Producing Isotopically Pure ^4He for Investigations of Quantum Turbulence

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Abstract

The study of quantum turbulence in helium-4 requires isotopically pure liquid. The present work will produce small quantities (tens of liquid milliliters) of such a liquid. The ratio of He-3 to He-4 in gas-well helium is around 10^-7. The purification method employed has routinely reduced this figure to 10^-9 and possibly as low as 10^-13. The method used is based upon techniques pioneered by Peter McClintock et. al. This paper describes design considerations and construction of a filtration apparatus similar to McClintock's heat flush, but with a design which is simpler to build and run. The apparatus was successfully tested.

Introduction

For several decades liquid helium has been used as a working fluid for fluid dynamics experiments. One would go to the trouble of working with a cryogenic fluid because helium's exceptionally low viscosity enables high Reynolds number experiments to be conducted with relative ease.

The desire to create ultra high purity helium-4 (He-4) originates in the study of it's superfluid nature at low temperatures. In particular, the experiments to be undertaken in the Ihas Lab here at the University of Florida seek to probe that dissipation of turbulent vortices in superfluid Helium at milliKelvin temperatures.

Because these experiments seek to investigate the fashion in which energy is dissipated by a superfluid, it is desirable to eliminate all other paths of energy dissipation. One such path is the viscosity of any normal fluid contaminants within the superfluid. It is relatively easy to filter out most contaminants because they freeze at cryogenic temperatures. The problem addressed in this paper is the removal of helium 3, which, being similar chemically and in most physical ways, is a particularly troublesome contaminant in the cryogenic regimine . In gas-well helium the ratio of He-3 to He-4 is around 10^-7, enough to introduce significant effects. We exploit the fact that at 1 K He-4 is a superfluid, while He-3 is a normal fluid.

Helium-3 acts as a contaminant because its quantum mechanical properties differ from those of He-4. In the particular: He-4 begins its transition to the superfluid state at 2.17 K, whereas He-3 does not do so until 2.6 mK. Therefore, when a mixture of He-3 and He-4 is cooled to, let us say 2 K, some fraction of helium-4 will be in the superfluid state, but all of the He-3 will remain a normal fluid. Therefore, the He-3 will serve to degrade the superfluid properties of interest.

Since filtration apparatus developed below exploits the gradual fashion in which helium undergoes the transition to the superfluid state, it will therefore be worthwhile to describe the nature of this transition explicitly. Tisza proposed that the phenomena involved can be theoretically understood within the framework of a "two-fluid" model of helium-4. [1] He models liquid helium below 2.17 K as a mixture of a normal fluid and a superfluid. The proportions in which these two components are present depend strongly upon temperature, such that when temperature is lowered from 2.17 K the fraction of normal fluid decays rapidly to an essentially pure superfluid below 1 K.

Early helium-4 purification schemes employed a "superleak" filter that, ideally, permits only a zero-viscosity fluid to pass. At temperatures below 2.17K, the superfluid component of He-4 will pass through the filter easily, leaving the non-superfluid He-4 and He-3 behind. The superleak is usually a porous material with pore sizes of 50 μm or less. The final product's purity is only as good as the superleak. Typically a He-3 to He-4 ratio of 10^-9 may be achieved.

More recently, Peter McClintock et. al. have developed a filtration technique which they call "heat flush". [2] Their technique is also based upon the two-fluid nature of superfluid He-4: heat flush makes use of the viscosity of the normal fluid to carry away the He-3 particles. A situation is created in which the normal fluid is made to flow in one direction while the superfluid flows in the other.

Fortunately, it is not difficult to create such a situation. Superfluid helium carries no entropy, therefore only the normal portion may conduct heat. Interestingly, in liquid helium the propagation of heat is accomplished by mass flow: the flow of normal fluid away from a heater. Therefore, if we turn on a heater in a pipe filled with liquid helium, the two fluid model predicts that the normal fluid will flow away from the heater in order to propagate entropy and the superfluid must flow toward the heater to conserve mass. Because the normal fluid has viscosity, it will tend to drag impurities (He-3) with it, leaving us with pure helium-4 near the heater, similar to the action of a diffusion pump. With such an apparatus, McClintock's product has only 1 part He-3 per 10^15 parts He-4. [2]

Because the heat flush produces dramatically superior product purity, without greatly increased complexity, it was decided that a heat flush filter would be built.

