### Design of a Thermal Conductivity Measurement Device for Cryogenic Applications

A technical report submitted to the Department of Mechanical and Aerospace Engineering Presented to the faculty of the School of Engineering and Applied Science

University of Virginia - Charlottesville, Virginia

In partial fulfillment of the requirements of the degree:

Bachelor of Science in Engineering

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On my honor as a University student, I have neither given nor received unauthorized aid on this assignment as defined by the Honor guidelines for Thesis-related assignments.

Advised by Ethan Scott, Department of Mechanical and Aerospace Engineering

#### **Problem Statement**

The objective of the capstone project was to design and fabricate a cryogenic thermal conductivity measurement insert for an evaporation fridge capable of achieving a base temperature of 1 Kelvin. The overall challenge has several subcategories. First, the insert must obtain accurate measurements while fitting within the size constraints of the refrigerator. Second, the insert must allow simple mounting and detachment of test samples. Third, wires need to run the length of the insert to obtain sample measurements and connect to external hardware. Fourth, the sample must be in a vacuum sealed environment to prevent unwanted heat transfer and liquid helium leakage. Fifth, heat transfer to the sample mount must be minimized. The temperature gradient from the surface (273K) to the refrigerator floor (1K) is large and must be accommodated in the design. The sample mount must have an approximately uniform temperature to ensure measurement integrity. The number of different materials needs to be minimized to maintain thermal stability and accurate measurements.

Quantum computing technologies operate at temperatures near absolute zero to reduce thermal noise. Thermal noise negatively affects the quantum phenomenons of superposition, interference, and entanglement. These behaviors enable quantum computers to perform simultaneous computations to solve complex problems. At these cryogenic temperatures, there are significant challenges in ensuring stable material properties and obtaining accurate measurements. This is due to significant changes in material properties and behaviors. The design of quantum computer systems therefore requires minimal heat transfer and heat production. The project aims to obtain the thermal conductivities of different materials at a range of cryogenic temperatures, especially crystals and minerals, which currently are not available in the literature due to the difficulty of achieving these temperatures.

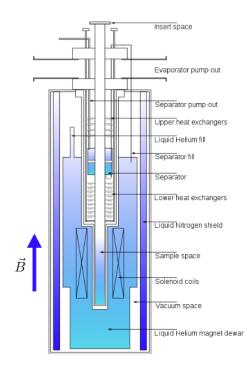
#### Research

There are many cryogenic sampling strategies, both commercially available and described in detail in academic journals. It is important to understand commonly used cryostat designs in order to understand the ideas utilized in other experiments that could be useful to solve the current problem. There are two common types of cryostats. The simplest and most common type are "immersion cryostats" or "dipper probes." For these, a sample is mounted on a copper block on the end of a low-thermal-conductivity tube, which is cooled by lowering the tube into cryogenic liquid. The more advanced category of cryostats are called "liquid-flow" cryostats. A sample is mounted onto a copper plate inside a vacuum, which is continuously cooled by transferring cryogenic fluid, lowering its temperature from room temperature (~300 K) to 1 K. Both of these cryostat designs are limited to the base temperature of the cryogenic fluid. In the case of liquid helium, this temperature is 4 K.

To achieve temperatures of 1 K, the evaporation fridge in this project combines aspects of both immersion and liquid-flow cryostats. A diagram of the fridge can be found in Figure 1. Similar to a liquid-flow design, the evaporation fridge transfers helium from a primary reservoir to a main sample reservoir. In the sample reservoir, the liquid helium cools the sample via conduction as well as intentionally pumping the liquid helium to evaporate and cool the sample further. Placing samples inside the evaporation fridge is more similar to an immersion cryostat than a liquid-flow, in that they must be inserted on the end of a low-thermal-conductivity tube. Research was conducted to explore existing inserts for both systems as there was limited information on evaporation refrigerator inserts (most of which are custom built). Much of the insight on the insert design was obtained from J.W. Ekin's 2006 book *Experimental Techniques for Low-Temperature Measurements*.

### Figure 1

## Diagram of Fridge Interior



Note. From Keller & Higgins (2019).

Most of the research involving liquid-flow cryostats was on their material selections, vacuum sealing, and wiring. Commercially-available products consist mainly of stainless steel. To allow easy access to the sample while still being able to hold a vacuum, the products use either an indium seal, a stainless steel conical seal, or an IVC (internal vacuum component) grease seal. Wiring also varied between products, but all designs either used a range of 5 to 12 twisted pairs and/or coaxial cables. Some designs from Cryo Industries of America referenced using flexible printed circuit boards (Kapton flex circuits) for wire installation and thermal anchoring.

A study conducted by Monchau et al. (2023) on the "Measurement of the Thermal Conductivity at Cryogenic Temperature" developed a device to measure the thermal conductivity of materials at low temperatures. The goal behind this project was to understand the thermophysical properties of materials for applications such as storing liquid hydrogen. The researchers designed a measurement apparatus based on an aluminum rod, with one end heated by an electrical resistor while the other end was cooled by liquid nitrogen. The resulting thermal gradient was monitored using thermocouples. To ensure reliable data, the entire setup was enclosed within a vacuum chamber to minimize heat loss through conduction and convection. Thermal conductivity was calculated based on the applied power, rod geometry, and the measured temperature difference. The device was tested using aluminum samples, with thermal conductivity measurements closely aligning with reference data from the National Institute of Standards and Technology (NIST).

The study found that annealing cycles led to variations in thermal conductivity exceeding 30%, highlighting the importance of accounting for a material's thermal history during characterization. The device achieved an uncertainty margin of less than 3%. The researchers emphasized that while bibliographic data can provide a baseline, direct measurements are necessary due to the variability introduced by thermal processes. Overall, both the study by Monchau et al. and the capstone project focus on measuring the thermal conductivity at cryogenic temperatures, yet there are some key differences in design and operational requirements. Both systems use the concepts of vacuums, various material samples, heaters to study temperature gradients, and devices to measure the temperature. However, this capstone project operates at a much lower base temperature of 1K, compared to 77K in the study. In addition, the heater in the capstone project is located on the cooling side of the rod whereas in Monchau et al.'s study, the heater was placed on the room temperature side. Although the goals and applications of these projects differ, this study serves as a baseline example of how similar

technical concepts, methodologies, and designs have been successfully applied in cryogenic testing.

