive components vulnerable to ESD events from your body or your tools.

2. After a TSC experiment is completed, the temperature will be 300 K. If an experiment has been aborted for any reason, the software will set the setpoint to 300 K, but the system may take a few minutes to reach that temperature. For safety reasons, and to minimize condensation within the cold chamber, wait until both outputs on the sample show at least 280 K before proceeding to vent or open the chamber.

3. Begin by unscrewing the collar at the chamber valve to vent the chamber to atmospheric pressure. Then carefully unscrew the brass collar around the cover and gently lift the cover straight upwards off of the system.

4. Remove the shroud by pulling up on it, taking care not to squeeze to hard to disrupt or scratch the sample inside.

5. Unwrap the tie down wires and pull up on the electrical contact wire, then the RTD. The thermal grease tends to form whiskers when pulled, so the tip of a needle or wire, or the corner of a lint-free wiper may be used to catch the excess from landing on the sample.

6. Carefully loosen the nylon screw holding down the ITO clamp and rotate the clamp away from the sample.

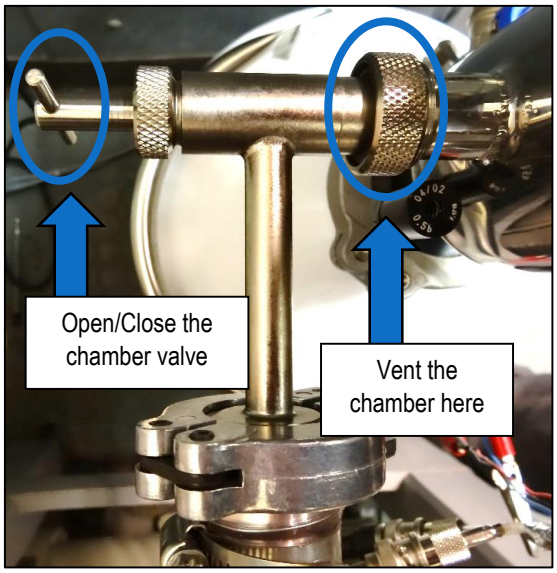


Figure 31: Chamber vacuum connector, showing the valve between the chamber and the pump, as well as the collar which can be used to vent the chamber.

A.9 Filling the Liquid Nitrogen Dewar

1. If the liquid nitrogen runs out, begin by removing any samples and disconnecting all of the connectors from the bottom half of the vacuum chamber.

2. Open the collar above the Dewar, but below the black safety-valve assembly, by loosening the wingnut, swinging the bolt out, and separating the two halves.

3. Carefully pull the vacuum chamber directly upwards until the entire tube is exposed. The tube is roughly two feet long. The tube may still be at a very low temperature, so care must be taken in handling it.

4. Place the blue insulating cap on the liquid nitrogen flask and take it out to be filled. Due to safety concerns, instructions for operating the liquid nitrogen source at the MRC are not included here. See your supervisor or other user of that system for training if you have not yet been trained.

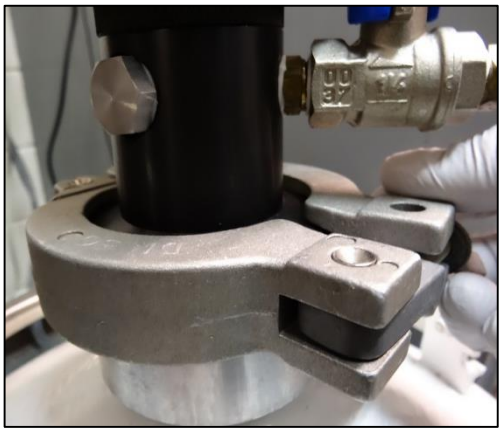


Figure 32: Opening the collar to remove the flask

5. After filling, remove the blue cap. Using several lint-free wipers to protect your hands from the potential cold, wipe any extra condensation from the tube before carefully reinserting it into the Dewar.

6. Reattach the chamber to the flask using the large collar, and then reconnect the vacuum tube, instrument connectors, etc. to prepare for another experiment.

Figure 33: Extracting the tube from the flask. The tube is still dangerously cold.

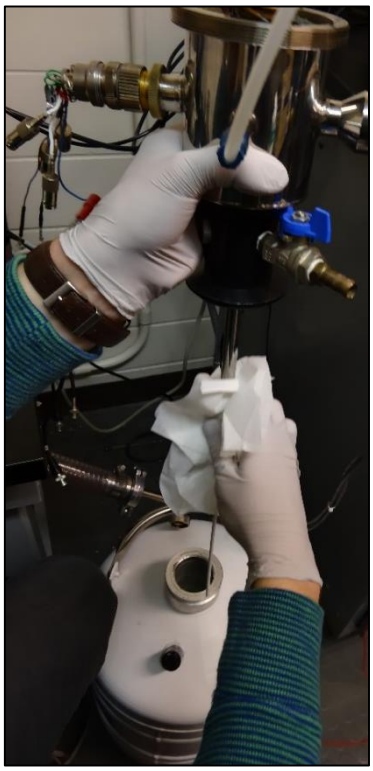


Figure 34: Wiping the condensation from the tube before reinserting it.

APPENDIX B: Software Help Manual

B.1 Thermally Stimulated Current Experiment Process

B.1.1 Initialization/Parameter Entry

Immediately upon opening the program, the front panel is initialized for parameter entry. Using the File Controls, Profile Controls, GPIB Settings, and Experiment Parameters (described in Sections 2.1-2.4) as well as the Advanced Settings dialog (Section 3), the user has complete control over designing TSC experiments. This phase ends when the user completes the setup and clicks the "Start" button. At that point, any parameters are locked in until the experiment completes, or is aborted by the user.

B.1.2 Cooling

When the experiment begins, it automatically places the system in the cooling phase, in which the heater is turned off and the temperature allowed to drop. Depending on the chosen charge profile, the voltage source may or may not turn on during this phase. Once the sample reaches the desired temperature, this phase ends and the soak phase begins.