Design

The following is a detailed description of the design and fabrication process.

A heat flush filtration system is quite simple in comparison to the other apparatus used to study superfluid helium. The apparatus essentially amounts to a pipe with a heater inside. Calculations regarding the amount of heat required and tube dimensions resulted in a practical design.

McClintock has published analyses of the governing equations and ideal operating parameters. [3] The proportion of superfluid required dictates that the heat flushing process be performed between 1 and 2K. Further analysis indicates that the optimal temperature lies between 1.2 and 1.6K. A spreadsheet can quickly apply the final governing equations to a wide range of design variables such as flush tube diameter and length before selecting the final geometry. This approach also allows speedy analysis of design changes necessitated by practical concerns. An example of the spreadsheets used is included in Appendix 1.

Next, we must obtain liquid helium at 1.4K. The condensation point of helium is T = 4.2 K, but the ideal operating range of the filter lies substantially lower. It is common practice to cool helium into this range by forced evaporation. Vacuum is applied over the liquid, which lowers vapor pressure and reduces the amount of energy need for an atom to escape the liquid. As these atoms evaporate they carry away energy and cool the liquid. Vigorous pumping may reduce the temperature to as little as 1K.

By the time the desired operating regime is reached, a substantial fraction of the original atoms will have been pumped away. To use the helium more efficiently, and because pumping capacity is limited, only a small amount of helium will be cooled. The apparatus will reside within a tube, 2 inches in diameter, which will be lowered into a 100L transport dewar of liquid helium. The tube is vacuum insulated, like a thermos bottle, and has a tiny hole at the bottom, which is sufficiently small that the pressure difference between the helium in the dewar (atmospheric), the superfluid inside (10Pascal) may be maintained. This is achieved using a long, narrow capillary. A large pump reduces the pressure inside the tube, allowing us to cool only the liquid inside the tube. Theoretically, as atoms evaporate and are pumped away, new atoms are drawn in, but do not raise the temperature because they are superfluid and carry no entropy. Actually, there is some normal fluid at 1.4 K, so a small amount of heat is introduced, requiring that we run the pump continuously. Of practical concern is that the filtration apparatus must fit within the 2 inch tube and must rest at the bottom of the tube, which will be the coldest region being the farthest from the surrounding room. An illustration of the suction refrigerator appears in Appendix 2.

As stated above, the dynamic flushing process will occur within a pipe. This is for several reasons: it simplifies the analysis to a one dimensional problem, and construction will be simplified because we can place the heater in the same tube which is required to extract the product.

The heat flush tube must also be vacuum insulated. Because the heat flush relies upon a temperature gradient that will be created by heating the helium inside the flushing pipe, the pipe must not be in contact with the surrounding, colder, helium. This requirement stipulates that the flushing tube and heater be within a vacuum sealed container. The standard method of constructing such a container is to build a short cylinder which will be coaxial with the 2-inch suction refrigerator. The top end of our cylinder will be fixed to the pipes leading out of the fridge. The wall and bottom of the cylinder (can) is removable to facilitate construction and maintenance. The two parts are sealed together with an indium wire crushed between two flanges. See Appendix 3 for a detail drawing of the vacuum can and Appendix 4 for an assembly drawing of the entire apparatus.

The temperature gradient to be established along the flushing tube is critical to the filter's success. Therefore, thermometers (Scientific Instruments calibrated Germaium and RuO_2 sensors were used) are necessary to monitor the temperature of the heater and surrounding bath of helium. A Linear Research LR-110 picowatt bridge was used to measuring the resistance thermometers. Bath temperature is dependent upon the suction applied, which may be regulated by valves in the pumping line. Temperature control circuitry (Linear Research LR-130 PID controller) is employed to maintain the heater at the desired temperature difference from the bath.