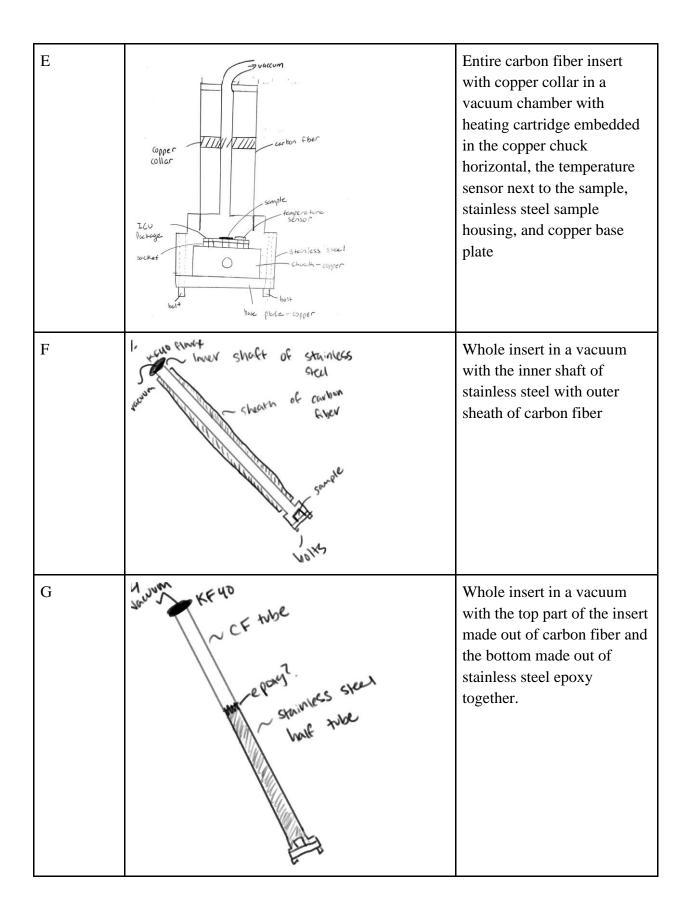
## Ideation

The group generated ten conceptual designs as part of the ideation process. Given the design requirements of a vacuum chamber fitting in a narrow 1.56" freezer opening, the design ideas are fairly similar outside of material changes and different arrangements of components at the bottom of the insert.

Idea	Conceptual Drawing	Description
Reference	Jocowith Ritor V-roled of e.F. graphike (verteed) of e.F.	Whole insert in a vacuum with the top part of the insert made out of pitch bonded graphite and the chamber is made out of stainless steel epoxy together
А	ICU ICU ICU ICU ICU ICU ICU ICU	Entire carbon fiber insert in a vacuum chamber with heating cartridge embedded in the copper chuck horizontal, the temperature sensor next to the sample, and stainless steel base plate

 Table 1 - Ideation

В	ICU Server S	Entire carbon fiber insert in a vacuum chamber with heating cartridge embedded in the copper chuck horizontal, the temperature next to the sample, and copper base plate
С	ICU Social Socia	Entire carbon fiber insert in a vacuum chamber with heating cartridge embedded in the copper chuck horizontal, the temperature sensor next to the sample, stainless steel sample housing, and copper base plate
D	source source source source but but but but but but but but but but	Entire carbon fiber insert with copper collar in a vacuum chamber with the sample mount set up from bottom for top as follows: copper base plate, insulative layer, heating layer, copper chuck, socket, ICU package, sample, and the temperature sensor next to the sample



Ι	20 stantess steel vod vod vod vecuuw tube prone tube vocuum control vocuum control vocuum control vocuum control	Whole insert in a vacuum by a vacuum sealed plastic tube with the top part of the insert made out of stainless steel and the sample mount chamber is weld to the top
J	Sounds to D Start to D Nuccount ford Pusche torre Auread T. Sourpe	Whole insert in a vacuum by a vacuum sealed plastic tube with the top part of the insert made out of stainless steel and the sample mount chamber is welded to the top with a removable base plate.

#### **Selection and Screening**

The group developed eight criteria for evaluating freezer insert designs. The most important criteria were ease of manufacturing with UVA facilities and accurate temperature readings (since the thermal conductivity measurements are unusable without the temperature). Vacuum sealability was also important for preventing liquid helium leakage into the electronics, which would ruin them. Cost was a less important constraint because most design ideas used similar materials but in different configurations. Since the sample mount will be quite small, the distance between the temperature sensor and the sample is also less important, because the copper chuck will ensure nearly uniform temperature distribution throughout the apparatus.

For the first round of selection, the group generated 10 design ideas with different materials, positions of the heater/temp. sensor/wires, and vacuum setup. After screening for requirements like manufacturability, price, and durability, designs A, B,C, D, and E were selected for further consideration. Pictures of designs of the reference as well as A-J are found in Table 3. For the second round of screening, a weighted score system was used to evaluate four hybrid designs. More important design requirements, like manufacturability or vacuum sealing, were given higher weights, while less important requirements, like price, had less weight. The sum of all weighting factors adds to 1. Scores were adjusted based on the +/0/- scoring for each hybrid design, leading to total scores of 0.3, 0.5, 0.7, and 0.6 for hybrids 1, 2, 3, and 4. The group opted to further pursue the copper base plate/collar/chuck design and the idea with a pitch-bonded graphite tube epoxied to a stainless steel sample housing chamber.

Reference: Carbon fiber to stainless junction with epoxy
A: Heater horizontal, stainless steel base
B: Heater horizontal, copper base
C: Stainless sample chamber, copper base
D: Stacked insulator/heater layers, carbon fiber
E: Copper collar, carbon fiber tube
F: Inner stainless sheath, outer carbon fiber
G: Pitch bonded graphite - stainless steel
H: narrow plastic chamber to stainless at top
I: electrical leads outside plastic tube

Table 3 - Selection 1 Criteria

	Refe renc									
Selection 1 Criteria	e	А	В	С	D	Е	F	G	Η	Ι
Ease of removing the chuck	0	1	1	1	1	1	1	1	1	1
Must be vacuum sealable	0	0	0	0	0	0	0	0	-1	-1
Ease of (dis)connecting cables	0	0	0	0	0	0	0	0	0	1
Ease of manufacturing	0	1	1	1	0	0	0	0	1	0
Temperature sensor in close proximity to sample	0	0	0	0	0	0	0	0	0	0
Accurate temperature reading	0	-1	-1	-1	0	0	0	0	-1	-1
Durability	0	1	1	1	1	1	0	1	1	1
Inexpensive Materials	0	0	0	0	0	0	0	-1	0	0
Pluses	0	3	3	3	2	2	1	2	3	3
Minuses	0	1	1	1	0	0	0	1	2	2
Sames	8	4	4	4	6	6	7	5	3	3
Net score	0	2	2	2	2	2	1	1	1	1
Rank	10	1	1	1	1	1	9	9	9	9
Continue?	No	Yes	Yes	Yes	Yes	Yes	No	No	No	No

 Table 4 - Selection 2 Criteria

		outer carbon fiber shell. Vacuum socket for		fiber shell with copper collar, insulative/heater		Hybrid 3: base plate, copper chu copper col carbon fib	ıck, lar,	<b>Hybrid 4:</b> Steel chamber epoxied to pitch bonded graphite tube, horizontally- mounted heater	
Selection 2 Criteria	weight	score	weight score	score	weight score	score	weight score	score	weight score
Ease of removing the chuck	0.1	1	0.1	1	0.1	1	0.1	1	0.1
Must be vacuum sealable	0.2	1	0.2	1	0.2	1	0.2	1	0.2
Ease of cable connections	0.1	0	0	0	0	-1	-0.1	1	0.1
Ease of manufacturing	0.25	-1	-0.25	0	0	1	0.25	0	0
Temperature sensor close to sample	0.05	1	0.05	1	0.05	1	0.05	1	0.05
Accurate temperature reading	0.15	1	0.15	1	0.15	1	0.15	1	0.15
Durability	0.1	1	0.1	0	0	0	0	0	0
Inexpensive Materials	0.05	-1	-0.05	0	0	1	0.05	0	0
	Net score	3	0.3	4	0.5	5	0.7	5	0.6
	Rank	4	4	3	3	2	1	1	2
Continue?		N	0	N	0	Y	es	Y	es