B.1.3 Soaking

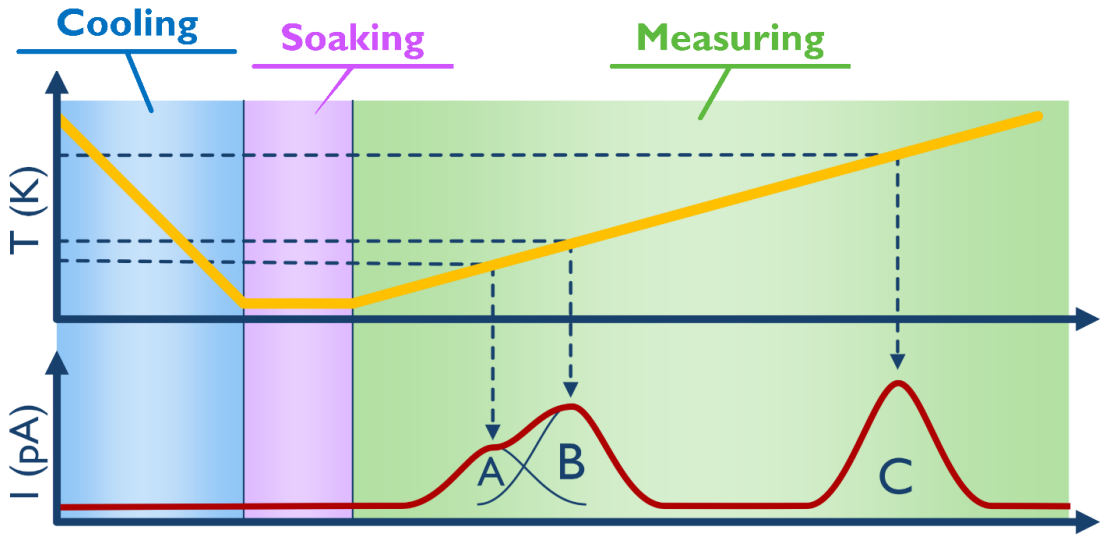
Reaching the desired temperature is not always enough to ensure a high-quality experiment. In order to minimize any thermal gradients, which may cause parasitic thermoelectric currents, the sample is allowed to soak at the desired temperature for a determined amount of time. This time period often also doubles as the charging period. The sample stays in this mode until the soaking time expires.

B.1.4 Measurement

Following the soak, the system enters the measurement phase. Here the system turns on the voltage source to the measurement voltage and proceeds to ramp to each input temperature in turn. When ramping up, the system controls the rate to match the desired ramp rate, and when ramping down the system does so as fast as possible by turning the heater off. The system forces the last setpoint to be 300 K, and the experiment is complete once this temperature is reached.

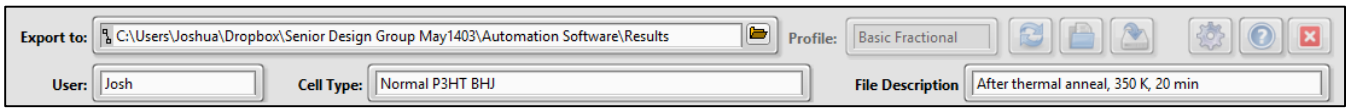
B.1.5 Completion

Once the system has completed its experiment, it will complete the export of the log file and reset the equipment to the defaults. It will then give the user an option to run another experiment or to close the program. If the user chooses to run another experiment, the system will by default keep the parameters from the last experiment.



B.2 Front Panel Anatomy

B.2.1 File Controls



B.2.1.1 Export Directory

The "Export To:" text box allows the user to specify the folder to which log files will be saved. Either type the name of the folder directly into the box, using the arrow keys to show wrapped text if the directory is too long, or click the small folder icon on the right side of the box. This opens up an explorer window, where the user can navigate to the desired folder. Once inside the folder, click on the "Current Folder" button on the bottom of the dialog. Log files will now be saved to this folder.

B.2.1.2 User

The "User" text box serves two purposes:

1. The name entered on the front panel is saved in the log file as a way to remind viewers of the file who ran the experiment and ease data analysis, which can often occur days or weeks after the experiment itself
2. During an experiment, the name remains visible on the front panel, such that if the user steps away from the system, others in the lab will know who is running the experiment.

Simply type a name in the box, then press <Enter> or click somewhere else on the front panel.

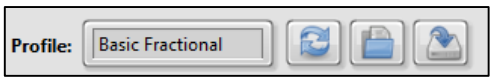
B.2.1.3 Cell Type

The "Cell Type:" text box allows the user to notate not only the cell type, but also any desired processing parameters or other unique identifiers. This identifier is the first part of the filename of the log file, as described more thoroughly in Section 4.1.1. Type the desired information into the box, then press <Enter> or click elsewhere on the front panel.

B.2.1.4 File Description

The "File Description:" text box serves only to add more information to the log file, to ease file identification at a later date. Simply type the desired information into the box, then press <Enter> or click elsewhere on the front panel.

B.2.2 Experiment Profile Settings



In order to decrease experiment setup time and improve repeatability, the program allows a user to save experiment profiles, which contain all of the parameters in the front panel (Section 2.4) and from the Advanced Settings panel (Section 3).
Experiment profiles are easily legible text files (*.txt) which are saved in the "Profiles" subfolder of the program's root directory. Please do not move or delete this folder.

The name of the current profile is displayed at the top of the front panel. If the name is followed by an asterisk (*) then one or more settings have been changed since the profile was last loaded. These changes are not automatically saved in memory, and will be lost if the user closes the program without saving them.

B.2.2.1 Save Experiment Profile

Once an experiment has been designed as desired and all of the parameters inputted, the user can save the experiment as a profile by clicking the Save button at the top of the front panel. This will open a small dialog in which the user is asked to input a profile name.
The filename cannot contain any of the following characters: \ / : * ? " < > |

Upon clicking the "OK" button in this dialog, all of the current values on the front panel and in the advanced setting dialog will be saved under that name. If a profile with the name already exists, the user will be asked if they are sure they want to overwrite the file, as any old settings within that file will be lost.