Upon submersion in the transport dewar, the heat flush tube will be flooded with mixed He-3 and He-4. When the helium in the suction refrigerator has been brought into the operating temperature range (1.4 K), the heater will be activated and begin to flush away the He-3. The initial cleansing of the heat flush tube will require several hours in order to be sure that all He-3 has been removed. Afterward, one is left with pure He-4 near the heater and the challenge is to bottle it.

McClintock's group uses a vacuum insulated siphon to remove the product as a liquid. They then route the liquid to a heat exchanger that warms the product and allows it to expand into a gas bag. They can then pump the product into cylinders at their leisure. However, their group is set up for high capacity (liquid liters per hour) production and their methods are not necessary in a small scale operation such as is required here.

It was therefore decided that the bottling strategy could be much simpler. The flushing tube terminates at a needle valve, which is initially closed and remains so during the initial cleansing of the flushing tube. When the operator is satisfied that only He-4 remains at the heater, the valve is opened. Hydraulic pressure forces He-4 into the extraction tube, which is stainless steel (not vacuum insulated), and causes it to rise out of the suction refrigerator. A detailed drawing of the needle valve is included in Appendix # and the Assembly drawing in Appendix# illustrates the needle valve's position in the assembled apparatus. The product will rise above the level of the helium in the suction refrigerator because of the fountain effect—the pressure produced by the counter-flowing normal and superfluid. This results in an excess pressure, so that the product will evolve from the tube and can be collected in a 50 liter gas cylinder by a stainless steel flexible line. One may also close the needle valve and raise the fridge in the transport dewar until the desired pressure is reached in the gas cylinder. One would then lower the suction refrigerator, attached a new gas cylinder and repeat until the desired amount of product is obtained. Or, more product may be added to the initial container at higher pressure, limited by the strength of the apparatus (about 2 bar).

Conclusion

The apparatus is easy to operate and will yield product rapidly. Set up, cool down, and several product extraction cycles may all be performed in a single day.

Unfortunately, as of this writing, product purity has not been determined. This is due to the scarcity of sufficiently sensitive instruments. Ideally, this work would yield an absolutely pure product and one therefore seeks the most sensitive means available to quantify its purity. Low-Z Isotope Ratio Mass Spectroscopy, which currently has a sensitivity as high as one part per 10^10, is the preferred analysis tool in the field. Formerly this service was available from the United States Bureau of Mines. However, since this agency was disbanded, such instruments are scarce and usually used in geological analysis, which does not require such sensitive measurements. Currently, A search is underway for a convenient analysis facility.