#### **Initial Specifications and Idea Development**

The specification for the insert originated with guidance from the project's technical advisor, Professor Scott, who briefed us on design constraints. The group also visited the UVA Physics Lab where the evaporation fridge is located and took measurements there. Specifications were deemed more important if the insert would not function if they were not met. If the insert could function, but would be more difficult to use, the importance was decreased. These specifications guided the many design iterations. Since holding a vacuum was such a crucial design aspect, many different seals were developed. One idea had a vacuum line going down the shaft, making only the sample mount area vacuum sealed. The final idea has the entire main structural tube vacuum sealed because the design was more straightforward.

Spec #	Metric	Measurement Method	Importance	Units
1	The insert must function between 1K- 300K maintaining consistent performance and structural integrity	Temperature sensors, pressure gauge	5	K Torr
2	The seals must hold a vacuum between 1K-300K.	Pressure gauge, thermometer, pressure checks throughout testing	5	K Torr
3	The vacuum must maintain a pressure of 1*10 <sup>-6</sup> Torr.	Pressure gauge	5	Torr
4	The cost of the insert must be less than \$1600.	Budget, detailed record of material costs	4	\$
5	The diameter of the entire shaft must be less than $1 9/16$ ".	Calipers	5	in.
6	The insert must be able to heat samples from 1K to around 50K for data collection.	Temperature sensor	4	K
7	The sample mount and its parts must be located about $\frac{1}{2}$ " from the end of the shaft.	Calipers	3	in.
8	Sample mount must be able to be easily taken apart to test each sample.	Time to assemble and disassemble, accessibility to the sample, ease of handling, durability	3	s
9	The product must allow accurate sample temperature measurements.	Temperature sensors	4	K
10	The insert must use a 40 KF flange seal on the top that mates with the existing fridge seal.	Verify that KF 40 mates with freezer, calipers, pressure gauge	5	in. Torr
11	The insert material must be insulative at cryogenic temperatures.	Thermal coefficient of expansion- material data, thermal conductivity	3	1/K W/mK

Table 5 - Initial Specifications
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12	The shaft must interface with a Pfeiffer HiCube vacuum pump.	Verify correct mating sizes, check seal with pressure sensor	5	Torr.
13	The fridge insert shall function as intended after repeated use in low K temperatures.	Cycle insert in low K temperatures to the sponsor's required number of times	3	Cycle
14	The wiring from shaft to sample mount shall be easy to disconnect/reconnect.	Time to connect/ disconnect, quality of the connection should remain the same after multiple uses (pressure gauge)	4	s Torr
15	The insert shall function as intended while succumbing to thermal expansion/contraction.	Ensure the shaft still fits/functions normally when repeatedly exposed to low K temperatures	4	in.
16	Shaft pieces must survive normal use of attachment and reattachment.	Attach and reattach shaft pieces the sponsor's required number of times to simulate their use case	5	Cycle
17	Samples must be easily connected to the IC package.	Time how long it takes to swap out material samples. Verify this is an acceptable time.	4	S
18	Electrical wiring must be able to carry an AC current of 300 mA for the $3\omega$ technique, and total electronics must not use more than 4 Watts of power.	Use properly rated wires, test current/voltage with an ammeter/multimeter	5	mA W

### **Technical Aspects**

Throughout the semester, many technical aspects were employed to gain perspective and insight into the problem. In order for the structure team to choose the best material given the unique application and budget constraints, the heat flux through tubes of various materials and diameters was analyzed. This analysis used applied knowledge from Heat and Mass Transfer (HMT), especially the one-dimensional, steady-state heat transfer equation. The sample mount team also used a concept from HMT to determine the thermal efficiency of the sample mount. Different resistances, cross-sectional areas, and thicknesses impacted the heat transfer and the efficiency of the system as a whole.

To calculate the heat transfer along the insert's length, it was treated as an unrolled plane wall with the circumference as its height, tube thickness as its width, and the tube height as the characteristic length. The side walls are insulated because they will be in a vacuum, so heat will only transfer laterally down the tube. With 1D, steady state heat transfer, the heat equation simplifies significantly to  $q=-k(T)*A_c*(dT/dx)$ . k(T) is the thermal conductivity of the material,  $A_c$  is the cross sectional area, and dT/dx is the temperature gradient, which is the slope of the temperature graph. Since it is in steady state, dT/dx is a constant equal to (300K -1K) divided by the length of the tube. 300K is the surface temperature and 1K is the floor temperature.

Since the thermal conductivity of materials depends on temperature, it changes down the length of the tube. The average thermal conductivity of the material was used, and found by integrating k(T) from 1K to 300K, then dividing by the operational temperature range. The group used previous research about k-T curves for various materials to find these average values, including a P. Duthil paper titled "Material Properties at Low Temperature" for stainless steel. Mathematical analysis highlighted that the thin tube would conduct less heat but the wide tube still had acceptably low heat transfer for the desired experiments. Therefore, the group selected stainless steel for ease of manufacturing, price, and its verified thermal properties.

To calculate the thermal efficiency of the chuck and base plate, the calculated the temperature difference between the top of the sample and the base (at 1 Kelvin) divided by the target temperature. Thermal efficiency represents how close the system is to obtaining the target temperature. The chuck is made out of copper, and the base plate is made out of aluminum. To simplify calculations, it was assumed that there would be no convective heat transfer since the

system is in a vacuum chamber. The temperature of the sample was calculated based on the heat transfer from the chuck to the socket to the sample through different resistivities, areas, and thicknesses of the given materials. Different resistivities, cross-sectional areas, and thickness greatly impacted the heat transfer and effectiveness of the system as a whole. Thermal efficiency determined whether the 50W cartridge heater would supply enough power to heat the sample so all temperatures from 1 to 100 K could be achieved. Therefore, higher values of efficiency imply closer adherence to the desired temperature range. Although the 50W heater had only 21.75% efficiency, the group purchased the 50W heater for the final design due to cost constraints.

The vacuum team determined the maximum external pressure of the selected stainless steel tube to ensure that the tube would not crack or fail while holding the vacuum. This required concepts from many mechanical engineering courses, including Aerospace Structures, Fluid Mechanics, and Machine Elements. Calculations verified that the stainless steel tube would not fail due to pressure differentials from the vacuum seal. 316 stainless steel (S.S.) was selected due to its yield strength at low temperatures. A safety factor of 5 ensured structural integrity. The max allowable pressure was related to yield stress using the hoop stress equation. The calculated max pressure of the 316 stainless steel tube is 3.248 MPa, significantly higher than the pressure of 1 atm found in the vacuum. This calculation verifies the fact that the insert will not fail in the vacuum due to pressure. The electronics team used mechatronics principles to develop the wiring diagrams and the connections between the different electrical components.