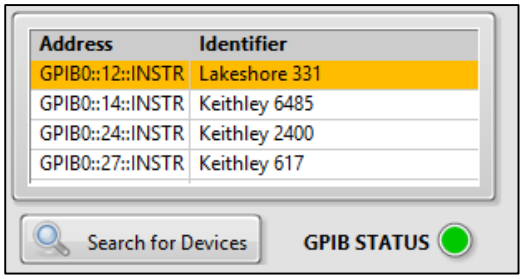
B.2.2.2 Load Saved Profile

Once a profile has been saved, it can easily be loaded by clicking the Load button at the top of the front panel. This will open a dialog showing the user all of the existing profiles in the Profiles folder. Select one by left-clicking on the name of the desired profile, then clicking OK. If any unsaved changes had been made to the parameters on the front panel or in the advanced settings, then they will be lost when the new profile is loaded.

B.2.2.3 Refresh profile

The "Refresh" button at the top of the front panel discards any changes that have been made to the settings on the front panel or in the advanced settings dialog and reloads the experiment profile named in the text box. Unsaved changes are irrevocably lost when this is done.

B.2.3 GPIB Addresses



The Dashboard program communicates with the necessary instrumentation through the GPIB protocol.

B.2.3.1 Connected Devices

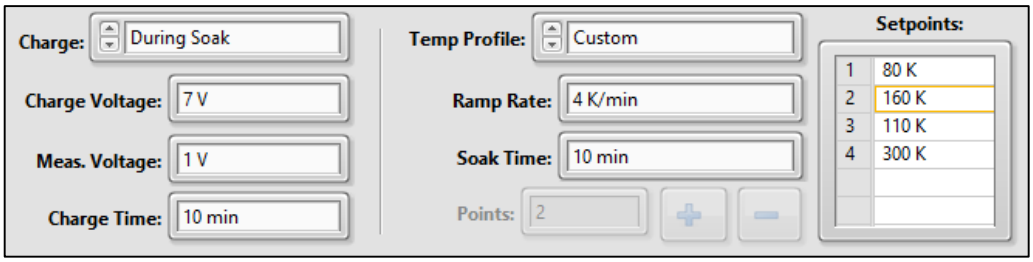
The "Connected Devices listbox" shows all of the devices which are compatible with the software.
The software is set up to communicate with the following devices: Lakeshore 331, Keithley 2400, Keithley 617, and Keithley 6485. If none of these are found, the listbox will display "GPIB Error—Check connection" in the first line. If at least one device is found, the system will still allow an experiment to take place, but will not report those data points which rely on the missing equipment.

The GPIB Status indicator light glows green if the GPIB bus is active and at least one compatible device has responded to the query. If no devices have been found, the indicator light turns red.

B.2.3.2 Search for Devices

The "search" button sends out a general identification query to every device on the GPIB bus. Every device responds with its bus address, manufacturer, model number, and other relevant information. This data is then used to update the Connected Devices listbox (Section 2.3.1) and the GPIB Status indicator.

B.2.4 Experiment Parameters



The experiment parameters section of the front panel allows the user to enter all of the desired settings to design the experiment. These parameters are only available up until the user begins the experiment, at which point they are locked in and become static on the front panel. The setpoints will remain visible, as a reminder to the user of what is being run.

B.2.4.1 Charge Types

During Soak

When "During Soak" is selected, the charging voltage will be applied to the system when the cooling phase ends and the soak phase begins. The charging voltage will be applied for the duration of the "Charging Time" (Section 2.4.4) which must be less than or equal to the "Soak Time" (Section 2.4.7). If the charging time is less than the soak time, no voltage will be applied during the time period after the charge time has expired and before the measurement begins.

While Cooling

When the "While Cooling" option is selected, the charging voltage will be applied at the beginning of the cooling phase, and will remain on until the soak phase begins. This allows for non-isothermal filling of trap states. Under this configuration, zero voltage bias will be applied to the sample during the soak phase. With this configuration, there is no option to input the charge time, as it depends on the cooling rates of the sample.

Until Measurement

Under the "Until Measurement" configuration, the charging voltage is applied during the entirety of both the cooling phase and the soak phase. With this configuration, there is no option to input the charge time, as it depends on the cooling rates of the sample.

B.2.4.2 Charge Voltage

The "Charge Voltage" setting on the front panel dictates the voltage applied to the cell during the charging phase, as explained in Section 2.4.1. The voltage is constant at this value and accurate to the rated accuracy of the voltage source instrument attached to the system.

B.2.4.3 Measurement Voltage

Once the temperature ramp-up has begun, the system applies the "Measurement Voltage" to the sample, which helps to collect de-trapping electrons and create the thermally stimulated currents. This voltage is constant from the end of the soak cycle until the end of the experiment.

B.2.4.4 Charge Time

When the "During Soak" option is selected for the charge type (see Section 2.4.1) the "Charge Time" option dictates the duration of the charging phase. The charging voltage is applied for that duration, starting at the beginning of the soak cycle. The charge time must be less than the soak time so that the measurement voltage can be applied at the beginning of the ramp-up.

B.2.4.5 Temperature Profiles

Basic Ramp

The "Basic Ramp" option simply lowers the sample to the desired temperature, soaks for the desired time, and then increases the temperature continuously at the desired rate until the temperature returns to 300 K. This mode supports no more than two temperature setpoints, and the second must always be 300 K for safety reasons.

Base/Peak

The "Base/Peak" option creates the option of conducting fractional emptying experiments, with the stipulation that each cooling cycle returns the sample to the original baseline temperature. This mode requires three or more setpoint values, the first of which is the baseline temperature, and each successive temperature is the height of a temperature peak. No charging or soaking will occur between peaks. While the height of each peak is variable, all of the ramp-up phases will occur at the same rate The number of peaks can be changed using the "+" and "-" buttons next to the "Points" indicator (Section 2.4.8) The final peak value must be 300 K for safety reasons.