Acknowledgements

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References

1 Tiza L 1938 *Nature* **141** 913

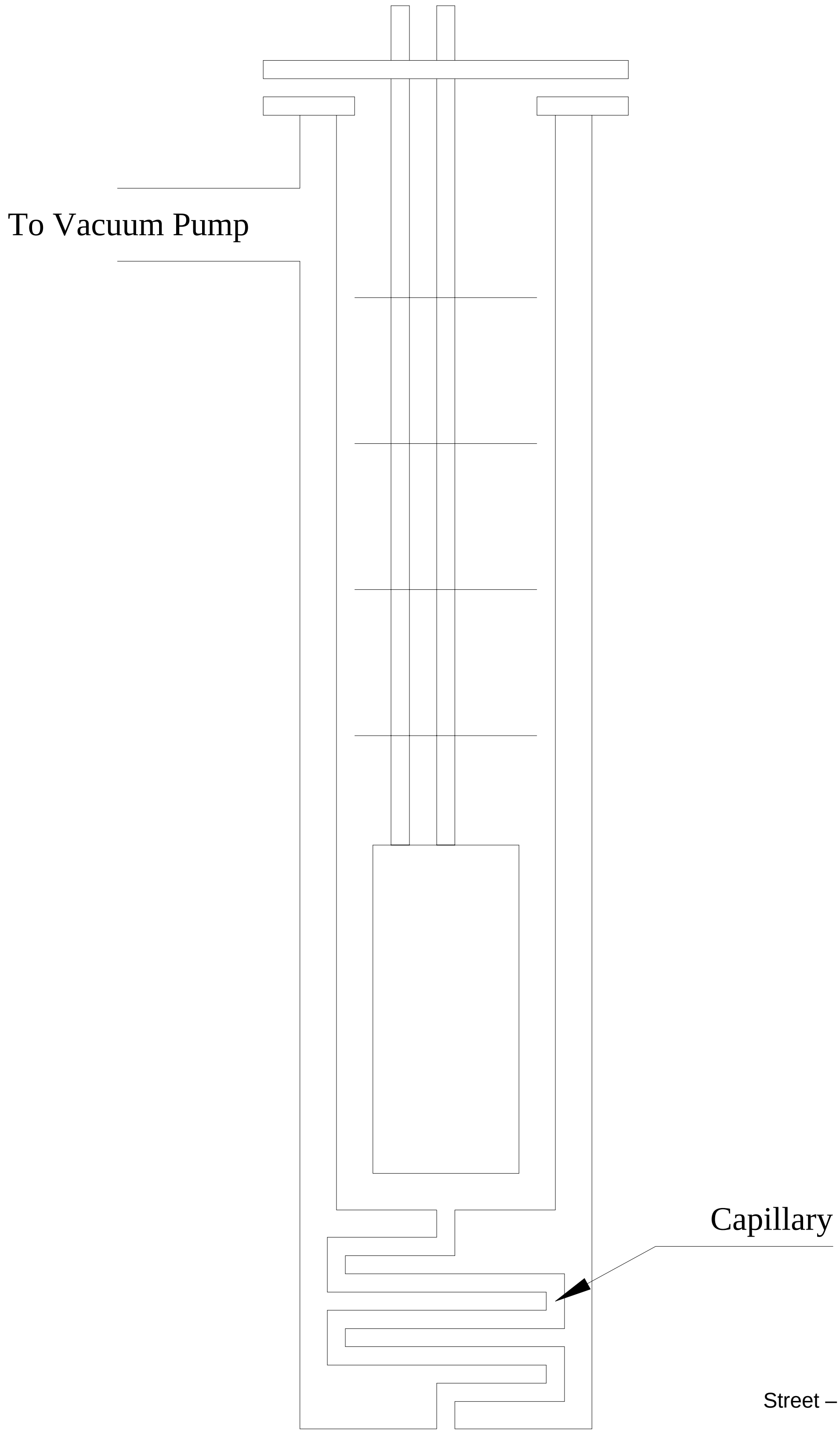
2 Scott, R.J. and McClintock P.V.E. 1977 *Physics Letters* **64A** 205

