Low Cost System for Test of Thru-Plane Thermal Transfer Coefficient

by

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ABSTRACT

Determining the thermal conductivity of carbon gas diffusion layers used in hydrogen fuel cells is a very active topic of research. The primary driver behind this research is due to the need for development of proton exchange membrane fuels with longer usable life cycles before failure. As heat is a byproduct of the oxygen-hydrogen reaction an optimized pathway to remove the excess heat is needed to prevent thermal damage to the fuel cell as both mechanical and chemical degradation is accelerated under elevated temperatures. Commercial systems used for testing thermal conductivity are readily available, but are prohibitively expensive, ranging from just over $10,000 to $80,000 for high-end systems. As this cost can exclude some research labs from experimenting with thermal conductivity, a low cost alternative system is a desirable product. The development of a low cost system that maintained typical accuracy levels of commercials systems was carried out successfully at a significant cost reduction. The end product was capable of obtaining comparable accuracy to commercial systems at a cost reduction of more than 600% when compared to entry level commercial models. Combined with a system design that only required some basic fabrication equipment, this design will allow many research labs to expand their testing capabilities without straining departmental budgets. As expected with the development of low cost solutions, the reduction in cost came at the loss in other aspects of system performance, mainly run time. While the developed system requires a significate time investment to obtain useable results, the system can be improved by the used of RTDs in place of thermocouples or incorporation of an isothermal cold plate. These improvements would reduce the runtime to less than that of a standard work day while maintaining an approximate reduction in cost of 350%.

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CHAPTER 1

INTRODUCTION

At the beginning of this project, it was determined that developing the capability of testing the thermal conductivity of carbon gas diffusion layers (GDLs) was a very desirable prospect for use in proton exchange membrane fuel cell (PEMFC) optimization work that was being conducted at the Arizona State University Polytechnic campus. The reasoning for developing this capability is that additives applied to GDLs, such as polytetrafluoroethylene (PTFE), have been found to reduce the thermal conductivity of GDLs in-both the thru plane and in-plane directions (Zamel, 2011) (Sadeghifar, 2013). Interestingly, it has also been shown that PTFE treatments may increase the thru-plane thermal conductivity of the GDLs at low compression loadings and decrease thermal conductivity as the load increases (Karimi, 2010). Characterization of the thermal conductivity of GDLs is a critical design requirement for future development in order to extend the usable lives of PEMFCs to a commercially viable level as high temperatures and local hot-spots accelerate degradation.

PEMFC are constructed as a symmetrical sandwich centered about a proton exchange membrane (PEM). Moving towards the outside layer of the PEMFC is the catalyst layer. The catalyst layer consists of carbon nanotubes with Pt nanoparticles distributed throughout to act as a catalyst. This layer is followed by the GDL which is used to deliver ether hydrogen gas or oxygen/air depending on if the anode or cathode side of the PEMFC is being used. The final layer of the PEMFC is the bipolar plate. This bipolar plate is used to transfer the load generated at the catalyst layer to be utilized and contains channels to facilitate delivery of the reactants and transportation of the byproducts, water and heat, away from the PEMFC. A diagram of a typical PEMFC may be seen in figure 1.

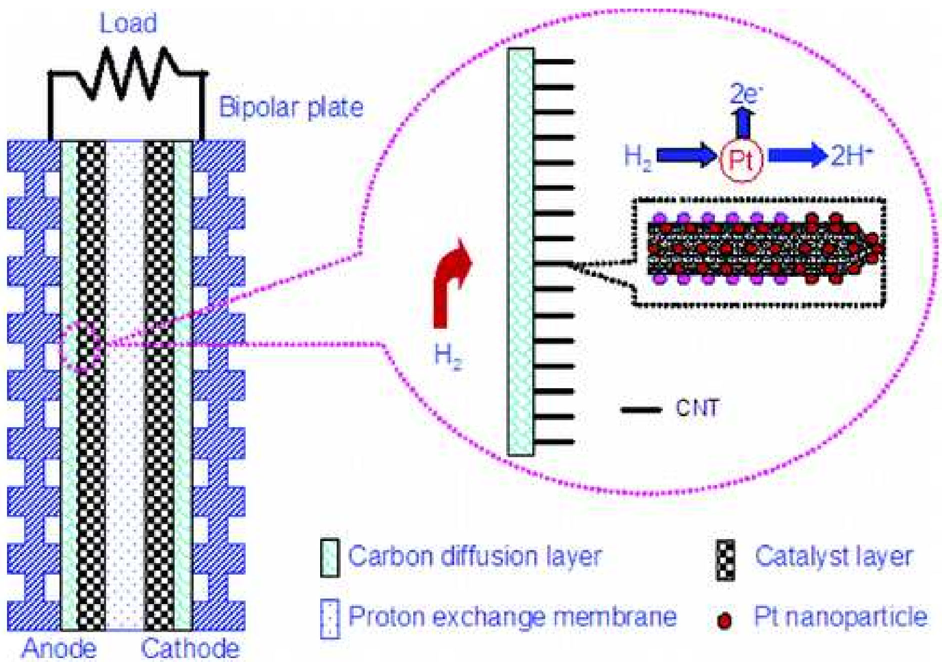


Figure 1. Construction of a typical PEMFC (Wang, 2004)

Over the lifetime of a PEMFC, degradation of performance can be found for a multitude of causes in each of the follow layers: the PEM, Catalyst layer, and the GDL. Causes of degradation and failure can be broadly defined into two categories that are chemical or mechanical in nature, both of which are accelerated by an increase of temperature within the PEMFC (Zhang, 2006). As the bipolar plate is utilized to carry the majority of the heat produced within the PEMFC away to be dissipated, increasing ability for the GDL to transfer heat to the bipolar plate should be expected to increase the usable life of the PEMFC. It has been shown that due to compression between the GLD and the bipolar plate, hotspots that accelerate the degradation of the fuel cell in these areas can form (Hottine, 2006).

Complete failure of the fuel cell typically occurs in the PEM due to development of macroscopic pin-hole and tear development, resulting in excessive gas transfer between the anode and cathode of the PEMFC (Huang, 2006). These tears and pin-holes develop over the course of cyclic usage of the PEMFC. It has been shown that under typical operating parameters the yield strength of the PEM is typically exceeded, resulting in plastic deformation of the PEM and thinning once the PEMFC is shut down. Typical operating parameters under which the PEM's yield strength is exceeded may also induce tensile stresses into the PEM (Kusolgu, 2006). As the PEM thins, performance of the PEMFC is expected to decline due to addition mass transportation occurring (Rama, 2006) (Seddiq, 2006). Where local hotspots exist within the PEMFC, it is anticipated that development of tears and pin holes will occur due to the reduction in break strength of the PEM (Tang, 2006). Development of the failure mechanism described above can typically be observed around 1000 hours of runtime on a PEMFC (Liu, 2006).

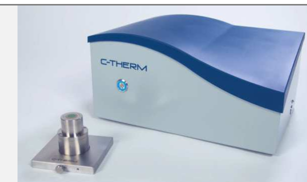
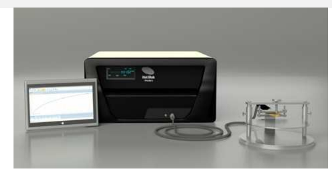
Degradation can also occur in the catalyst layer and the GDL itself. Within the catalyst layer, it has been observed that over time Pt nanoparticles increase in size and dispersion of the particles decreases (Zhang, 2006). The cause of this is due to dissolution of the Pt nanoparticles followed by re-disposition of particles within the catalyst layer, a chemical process that is accelerated at elevated temperatures (Zhang, 2006). As for degradation of the GDL, this is typically caused by oxidation of the carbon paper used. At elevated temperatures, the formation of carbon monoxide and carbon dioxide occurs causing mechanical failure of the GDL (Zhang, 2006).

With thermal characteristic having such impact on the long term operation of PEMFCs, it is critical to develop and optimize parameters of GDLs for maximal thermal transfer. Commercial systems for testing the thermal conductivity of materials are readily available from a multitude of manufacturers. The main barrier of entry to obtaining such a system is the initial price point. Entry level systems start around $10,000 and upper end testing systems range in cost starting from $80,000. System specifications may be seen in Table 1 on page 6. The specifications shown were tabulated using published information available from the manufactures with the expiation of price, which was obtained from correspondence with the various manufactures which can be seen in Appendix C. Due to this price point, it may be difficult for some research labs to expand and introduce the ability to test thermal conductivity without writing the purchase of equipment into a research grant or departmental approval. In addition, many commercial systems on the market today lack the ability to apply a compressive load to the specimen being tested. As it has been widely documented that the thermal conductivity of GDLs is dependent on the applied pressure, it is of critical importance that the ability to test under varying loads is available (Hamour, 2011) (Zamel, 2011) (Sadeghi, 2011). This bars some of the lower cost entry level models from being utilized for GDL testing. It should also be noted that at a point, additional pressure on the GDL with in the PEMFC will have a negative impact on overall performance (Ge, 2006).

Considering the expense associated with the procurement of thermal conductivity testing equipment, an alternative low cost solution is desirable to allow for additional research into thermal optimization of PEMFCs. This document will evaluate possible solutions to this issue by attempting to construct a simple, low cost table top alternative system. In this document possible test methods will be outlined, and requirements for constructing and utilizing such a system will be described along with experimental results developed using the final product.

Table 1

Commercial Thermal Conductivity Test System Specifications

Manufacture	*Thermal Conductivity test range*	*Temperature testing range*	*Sample Limitations*	*Testing Specification*	*Test run time*	*Price Range*	*System Photo*
C-Therm Technologies Ltd	0 to 500 W/mK, Uncertainty better than 5%, typically 1%	-50º to 200ºC (Standard Sensor), 300 ºC option available	None, unlimited sample size	Modified Transient Plane Source (Conforms to ASTM D7984)	0.8 to 3 seconds	$30,000 to $40,000	
Thermtest	0.005 to 1800 W/mK, Uncertainty better than 5%, typically 2%	With Kapton sensor (-160ºC to 400ºC) With Mica sensor (up to 1000°C)	Smallest:0.01 mm Thick, 2 mm Diameter or Square No upper limit	Transient Plane Source (ISO/DIS 22007-2.2)	0.1 to 1280 seconds	$15,000 to $80,000	
Hukseflux Thermal Sensors B.V.	Unpublished, 3%-6% uncertainty	-30 to +120 °C	preferred: 70 x110 mm, always > 50 x 50 mm, 0.1 - 6 mm Thick	Thin Heater Apparatus ASTM C 1114-98	Unpublished	$11,475 to $20,985	

CHAPTER 2

AVALABLE METHODS FOR THERMAL CONDUCTIVITY TESTING

Depending on the material being researched, there are various methods for testing the thermal conductivity ranging from commercial methods to systems specified by industrial standards published by the American Society for Testing and Materials (ASTM). As commercial suppliers do not readily provide specific details on how exactly their systems work, ASTM specifications for testing of thermal conductivity were primarily used for selection of a method for testing of materials. Documentation supplied by commercial entities was primarily used for comparison of system performance.

The first ASTM method evaluated for utilization in development of the low cost thermal conductivity was C111M-09 "Standard Test Method for Thermal Conductivity of a Refractories by Hot Wire (Platinum Resistance Thermometer Technique). This test method uses a platinum wire embedded into the test sample with a constant applied voltage that acts as a heating element. By measuring the rate at which the platinum wire increases in temperature, the thermal conductivity of the sample surrounding the wire can be calculated using Fourier's Law. This test method is suitable for materials with conductivities below 16 W/mk (ASTM C113M-09) which is suitable for GDLs when looking at the expected thermal conductivity. However, there are two major downsides that would prevent this method from being used for testing of GDLs from the perspective of a low cost system. The initial concern is that the requirement of using a platinum wire as the heating element. While it may be possible to use this method and still be under the cost of a commercial system, the cost of the platinum wire is in direct conflict with the goals and objectives for the development of a low cost system. The largest issue with using this test method is due to the anisotropic nature of GDLs. Fibrous materials introduce significant errors in the thermal conductivity as stated in section 1.5 of ASTM C1113M-09 (2013).

After the determination was made that ASTM C1113M-09 was not suited to the goals and objectives of this development cycle, another ASTM specification was found and evaluated. ASTM E2584-14, Standard Practice for Thermal Conductivity of Materials Using a Thermal Capacitance (Slug) Calorimeter was initially found to be a very promising method for testing of GDLs. This process uses an AISI 304 Stainless steel calorimeter sandwiched between samples that are being tested. A diagram of the test set up may be seen in Figure 2 seen below.

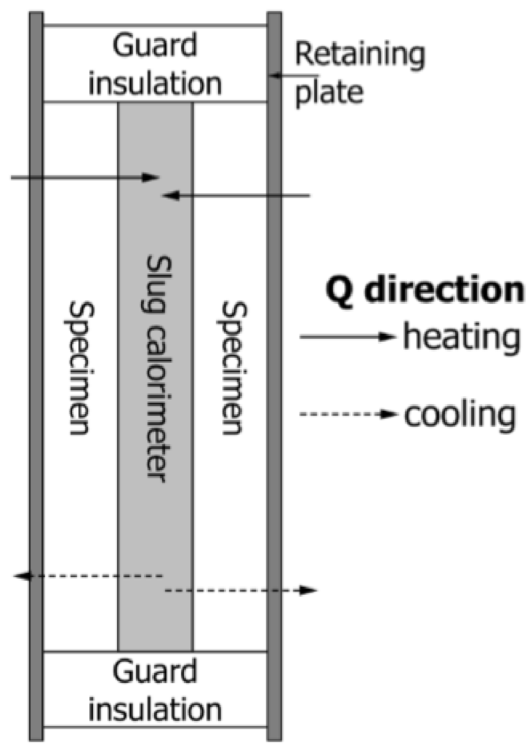


Figure 2. Test setup outlined in ASTM E2584-14 (2014)

By applying a uniform heat source to the sandwich stack and measuring the temperature increase in the calorimeter, a heat flux can be calculated by using the known thermal capacitance of the AISI 304. From this point, Fourier's law is applied to determine thermal conductivity. This test method is suitable for materials with a thermal conductivity between .02 W/mk and 2 W/mk (ASTM E2584-14, 2014). The only undesirable requirement of this specification is the limitation on the compressive load applied to the tested materials. A maximum torque of 1 kg-m may be applied to the screws may be used to hold the test sandwich together (ASTM E2584-14). This may result in thermal resistance due to the porosity of GDLs dominating the actual thermal conductivity and removes the ability to adjust the compressive load on the samples.

Further research identified ASTM E1225-13, Standard Test Method for Thermal Conductivity of Solids Using the Guarded-Comparative-Longitudinal Heat Flow Technique, as a prime candidate for the method utilized in development of this low cost thermal conductivity test bed. This method uses two heat flux gauges with a test sample compressed between them. This test stack is surrounded by an insulator to minimize heat losses. Reference Figure 3 below for a diagram of the test setup describe by ASTM E1225-13.

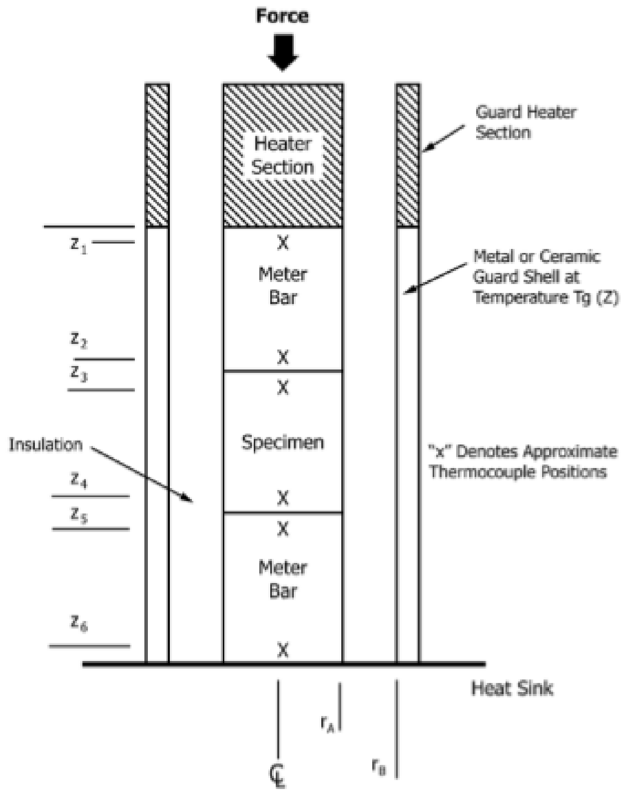


Figure 3. Test setup describe by ASTM E1225-13 (2013)

Once side of the test stack is heat and the other cooled to create a temperature gradient. After letting the system reach a steady state, Fourier's Law is applied to determine the thermal conductivity of the sample. This test method can be used for a wide range of thermal conductivities, from .2 W/mk to 200 W/mk, and a wide range of temperatures, from 90 k to 1300 k (ASTM E1225-13, 2013). Taking this into consideration and the fact that a compressive load can be easily adjusted, it was determined that this standard is best suited for testing the thermal conductivity of GDLs. One specific section of this standard worth noting is 5.2 which reads as follows:

> Proper design of a guarded-longitudinal system is difficult and it is not practical in a method of this type to try to establish details of construction and

> procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, temperature measurements, and general testing practices. Standardization of this test method is not intended to restrict in any way the future development by research workers of new or methods or improved procedures. However, new or improved techniques must be thoroughly tested. (ASTM E1225-13, p. 2)

This statement shows that there is value and need for documenting the development of a test bed that is compliant with this specification.

Available commercial systems are typically compliant with the specifications mentioned previously or other specifications depending on what material is to be tested with the equipment. One such manufacture of thermal conductivity testing devices is C-Therm Technologies. Equipment offered by this company comes with the test apparatus and software that performs all necessary calculations for the user. The user only needs to ensure that all proper steps are followed when setting up the equipment. The performance of systems supplied by C-Therm can be seen in the table below.

Table 2

C-Therm system specifications. (Simplifying Thermal Conductivity (k) [Brochure], n.d.)