Custom

The "Custom" option allows users complete control over the temperature profile of the experiment, and requires at least 4 setpoints. The first setpoint should be the lowest, as this is where the soak will take place. Each successive point can be the height of a peak or the depth of a valley between peaks. No soaking or charging will take place between successive peaks, and all ramp-up phases will occur with the same ramping rate. The number of points can be changed using the "+" and "-" buttons next to the "Points" indicator (Section 2.4.8)

B.2.4.6 Ramp Rate

The "Ramp rate" field allows the user to define the rate at which the temperature ramps up during the measurements. The number chosen here will have no effect on the cooling sections, as temperature rate controller is disconnected and the heater forced off for cooling phases. In temperature profiles with more than one heating phase, all phases are subject to the same heating rate.

B.2.4.7 Soak Time

The "Soak time" defines how long the temperature will be held at the first setpoint once it is reached. If the "during soak" charging option is selected, the soak time must be greater than or equal to the charge time. Regardless of the number of peaks in the temperature profile, the soak time is only applied after the cooling phase.

B.2.4.8 Points

The numerical "Points" input dictates how many different temperature setpoints are used in the experiment. Under the Basic Ramp profile (Sec. 2.4.5.1), only two setpoints are used, so the "points" indicator is disabled. In the Base/Peak and Custom modes, any number of points above three and four, respectively, are allowed. Adding and subtracting points is accomplished by clicking the "+" and "-" buttons next to the numerical box.

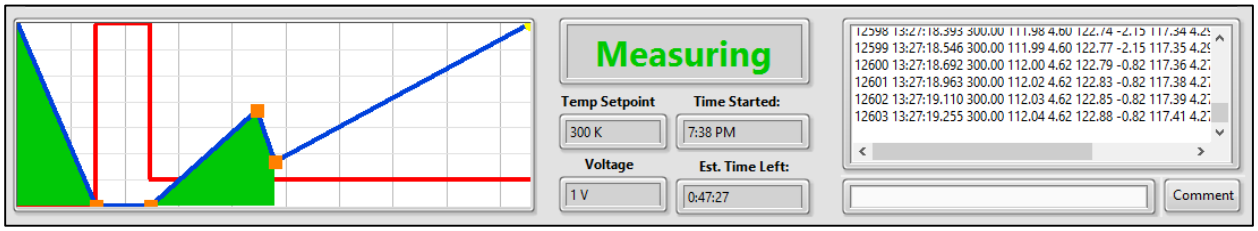
B.2.4.9 Setpoints

The "Setpoints" box allows the user to type in temperatures (in Kelvin) to use in designing the experiment. The entries in the box are limited by the following constraints:

- The number of boxes allowed to have values is limited by the "points" indicator (Section 2.4.8)
- The value of each setpoint must be within the maximum and minimum temperature ranges as defined within the advanced settings dialog.
- The last point must always be 300 K for safety reasons

The impact each setpoint has on the experiment as a whole will be defined by the chosen profile type (Section 2.4.5)

B.2.5 Experiment Progress Section



B.2.5.1 Schematic Timeline

Before an experiment is commenced, the schematic timeline gives the user a quick overview of the parameters, making it quick and easy to find and correct small errors or typos before the experiment takes place. The red line shows the voltage to be applied, while the blue line shows the temperature ramps to be conducted. While the timeline is not clickable and has no direct interactivity, the orange squares denote which vertices on the graph may be altered under the selected temperature and charging schemes. The timeline updates after every change of settings.

During an experiment, the progress of the experiment is shown by a small yellow dot, which is located at the present temperature setpoint on the graph. A green fill line moves across the graph as well, showing all of the setpoints which have already been achieved. The point does not move linearly as a progress bar would, but rather moves only at the transitions between setpoints.

The timeline is not to scale. The axes are arbitrary, and the y-axis for the temperature curve is in fact logarithmic. Do not attempt to read parameters from the timeline--read them from the numerical inputs instead.

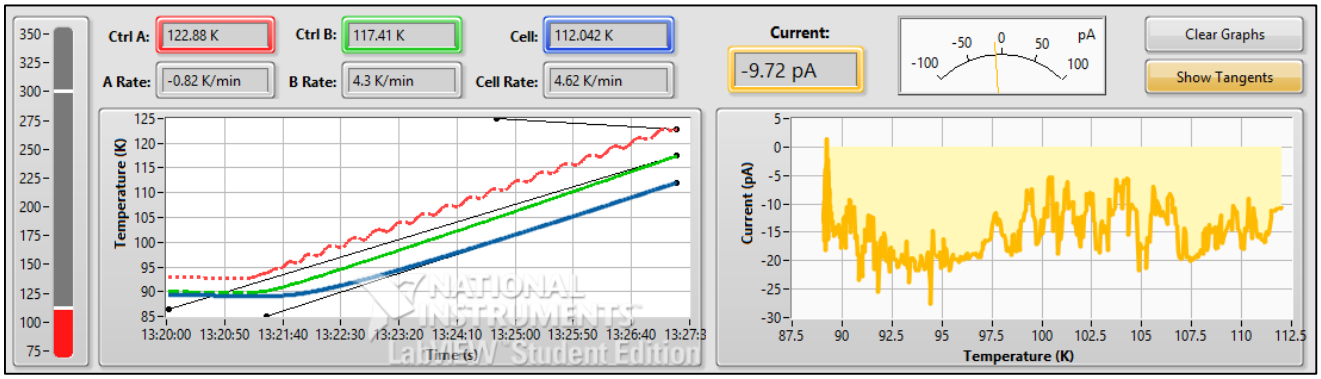
B.2.5.2 Progress Indicators

The five progress indicators between the schematic timeline and the log output window indicate present values for the experiment phase (Cooling, Soaking, and Measuring), the temperature setpoint, and the voltage. Also, the time when the experiment was started (measured at the moment the “Start” button was pressed) and a rough estimate of the remaining time until the end of the experiment.

B.2.5.3 Log File Readout and Comment Button

The log file window shows in real time the data being written to the log file. To save processing power and improve computation time, the log window only displays the last 800 lines. Using the scroll bar, the user can pause the log temporarily and examine any events of interest, until those lines are out of range or until the mouse pointer leaves the log window. To ease analysis, th user also has the ability to insert comments into the log file by typing a message into the input box beneath the log output and pressing the “Comment” button. The text of the comment is appended to the end of the next line to be recorded after the button was pressed, so it should be noted that these comments are qualitatively, not quantitatively useful.