3 Hendry P.C. and P.V.E. McClintock 1987 *Cryogenics* **27** 131

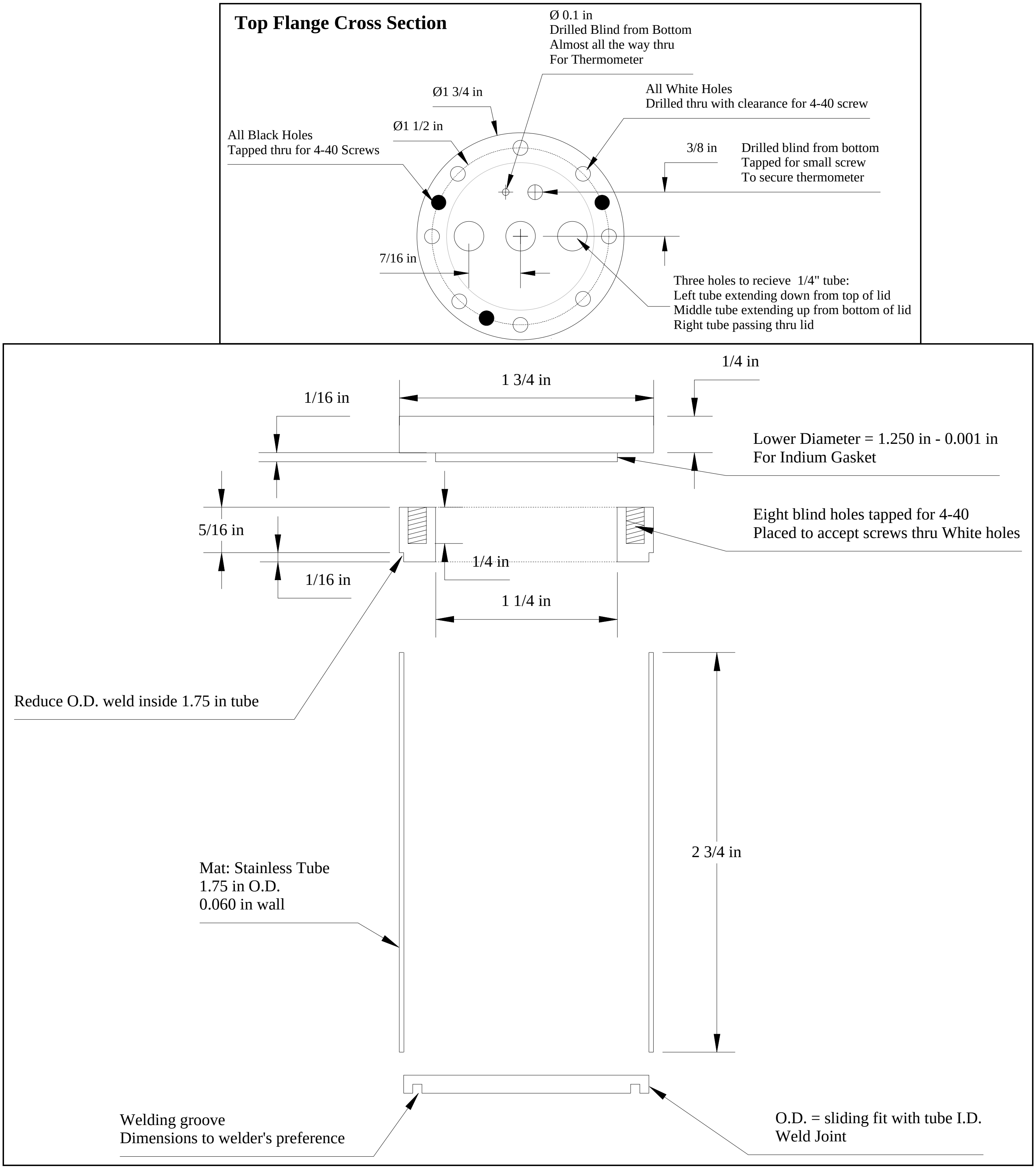
Appendix 1 – Design Spreadsheet

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	He-3 Concentration						Flush Tube				Heater		
2					target = 10^-31								
3	x (m)	Q (W/m^2)	T (K)	Vn/D	u(x)/uo		Dia (m)	C.S. area (m^2)	Q required (W)		Current (mA)	Voltage (V)	R (Ohms)
4	0.001	150	1.4	41667	8.0241E-19		0.0064	3.16692E-05	0.0048		3.8529	1.2329	320
5		175		48611	7.7350E-22				0.0055		4.1616	1.3317	
6		200		55556	7.4564E-25				0.0063		4.4490	1.4237	
7		225		62500	7.1878E-28				0.0071		4.7188	1.5100	
8		250		69444	6.9288E-31				0.0079		4.9741	1.5917	
9		275		76389	6.6792E-34				0.0087		5.2169	1.6694	
10		300		83333	6.4386E-37				0.0095		5.4488	1.7436	
11		325		90278	6.2067E-40				0.0103		5.6713	1.8148	
12		350		97222	5.9831E-43				0.0111		5.8854	1.8833	
13													
14													
15	Constants												
16													
17	1.38E-23	Boltzmann's constant (m^2 kg / (s^2 K))											
18	9.9697E-28	Mew (Kg)											
19	1.05E-34	H-bar (m2 kg / s)											
20	1.20E-22	Delta (m^2 Kg / s^2)											
21	2.05E-44	Po (m kg / s)											
22	124.8	Density (kg / m^3)											
23	2.40E+19	Alpha 1/(ms)											
24													
25													
26													