Spec #	Metric	Importance	Units	Marginal Value	Ideal Value
1	The insert must function between 1K- 300K maintaining consistent performance and structural integrity	4	K Torr	50K-300K* 10 <sup>-5</sup> -10 <sup>-7</sup> Torr	1K-300K <10 <sup>-6</sup> Torr

 Table 6 - Final Specifications

2	The seals must hold a vacuum between 1K-300K.	5	Torr	10 <sup>-5</sup> -10 <sup>-7</sup>	<10-6
3	The vacuum must hold a high vacuum.	5	Torr	10-5-10-7	<10 <sup>-6</sup>
4	The cost of the insert must be less than \$1600.	3	\$	<1650	<1600
5	The diameter of the entire shaft must be less than 1 9/16".	5	in.	1 1/2 - 1 5/8	<1 9/16
6	The insert must use a 40 KF flange seal on the top that mates with the existing fridge seal.	5	Torr	10 <sup>-5</sup> -10 <sup>-7</sup>	<10-6
7	The shaft must interface with a Pfeiffer HiCube vacuum pump.	5	Torr.	10 <sup>-5</sup> -10 <sup>-7</sup>	<10-6
8	Shaft pieces must survive normal use of attachment and reattachment.	4	Cycles	>20	>50
9	The wiring from shaft to sample mount shall be easy to disconnect/reconnect.	3	min.	<5	<3
10	Electrical wiring must be able to carry an AC current of 300 mA for the 3 $\omega$ technique, and total electronics must not use more than 4 Watts of power.	5	mA W	250-350 mA <4 W	300 mA <3 W

#### **Final Design Reasoning**

The initial design utilized a main shaft made of carbon fiber reinforced plastic (CFRP), instead of the final design's stainless steel shaft. This CFRP shaft would require epoxy connections to the upper and lower seals of the insert. CFRP was ideal for its low thermal conductivity compared to other materials and easy purchase in pre-manufactured parts. However, CRFP was abandoned because epoxy has not been verified for vacuum seals at cryogenic temperatures (Ekin, 2006), and the vacuum performance could be inconsistent due to irregular fiber weaves (Bechel, 2006). The final design utilizes a 316 stainless steel tube brazed to the top and bottom seals, maintaining a vacuum of 10^-6 Torr (Ekin, 2006) at cryogenic temperatures. The top of this tube is brazed to two KF flanges, which are bored to fit the diameter of the tube. One of the flanges is a KF50 flange and is for creating the seal between the top of the fridge and the area around the insert. The other flange is of size KF40 and is for creating a sealed interface for other standard KF components at the top of the insert. KF seals are standardized and commonly used for room-temperature sealing (Ekin, 2006). Since rubber seals will become brittle and eventually crack at temperatures nearing absolute zero, the seal at the bottom of the insert uses indium wire compressed via screws between a top and bottom flange. Indium wire is commonly used to make compact cryogenic seals because of its malleability and ability to withstand thermal cycling (Ekin, 2006). Indium wire can also be easily melted down and re-extruded for further use (HYPER Center, 2016). The bottom of the insert is brazed to a stainless steel ring with six tapped holes that are aligned with holes on the baseplate. #2-56 screws secure these two parts together with the ring of indium wire compressed between them to create the seal.

To conduct  $3\omega$  measurements, electrical leads extend from each of the four pins of the socket for +/- voltage and current. The temperature sensor also requires four leads and the cartridge heater needs two. The  $3\omega$  leads are the most sensitive and route through a BNC four-pin feedthrough that connects to the KF-40 flange. The other six wires are soldered to a 6-pin vacuum feedthrough on the surface. One of the most important design considerations for the electrical system is that it can be easily disconnected/reconnected. To ensure this is possible, two small pin terminals are soldered to a breadboard that attaches to the baseplate. These pin terminals will have screw connections to easily connect wires. After much deliberation, the group purchased Quad-Lead wires from Lakeshore Cryotronics, because they are pre-wrapped to prevent electrical noise and are rated for the extremely low operational temperatures. Surface connections were soldered to wires to connect to the Lakeshore 335 temperature controller and a computer to collect  $3\omega$  measurements.

The design of the sample mount from bottom to top is as follows: base plate, chuck, heater in bedded in the chuck, temperature sensor embedded in the chuck, socket, ICU package, and sample. The base plate is made of copper and the chuck is made of copper due to its high thermal conductivity, which is critical for uniform heat transfer to effectively heat the sample. The challenge was to manage heat flow precisely so the heater could increase the sample's temperature without affecting the sensitive socket and integrated circuit (ICU) package that houses the sample. This required a notched design, so that only the sample contacted the chuck directly. A regularly-shaped connection between the base plate and chuck ensures even heat flow and prevents any hot spots that may cause localized thermal expansion or stress concentrations. Discrepancy in thermal conductivity between different materials may produce uneven temperature distributions forming thermal stresses, which affects the sample's quantum properties and lead to inaccurate measurements.

The capstone project addressed the engineering challenge of designing a stable cryogenic environment to obtain precise material property measurements. This required minimizing heat transfer of the outer structure, ensuring material compatibility, maximizing heat transfer of the sample mount, and maintaining a pure vacuum. A successful design would provide accurate and precise data collection for understanding material properties at cryogenic environments useful for quantum computing.

#### Testing

First, the cartridge heater was tested with open loop control from the Lakeshore 335 controller set to 0.5A constant current output to ensure wiring continuity and functionality. The heater quickly heated up when tested. The insert was also tested for vacuum sealability. To complete this, a HiCube 300H Neo Vacuum pump was used. The first step to this process was to rough pump the insert to remove the dirt and debris. Once the pressure was low enough, a turbo pump activated to achieve a stronger vacuum. The turbo pump successfully got up to speed, and the pressure consistently dropped. After 30 minutes, the system achieved ~300e-5 millibars of pressure, and would likely continue to drop to the ideal 100e-6 range if the pump was left on. A picture of the insert connected to the vacuum pump can be seen in Figure 2.

#### Figure 2

Insert Connected to Vacuum Pump



The next step was to test the strength of the soldered and pin connections using a wire continuity test. Using a digital multimeter, leads were attached to leads of the multimeter to check connections from the surface to the baseplate, also ensuring that none of the wires shorted to ground. All of the connections functioned properly, and the leads of the Cernox sensor yielded measurements of  $41\Omega$ , in line with the expected 40.7 Ohm reading expected of the sensor at room temperature (based off of the datasheet).

The cartridge heater was later tested with a closed-loop feedback system to verify the Cernox sensor and heater worked in tandem. The Cernox temperature sensor purchased was uncalibrated due to cost constraints. The two Temperature-Resistance curves provided to the Lakeshore 335 temperature controller only ranged from 0 Kelvin to around 40K for the temperature sensor, but the insert first needed to be tested at room temperature. Therefore, a thermocouple was substituted for the Cernox sensor to ensure the heater worked in a closed loop at room temperature. The heater successfully utilized the feedback from the thermocouple to reach 310K and stabilize there. The thermal time constant of the heater was also determined to better understand how fast the heater could reach a given temperature.