B.2.6 Data Indicators



B.2.6.1 Numerical Temperature and Current Indicators

The numerical indicators along the top all display the most recent values of their labeled quantity.

Ctrl A/B: These indicators show the temperature measured by the A and B sensors from the Lakeshore 331 controller.
A/B Rate: These indicators show the calculated time rate of change of the A and B Lakeshore inputs.
Cell: The temperature measured on the surface of the cell
Cell Rate: The calculated time rate of change of the temperature of the cell
Current: The measured current through the cell

B.2.6.2 Thermometer Indicator

The thermometer indicator on the far left is a graphical depiction of the present cell temperature (the white marker at the top of the red fill) and the present temperature setpoint (the other white marker).

B.2.6.3 Temperature Graph

The temperature graph displays three different curves, which are color-coded with the numerical indicators they represent (Section 2.6.1). The red line is the temperature of the A sensor, the green line is the B sensor, and the blue line is the cell temperature. When the tangent lines are activated, three black lines appear and match the slopes of each of the three curves.

B.2.6.4 Current Graph

The yellow graph on the right displays the measured current through the sample. A faint yellow fill appears between the current and the line denoted by zero current. The X axis of the graph is temperature, not time, so the line appears to move backwards when cooling and may even move in loops depending on experiment conditions.

B.2.6.5 Clear Graphs Option

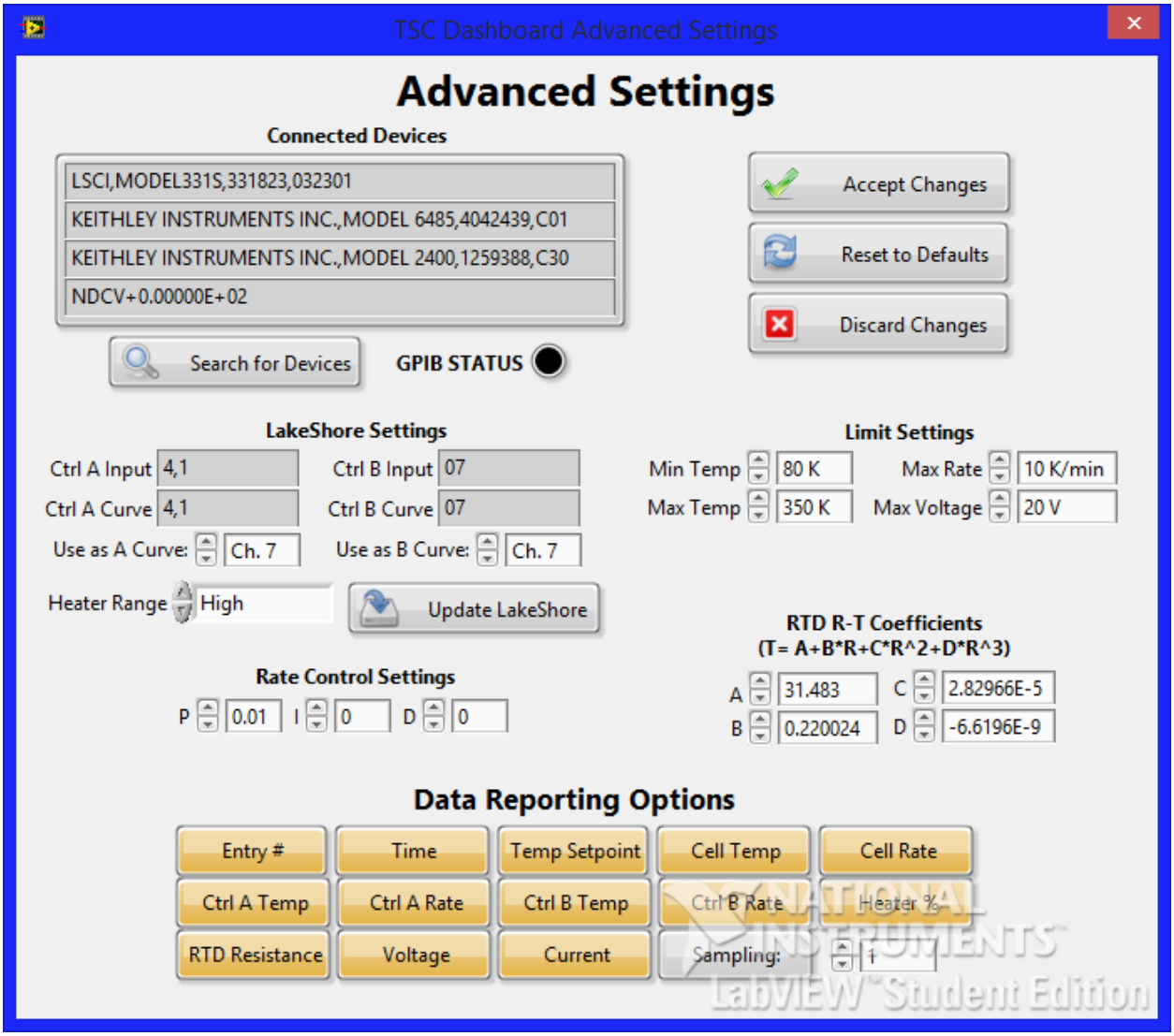
Pressing the "Clear Graphs" button irrecoverably deletes the data in the temperature and current graphs. No data is lost, and ALL of the data, before and after the clear, will still be saved into the log file, but that data cannot be retroactively brought back into the graphs on the software. Clearing the graphs is useful whenever the system transitions to a new phase, or to exclude large transients.

B.2.6.6 Show Tangents Option

Activation the "Show Tangents" button will draw thin tangent lines on the temperature graph, allowing for a graphical comparison of the ramping rates between inputs and over time. The presence or absence of these graphs does not affect the data collection nor the data logging.

B.3 Advanced Settings Dialog

Clicking on the gear icon on the front panel before starting an experiment brings up a new pane, which gives the user access to a number of new parameters as can be seen below.