C-THERM TCI SPECIFICATIONS	
Thermal Conductivity Range	0 to 500 W/mK
Test Time	0.8 to 3 seconds
Minimum Sample Testing Size	17 mm diameter
Maximum Sample Testing Size	Unlimited
Test Method	Modified Transient Plane Source (MTPS)
ASTM	D7984
Minimum Thickness	Nominally 0.5 mm, dependent on thermal conductivity of material
Maximum Thickness	Unlimited
Temperature Range	-50 °C to 200 °C With option to extend to 500 °C
Precision	Typically better than 1%
Accuracy	Better than 5%
Extra Hook-Ups Required	None
Software	Intuitive Windows®-based software interface. Easy export to Microsoft Excel®. Additional functionality offers indirect, user-input capabilities for a number of other thermo-physical properties including: • Thermal Diffusivity • Heat Capacity • Density
Input Power	110-230 VAC 50-60 Hz
Certifications	FCC, CE, CSA

Correspondence with C-Therm about the cost of these systems indicated the cost of such equipment runs from $30,000 to $50,000 depending on the desired system. This correspondence can be found in Appendix C along with other correspondence with other manufacturers of testing equipment that show a cost ranging from $10,000 to $80,000.

CHAPTER 3

SYSTEM DEVELOPMENT

System Properties

When system development was first initiated, the primary intent was to test carbon gas diffusion layers for use in hydrogen fuel cells. With this in mind, research was conducted into what ranges of temperatures and pressures were typically investigated for GDLs. It was also determined from the initial program development that having the possibility to vary the areas of the specimen was of importance for the final configuration. Other limitations were established, including the limited funding available from Arizona State University. The system was intended utilize as much existing equipment as possible, and the final system was to follow published specifications on how to perform thermal conductivity testing. As stated in Chapter 2, ASTM 1225-13 was selected for this purpose.

It was determined that temperature ranges are typically limited to the operating ranges of hydrogen fuel cells. Once a cell passes an internal temperature of 100 °C, materials and membranes within the cell begin to sustain damage. Based off of this information a factor of safety of 1.2 to the maximum operating temperature was applied in order to develop a target minimum design temperature of 120 °C. The reason for selecting this as a minimum temperature rather than a maximum is to allow for the system to be utilized for other materials at a later date should interest in performing such studies develop.

The second design parameter to be developed was the target pressure for testing of the GDLs. Various sources were used for development of a target pressure, including research papers and industry specifications for testing thermal conductivity. Typical journal papers on the topic of GDL thermal conductivity tested at 1.4 Mpa (Sadeghifar, 2013) or below. In contrast, the ASTM specification, ASTM D5470-95 (2001), reports a minimum test pressure of 3.0 Mpa at the sample to reduce any additional thermal resistance due to interface between the apparatus and the test sample. As there was interest in utilizing this system for more than just GDLs, 3.0 Mpa was selected as the minimum pressure the system should be able to apply. It should be noted that testing of GDLs has been conducted at pressures as high as 5.5 MPa (Nitta, 2008), so the ability to apply compressive loads beyond the 3.0 Mpa target is acceptable.

As the effective pressure at the sample is dependent on the area of the sample, the size of the sample holders was defined to have a maximum diameter of 1" and a minimum diameter of .5". With this defined, it was possible to determine that the minimum compressive force to be applied to the test column was 1500 N to provide the established target pressure.

System Configuration

With the target testing parameters defined, system development could proceed. To meet the requirements of ease of fabrication and utilization, a method similar to the test apparatus outlined in ASTM E1225-13 was selected. Two heat flux gauges would be fabricated and the sample would be placed in between the gauges. To allow for variable sample areas, the flux gauges would have pockets machined on the inside faces to allow for installation of a small aluminum disk. This disks would be the surfaces that contact the sample and would easily be removed and replaced with disks of different sizes. In contrast to ASTM E1225-13, multiple thermocouples would be used along the length of the flux gauges as opposed to two per flux gauge to reduce the amount of error in the heat flux calculations. One flux gauge would have a heating element installed and the other would be placed upon a heat sink to generate the required heat flux. Another distinction between ASTM E1225-13 and this method is that the physical dimensions of the samples to be tested are not held to be identical in dimension to the flux gauges.

To apply a compressive load to the test stack, flux gauges and sample, a screw would be used to apply the necessary load and the actual compressive force would be measured by using a load cell. One limitation of this design selection is that the load will not automatically adjust for account for increases due to thermal expansion. A method for accounting for this thermal expansion can be seen in by utilizing a computer controlled linear actuator to apply a compressive force Culham (2002). However, to avoid the cost incurred by the use of a computer controlled actuator, this short-coming can easily be addressed by setting the load just under the target as it heats up. Once at the desired test temperature, a final adjustment can easily be made. Reference Figure 4 for a photo of the constructed test stack.

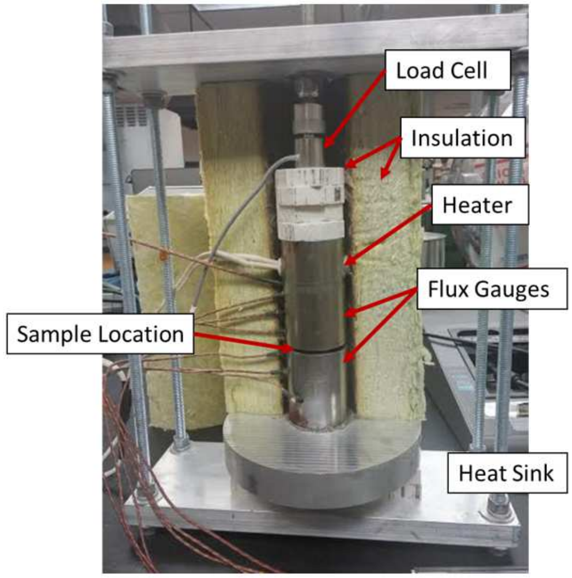


Figure 4. Photo of constructed test stack.

The next component of the system to be selected was the heating element. Sizing of the heating element was determined using Fourier's Law of heat flux to determine the minimum wattage the heating element needed to produce. To perform this calculation, the following parameters were used: an assumed thermal conductivity of .5 W/mk which is slightly greater than reported thru-plane conductivity values reported for GDLs, a sample thickness of .0005 meters to represent a sample consisting of three GDLs laid upon each other, the area of .0005 m2 as that is the largest size sample to be used in the system, and finally the temperature gradient was assumed to be the maximum test temperature minus room temperature conditions resulting in a gradient of 90 K. Such an unrealistically large temperature range was selected to provide a conservative estimate of the required heating element output. The 90 K temperature gradient was taken with consideration for the requirement that the cooler flux meter needed to be at room temperature and the hot flux meter needed to be at the minimum design temperature. By the application of Fourier's Law of heat conduction, this resulted in a required output of 45 Watts for the heating element. Upon review of available heating elements on the market, it was determined that there is no significant price difference from a 50-watt element to a 100-watt element. For this reason, a 100 Watt 120 Volt AC cartridge heater was selected. The extra available wattage will allow for the system to be used for a wider range of materials rather than just carbon GDLs.

With the major components selected, the remaining system components were chosen. For construction of the heat flux gauges, 1012 steel was selected for its low cost, high machinability, and reasonably well documented thermal conductivity to allow for accurate calculation of heat flux. An alternative material that can be used as an in place of the 1012 steel would be Austenitic Stainless steels such as 304 as recommended by ASTM E1225. The tradeoffs for selecting the stainless over the 1012 steel would be a small increase in cost, a significant increase in difficulty of machining, and a slight increase in the accuracy of the thermal conductivity of the material. For the heat sink and frame, 6061-T6 aluminum was selected for its low cost and superb machinability. The remainder of the frame was assembled using 3/8-16 by 2-foot-long all-thread rod for ease of assembly. A major consideration for safety of the system was that all materials that would come into contact, or at least close proximity to the test stack. These materials would need to hold a high temperature rating to mitigate any fire risk. The risk items identified were the insulation materials and the thermocouples. Insulation materials included mineral wool and ridge calcium silicate insulation, both of which are suitable for temperatures up to 1200 degrees Fahrenheit and 1700 degrees Fahrenheit respectively. Thermocouples used in the test stack utilize a fiberglass insulation that permits them to be used up to a maximum temperature of 800 degrees Fahrenheit without issue.

A full set of blueprints detailing the configuration, materials use, and assembly may be seen in appendix A of this report. The lab provided components used in this build are a Watlow Series 808 temperature controller, a Circuit Specialist CSI3010X DC power supply, a FLUKE 8842A multi-meter, and a AccSense VersaLog Model TC data logger. Design of the blueprints and fabrication of the test bed was assisted by the use of a computer model of the system which can be seen in figure 5.

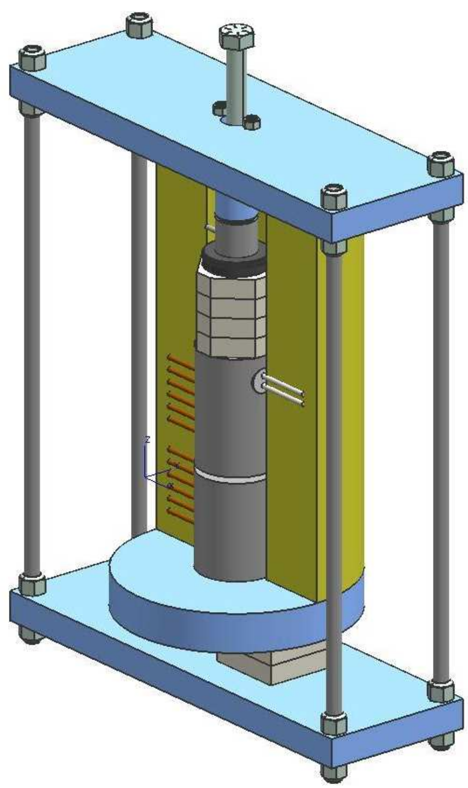


Figure 5. Computer model of testbed.

Usage Instructions

Prior to testing, the user of the system is to obtain a copy of the fabrication document located in Appendix A of this paper. The user is to ensure that all details of the system are available and undamaged. Should a detail be damaged, it is to the user's discretion if it is possible to repair the damaged detail, or replace it in its entirety. Critical features to be inspected are as follows: Sample holder surface finish, Thermocouple wire shielding damage, damage to the heating element, and damage to the load cell.

Should it be necessary to replace any detail of the system, the system user is to ensure that proper documentation of replacement details is provided. This documentation is to include actual measurements of features that will have an impact on the measurements obtained by the system. These include features such as thermocouple spacing, sample holder thickness and diameter, heating element size, etc. It is critical to verify that replacement materials are suitable for temperature ranges that are to be tested in order to mitigate fire risks.

Once all details have been located, inspected, and determined to be acceptable, the user is to assemble all details per the fabrication document. Apply thermal paste at locations specified by the fabrication document. Once assembled, wire the load cell to a 10 V DC power supply and a multi-meter capable of readings between 0.00 mV to 20.00 mV or better per manufacturer's instructions. Apply compressive load to the test stack with no sample present until the load cell shows a minimum reading of 12 mV and let the system stand as is for a minimum time of 30 minutes. This forces air pockets trapped by the thermal paste out from test stack interfaces. Reference the visual work instructions contained in

Appendix B for additional information regarding compressive load application. Once the above steps have been completed, testing with the system may begin.

Testing Process

The system user must first define the properties with which they would like to test the samples of interest. These properties include temperature, pressure to the sample, and sample area. Once the values of interest have been determined, the user is to develop a baseline system performance with no sample present in the test stack. Apply the desired load to the test stack, set the power supply to the desired temperature, and set the data acquisition system to collect at a minimum 24 hours of data with a low sample time interval. A sample time interval of 5 to 10 seconds is recommended. Install the insulation around the test stack and secure it in place using twine or string wound around the insulation.

The system user must then verify that the desired temperature at the sample interface has been achieved by first downloading and averaging the readings of each thermocouple over the duration of the run once a steady state has been achieved. The user is to use proper engineering judgement to determine when steady state has been achieved. Reference Figure 6 for an example of what data to select for system steady state. The user is to then take an average of the two thermocouples nearest the sample interface to determine the temperature at the sample interface. If the desired temperature has not been achieved, adjust the temperature of the power supply up or down as required and re-run the system for another 24 hours.

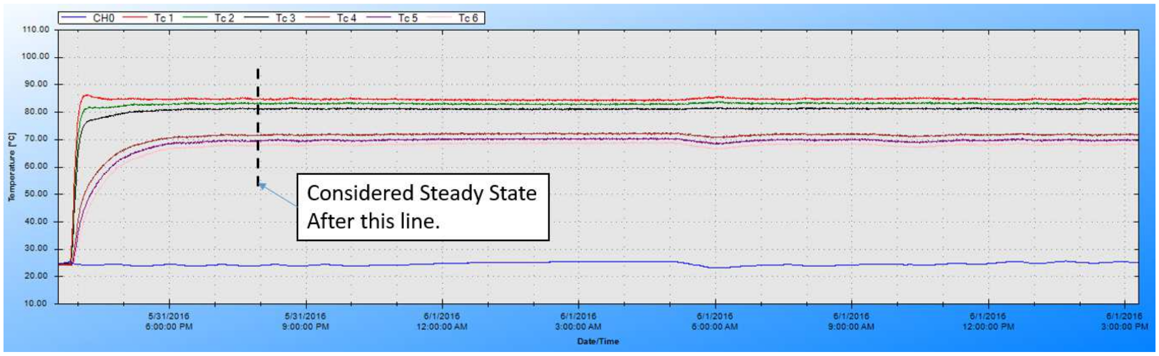


Figure 6. Steady State Output Example

After one baseline run is collected, the system user shall shut off the power supply, remove the insulation, release the compressive load, and let the test stack cool to ambient temperatures. Once cooled, the system user is to reset the system and collect another baseline run. Reference the visual work instructions contained in Appendix B for a demonstration of how to set the system up for testing. It is recommended to collect a minimum of five runs prior to performing any necessary analysis; however, if schedule does not permit sufficient time for multiple runs, it is possible to obtain a useable measurement with a larger uncertainty. The system user will need to exercise engineering discretion when reducing the number of sample runs.

After the baseline data has been collected, the system user is to calibrate the system by using a certified material with well documented thermal conductivity tables. It is preferable to select a calibration sample that is similar in thermal conductivity to the anticipated thermal conductivity of the material that is under investigation. It is required that the sample to be a solid isotropic material to allow for proper preparation of the calibration sample. The calibration sample needs to be the same diameter as the sample holders installed in the system and shall have a surface finish of .41 microns. The same process is to be used during calibration for collection of multiple experimental runs.

After system calibrations are complete, the user may move forward with testing of the sample material of interest. Depending on the type of sample being tested, it is strongly advised that, if possible, it is prepared in a similar manner as the calibration sample. This requires the same sample finish of .41 microns or better on both faces of the sample. Should the sample in question not be suited for such a surface finish—for example, if the material is highly porous—then surface finish requirements do not apply. Should the sample be flexible, such as carbon GDLs, then the flatness requirements do not apply. It is the responsibility of the user to determine if the sample will show a non-negligible deflection at the target compressive load. Should a non-negligible amount of deflection be anticipated, the user shall account for this deflection by calculation using available material properties or empirically by use of precision height gauges and feeler gauges to determine the amount of deflection after the load has been applied. It is to be expected that empirical measurements utilizing height gauges will provide a better level of accuracy and is the preferred method of calculation. The operator is to document the thickness of the sample prior to the test, the amount of compression achieved at the testing load, and the sample thickness after test conclusion. The process for testing the samples is to follow the same procedure as the baseline development. Once all the required data has been collected, the operator may move onto analyzing the data to determine the thru-plane thermal conductivity of the test sample.

The operator is to go through each data file for every run of the baseline, calibration, and sample test and select the time frame in which the system was running at a steady state. The operator is to then copy all data into an Excel file for analysis. Each run is to be located on its own tab, and be named accordingly. It is advised that the raw data file source is referenced within each data tab. Next, calculations for the following of each thermocouple reading within the run tab should be conducted: total number of data points for each thermocouple, average reading of each thermocouple, and the standard deviation of each thermocouple. Once this is calculated, the operator is to verify that the readings of the thermocouples are normally distributed as this is a critical assumption for the method used of calculating the error on the mean reading of each thermocouple. Using this data, the operator may now calculate the error on the mean of each thermocouple using the equation below:

$$E_{tc} = \sigma_{tc}\sqrt{\frac{1}{n_{tc}}} \tag{1}$$

Where σ_{tc} is the standard deviation of the thermocouple and n_{tc} is the number of samples collected for that thermocouple. This equation will report a percent error about the mean reading of the thermocouple. Multiply the average thermocouple reading by this mean error to produce the +/- error about the mean reading. For a test run, this +/- error should be .2 degrees Celsius or lower to be considered acceptable. A sample portion of this Excel spreadsheet set-up may be seen in Figure 7.

	A	B	C	D	E	F	G	H
1		The below data was taken from Carbon Samples Run 1 70-3.0-1.0						
2								
3	Time Stamp	Room Temp	Tc 1	Tc 2	Tc 3	Tc 4	Tc 5	Tc 6
4	6/14/2016 19:56	24.09	80.65	79.69	77.82	53.24	51.46	50.12
5	6/14/2016 19:56	24.1	80.69	79.25	77.61	53.15	51.67	49.93
6	6/14/2016 19:56	24.09	80.79	79.4	77.96	52.87	51.58	50.2
7	6/14/2016 19:57	24.1	80.72	79.44	77.94	53.14	51.42	50.02
8	6/14/2016 19:57	24.1	80.83	79.53	77.86	52.88	51.29	50.19
9	6/14/2016 19:57	24.1	80.74	79.27	77.87	52.77	51.58	49.91
10	6/14/2016 19:57	24.11	80.56	79.57	77.79	53.18	51.28	50.21
11	6/14/2016 19:57	24.11	80.71	79.71	78.06	53.06	51.49	50.21
12	6/14/2016 19:57	24.11	80.91	79.46	77.79	53.14	51.63	50.33
13	6/14/2016 19:57	24.11	80.94	79.19	77.77	53.04	51.18	49.9
14	6/14/2016 19:57	24.12	80.72	79.45	77.86	53.17	51.41	49.93

	I	J	K	L	M	N	O	P	Q
3			Average	Std Dev	Mean Error	+/- Error			
4		Room Temp	24.17	0.23	0.19%	0.05			
5		Tc 1	80.75	0.15	0.12%	0.10	Number of Samples		14184
6		Tc 2	79.38	0.14	0.12%	0.09			
7		Tc 3	77.84	0.13	0.11%	0.08			
8		Tc 4	53.17	0.16	0.14%	0.07			
9		Tc 5	51.58	0.16	0.13%	0.07			
10		Tc 6	50.18	0.18	0.15%	0.07			

Figure 7- Sample Set-up for Calculating Thermocouple Values.