Appendix 2 – Schematic Diagram – Suction Refrigerator



Appendix 3 – Construction Drawing – Vacuum Can



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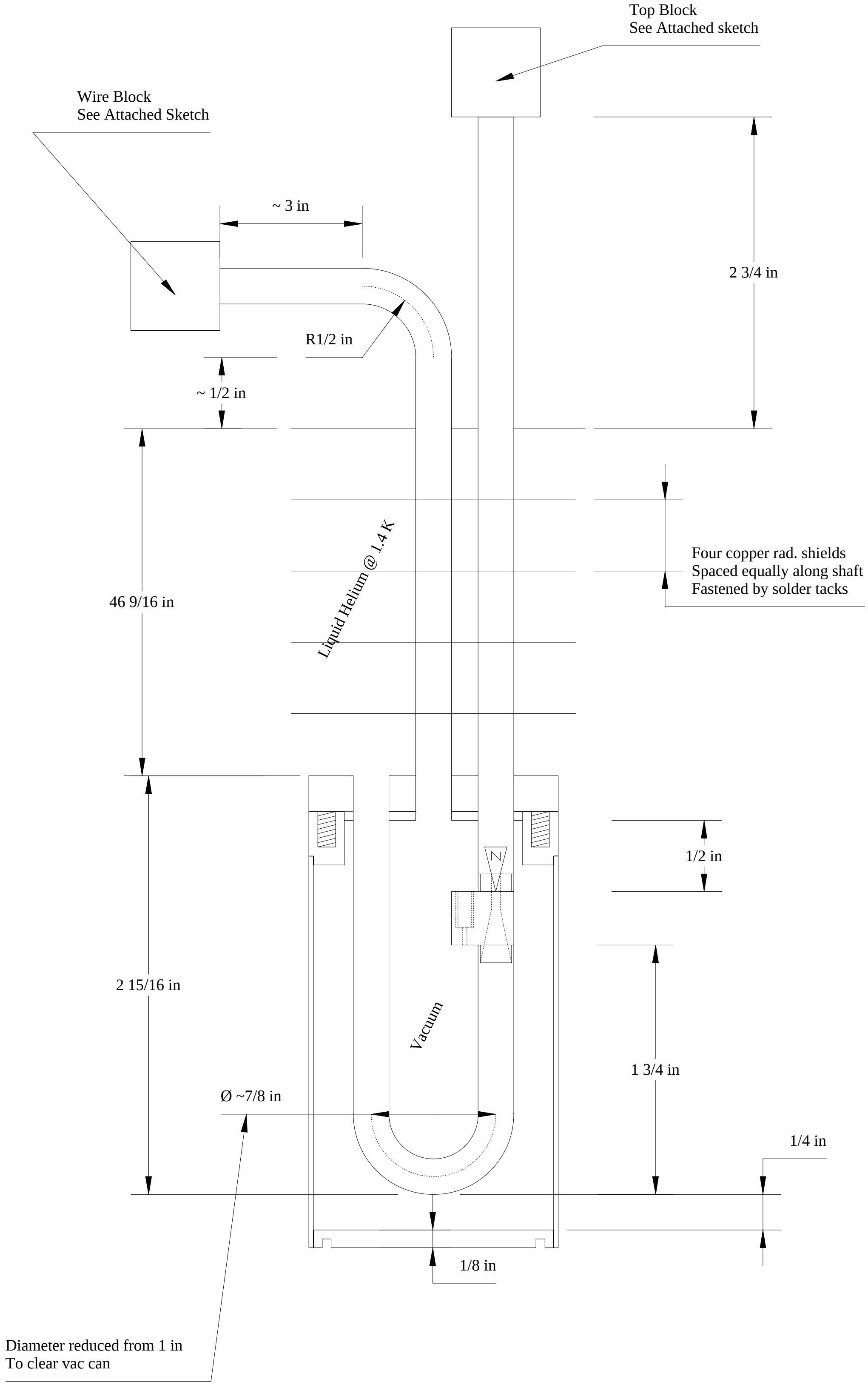
Appendix 4 – Schematic Diagram – Assembly



Appendix 1 – Construction Drawing – Needle Valve