B.3.1 GPIB Controls

The GPIB section of the advanced settings pane is similar to the section on the front panel (Section 2.3) but instead contains the raw GPIB identification strings from the connected instruments. Similar to that section, the refresh button resends the IDN command to see what devices are connected.

B.3.2 LakeShore Settings

The LakeShore temperature controller has several important settings which are integral to the operation of the TSC chamber.

The “Input” value for the LakeShore tells the LakeShore the type of sensor which is connected to its input. The “curve” number tells the Lakeshore how to convert the resistive or voltage values its measures into current. The input designators will only change if the cryostat system has to be rewired with new sensors, and can be done on the front panel of the LakeShore in that case. The curves are also fairly unlikely to change, and a new calibration curve must be entered manually. However, the advanced settings

option makes it possible to change the calibration curve. To do so, simply change the "Use as A Curve" and "Use as B Curve" inputs to the proper value, and then click the "Update LakeShore" button.

The "Heater Range" option dictates the highest value of the heater power which will be allowed. High corresponds to 25 W, Medium corresponds to 2.5 W, and Low corresponds to 0.25 W. Regardless of this setting, the heater will still be turned off during cooling phases.

B.3.3 Rate Control Settings

In order to accurately control the ramp rate during the measurement cycle, a PID (Proportional, Integration, Differentiation) control loop has been programmed into the TSC Dashboard. Any change in the physical setup may cause either an offset in the ramp rate or rate oscillations. Either case can be addressed by properly retuning the PID coefficients. By default, they are P=0.2, I=0.1, D=10, as determined by a series of tests. Because of the nature of PID controllers, excessive deviations from these default numbers may make the symptoms significantly worse.

B.3.4 Limit Settings

In order to protect the sample, as well as to ensure that experiment setpoints remain within reasonable bounds, the limit settings on the advanced settings pane allow the user to define extreme values. When entering values on the front panel, the software will automatically coerce the value to be in the ranges defined by the limit settings.

B.3.5 RTD R-T Coefficients

The RTD used in this design to make contact to the sample surface has R-T characteristics governed (in the important TSC range) by a third order polynomial function:

$$T = A + BR + CR^2 + DR^3$$

Our calculated line of best fit between 80 and 350 K is defined by:

$$A = 31.483$$
$$B = 0.220024$$
$$C = 2.82966 \times 10^{-5}$$
$$D = -6.6196 \times 10^{-9}$$

If a new calibration is required, these parameters can be tweaked by changing these values here.

B.3.6 Data Reporting Options

The Data Reporting section of the Advanced Settings pane consists of 14 buttons and one numerical input. The first 13 buttons each represent columns of data in the log file. If the button is activated (turned orange) then that column will appear in the TSC log file, and it will be excluded if it is deactivated (turned grey).

Activating the final button, "Sampling", will configure the log file only to contain those records for which the entry number is a multiple of the integer entered in the numerical box directly next to the button. Those other records will not be saved, so this function is only recommended for extremely long TSC measurements, for which the time-resolution is non-critical and the un-sampled log file is prohibitively large.

B.3.7 Saving Advanced Controls

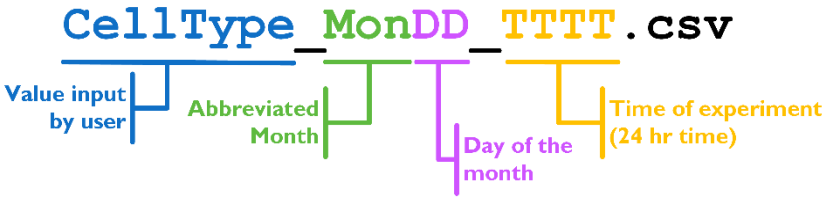
When the advanced settings have been configured as desired, press the "Accept Changes" button. Just like the inputs on the front panel, the advanced settings are not saved in memory until a profile is saved with those changes (see Section 2.2.1). Unless such a profile is created and loaded, the advanced settings will remain at the default values.

B.4 Log Files

At the completion of each experiment, the TSC Dashboard system exports a CSV file containing all of the collected data, as well as a header containing all of the experimental parameters.

B.4.1 Filename

The log file is automatically given a name which has the following format:



B.4.2 Header

The first section of each log file states clearly the parameters used for the experiment. The header is reproduced here, where the content of the orange boxes will vary depending on the specific parameters of the experiment.

THERMALLY STIMULATED CURRENT	
MEASUREMENT CONTROL PANEL	

ISU ECpE Senior Design Group May14-03	
Joshua Straquadine	Martin Andersen
Mouhamadou Diallo	Nicholas Rodriguez

Version 1.0	January 2014

Experiment Settings:	
Profile Name:	Default
Export Folder:	C:\Desktop\TSC\...
User:	Josh
Cell Type:	P3HT:PCBM
Comment:	
Charge:	During Soak
Charge Time (min):	2
Charge Voltage (V):	5
Meas. Voltage (V):	1
Temp Profile:	Basic Ramp
Ramp Rate (K/min):	5
Soak Time (min):	2

No. of Setpoints:	2
Setpoints (K):	80, 300
Output Format:	T,T,T,T,T,T,T,T,T,T,T
Sampling Rate:	1
Heater Range:	High
P:	0.2
I:	0.1
D:	10
A:	31.483
B:	0.220024
C:	0.000028
D:	0
Max Rate:	10
Min Temp:	80
Max Voltage:	20
Max Temp:	350

Run Started at 14:33 on Sunday 04 13 2014	

B.4.3 Data Format

Depending on the data output settings chosen by the user on the advanced settings pane, these columns can be excluded or included at will. All of the options, in the order in which they may appear if included, are shown below.

#	Time	Set T (K)	Cell T (K)	Cell Rate (K/min)	LS Ta (K)	Ta Rate (K/min)	LS Tb (K)	Tb Rate (K/min)	Heater (%)	RTD Res (Ohm)	V (V)	I (pA)
0	2:33:25 PM	80	299.2	0	298.65	0	297.13	0	0	1100.999	5	512.557
1	2:33:25 PM	80	299.16	-7.66	298.64	-2.3	297.1	-6.9	0	1100.87	5	489.099
2	2:33:25 PM	80	299.14	-7.84	298.63	-2.91	297.08	-7.42	0	1100.796	5	452.626

If the "Sampling" option is activated, then only lines in which the "#" column is a multiple of the chosen sampling rate will be reported in the log file, and the other data will be lost. For this reason, using a large sampling rate is not recommended, and sampling should only be used if the log files become too large to be used.