With the nominal values determined for the thermocouple reading for each experimental run, the next step the operator must complete is the determination of the existence of statistical outliers. This is done by setting up a table for all runs of each category: baseline, calibration, and sample test. Once the table is created, the operator shall calculate the average value and the interquartile range for each thermocouple in the flux gauges. Once these values are calculated, the outlier bounds are calculated using the following equation:

$$Outlier\ Boundary = \overline{Tc} \pm 1.5IQR_{Tc} \tag{2}$$

Where $\overline{Tc}$ is the average reading of the thermocouple across all test runs and IQR_{Tc} is the interquartile range of the data set. If any runs show a thermocouple reading beyond the outlier boundary, the operator is to review their notes to attempt to identify the cause of the errant reading. This erroneous reading is to be omitted from further calculations. Reference Figure 8 for an example Excel set-up for this step of the analysis. The outlier matrix shown in the figure utilizes an *If* statement that will return a “1” if the corresponding data set exceeds the outlier boundary and a “0” if the reading is within the boundary. As a note, additional runs beyond the required 5 will significantly increase the fidelity of outlier identification.

ABS Runs	Run 1	Run 2	Run 3	Run 4	Run 5	Mean	IQR	Outlier Matrix	Run 1	Run 2	Run 3	Run 4	Run 5
Room Temp	24.54	24.00	24.89	24.58	24.14	24.43	0.660	Room Temp	0	0	0	0	0
Tc 1	84.16	80.54	80.41	80.69	80.02	81.16	2.211	Tc 1	0	0	0	0	0
Tc 2	83.54	80.14	79.96	80.12	79.39	80.63	2.162	Tc 2	0	0	0	0	0
Tc 3	83.07	79.23	79.14	79.29	78.67	79.88	2.270	Tc 3	0	0	0	0	0
Tc 4	40.91	41.56	42.24	42.14	41.94	41.76	0.953	Tc 4	0	0	0	0	0
Tc 5	40.24	40.84	41.54	41.42	41.23	41.05	0.937	Tc 5	0	0	0	0	0
Tc 6	39.61	40.13	40.88	40.75	40.54	40.38	0.949	Tc 6	0	0	0	0	0

Figure 8 – Sample Outlier Identification Set-Up

Once the outliers have been identified, the next phase for the operator to complete is calculation of the heat flux traveling through the system. This is done by tabulating the results of each run not identified as an outlier to the thermocouple's position within the heat flux gauge. Once completed, a linear regression of the data is to be developed. The resulting equation is to be used to determine the temperature difference from the top of the heat flux gauge to the bottom the flux gauge. As the area of the heat flux gauge and the length are known, the remaining value to be determined is the precise thermal conductivity of the 1012 steel used to fabricate the flux gauges as the thermal conductivity is a function of the material temperature. By using tabulated data that shows the thermal conductivity and the corresponding material temperature, a simple linear interpolation is to be conducted using the average of all thermocouple readings installed in the flux gauge. With this information, a simple calculation of unidirectional Fourier's Law produces the heat flux through the flux meter.

$$Q = -kA\left(\frac{dT}{dx}\right) \tag{3}$$

The results of the calculated heat flux through the upper and lower flux meters are to be compared, as any significant discrepancies between the two indicate a problem with the data requiring further investigation. If the two calculated heat fluxes are in agreement with each other, the two values are to be averaged to produce the heat flux through the sample interface.

Calculation of a calibration factor is to be performed at this point. The operator is to select a material with well-known thermal conductivity properties and that is readily available. For reference, the materials listed in Table 1 of ASTM E1225 may be used as a suitable starting point for the selection of a calibration material. Once a material has been selected, it is to be tested as specified earlier in this document. To calculate the calibration factor, the operator shall first calculate the sample temperature by averaging the all average thermocouple readings located adjacent to the sample interface. With this value obtained, the operator calculates the expected thermal conductivity of the sample by the method deemed most appropriate based upon the source data, such as linear interpolation for tabulated data. To proceed, the operator shall apply Fourier's Law of Heat Conduction using the calculated heat flux, physical sample dimension, and the calculated thermal conductivity of the sample to determent the expected temperature gradient. The operator then determines the actual temperature gradient by comparing the baseline data to the calibration data to find a measured change in temperature. The final step in this process is to subtract the anticipated temperature gradient from the expected to produce a calibration factor. This factor will be applied when calculating the thermal conductivity of the sample of interest to account for fabrication and set-up errors inherent to the system.

The only two steps remaining at this point are to calculate the thermal conductivity of the sample of interest, and the overall uncertainty in the calculation. The thermal conductivity is calculated in a similar manner as the calibration factor, except the value to be calculated is the thermal conductivity rather than the temperature gradient by the application of unidirectional Fourier's Law. The temperature gradient is calculated by comparison on the sample runs to the baseline runs. Once the thermal conductivity based on the physical dimension of the sample is calculated, the total uncertainty of the calculation can be determined by using the equation below.

$$u_{total} = \sqrt{u_{Thickness}{}^2 + u_{Area}{}^2 + u_{Heat\ Flux}{}^2 + u_{Thermocouples}{}^2} \tag{4}$$

The error in the thickness and area of the readings is dependent upon what measurement equipment was used. Error in the calculated heat flux is determined by using the regressions developed earlier in the process. The error in the thermocouples was calculated when the error of the mean was calculated. By comparing a run to a baseline run without a sample, errors due to environmental causes and radiation losses can be neglected.

CHAPTER 4

RESULTS AND DISSCUSSION

System Performance

Experiments were performed on carbon GDL's and an Acrylonitrile-Butadiene-Styrene (ABS) sample with a 3% black carbon additive by weight. All testing was conducted per the process outlined in Chapter 3. To increase the fidelity of readings utilizing the carbon GDL's, three samples were stacked on top of each other to produce an overall thickness of .685 millimeters and the samples were 25.400 millimeters in diameter. The ABS sample used in testing was measured to be .863 millimeters thick and 24.130 millimeters in diameter. End results of the testing of the two samples produced the results found in Table 3 below, which includes a summary of the data collected and the results found with a system setting of 70 °C and a pressure of 1.4 MPa applied.

Table 3

Properties and results of tested sample.

	ABS	GDL's
Thickness (mm)	0.863	0.685
Diameter (mm)	24.130	25.400
Calculated Heat Flux (W)	5.74	7.33
Calibrated Temp. Gradient (k)	30.94	11.74
Calculated Thermal Cond. (W/mk)	0.35	0.44
Total Uncertainty	5.48%	11.38%
Number of Data Points used in Calculation	146923	122454

Results found for the carbon GDLs were consistent with published documentation for the thru-plane conductivity, which showed 0.43 W/mk (P.T Nguyen, 2004) and .34

W/mk (M. Wohr, 1998). Published thermal conductivities for ABS with a 3% carbon additive were not available; however, manufacturers report a thermal conductivity for ABS with no additives ranging from .12 W/mk to .2 W/mk. This suggests that the addition of carbon black raised the thermal conductivity of the system.

As can be seen in the summary results, the calculated thermal conductivity for the GDLs is associated to a higher total uncertainty than the ABS. This is due to the non-negligible compression of the GDLs that occurs with the amount of pressure that was applied to the samples during the experimentation. A reduction in thickness of 28% was anticipated based upon data published by Sadeghifar (2013). Using this approximation is the largest source of uncertainty in the GDL calculations. If a more accurate method was used, such as a combination of height gauges and feeler gauges, the total uncertainty would reduce to approximately what was seen in the ABS calculations. The overall accuracy obtained by this system is deemed reasonable as a comparable system developed by Karimi et. Al. in 2010 was able to maintain an accuracy of 4% to 11%, depending on test parameters.

The total cost for fabrication of this system can be seen in Table 4 below; items with asterisks next to them denote that the item was already owned by Arizona State University prior to initiation to this project.

Table 4

Bill of materials and price of system.

System BOM		Price
Cartridge Heater	$	19.14
Calcium Silicate Insulation	$	32.07
Mineral Wool Insulation	$	38.89
6.5" Dia X 1" 6061-T6 Rod	$	37.05
2" Dia X 6" 1012 Rod	$	19.47
1" Dia X 3" 6061-T6 Rod	$	4.32
3/4" X 4" X 1' 6061-T6 (2)	$	50.92
3/8-16X 2' All-Thread (4)	$	7.16
20' Tyke K, 20 Gauge Tc Wire	$	32.80
.007" X 2" X 5' 304 Stainless Foil	$	12.77
Omega LCM304-5KN Load Cell	$	315.00
Hardware	$	20.00
Watlow Temp. Contoller Serier 808*	$	250.00
Circuit Specialist CSI3010X*	$	169.00
Fluke 8842A Multimeter*	$	189.00
Accsense Versalog Data Logger*	$	568.00
Total	$	1,765.59

As can been seen, the majority of the total cost associated with this system were already available to the school, leaving a startup cost of approximately $550 dollars to expand the testing capabilities of the fuel cell testing lab on the ASU Polytechnic campus. Compared to entry level commercial models, this system represents a reduction in purchase cost of more than 600% over commercial models. Compared to a similar system developed internally by a university, this system has a 1300% reduction in cost compared to the $23,700 spent by Culham (2002); however, this reduction in price comes at the expense of other areas that may be of concern.

Commercial systems can provide a guaranteed accuracy of 5% or better, foolproof operation, and nearly instantaneous results. The testing system detailed in this report requires a significant amount of user interaction. This forces the user selected to be very skilled and methodical as carelessness can produce errant results or a significant level of uncertainty in the final calculated thermal conductivity. There is an additional increase to the amount of time testing needs to occur in order to achieve acceptable results. To get a minimum level of data to produce a workable calculation, two weeks are required to get a baseline and calibration, followed by a week for each sample to be tested. A change in the testing parameters, temperature, pressure, or area, would require additional weeks to develop a baseline and calibration data, although such drawbacks are expected when attempting to develop a low cost test bed.

Future Development and Improvement

The major drawback to utilizing the system that has been detailed and developed above is the time required to produce a usable result. Future design modifications should be focused on reducing the run time with minimal increases to system cost. One such opportunity for improvement would be the modification from performing calculations from steady state condition to utilizing quasi-steady state methods. This method would reduce runtimes by 10-100 percent when compared to steady state methods (Zamel, 2011). This could potentially reduce the run time from 24 hours to a more reasonable run time of less than 5 hours. One requirement for implementation of this method would be to replace the thermocouple with a more accurate temperature reading sensor, such as resistance temperature detectors (RTD), to remove the need to lower the error about the mean utilizing a large number of data points. RTDs are typically available from various manufactures with a rated accuracy of +/-0.12%, which is on par with the mean accuracy level of the K type thermocouples used in the experiments detailed in this document. This would allow the operator to collect less than 100 data points and make a useable calculation as opposed to the 13,000 plus data points required by the use of thermocouples. This would increase the overall system cost by approximately 34%.

To reduce the need for multiple runs, it will be necessary to reduce variability in setup and environmental impacts. To reduce variability in setup, installing slip fit, precision locating pins in both the lower and upper flux meter will ensure that they are properly aligned in the testbed. These pins can be installed into solid insulation and then bonded onto the flux meters to prevent heat shunting occurring in the test stack, which would result in errant readings. To reduce environmental impacts, replacement of the heat sink with an isothermal cold plate is a potential solution. By using a cold plate in conjunction with the insulation already in use, the temperature on the cold side of the test stack will be maintained regardless of ambient conditions. With the current configuration, if ambient temperature fluctuates, the temperature of the heat sink fluctuates in turn impacting the temperature gradient within the test stack. If the input temperatures and cold plate temperatures are configured properly, it will be possible to achieve steady state operating conditions in approximately 10 minutes (Burheim, 2010). Utilizing a vacuum to further insulate the test stack is not necessary as it has been shown that the small amount of atmosphere within the GDL will not significantly influence the measured thermal conductivity (Sadeghi, 2011).

By implementing these improvements, it will be possible to reduce the turn-around time for a thermal conductivity measurement from 3 weeks down to approximately 1 or 2 days. These changes would increase the overall cost of this system; however, it would still be anticipated to be at least a 350% reduction in cost to commercial testing systems. By utilizing the designs in this document and leveraging existing equipment available within other research laboratories, a further reduction in cost would be anticipated should they undertake fabrication of this system.

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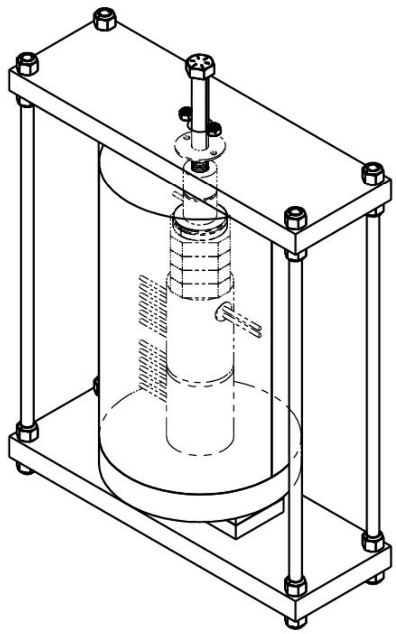
APPENDIX A

SYSTEM BLUE PRINTS

ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	15-09-14



THERMAL CONDUCTIVITY TEST-BED
FABRICATION DOCUMENT

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	SCALE: NA	UNIT: IN	DRAWN: BMS	CHECKED: -	APPROVED: -
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ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	6-7-2016

2	20	9364K56	INSULATION	MCMASTER
AR	19	6579T25	THERMOCOUPLE WIRE	MCMASTER
1	18	3618K12	CARTRIGE HEATER	MCMASTER
2	17	#10-32 NYLOCK	LOCKNUT	COMMERCIAL
2	16	#10-32 X 1.00 SHCS	SCREW	COMMERCIAL
16	15	Ø3/8-16 NYLOCK	LOCKNUT	COMMERCIAL
4	14	Ø3/8-16 X 15.00	ALL-THREAD ROD	COMMERCIAL
4	13	2.00 X 2.00 X 0.50	RISER	PLYWOOD
1	12	12.00 X 4.00 X 0.75	LOWER FRAME PLATE	6061-T6511
1	11	12.00 X 4.00 X 0.75	UPR FRAME PLATE	6061-T6511
1	10	Ø1/2-13 X 4 LN HHCS	LOAD BOLT	COMMERCIAL
1	9	Ø1.00 X 1.50	ADAPTER	6061-T6511
1	8B	Ø1.5 OD X Ø 17/32 X 1/16	WASHER	COMMERCIAL
1	8A	Ø1/2-13 NYLOCK	LOCKNUT	COMMERCIAL
1	8		WELDED ASSY	
1	7	LCM304-5KN	LOAD CELL	OMEGA
1	6	91131A090	ALIGNMENT WASHER	MCMASTER
4	5	93505K12	STACK INSULATION	MCMASTER
1	4	Ø6.00 X 1.00	HEAT SINK	6061-T6511
1	3	Ø2.00 X 3.00	LWR FLUX METER	1018
1	2	Ø2.00 X 3.00	UPR FLUX METER	1018
2	1	Ø1.00 X 0.50	SAMPLE HOLDER	6061-T6511
QTY	DET #	MATERIAL	PART NAME	NOTES

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ARIZONA STATE UNIVERSITY

SHEET 2 OF 12

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ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	6-7-2016

10 RECOORD MANUFACTOR CALIBRATION DATA FOR DETAIL 7 BELOW
mV _________ LOAD _________
mV _________ LOAD _________
mV _________ LOAD _________
mV _________ LOAD _________

9 RECOORD ACTUAL SPACEING OF 12 DIA HOLES IN DETAIL 3 BELOW
SPACING _________ ± _________

8 RECOORD ACTUAL SPACEING OF 12 DIA HOLES IN DETAIL 2 BELOW
SPACING _________ ± _________

7 RECOORD ACTUAL DIAMETER OF DETAIL 1 AFTER FABRICATION BELOW
PART 1 _________ PART 2 _________

6 SUBSTITUTION OF MATERIALS AND COMPONETS ACCEPTABLE WITH ENGINEERING CONCURANCE SUBSTITUIONS MUST BE FUCTONALLY IDENTICAL

5 CALIBRATE THERMOCOUPLES USING AN ICE/WATER SLURRY

4 CUT DETAIL 19 TO DESIRED LENGTH AND PREPAIR THERMOCOUPLE WIRE PER MANUFACTOR'S INSTRUCTIONS ENSURE ENDS OF THERMOCOUPLES ARE PROPERLY INSULATED AFTER FABRICATION UTILIZING SHRINK TUBING