#### **Summary and Conclusions**

After completing preliminary research, design ideation, and specification development the capstone group completed the first semester with parts ordered for a final design. Accompanying this final design is a fully assembled CAD drawing of the Kelvin Fridge insert and detailed drawings of individual parts. As mentioned in the Final Design Reasoning section of the report, this final design developed from constant iteration. The second semester was spent manufacturing and testing the design. The Gantt chart that was created in the beginning of the Spring semester was followed pretty closely, with some minor changes when manufacturing challenges came up. Many design challenges were overcome throughout the first semester. An early design change was to eliminate a CFRP shaft originally designed to provide structural support to the fridge insert. The CFRP shaft was removed due to its inability to maintain a vacuum seal at cryogenic temperatures. Furthermore, the sample mount orientation was modified from a horizontal position to a vertical one. This change was made to account for the extremely tight clearances for the sample mount region of the insert. A major challenge with this project has been accommodating the tight dimensions of the Kelvin fridge cavity. Since an appropriately sized flange is required to secure screws to the sample mount, all components placed within the sample mount and chuck needed to maintain a diameter of less than 1.25 inches. On the other hand, constrictive constraints, such as the maximum diameter of the insert, made it easy for the capstone team to converge on a final design. The numerous constraints provided by the problem statement led to preliminary designs that were similar in nature and limited design evolution once a design had been selected.

Our Capstone team constructed the Kelvin Fridge Insert after completing machine shop and soldering training including general, milling, and electrical. The small usable baseplate for the electronics was a challenge due to the difficulty of fitting all of the necessary components on the surface. To test different orientations, 3D printed chuck prototypes were tested for fit on the baseplate. These helped to visualize the design submitted in the fall. Testing included the heating cartridge fitting in the sample mount and properly securing it to the base plate, heating cartridge working, vacuum testing, and testing the heating cartridge with a thermocouple.

After completing this project, there is still future testing to be done and improvements to be made. Due to the Kelvin Fridge in the physics lab not being set-up and functioning this semester, the group was unable to test the insert at the temperature it was designed to function at. First, the temperature sensor must be calibrated to create a curve for the PID controller box that converts the resistance measurement to temperature. Testing at various temperatures down to 1K in the evaporation fridge is necessary for this calibration. This will allow the project advisor, Professor Scott, to utilize the  $3\omega$  method for measuring the thermal conductivity of his samples, which is the purpose of this project. For future design improvements, the current design could be improved upon given the size constraints to allow for horizontal sample mounting and heater placement to reduce the inaccurate measurements from uneven heating distribution.

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# Appendix A - BOM

Team	Item	Vendor	Part number	Cost/unit	Quantity	Fina	al Cost
Vacuum							
V001	KF 40 to KF 25 4 way flange	Amazon	UPC 64465067	\$ 69.99	1	\$	69.99
V002	10g High Purity Indium Wire 99.995% 1mmdiameterX1.8m Length	Amazon	7440-74-6	\$ 18.00	2	\$	36.00
V003	18-8 SS Screw, #2-56, 0.5 in. length (Pack of 100)	McMaster	92196A140	\$ 8.68	1	\$	8.68
V004	Electrical Coaxial Feedthrough, BNC, 4 Pin	LabIdeal.com	SKU: 400501	\$ 98.00	1	\$	98.00
V005	KF 40 Blank Centering Ring	IdealVac	P108633	\$ 43.25	1	\$	43.25
V006	KF25 Pressure Release Valve	Amazon	SKU: BMT-PRV	\$ 59.50	1	\$	59.50
V007	KF 40 Hollow Centering Ring + Clamps	Amazon	UPC 63111206	\$ 13.00	2	\$	25.99
V008	KF 25 Centering Ring with Clamps	Amazon	UPC 63111206	\$ 8.00	5	\$	39.99
V009	KF 25 3 way tee	Amazon		\$ 32.30	1	\$	32.30
V010	Brazing Flux, 8 oz.	McMaster	7693A1	\$ 8.60	1	\$	8.60
V011	High-Strength Brazing Alloy without Cadmium 1.1 oz., 1/16" Diameter, 5 Feet Long	McMaster	76955A72	\$ 57.87	1	\$	57.87
V012	KF40 Blank Flange	Amazon	631112071130	\$ 10.49	1	\$	10.49
V013	KF50 Blank Flange	Amazon	631112071147	\$ 11.49	1	\$	11.49
					Team Cost	\$	502.15
Structural							
S001	316 SS Tube - 1.25" OD, 0.035" Wall Thickness, 6 ft Length	McMaster	89995K618	\$ 59.71	1	\$	59.71
S002	Corrosion-Resistant 316 Stainless Steel Disc, 2" OD, 0.5" Height	McMaster	9260K1	\$ 15.33	1	\$	15.33
S003	Multipurpose 110 Copper Disc, 2" OD, 1" Height	McMaster	9103K2	\$ 42.11	1	\$	42.11
					Team Cost	\$	117.15
Electrical							
E001	Cartridge Heater	Lakeshore	HTR-50	\$ 78.00	1	\$	78.00
E002	Cernox Temperature Sensor (BO)	Lakeshore	CX-1030-BO	\$ 346.00	1	\$	346.00
E003	Quad-Lead Wire	Lakeshore	WQL-36-25	\$ 138.00	1	\$	138.00
E004	Dual Banana plug	Lakeshore	106-009	\$ 10.00	1	\$	10.00
E005	5 Pin Terminal Block	Pololu	#2494	\$ 3.45	1	\$	3.45
E006	6 Pin connector to Lakeshore control box	Lakeshore	G-106-233	\$ 13.00	1	\$	13.00
E007	KF40 - KF25 Reducer	Amazon	FRK011K40K25	\$ 20.99	1	\$	20.99
E008	8 pin wire feedthrough	Solid Sealing Tee	FA15103	\$ 184.00	1	\$	184.00
E009	Breadboard	Pololu	#2765	\$ 8.50	1	\$	8.50
E010	Thin rubber sheet	Amazon	B0C7T54NHL	\$ 6.99	1	\$	6.99
					Team Cost	\$	808.93
Sample Mount							
SM001	Alu. Bar, 1 ft Length, 1 in x 1 in	McMaster	9008K14	\$ 12.05	1	\$	12.05
SM002	ICU Package		CSB01652		1	1 No Cost	
SM003	Socket		110-43-316-41	\$ 2.69	5	No (	Cost
					Team Cost	\$	12.05
					Total cost	\$	1,440.28
					Budget	\$	1,600.00
					Over/Under Bud		159.72

# **Appendix B - Technical Aspects**

I. Mathematical Analysis of Structural Design

https://www.matweb.com/search/datasheet.aspx? MatGUID=abc4415b0f8b490387e3c922237098da&ckck=1