B.5 Interfacing with Hardware

The software interfaces with four different instruments:

- Keithley 6485
- Keithley 617
- Keithley 2400
- Lakeshore 331

To connect these four instruments together, GPIB stacking cables are used. The exact configuration of these cables does not matter, as long as every instrument is connected to the same daisy chain.

The software also uses a KUSB-488A USB-GPIB adapter to communicate between the computer and these instruments. Unfortunately, the drivers for the KUSB-488A adapter are written by Keithley, and are therefore not compatible with the LabVIEW built in drivers. If your computer is not recognizing the KUSB-488A:

- Uninstall the National Instruments GPIB drivers on your system
- Navigate to the Keithley website and download the newest KUSB-488A drivers
- Install the Keithley drivers on your system
- Reboot your system and try again

B.6 Still stumped?

Drop the design team a line by emailing jstraquadine@gmail.com!

APPENDIX C: Initial Designs and Design Process

C.1 Thermal Designs

C.1.1 Temperature Sensors

The Lakeshore device has two built in temperature sensors, both within the cryostat. Due to non-ideal effects of within the vacuum chamber, however, the temperature of the copper cryostat itself is not necessarily the same as that of the device under test. In order to accurately determine the temperature of the device, it is necessary to implement a method of real-time temperature measurement directly on the surface of the sample. Such a device must be small in order to minimize thermal mass, must be operational down to or beyond 80 K (-193 °C), and would ideally have a linear response with temperature. Several different technologies were considered to fulfill this need.

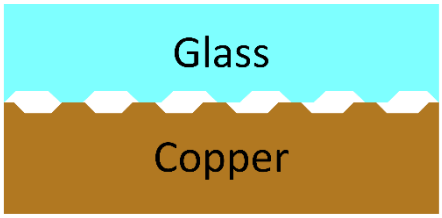
The first, and easily the cheapest solution considered was a semiconductor thermistor. These devices are readily available in easy-to-use, small packages. While most models available are designed for room temperature use, some specialized thermistors are available which operate in the required temperature regime. The major drawback to all of these devices, however, is that their temperature-resistance dependence is highly non-linear and follows the Steinhart-Hart equation:

$$\frac{1}{T} = A + B\ln(R) + C\ln^3(R)$$

While these devices would fulfill the need, however, a linear solution would be more desirable. Another, more linear technology which was considered was a thermocouple. Thermocouples consist of two dissimilar metals placed in contact with each other. When two such junctions are held at different temperatures, a measureable voltage is developed with a linear response. Thermocouples have historically been used many applications requiring a wide temperature range. We decided, however, that the requirement of a reference junction as well as the requirements of using specialized thermocouple wire to hook it up were both grounds enough to avoid this solution.

As explained in section 4.2.3, the final choice for the sensor was the PPG102A6 1 kΩ platinum RTD from US Sensor. One known drawback of all of the PPG102A6 devices is the relatively fragile platinum-nickel leads. In communicating with engineers at US Sensor and a few other companies, they said that all of their low temperature sensors come with similarly fragile leads. We looked into assorted assemblies, but the thermal mass of all of these units was far too large to be useful for our application.

C.1.2 Thermal Interface



The roughness of the cryostat means that it doesn't touch the sample that much, even though both surfaces were designed to be completely flat. And since there is no air to transfer heat through convection, and radiation is almost nothing at these temperatures, we need to create a better interface between the cryostat and the sample. Several methods have been tried.

C.1.2.1 Thermally Conductive Pads

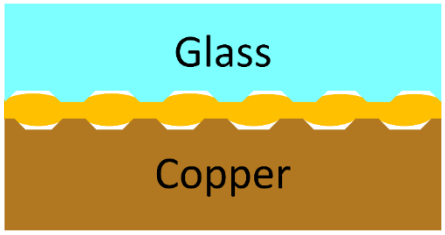
Traditionally, a thermal pad is a thermally conductive, electrically isolating pad to put between a heat source and a cooler. It is meant to replace the slightly better, but more messy thermal paste. This is easy to come by if the purpose is to get heat away from an active chip or transistor, but won't work as well with low temperatures, because the material tends to freeze.

The pads that we did use all had a soft side that looks like paste and a side covered in plastic. They are about half an mm thick.

The chart below shows the non-ideal characteristics of all the three thermal pads tested: the 5591S and 5519S from 3M and the Therm-A-Gap pad from Parker Chomerics.

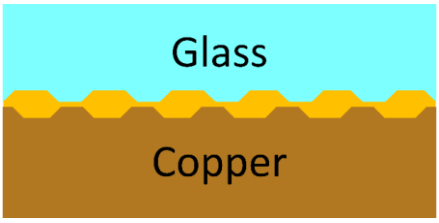
C.1.2.2 Encapsulation in Cryogenic Epoxy

Another idea was to cover the entire sample in an epoxy with high thermal conductivity. The epoxy will dry for 18 hours, the purpose is not to actually glue the sample to the cryostat. There are two sides to this idea. One is that that this saves us the mess of using grease, because the epoxy is softer and more flexible than glass (at least at room temperature, it might harden at lower temperatures). Another way to use this is to conduct heat away from the top side of the sample, around the edges and to the cryostat. While this works great with just samples of glass, it also has an undesired reaction with the solar cell. The epoxy used is a two component, low viscosity epoxy encapsulant; Stycast 1266.