3 FILL THERMOCOUPLE PORTS IN DETAILS 2 AND 3 WITH THERMAL PASTE PRIOR TO INSERTION OF THERMAL COUPLES

2 APPLY THERMAL PASTE BETWEEN THE FOLLOWING DETAILS UPON ASSEMBLY DETAIL 3 AND 4, DETAIL 2 AND 1, DETAIL 3 AND 1

1 TOOL FUNCTION MEASURING AND DETERMINATION OF THERMAL CONDUCTIVITY OF SOLID MATERIALS AT VARIOUS TEMPS AND PRESSURES

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ARIZONA STATE UNIVERSITY			SHEET 3 OF 12	
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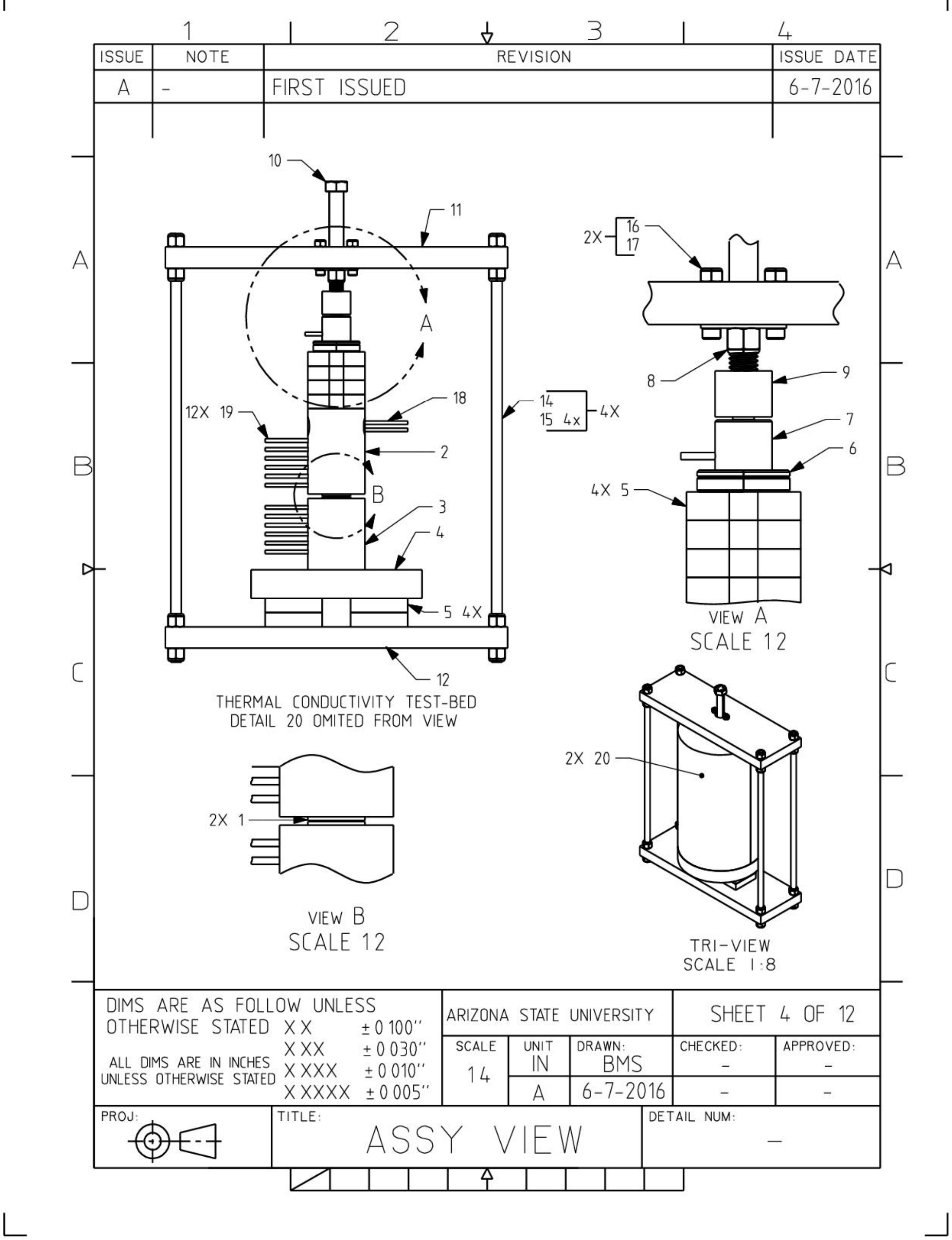
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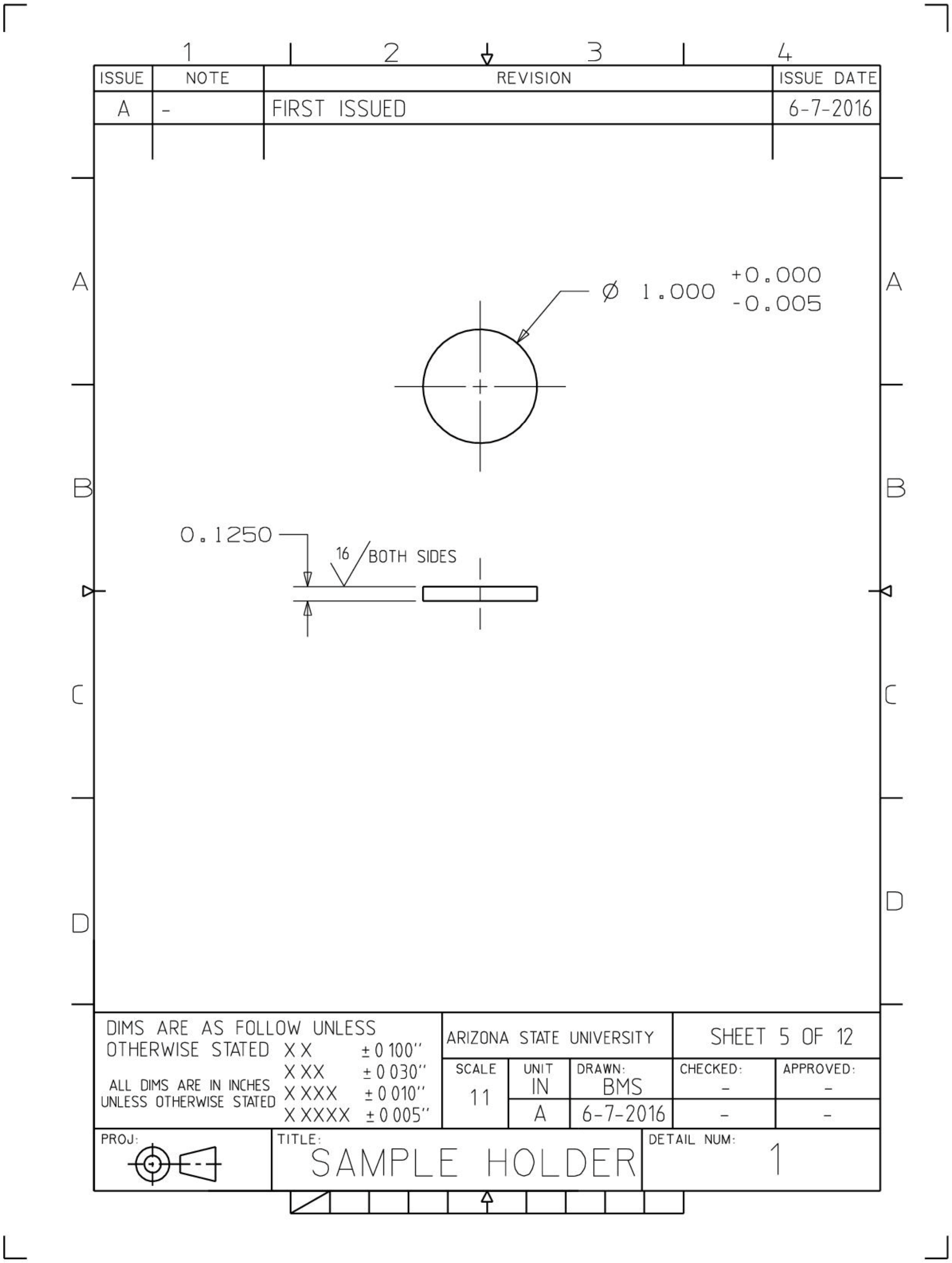
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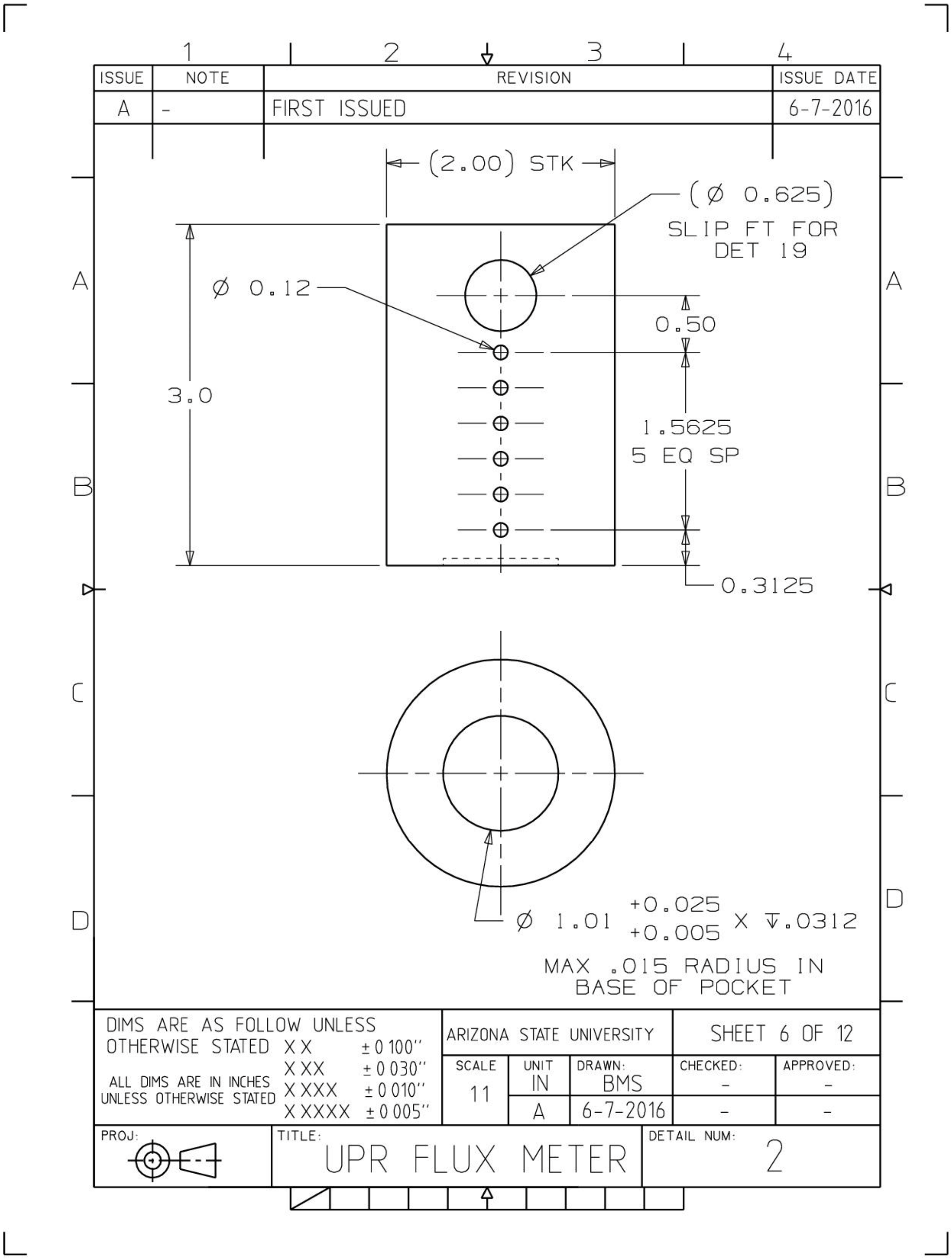
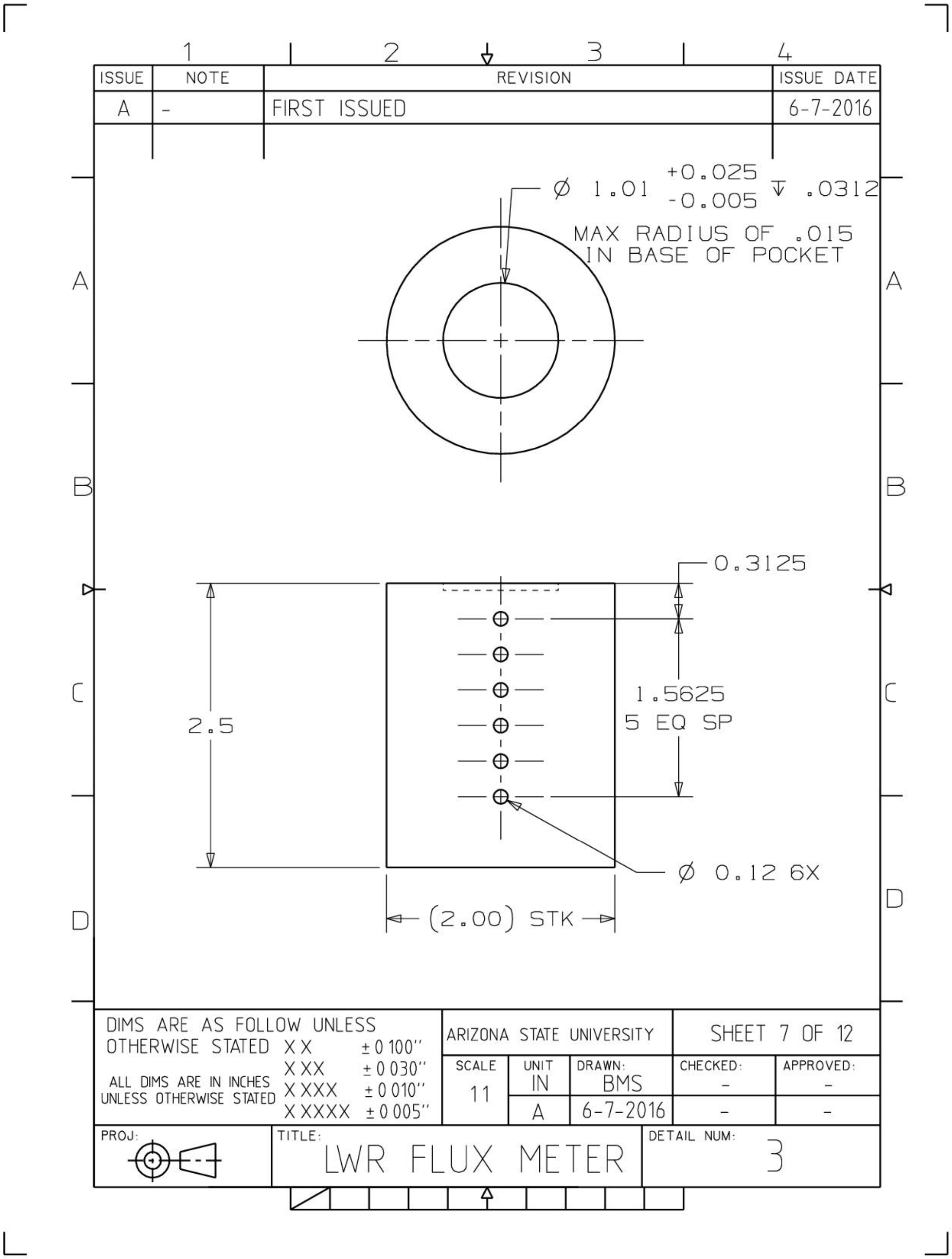
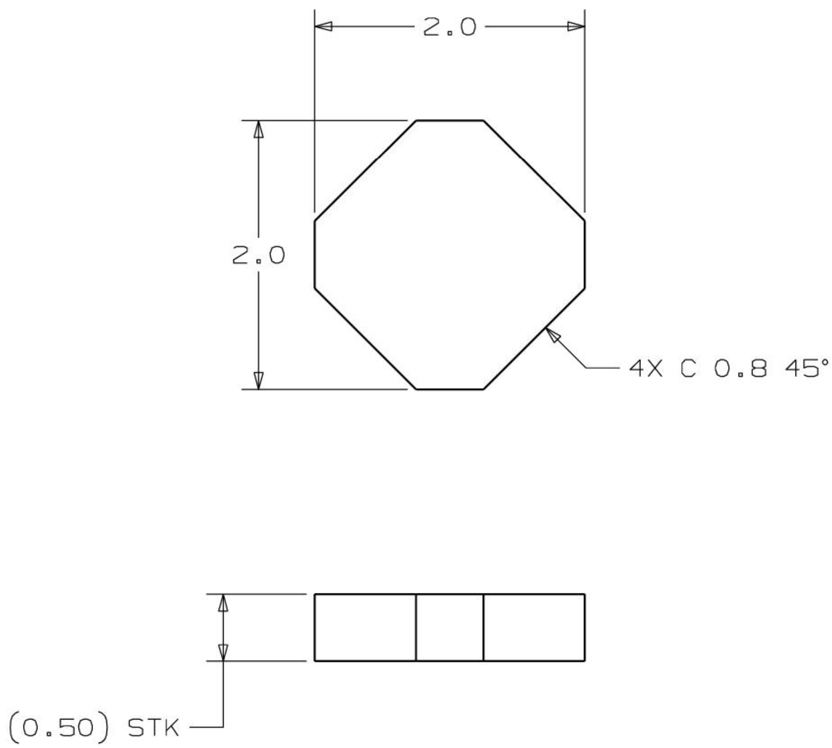
DETAIL NUM:
1



1
2
3
4
ISSUE
NOTE
REVISION
ISSUE DATE
A
-
FIRST ISSUED
6-7-2016
(2.00) STK
(Ø 0.625)
SLIP FT FOR DET 19
Ø 0.12
0.50
3.0
1.5625
5 EQ SP
0.3125
A
B
C
D
Ø 1.01 +0.025 +0.005 X ↧.0312
MAX .015 RADIUS IN BASE OF POCKET
DIMS ARE AS FOLLOW UNLESS OTHERWISE STATED
X.X ±0.100"
X.XX ±0.030"
X.XXX ±0.010"
X.XXXX ±0.005"
ALL DIMS ARE IN INCHES UNLESS OTHERWISE STATED
ARIZONA STATE UNIVERSITY
SHEET 6 OF 12
SCALE
1:1
UNIT
IN
DRAWN:
BMS
CHECKED:
-
APPROVED:
-
A
6-7-2016
-
-
PROJ:
TITLE:
UPR FLUX METER
DETAIL NUM:
2

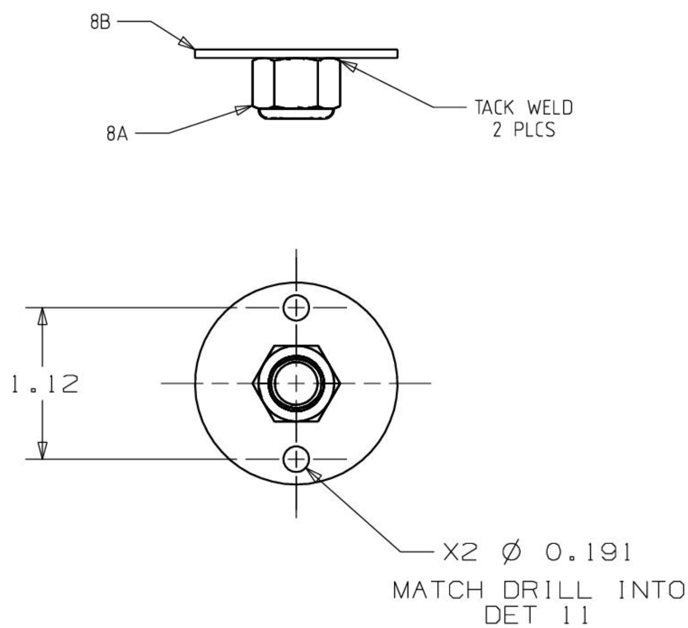

ISSUE
NOTE
REVISION
ISSUE DATE
A
-
FIRST ISSUED
6-7-2016
Ø 1.01 +0.025 -0.005 ↧ .0312
MAX RADIUS OF .015
IN BASE OF POCKET
0.3125
1.5625
5 EQ SP
2.5
Ø 0.12 6X
(2.00) STK
DIMS ARE AS FOLLOW UNLESS
OTHERWISE STATED X.X ±0.100″
X.XX ±0.030″
X.XXX ±0.010″
X.XXXX ±0.005″
ALL DIMS ARE IN INCHES
UNLESS OTHERWISE STATED
ARIZONA STATE UNIVERSITY
SHEET 7 OF 12
SCALE
1:1
UNIT
IN
A
DRAWN:
BMS
6-7-2016
CHECKED:
-
-
APPROVED:
-
-
PROJ:
TITLE:
LWR FLUX METER
DETAIL NUM:
3

ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	6-7-2016

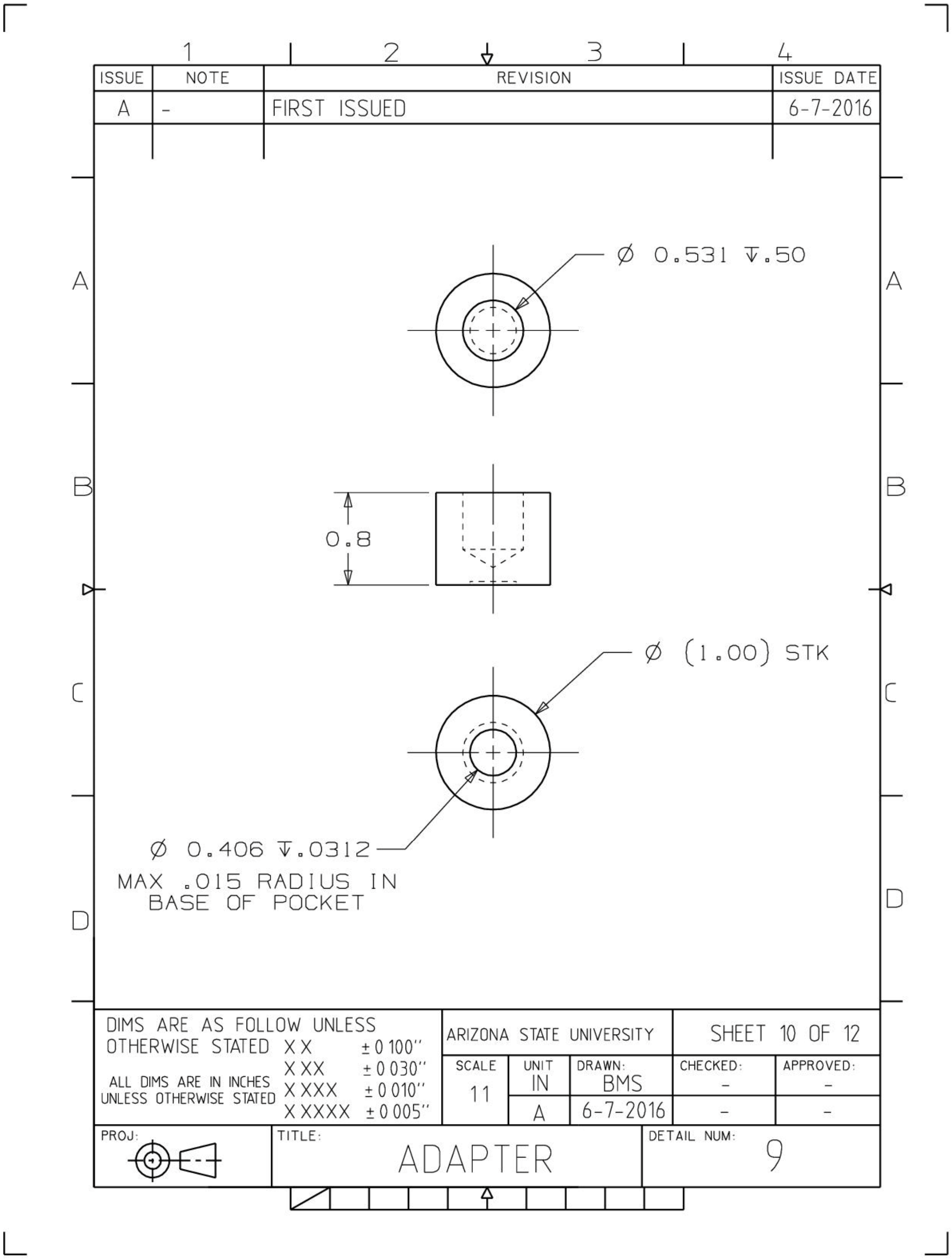


DIMS ARE AS FOLLOW UNLESS OTHERWISE STATED X X ± 0 100'' X XX ± 0 030'' X XXX ± 0 010'' X XXXX ± 0 005'' ALL DIMS ARE IN INCHES UNLESS OTHERWISE STATED	ARIZONA STATE UNIVERSITY			SHEET 8 OF 12	
	SCALE 11	UNIT IN A	DRAWN: BMS 6-7-2016	CHECKED: - -	APPROVED: - -
PROJ:	TITLE: STACK INSULATOR			DETAIL NUM: 5	

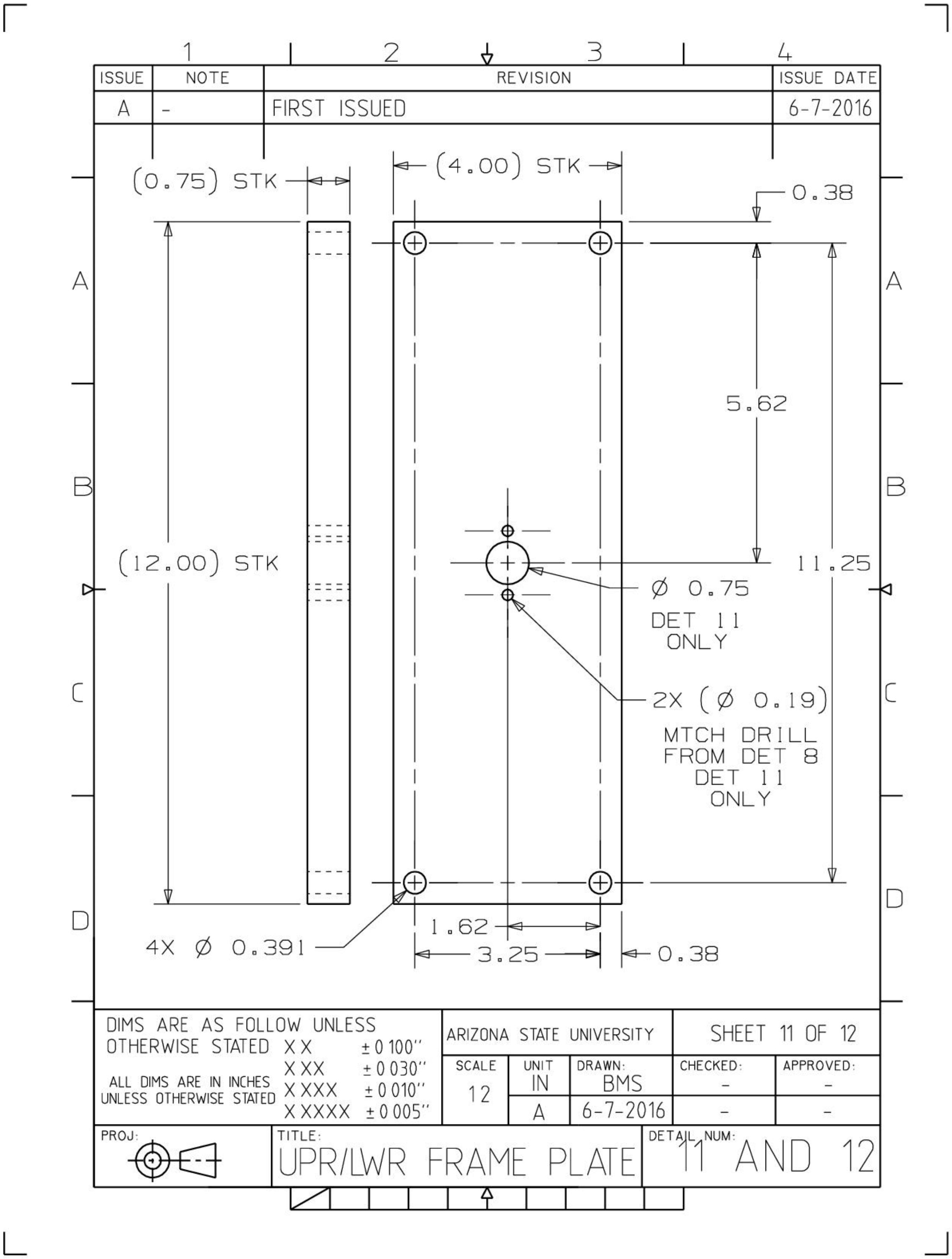
ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	6-7-2016



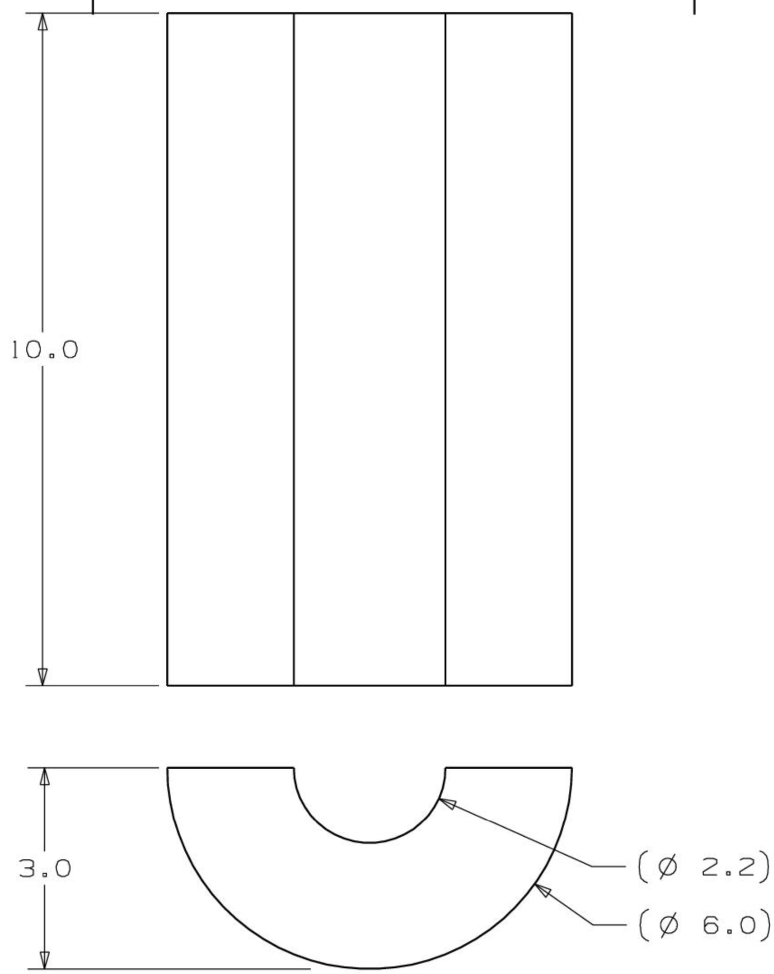
DIMS ARE AS FOLLOW UNLESS OTHERWISE STATED X.X ±0.100" X.XX ±0.030" X.XXX ±0.010" X.XXXX ±0.005" ALL DIMS ARE IN INCHES UNLESS OTHERWISE STATED	ARIZONA STATE UNIVERSITY			SHEET 9 OF 12	
	SCALE: 1:1	UNIT: IN	DRAWN: BMS	CHECKED: -	APPROVED: -
		A	6-7-2016	-	-
PROJ:	TITLE: WELDED ASSY			DETAIL NUM: 8	



ISSUE
NOTE
REVISION
ISSUE DATE
A
-
FIRST ISSUED
6-7-2016
Ø 0.531 ↧.50
0.8
Ø (1.00) STK
Ø 0.406 ↧.0312
MAX .015 RADIUS IN
BASE OF POCKET
DIMS ARE AS FOLLOW UNLESS
OTHERWISE STATED
X X ±0 100''
X XX ±0 030''
X XXX ±0 010''
X XXXX ±0 005''
ALL DIMS ARE IN INCHES
UNLESS OTHERWISE STATED
ARIZONA STATE UNIVERSITY
SHEET 10 OF 12
SCALE
11
UNIT
IN
A
DRAWN:
BMS
6-7-2016
CHECKED:
-
-
APPROVED:
-
-
PROJ:
TITLE:
ADAPTER
DETAIL NUM:
9


ISSUE
NOTE
REVISION
ISSUE DATE
A
-
FIRST ISSUED
6-7-2016
(0.75) STK
(4.00) STK
0.38
5.62
(12.00) STK
11.25
Ø 0.75
DET 11
ONLY
2X (Ø 0.19)
MTCH DRILL
FROM DET 8
DET 11
ONLY
4X Ø 0.391
1.62
3.25
0.38
DIMS ARE AS FOLLOW UNLESS
OTHERWISE STATED
X.X ±0.100″
X.XX ±0.030″
X.XXX ±0.010″
X.XXXX ±0.005″
ALL DIMS ARE IN INCHES
UNLESS OTHERWISE STATED
ARIZONA STATE UNIVERSITY
SHEET 11 OF 12
SCALE
12
UNIT
IN
A
DRAWN:
BMS
6-7-2016
CHECKED:
-
-
APPROVED:
-
-
PROJ:
TITLE:
UPR/LWR FRAME PLATE
DETAIL NUM:
11 AND 12

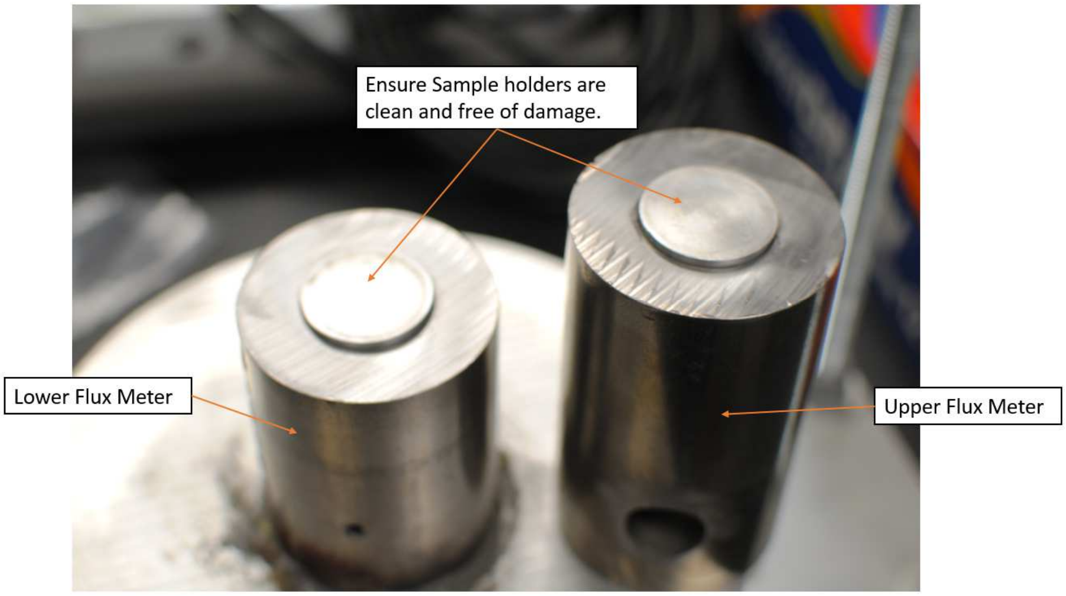
ISSUE	NOTE	REVISION	ISSUE DATE
A	-	FIRST ISSUED	6-7-2016

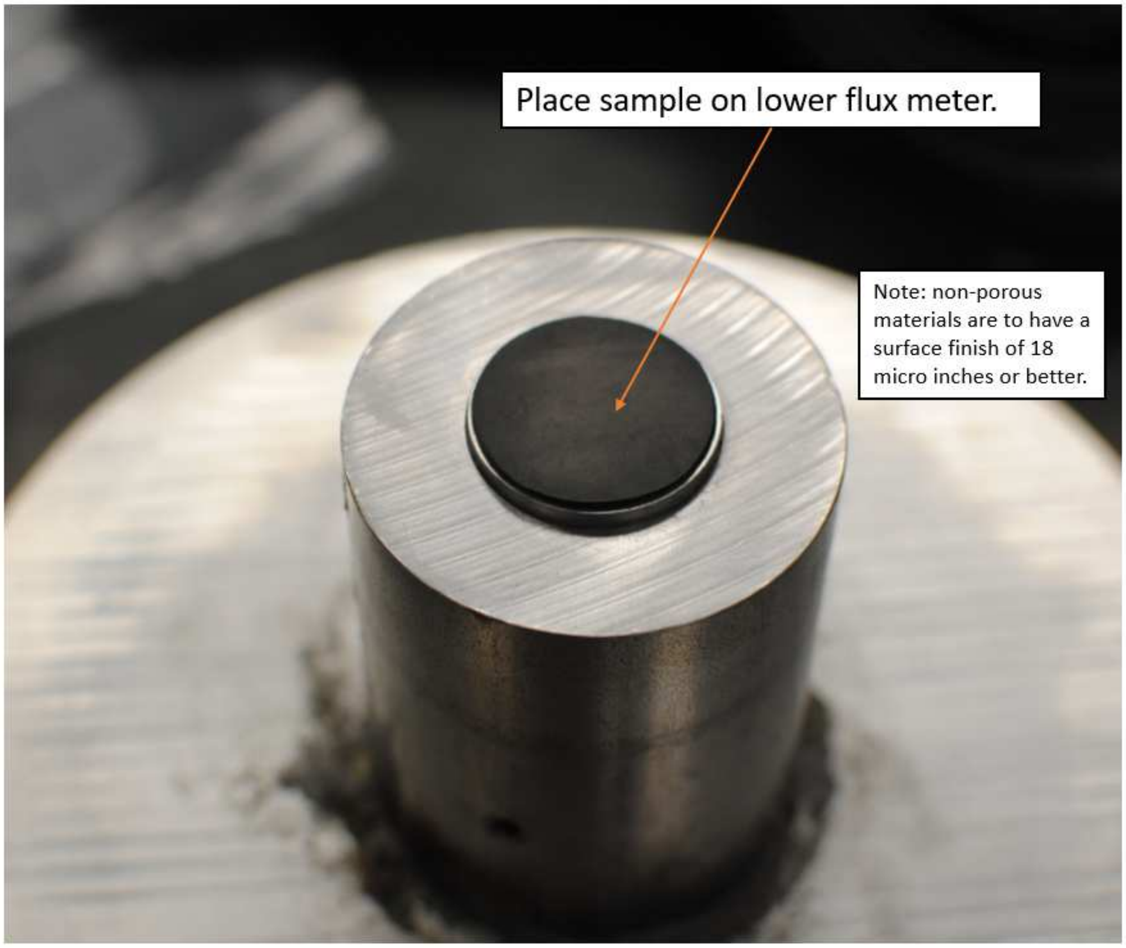


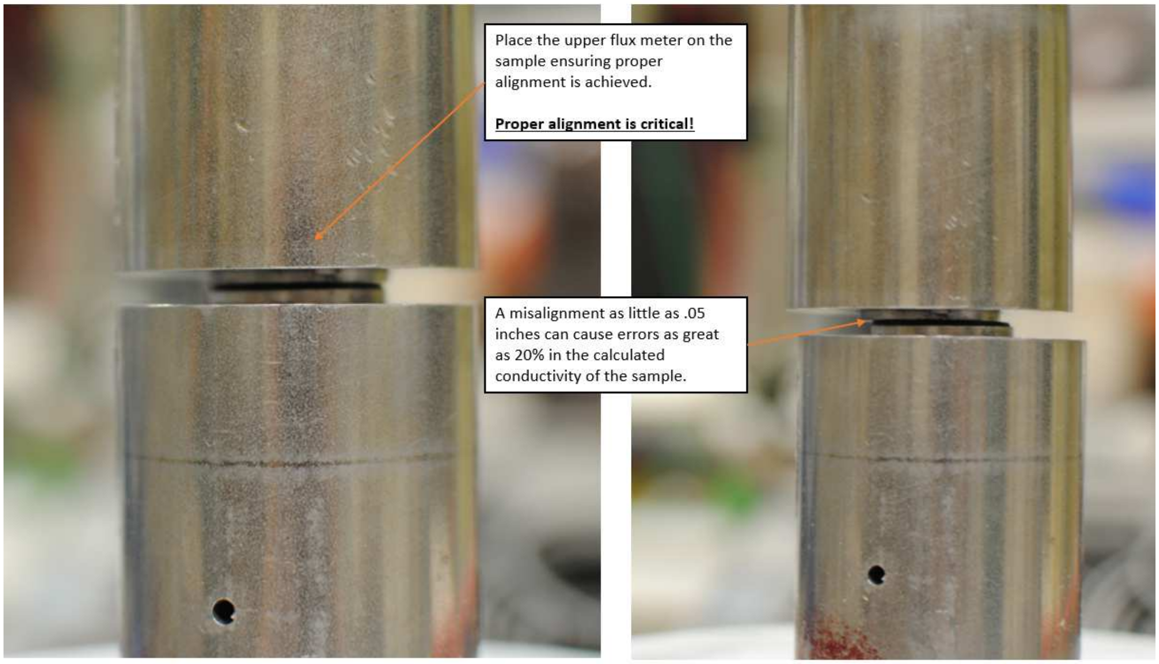
DIMS ARE AS FOLLOW UNLESS OTHERWISE STATED X.X ±0.100" X.XX ±0.030" X.XXX ±0.010" X.XXXX ±0.005" ALL DIMS ARE IN INCHES UNLESS OTHERWISE STATED	ARIZONA STATE UNIVERSITY			SHEET 12 OF 12	
	SCALE 1:2	UNIT IN	DRAWN: BMS	CHECKED: -	APPROVED: -
		A	6-7-2016	-	-
PROJ:	TITLE: INSULATION			DETAIL NUM: 20	