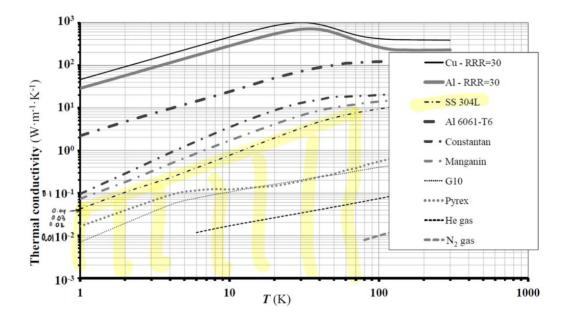
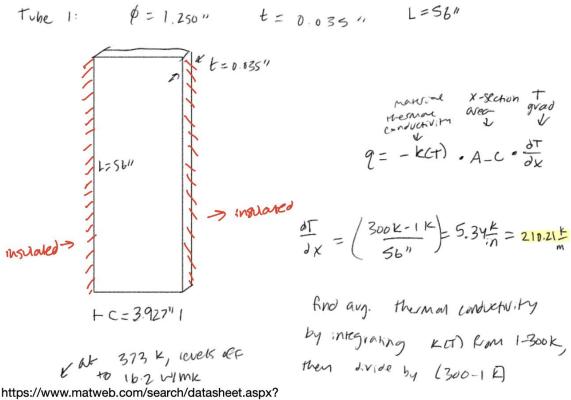


Figure 1: Thermal conductivity graph for selected materials, with stainless steel highlighted.



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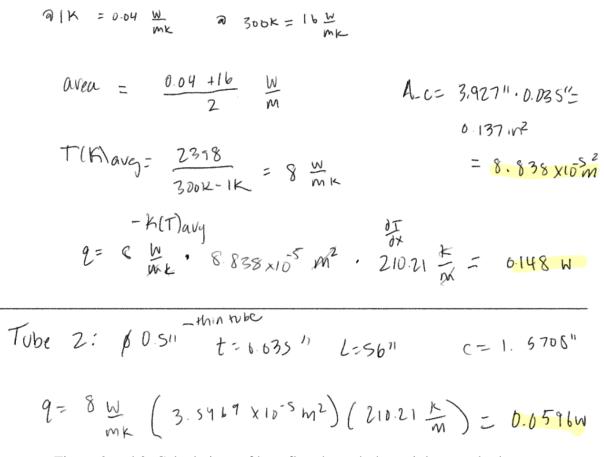


Figure 2 and 3: Calculations of heat flux through the stainless steel tube.

# II. Mathematical Analysis of Sample Mount Thermal Efficiency

Final design heat transfer calculations with aluminum chuck and copper base plate-50 Watt heater

```
The base temperature we wated to obtain was 14.

Temperature at the top of the sample

Temperature at the base + charge in temperature (AT)

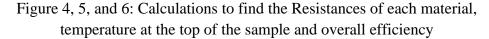
i

1h + AT = 1h + Q • R total = 1h + 50W (R total)

Efficiency = [Temperature at the top-Temperature at the base

+ target temperature we want to heat the sample to is

200 h once the sample reaches 2h
```



import numpy as np

# Constants for thermal conductivities (W/m\*K)
k\_aluminum = 237 # W/(m\*K)
k\_ICU = 0.5 # Black alumina W/(m\*K)
k\_socket = 121 # Brass alloy (360) W/(m\*K)
k\_copper = 401 # Copper (W/m\*K)

# Heat input (in watts)Q = 50 # Heater power in W

# Conversion factor from inches to meters inches\_to\_meters = 1 / 39.37

# Geometry
d\_base = 1.563 # Diameter of base in inches
d\_base\_meter = d\_base \* inches\_to\_meters # Diameter in meters
r\_base = d\_base\_meter / 2 # Radius in meters

# Dimensions for the chuck
l\_aluminum\_1= 1 \* inches\_to\_meters #length 1
l\_aluminum\_2= 0.55 \* inches\_to\_meters #length 2
w\_aluminum\_1 = 0.325 \* inches\_to\_meters # Width 1
w\_aluminum\_2 = 0.18 \* inches\_to\_meters # Width 2

# Dimensions for ICU package and socket (in meters)
1\_ICU = 0.8 \* inches\_to\_meters
w\_ICU = 0.31 \* inches\_to\_meters
1\_socket = 0.8 \* inches\_to\_meters
w\_socket = 0.4 \* inches\_to\_meters
# Area calculations
A\_base = np.pi \* r\_base\*\*2
A\_aluminum\_below\_heater = 1\_aluminum\_1 \* w\_aluminum\_1
A\_aluminum\_1 = 1\_aluminum\_1 \* w\_aluminum\_1
A\_aluminum\_2 = 1\_aluminum\_2 \* w\_aluminum\_2
A\_ICU = 1\_ICU \* w\_ICU
A\_socket = 1\_socket \* w\_socket

# Thicknesses of various layers
t\_base = 0.250 \* inches\_to\_meters
t\_aluminum\_below=.535 \* inches\_to\_meters
t\_aluminum\_top\_1 = (.140-.246\*.5 )\* inches\_to\_meters
t\_aluminum\_top\_2 = 0.200 \* inches\_to\_meters
t\_ICU = 0.135 \* inches\_to\_meters
t\_socket = 0.11 \* inches\_to\_meters
t\_heater = 0.246 \* inches\_to\_meters # Heater thickness

```
# Function to calculate thermal resistance (R = t / (k * A))
def thermal_resistance(t, k, A):
  return t / (k * A)
```

# Function to calculate the temperature at the top def calculate\_temperature\_at\_top(Q, R\_total, T\_base): delta\_T = Q \* R\_total  $return \ T\_base + delta\_T$ 

# Calculate individual thermal resistances
R\_base\_plate = thermal\_resistance(t\_base, k\_copper, A\_base)
R\_aluminum\_bottom = thermal\_resistance(t\_aluminum\_below, k\_copper,A\_aluminum\_below\_heater)
R\_aluminum\_top\_1 = thermal\_resistance(t\_aluminum\_top\_1, k\_aluminum,A\_aluminum\_1)
R\_aluminum\_top\_2 = thermal\_resistance(t\_aluminum\_top\_2, k\_aluminum, A\_aluminum\_2)
R\_ICU = thermal\_resistance(t\_ICU, k\_ICU, A\_ICU)
R\_socket = thermal\_resistance(t\_socket, k\_socket, A\_socket)

# Total resistances for top and bottom
R\_top = R\_aluminum\_top\_1+ R\_aluminum\_top\_2+ R\_socket + R\_ICU
R\_bottom = R\_base\_plate + R\_aluminum\_bottom

# Total resistance
R\_total = R\_top + R\_bottom

# Calculate the temperature at the top
T\_base = 1 # Bottom temperature in K
T\_top = calculate\_temperature\_at\_top(Q, R\_total, T\_base)