C.1.2.3 Thermally conductive cryogenic vacuum grease

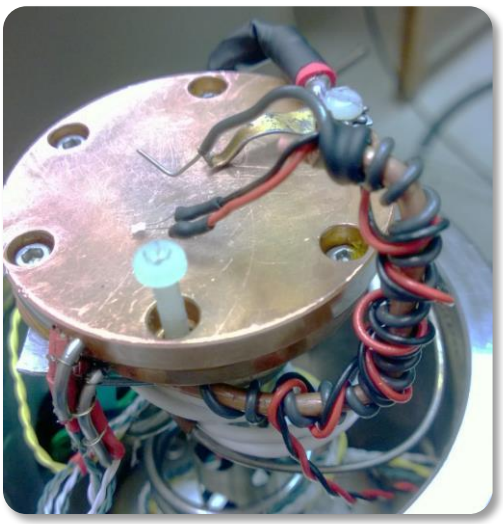
To reduce the thermal resistance from the sample to the cryostat, we need to use a thermal paste of some sort. Turns out, they are not available for temperatures down to 80 K, because the market for this isn't very big. Furthermore, it needs to be useable at very low pressure. A vacuum grease is an acceptable but not perfect substitute. This grease, Apiezon N, is designed to seal lids for container that are under vacuum. It is also designed to work down to 4 K. It isn't ideal though, because it has a thermal conductivity of only $0.194\ W \cdot m^{-1}K^{-1}$,, so that means we have to use absolutely as thin a layer as possible.

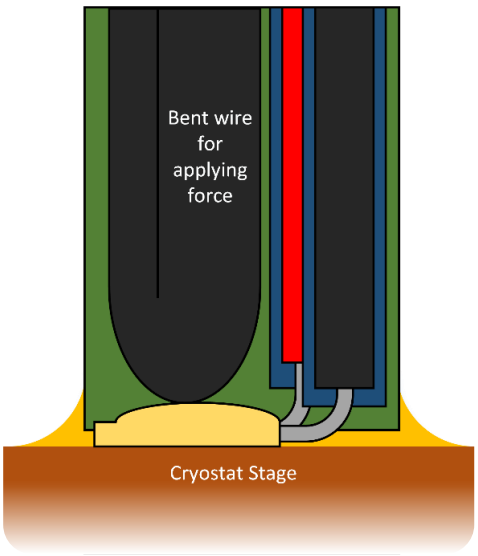


Because of the vacuum, however, thermal conductivity isn't quite as important as the thermal interface itself. Glass has approximately the same thermal conductivity as the grease, copper has up to $400\ W \cdot m^{-1}K^{-1}$, and vacuum has less than $0.002\ W \cdot m^{-1}K^{-1}$, and since the cryostat, grease and glass isn't that thick compare to the vacuum around it, nearly all the temperature difference is across the vacuum.

C.1.3 Probe Effects

The thermal mass on the end of the probe had a significant ability to warm the surface of the sample. Our first few iterations were based on a large copper wire which could be raised and lowered using a screw, but this mass was so large that the sample simply never came down to the right temperature. We also tried using a small wire to apply force to the RTD to hold it down and ensure good contact. All of these issues were doomed to failure, however, because there was simply too much material to cool efficiently. Below are some images of our unsuccessful attempts at probe design.





C.2 Electrical Designs

C.2.1 Current Measurement Instruments

Thermally stimulated current tests are fairly simple to implement because they only require monitoring of the current through the device as the temperature changes. The measurement of those currents is complicated, however by the very low amplitude of those currents. Those currents are in the range of femtoamperes, and therefore require a high resolution ammeter. One of the tasks within this design project was to determine which instrument was the best choice, while still fitting within our client's budget. The first thing which was determined was that the circuit with the ammeter must also be able to apply an excitation voltage to the cell. In order to simplify the wiring and to cut down the number of instruments required for the system, the decision was made to find a source measurement unit (SMU) capable of both sourcing voltage and measuring high accuracy current. Keithley, a division of Tektronix, is widely considered to be one of the most popular producers of SMUs.

Measuring very low currents accurately becomes a serious problem in the face of noise, coupling, and leakage. Without careful consideration of these issues, it is very likely that those very low currents would become drowned out by noise and other effects. To combat this, we determined that a triaxial connection would be the best option between the sample and the instrument. This limited us to SMUs which are designed with such triaxial hookups.

With these criteria in mind, we examined Keithley's SMU product line. All potential solutions are marked in the table. Based on the requirements of the project, we decided that the Model 2450 SMU would be the most cost effective choice. However, our budget simply could not afford any of the available SMUs, so we went back to the drawing board and decided to look for a Picoammeter instead.

Model	Min I	Max I	Min V	Max V	Read/sec	I Acc.	V Acc.	Chan.	Banana	Price
2634B	1 fA	10 A	100 nV	200 V	20K	0.020%	0.015%	1	No	$ 11,400
2635B	0.1 fA	10 A	100 nV	200 V	20K	0.020%	0.015%	1	No	$ 9,200
2602B	100 fA	10 A	100 nV	40 V	20K	0.020%	0.015%	2	Yes	$ 9,060
2612B	100 fA	10 A	100 nV	200 V	20K	0.020%	0.015%	2	Yes	$ 9,060
2601B	100 fA	10 A	100 nV	40 V	20K	0.020%	0.015%	1	Yes	$ 6,180
2611B	100 fA	10 A	100 nV	200 V	20K	0.020%	0.015%	1	Yes	$ 6,180
2604B	100 fA	10 A	100 nV	40 V	20K	0.020%	0.015%	2	Yes	$ 7,260
2614B	100 fA	10 A	100 nV	200 V	20K	0.020%	0.015%	2	Yes	$ 7,260
6430	10 aA	0.105 A	1 uV	200V	256	0.035%	0.012%	1	No	$ 13,000
2450	10 fA	1.05 A	20 mV	200 V	3,100	0.020%	0.012%	1	Yes	$ 5,400

C.3 Software Designs

Our first attempts at building the LabVIEW Dashboard took on a very linear approach, not unlike a single-threaded procedural program such as one might write in C or some other similar language. This system did produce the basic functionality, but was not very responsive to input nor flexible or fast in operation. This is because the code did not take full advantage of LabVIEW's inherently parallel architecture, nor did it use the highly interactive event-based model to enable quick responses. As additional features and flexibilities were added to this system, it became more and more ad hoc, and worked less and less well, so the second version started over completely from scratch to solve these issues.