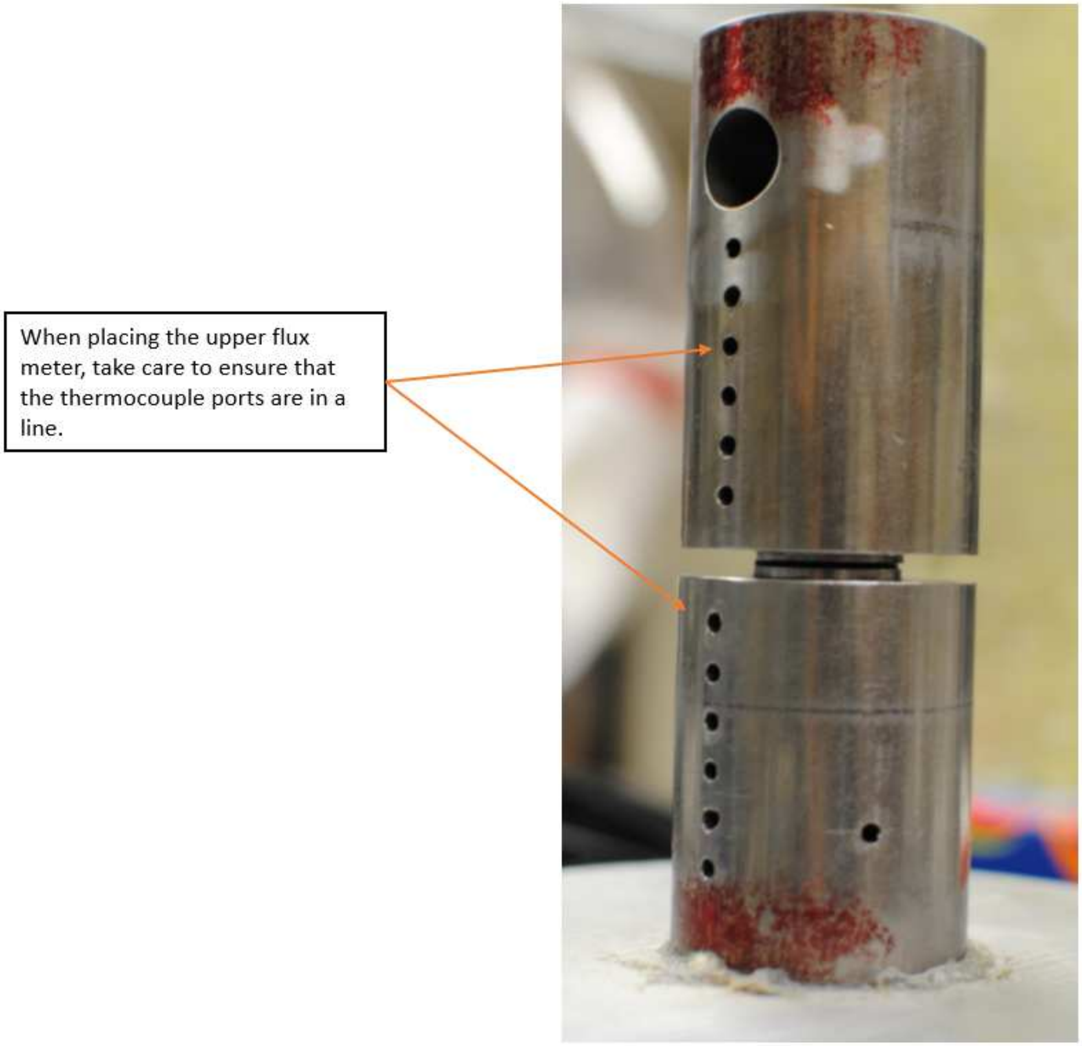
APPENDIX B

VISUAL WORK INSTRUCTIONS

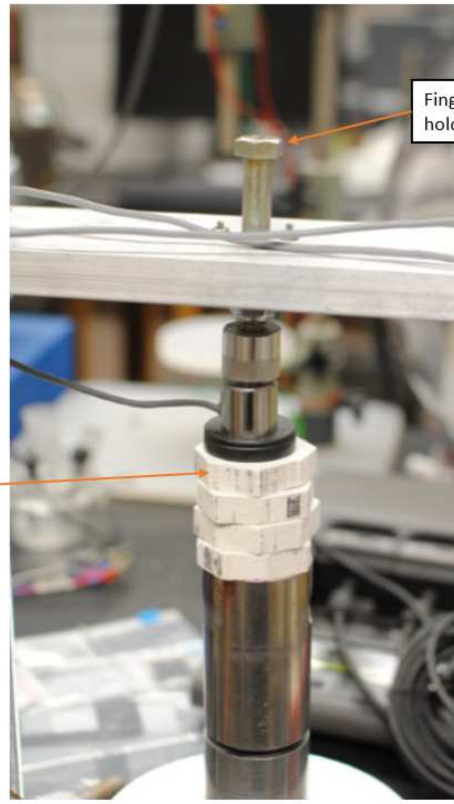

Ensure Sample holders are clean and free of damage.
Lower Flux Meter
Upper Flux Meter


Place sample on lower flux meter.
Note: non-porous materials are to have a surface finish of 18 micro inches or better.


Place the upper flux meter on the sample ensuring proper alignment is achieved.
Proper alignment is critical!
A misalignment as little as .05 inches can cause errors as great as 20% in the calculated conductivity of the sample.


When placing the upper flux meter, take care to ensure that the thermocouple ports are in a line.

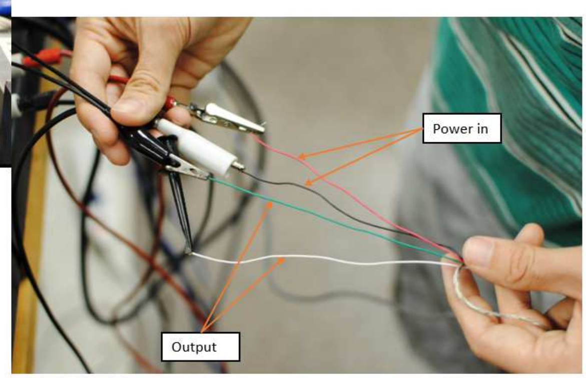
Finger-tighten the load screw to hold the test stack in place.

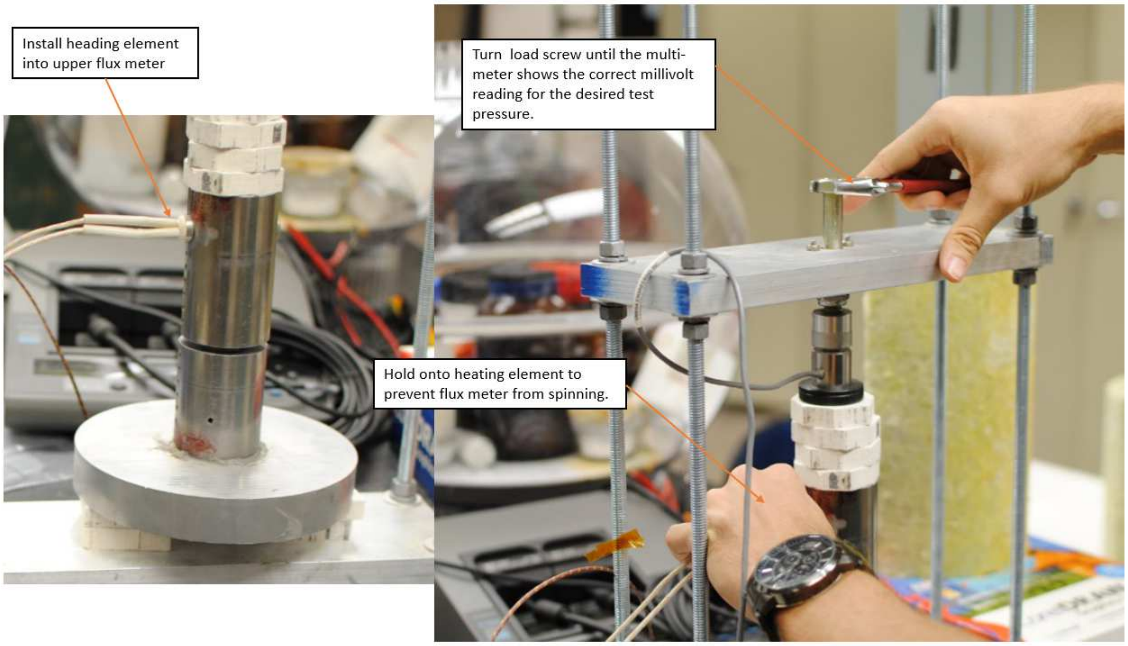


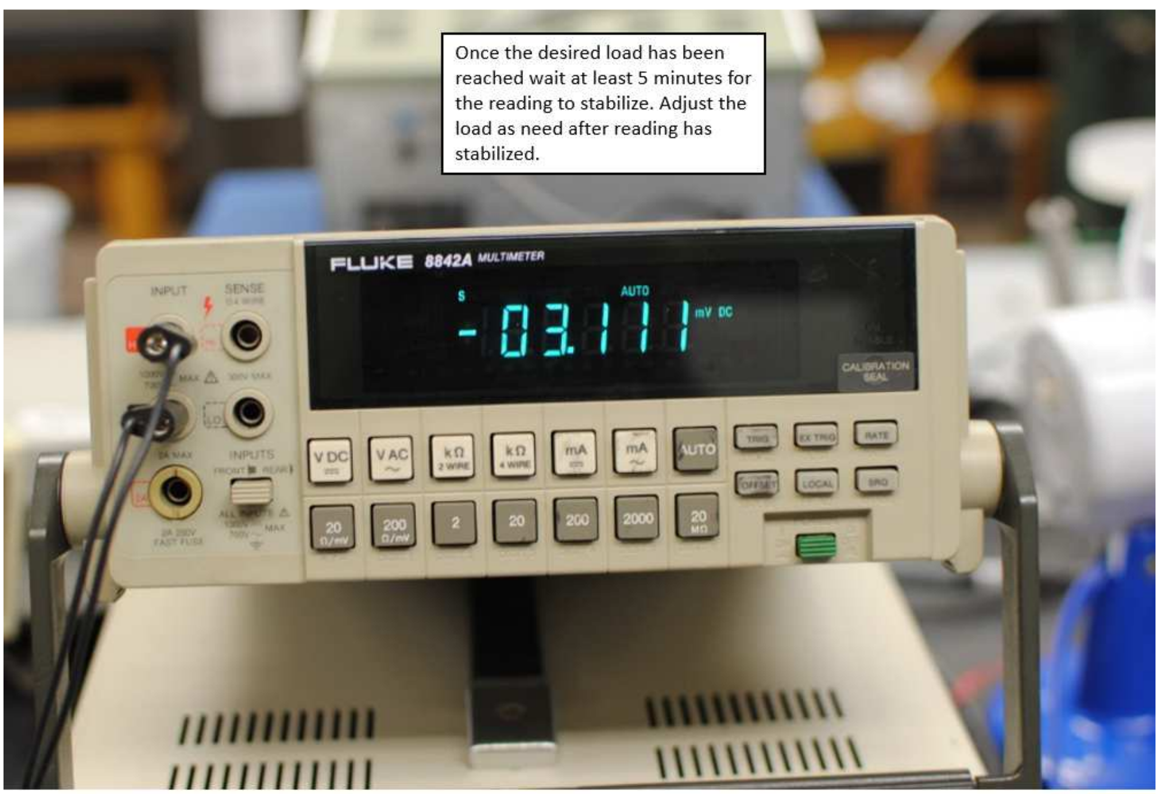
Place the insulation, alignment washer, load cell and load screw adapter on top of the test stack.

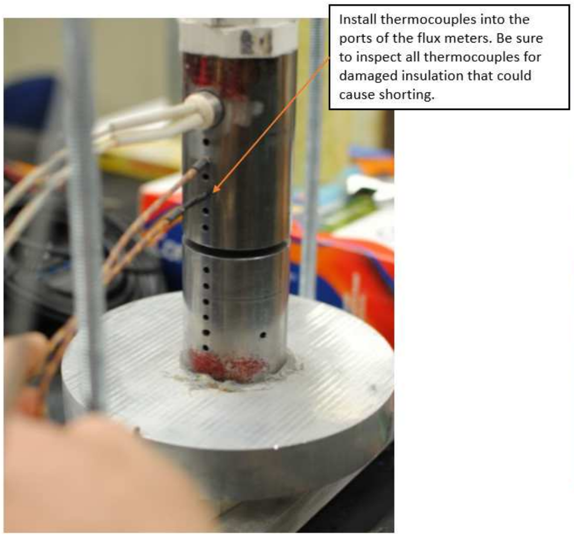
Set up a DC power supply and multi-meter and connect to the load cell. The load cell is to be supplied with 10 Volts DC.

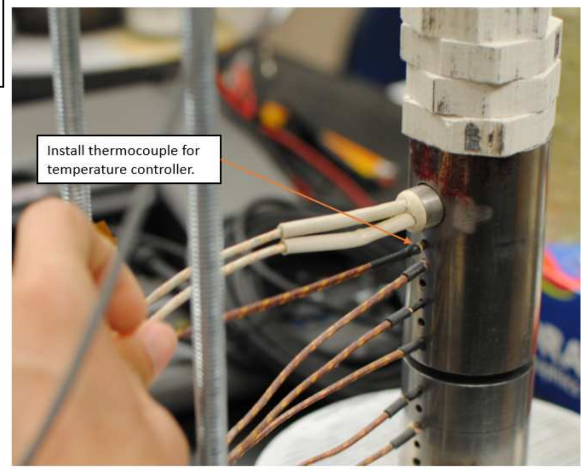
Wait at least 5 minutes after turning on power supply to allow the load cell to warm up.