# Calculate efficiency and heat percentages
efficiency = abs(T\_top - T\_base) / 100 # 100K target temperature

# Print results
print(f"Efficiency: {efficiency:.5f}")

**RESULTS** Efficiency: 21.74652

### **III.** Maximum External Pressure

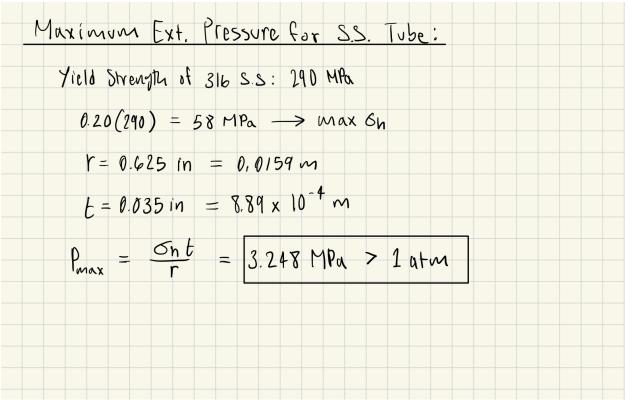
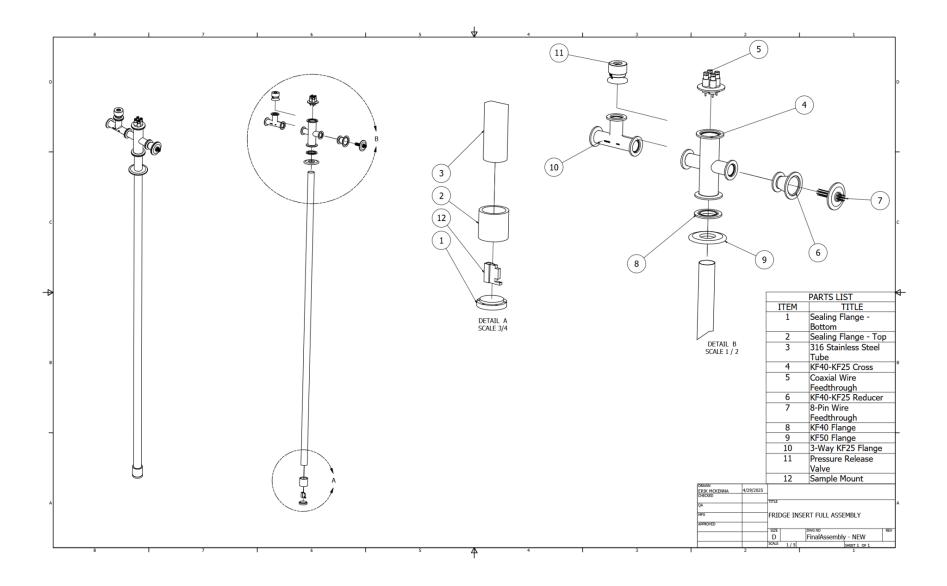
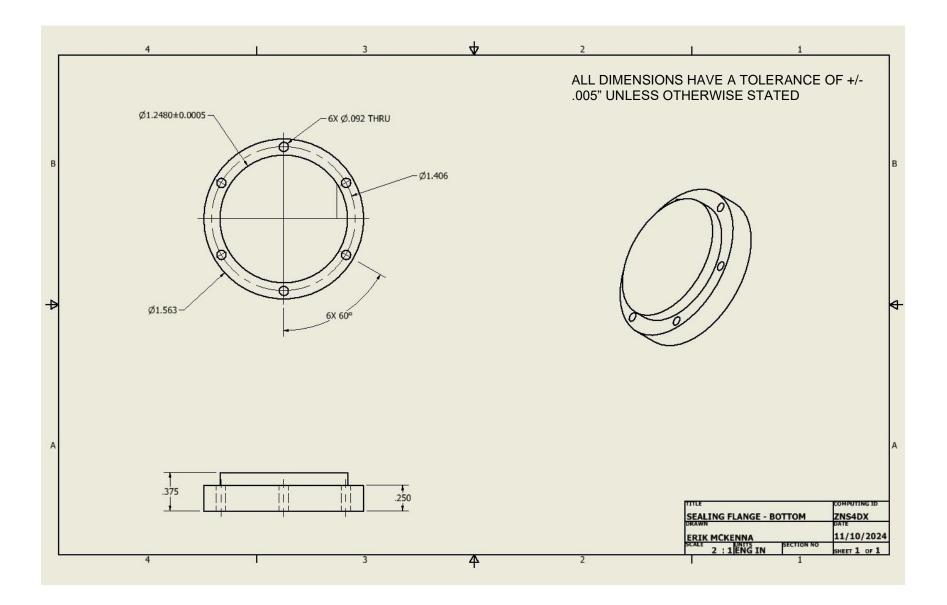
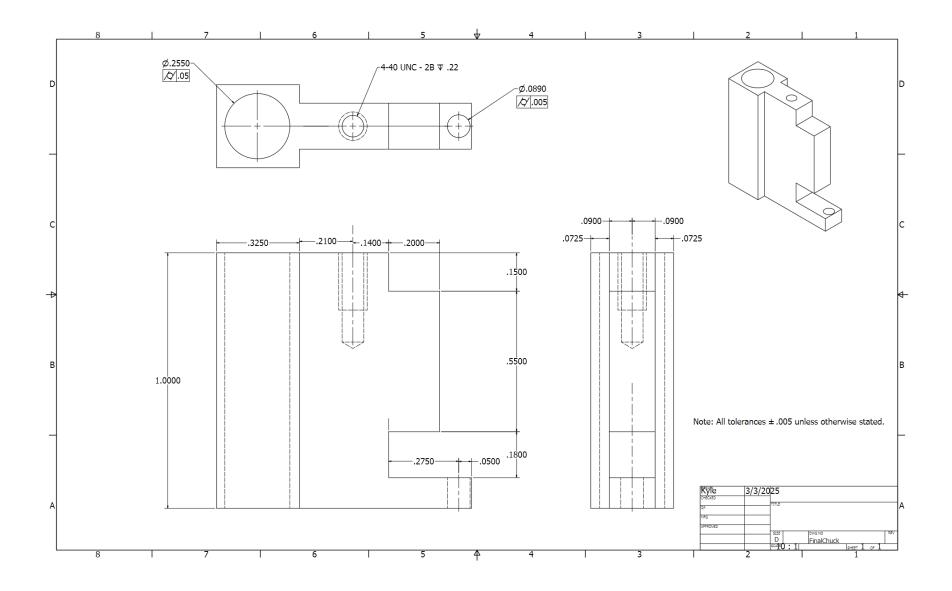