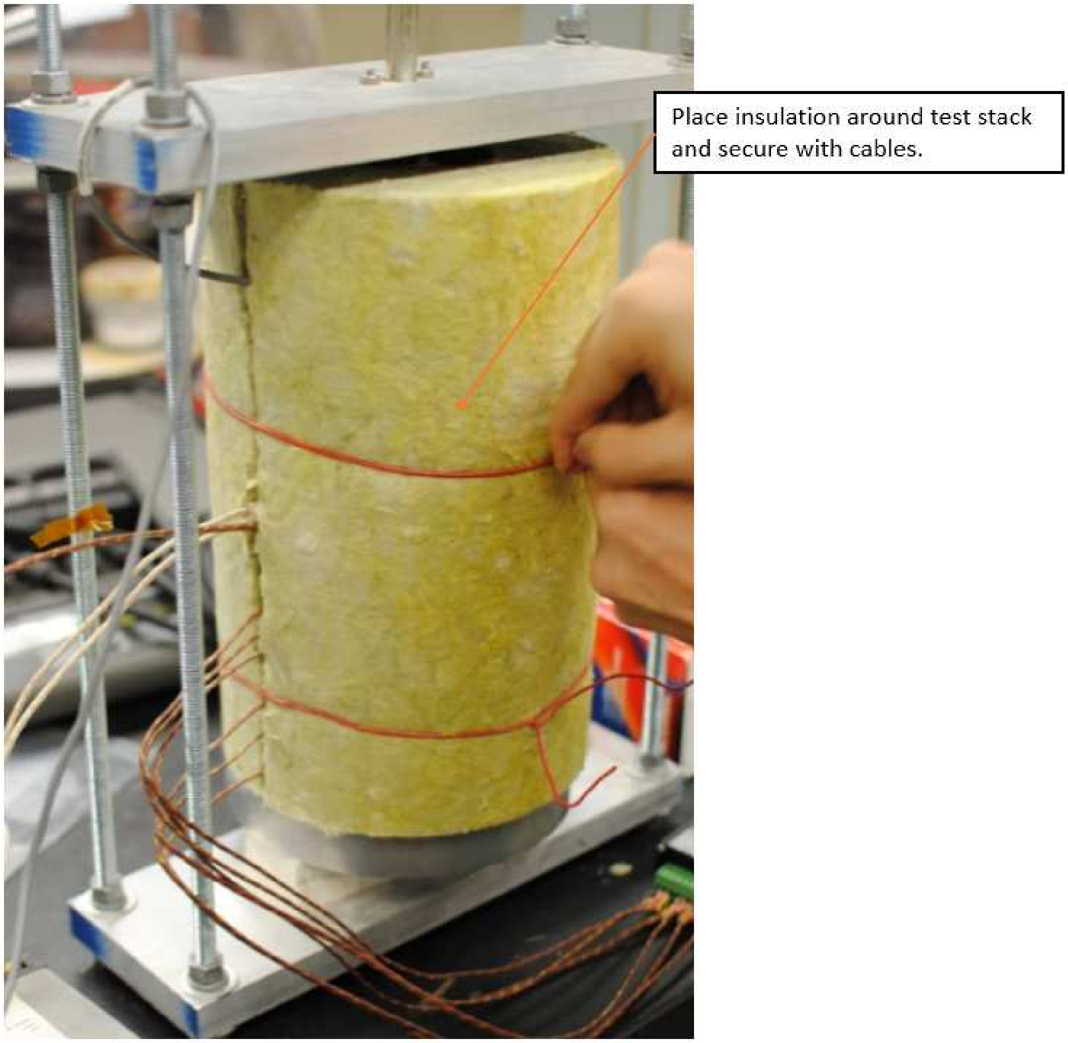


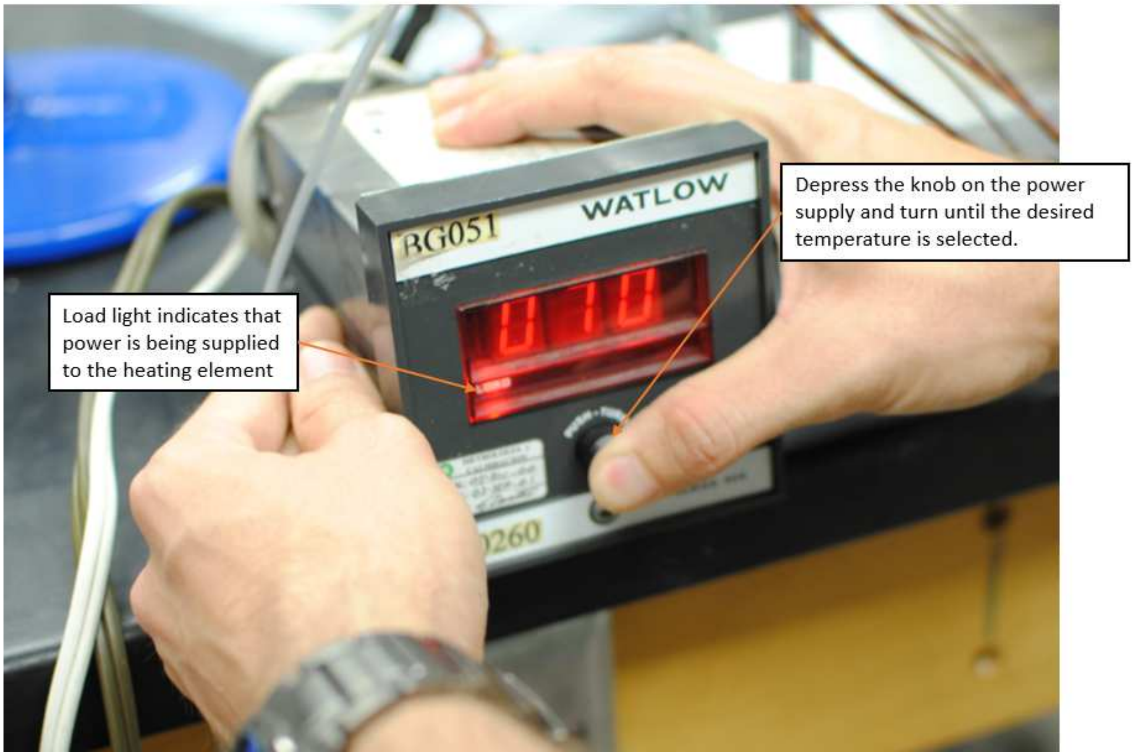

Install heading element into upper flux meter
Turn load screw until the multi-meter shows the correct millivolt reading for the desired test pressure.
Hold onto heating element to prevent flux meter from spinning.

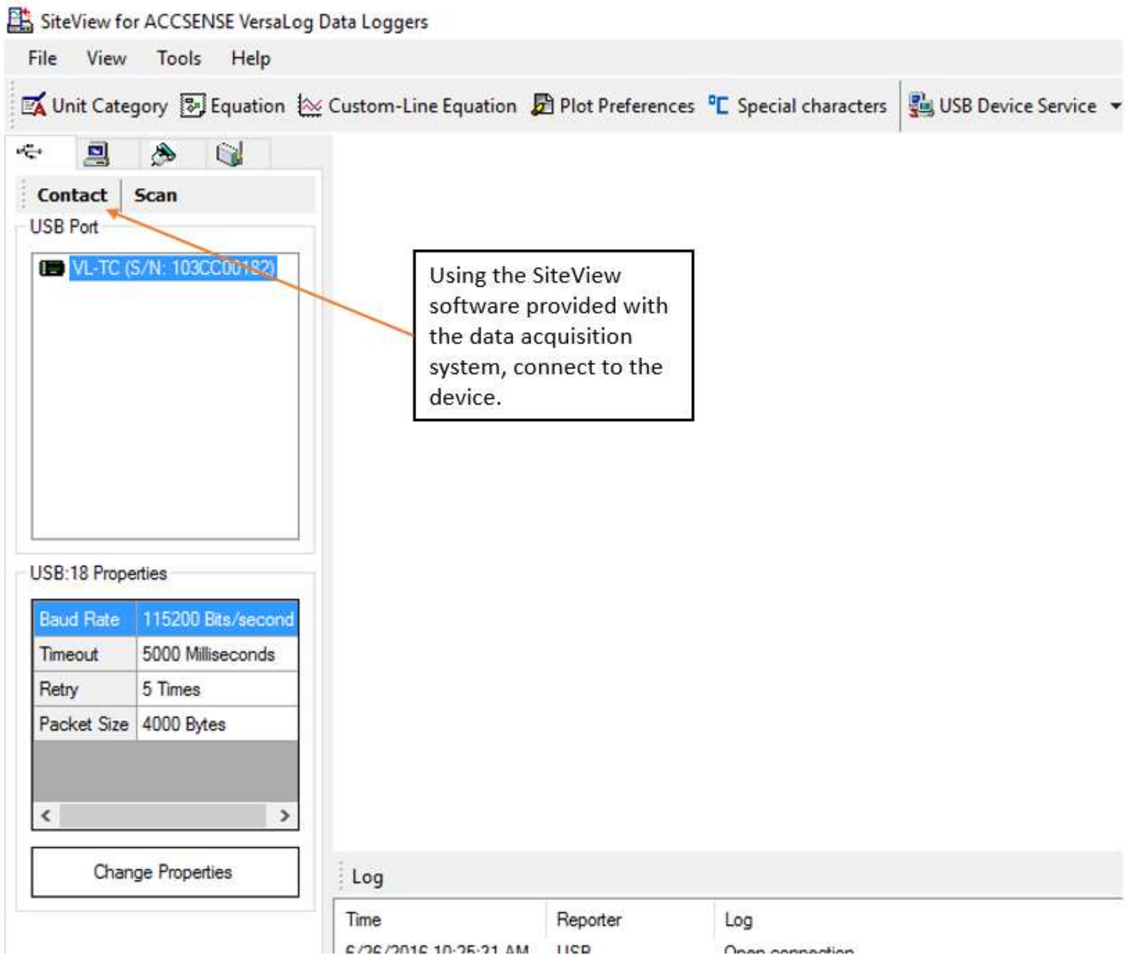

Once the desired load has been reached wait at least 5 minutes for the reading to stabilize. Adjust the load as need after reading has stabilized.
FLUKE 8842A MULTIMETER
-03.111 mV DC

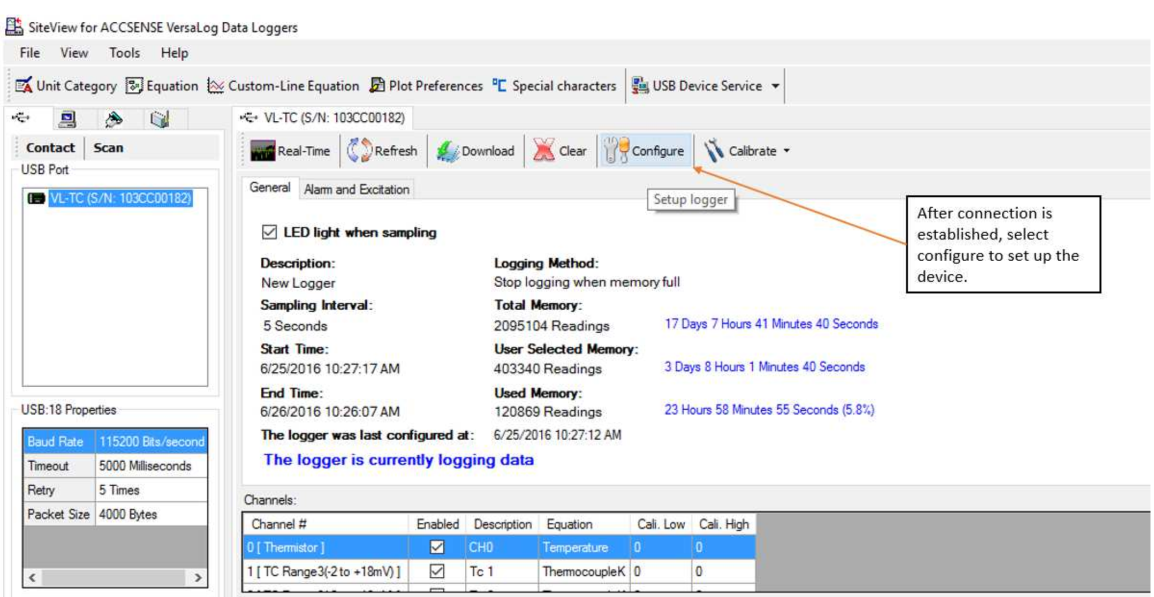

Install thermocouples into the ports of the flux meters. Be sure to inspect all thermocouples for damaged insulation that could cause shorting.

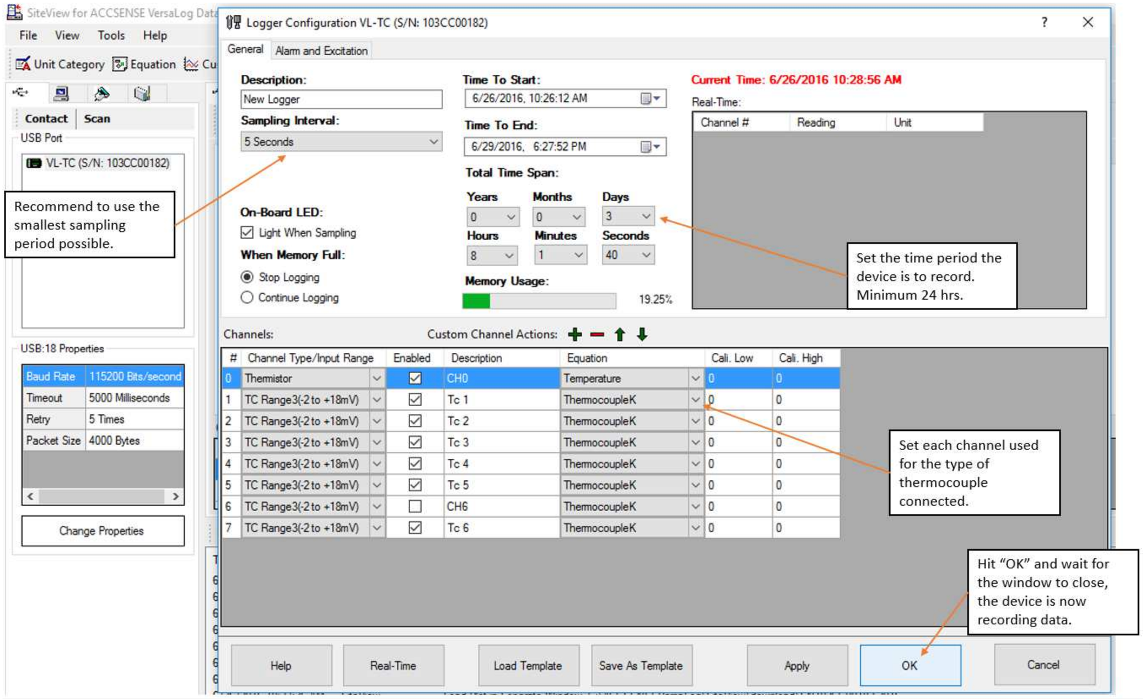

Install thermocouple for temperature controller.

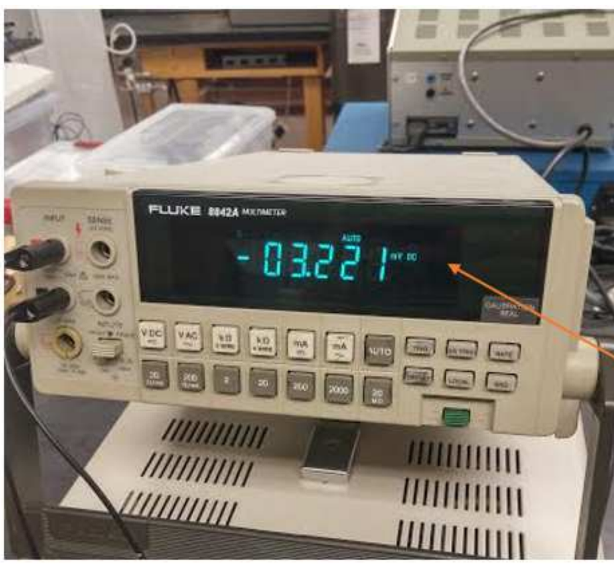

Place insulation around test stack and secure with cables.


Depress the knob on the power supply and turn until the desired temperature is selected.
Load light indicates that power is being supplied to the heating element
WATLOW
BG051
010


SiteView for ACCSENSE VersaLog Data Loggers
File View Tools Help
Unit Category Equation Custom-Line Equation Plot Preferences Special characters USB Device Service
Contact Scan
USB Port
VL-TC (S/N: 103CC00182)
Using the SiteView software provided with the data acquisition system, connect to the device.
USB:18 Properties
Baud Rate 115200 Bits/second
Timeout 5000 Milliseconds
Retry 5 Times
Packet Size 4000 Bytes
Change Properties
Log
Time Reporter Log

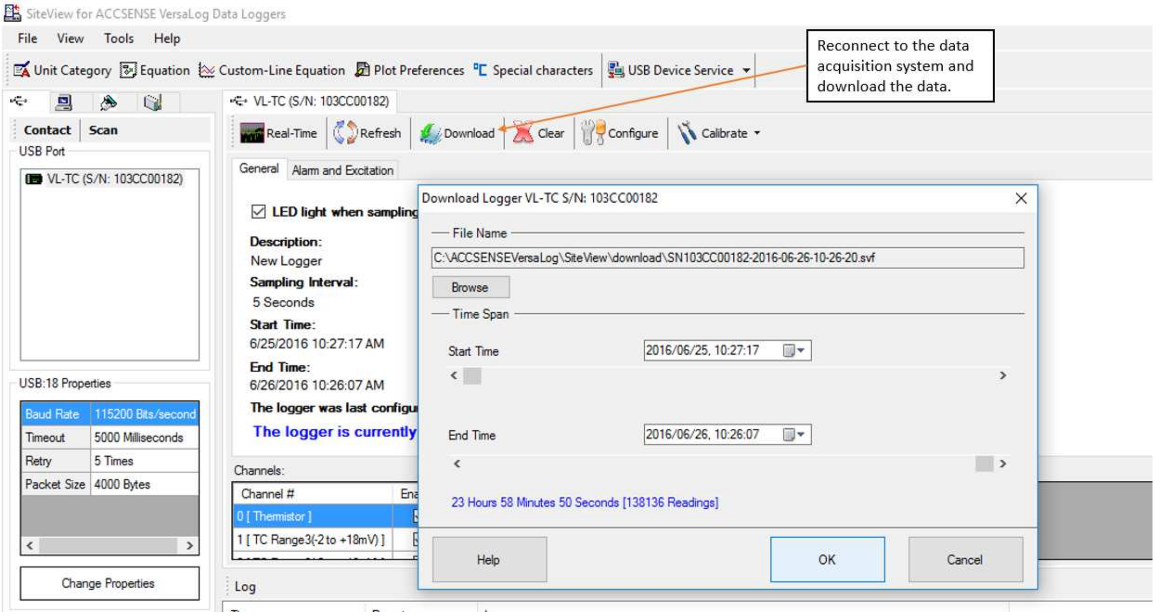

SiteView for ACCSENSE VersaLog Data Loggers
File View Tools Help
Unit Category Equation Custom-Line Equation Plot Preferences Special characters USB Device Service
Contact Scan
USB Port
VL-TC (S/N: 103CC00182)
Real-Time Refresh Download Clear Configure Calibrate
General Alarm and Excitation
Setup logger
After connection is established, select configure to set up the device.
LED light when sampling
Description: New Logger
Sampling Interval: 5 Seconds
Start Time: 6/25/2016 10:27:17 AM
End Time: 6/26/2016 10:26:07 AM
Logging Method: Stop logging when memory full
Total Memory: 2095104 Readings 17 Days 7 Hours 41 Minutes 40 Seconds
User Selected Memory: 403340 Readings 3 Days 8 Hours 1 Minutes 40 Seconds
Used Memory: 120869 Readings 23 Hours 58 Minutes 55 Seconds (5.8%)
The logger was last configured at: 6/25/2016 10:27:12 AM
The logger is currently logging data
USB:18 Properties
Baud Rate 115200 Bits/second
Timeout 5000 Milliseconds
Retry 5 Times
Packet Size 4000 Bytes
Channels:
Channel # Enabled Description Equation Cali. Low Cali. High
0 [Thermistor] CH0 Temperature 0 0
1 [TC Range3(-2 to +18mV)] Tc 1 ThermocoupleK 0 0

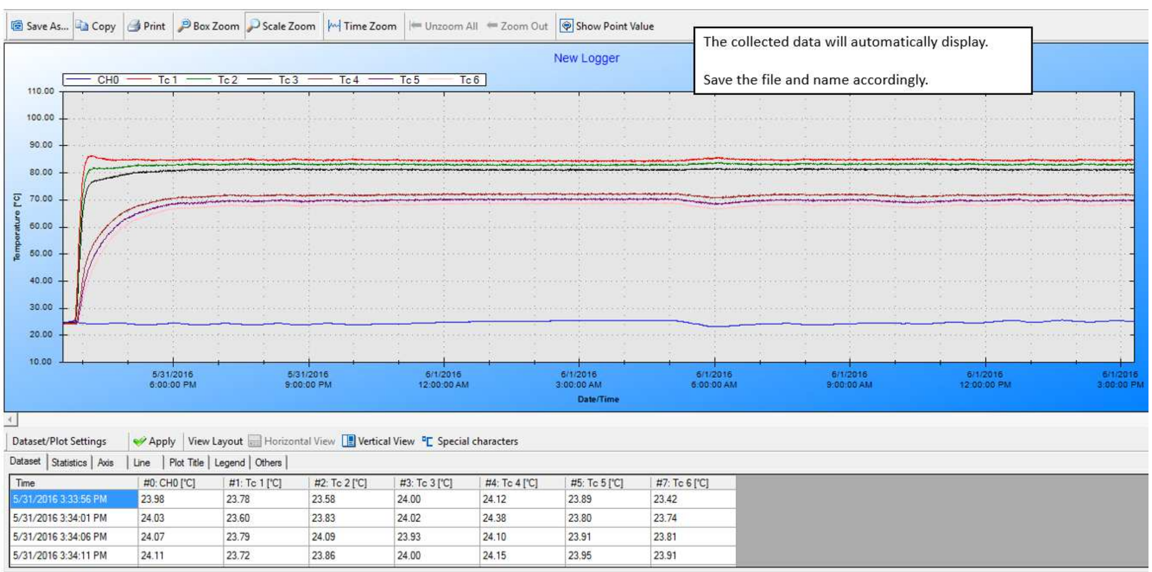


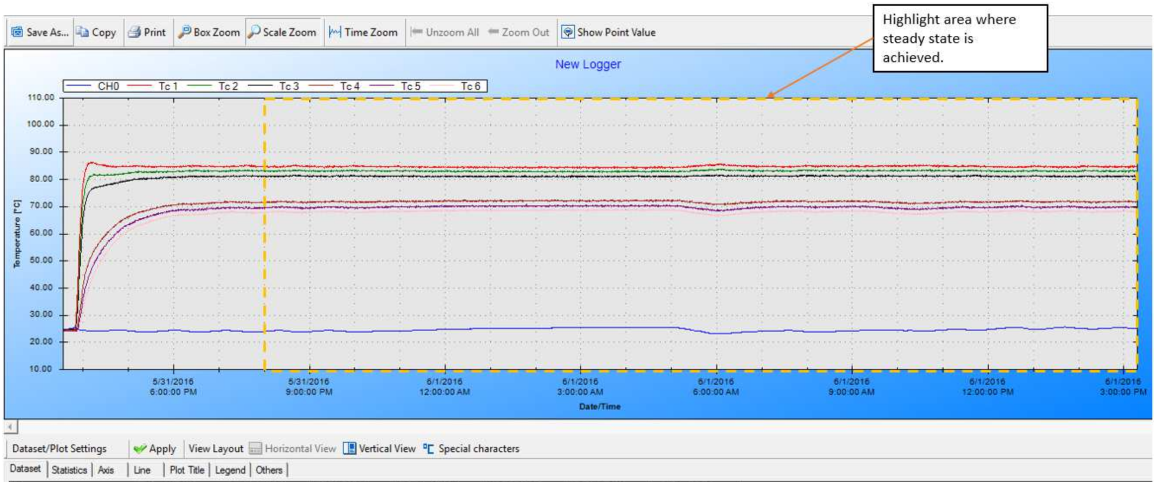


After the test time period has past, set up the load cell as shown earlier in this document.

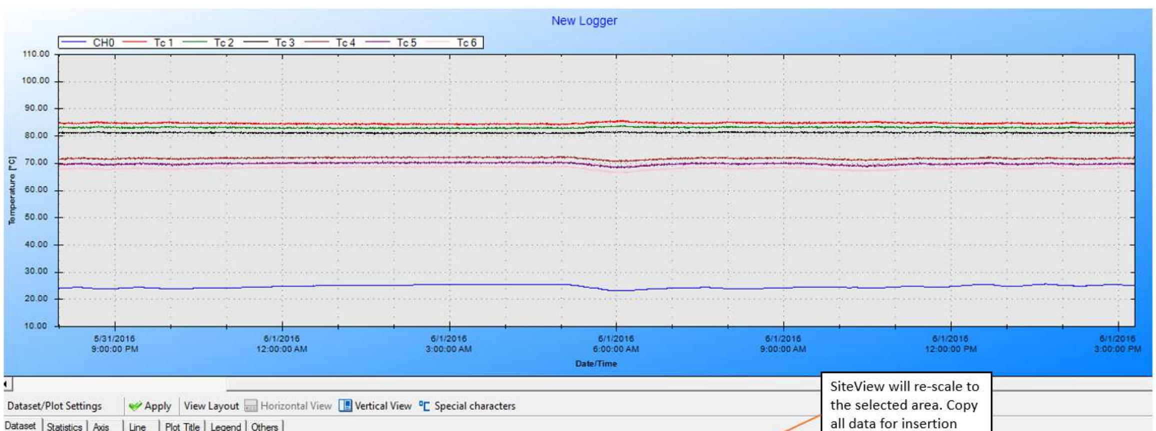
Due to thermal expansion an increase of load is expected. Record this amount as the test load.


Reconnect to the data acquisition system and download the data.


The collected data will automatically display.
Save the file and name accordingly.



Time	#0: CH0 [°C]	#1: Tc 1 [°C]	#2: Tc 2 [°C]	#3: Tc 3 [°C]	#4: Tc 4 [°C]	#5: Tc 5 [°C]	#7: Tc 6 [°C]
5/31/2016 3:33:56 PM	23.98	23.78	23.58	24.00	24.12	23.89	23.42
5/31/2016 3:34:01 PM	24.03	23.60	23.83	24.02	24.38	23.80	23.74
5/31/2016 3:34:06 PM	24.07	23.79	24.09	23.93	24.10	23.91	23.81
5/31/2016 3:34:11 PM	24.11	23.72	23.86	24.00	24.15	23.95	23.91



Time	#0: CH0 [°C]	#1: Tc 1 [°C]	#2: Tc 2 [°C]	#3: Tc 3 [°C]	#4: Tc 4 [°C]	#5: Tc 5 [°C]	#7: Tc 6 [°C]
6/1/2016 3:17:41 PM	25.00	84.86	83.33	81.11	72.18	69.57	68.02
6/1/2016 3:17:46 PM	25.03	85.03	83.23	81.18	71.91	69.61	67.88
6/1/2016 3:17:51 PM	25.07	85.06	83.25	81.29	71.94	69.60	68.33
6/1/2016 3:17:56 PM	25.10	85.06	83.34	81.19	72.03	69.80	68.36

The below data was taken from ABS Run 1 70-3.0-1.0

Time Stamp	Room Temp	Tc 1	Tc 2	Tc 3	Tc 4	Tc 5	Tc 6
6/21/2016 21:11	24.51	84.34	83.55	83.21	40.35	39.99	39.14
6/21/2016 21:11	24.51	84.09	83.65	82.92	40.45	39.78	39.31
6/21/2016 21:11	24.51	84.26	83.65	83.27	40.56	39.88	39.19
6/21/2016 21:11	24.51	84.2	83.6	83.06	40.51	40.16	39.06
6/21/2016 21:12	24.51	84.32	83.48	83.21	40.63	39.88	39.34
6/21/2016 21:12	24.51	84.3	83.69	83.27	40.92	39.71	39.27
6/21/2016 21:12	24.51	84.08	83.49	83.17	40.75	39.9	39.09
6/21/2016 21:12	24.51	84.14	83.4	82.94	40.57	39.95	39.28
6/21/2016 21:12	24.51	84.17	83.54	83.02	40.58	39.96	39.17
6/21/2016 21:12	24.51	84.14	83.71	83	40.63	40.1	39.24
6/21/2016 21:12	24.51	84.25	83.53	83.27	40.67	40.09	39.45
6/21/2016 21:12	24.51	84.22	83.52	83.25	40.49	40.17	39.31
6/21/2016 21:12	24.51	84.24	83.72	82.99	40.41	40	39.19
6/21/2016 21:12	24.51	84	83.83	83.06	40.47	39.61	39.42
6/21/2016 21:12	24.51	84.23	83.73	83.16	40.47	39.98	39.34
6/21/2016 21:12	24.51	84.09	83.67	83.15	40.64	40.04	39.33
6/21/2016 21:13	24.51	84.12	83.84	83.18	40.76	40.13	39.33
6/21/2016 21:13	24.51	84.02	83.62	83.17	40.42	39.69	39.17
6/21/2016 21:13	24.51	84.28	83.68	82.96	40.64	40.07	39.44
6/21/2016 21:13	24.51	84.31	83.71	83.2	40.48	39.9	39.25

	Average	Std Dev	Mean Error	+/- Error
Room Temp	24.54	0.05	0.05%	0.01
Tc 1	84.16	0.14	0.12%	0.10
Tc 2	83.54	0.14	0.12%	0.10
Tc 3	83.07	0.13	0.12%	0.10
Tc 4	40.91	0.19	0.17%	0.07
Tc 5	40.24	0.19	0.17%	0.07
Tc 6	39.61	0.20	0.17%	0.07

Number of Samples 12968

Calibration Run 2 | Calibration Run 3 | ABS Run 1 | ABS Run 2 | ABS Run 3 | Heat Flux Calcs Bas ...

Paste into Excel for analysis, repeat for each run conducted.

After inputting all runs, check for statistical outliers.

ABS Runs	Run 1	Run 2	Run 3	Run 4	Run 5		Mean	Stdev
							q	1.79
Room Temp	24.54	24.00	24.89	24.58	24.14		24.43	0.356
Tc 1	84.16	80.54	80.41	80.69	80.02		81.16	1.692
Tc 2	83.54	80.14	79.96	80.12	79.39		80.63	1.655
Tc 3	83.07	79.23	79.14	79.29	78.67		79.88	1.800
Tc 4	40.91	41.56	42.24	42.14	41.94		41.76	0.541
Tc 5	40.24	40.84	41.54	41.42	41.23		41.05	0.526
Tc 6	39.61	40.13	40.88	40.75	40.54		40.38	0.519

Outlier Matrix	Run 1	Run 2	Run 3	Run 4	Run 5
Room Temp	0	0	0	0	0
Tc 1	0	0	0	0	0
Tc 2	0	0	0	0	0
Tc 3	0	0	0	0	0
Tc 4	0	0	0	0	0
Tc 5	0	0	0	0	0
Tc 6	0	0	0	0	0

Heat Flux Calcs GDLS | Thermal Conductivity GDLS | Mat. K Tables | Outlier Check | Total Error (...

If the cell shows a "1" the corresponding measurement is to be omitted from further calculations.

Lower Flux Meter

Location (Tc)	Temp	
0	41.56	Run 2
0	42.24	Run 3
0	42.14	Run 4
0	41.94	Run 5
0.016	40.84	Run 2
0.016	41.54	Run 3
0.016	41.42	Run 4
0.016	41.23	Run 5
0.032	40.13	Run 2
0.032	40.88	Run 3
0.032	40.75	Run 4
0.032	40.54	Run 5

Calculated Delta T

2.40 C

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.989065
R Square	0.978249
Adjusted R Squar	0.972811
Standard Error	0.178722
Observations	6

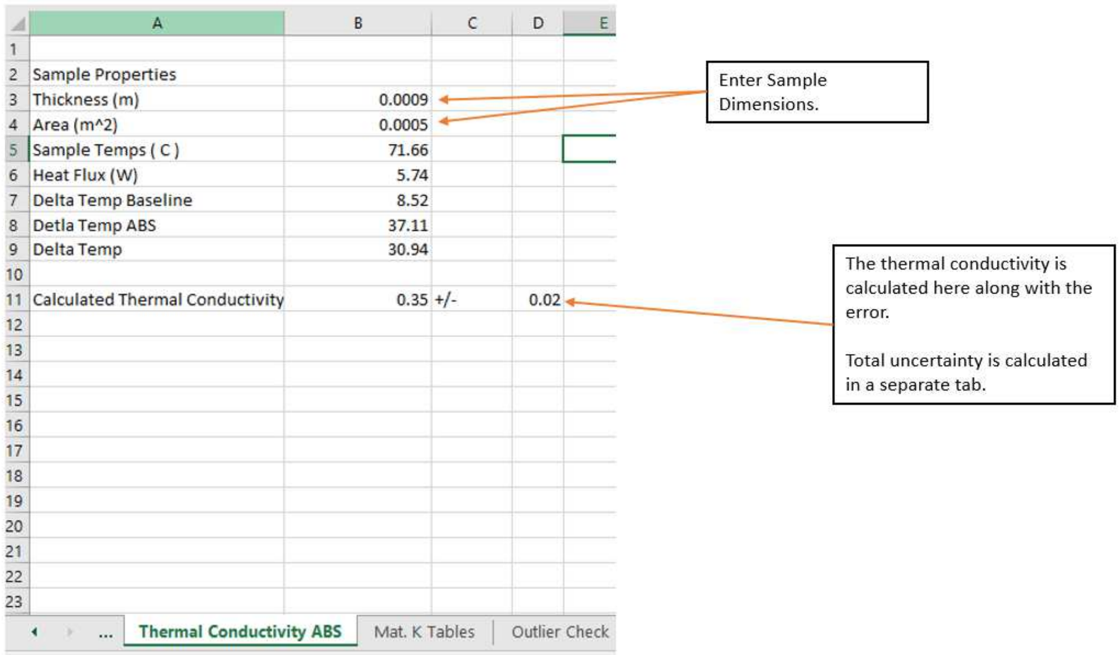
ANOVA

	df	*SS*	*MS*	*F*	*gnificance F*
Regression	1	5.746182	5.746182	179.8971	0.000179
Residual	4	0.127766	0.031941		
Total	5	5.873948			

	Coefficients	*tandard Err*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*ower 95.0%*	*pper 95.0%*
Intercept	57.29142	0.115364	496.6125	9.86E-11	56.97112	57.61172	56.97112	57.61172
X Variable 1	-75.5046	5.629388	-13.4126	0.000179	-91.1343	-59.8749	-91.1343	-59.8749

Preform a regression for both the lower and upper flux meters to calculate the heat flux of the runs.

Repeat these steps for the baseline runs and calibration runs.



A
B
C
D
E
Sample Properties
Thickness (m)
0.0009
Area (m^2)
0.0005
Sample Temps (C)
71.66
Heat Flux (W)
5.74
Delta Temp Baseline
8.52
Detla Temp ABS
37.11
Delta Temp
30.94
Calculated Thermal Conductivity
0.35
+/-
0.02
Thermal Conductivity ABS
Mat. K Tables
Outlier Check
Enter Sample Dimensions.
The thermal conductivity is calculated here along with the error.
Total uncertainty is calculated in a separate tab.

APPENDIX C

CORRESPONDANCE WITH MANUFACTORS

Re: Contact Form Submission | C-Therm

1 message

Adam Harris <aharris@ctherm.com> Sat, May 28, 2016 at 12:10 PM
To: Brent Sucher <bsucher@asu.edu>, Info <info@ctherm.com>

Hi Brent - these types of systems typically range in cost between 30K - 50K USD.

Best regards,

Adam

Sent from my BlackBerry - the most secure mobile device - via the Rogers Network

---

From:bsucher@asu.edu
Sent: May 28, 2016 8:07 PM
To:harris.adam.c@gmail.com; info@ctherm.com
Subject: Contact Form Submission | C-Therm

This entry was submitted on Saturday, May 28, 2016

First Name: **Brent**

Last Name: **Sucher**

Email: **bsucher@asu.edu**

Phone: **4802293953**

Company: **Arizona State University**

Street: **7001 E Williams Field Rd**

City: **Mesa**

State: **Arizona**

Zip: **85212**

Country: **United States**

AdWords Lead:

Information Package: **TCi: Thermal Conductivity Analyzer**

Hi,

I\'m currently a student at Arizona State University working on my degree in engineering. For one of my course this summer I\'m working on a paper about thermal conductivity and how systems determine what the conductivity of a material is. I would like to include some data on how much this kind of lab equipment cost. Would it be possible to get a ball-park on what your systems sell for so I may include that information in my paper?

Thank you for you time,

Brent Sucher

RE: Contact Form Submission

1 message

Dale Hume <dhume@thermtest.com> Mon, May 30, 2016 at 7:50 AM
To: "bsucher@asu.edu" <bsucher@asu.edu>

Hello Brent,

Sounds like a great paper. I would love to read it…be happy to provide thought if interested.

Price range: $15K to 80K.

Regards,

Dale Hume

Thermtest Inc

E-mail: dhume@thermtest.com

Office: 506-458-5350

Fax: 866-274-5269

Website: www.Thermtest.com

From: Thermtest [mailto:thermtest-noreply@thepulsegroup.ca]
Sent: Saturday, May 28, 2016 3:00 PM
To: Dale Hume <dhume@thermtest.com>
Subject: Contact Form Submission

First Name: Brent
Last Name: Sucher
E-mail: bsucher@asu.edu
Phone Number: 4802293953
Company:
Address:
City:
State/Province:
Postal Code:
Country:

Products: TPS Thermal Conductivity System
Comments: Hi, I'm currently a student at Arizona State University working on my degree in engineering. For one of my course this summer I'm working on a paper about thermal conductivity and how systems determine what the conductivity of a material is. I would like to include some data on how much this kind of lab equipment cost. Would it be possible to get a ball-park on what your systems sell for so I may include that information in my paper? Thank you for you time, Brent Sucher

RE: Request for information webcontact

1 message

Robert Dolce <rdolce@huksefluxusa.com> Mon, May 30, 2016 at 7:14 PM
To: "bsucher@asu.edu" <bsucher@asu.edu>

Brent,

Please see pricing below for the various thermal conductivity system models available.

THISYS System: $20,985.00

THASYS System: $19,589.00

TPSYS02 System: $10,500.00

FTN01 System: $12,595.00

MTN01 System: $11,475.00

TNS01 System: $16,235.00

Regards,

Robert Dolce

Managing Director, HukselfluxUSA Inc.

HukselfluxUSA

15 Frowein Road, Suite E-3

Center Moriches, NY 11934

631-251-6963 (office)

631-618-1702 (mobile)

631-657-0364 (fax)