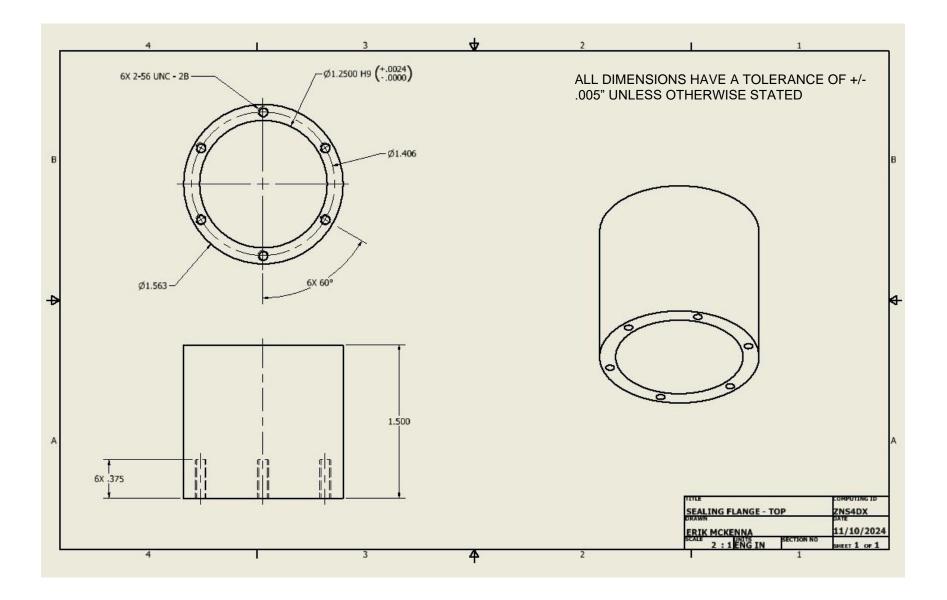
Figure 4: Calculations to find maximum external pressure on stainless steel tube.

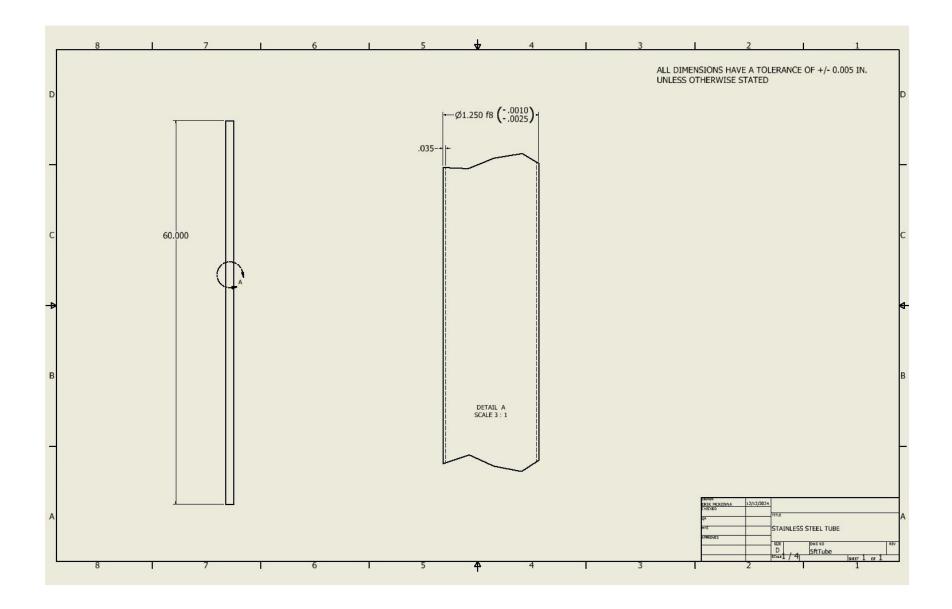
Appendix C - Detailed drawings.

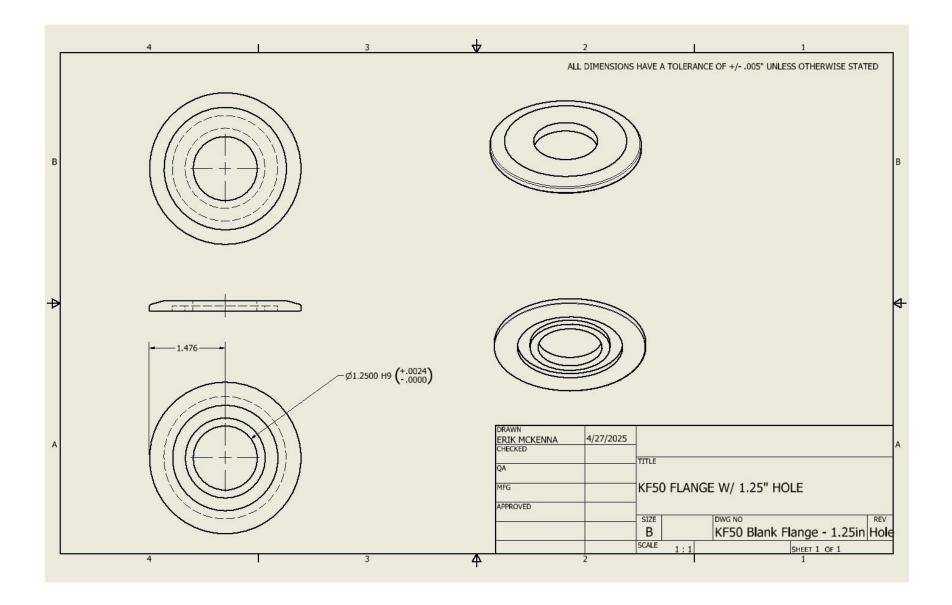


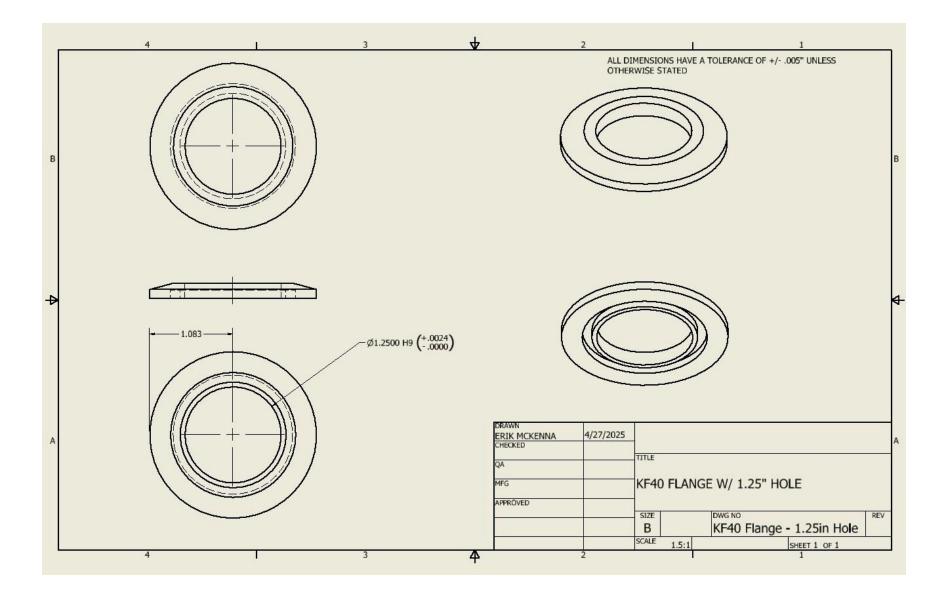












# Appendix D - Additional Figures



# Figure 3

Picture of Fridge in Physics Lab

# Figure 4

Wire-Bonded Samples



Examples of wire-bonded samples. We will use similar sample mounts in our fridge insert.