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Shock Compression of Liquid Helium and Helium-Hydrogen Mixtures

Development of a Cryogenic Capability for Shock Compression of Liquid Helium on Z

Final Report for LDRD Project 141536

David L. Hanson, Andrew J. Lopez, Keegan P. Shelton, Marcus D. Knudson

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Abstract

This final report on SNL/NM LDRD Project 141536 summarizes progress made toward the development of a cryogenic capability to generate liquid helium (LHe) samples for high accuracy equation-of-state (EOS) measurements on the Z current drive. Accurate data on He properties at Mbar pressures are critical to understanding giant planetary interiors and for validating first principles density functional simulations, but it is difficult to condense LHe samples at very low temperatures (<3.5 K) for experimental studies on gas guns, magnetic and explosive compression devices, and lasers. We have developed a conceptual design for a cryogenic LHe sample system to generate quiescent superfluid LHe samples at 1.5-1.8 K. This cryogenic system adapts the basic elements of a continuously operating, self-regulating ^{4}He evaporation refrigerator to the constraints of shock compression experiments on Z. To minimize heat load, the sample holder is surrounded by a double layer of thermal radiation shields cooled with LHe to 5 K. Delivery of LHe to the pumped-He evaporator bath is controlled by a flow impedance. The LHe sample holder assembly features modular components and simplified fabrication techniques to reduce cost and complexity to levels required of an expendable device. Prototypes have been fabricated, assembled, and instrumented for initial testing.

Acknowledgements

The authors would like to thank Dr. Michael E. Cuneo and Dr. Michael P. Desjarlais for encouragement in proposing this work for Sandia LDRD support. We also would like to extend our thanks to Dr. James R. Asay for originally stimulating our interest in the problem of He shock compression measurements, and to Robert R. Johnston and Kevin Youngman for many valuable contributions to the development of cryogenic techniques and infrastructure currently in use on Z and for contributions to earlier explorations on the path to developing a LHe sample capability for Z.

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I. Motivation and Scope of LDRD

Accurate data on He properties in the Mbar pressure regime are critical to understanding the structure, origin, and evolution of the Jovian and extrasolar giant planets [1-3], and for comparison with first principles density functional simulations [4, 5]. Liquid He (LHe) data have been obtained at lower pressures using conventional gas gun techniques, but recent first principles calculations suggest a significant increase in compressibility (max 5.5 fold compression) just beyond the available data. More recent results using the Omega laser suggest even higher compressibility (max 7-fold at ~100 GPa). This pressure regime is well within the range of dynamic experiments at the SNL/NM Z facility. The refurbished Z accelerator is a pulsed-power-based current driver (26 MA) capable of producing magnetically-generated high pressure pulses, as well as energetic, intense pulsed x-ray sources. High-current-density short circuit geometries and current pulse-shaping techniques have been developed to provide magnetically-driven high pressure capabilities for launching hypervelocity flyer plates for dynamic material properties and equation-of-state (EOS) experiments. Given the long standing controversy surrounding the compressibility of liquid D_2 [6-9], it would be highly beneficial to obtain similar experimental results on LHe with the mature Z shock physics platform developed over the last decade. Therefore, there is a crucial need for a cryogenic capability to generate large area LHe samples for high accuracy shock compression experiments on Z.

The primary problem addressed by this LDRD is the development of a cryogenic capability to generate LHe samples for high accuracy EOS measurements using the Z accelerator current drive. The specific goals of this LDRD are: (1) to identify a suitable cooling method for condensing LHe samples in an appropriate geometry for shock physics measurements on Z; (2) to develop a detailed cryogenic system design; and (3) to fabricate, assemble, and instrument prototype test hardware to demonstrate the feasibility of generating LHe samples on Z experiments.

Very limited high pressure He EOS data exist because of the difficulty of condensing LHe samples at very low temperatures (below 3.5 K) for measurements on gas guns, magnetic and explosive compression devices, and laser facilities. The cryogenic target system currently in use on Z [10] is capable of condensing liquid samples for a variety of permanent gases with normal boiling points above 15 K, including H_2, D_2, Ne, N_2, O_2, Ar, Kr, and Xe. This system cools an unshielded cryocell by conduction through a thermal link to a LHe or LN_2 reservoir. The thermal link provides sufficient standoff to allow survival of the cryostat containing the liquid cryogen reservoir. This approach will not work for LHe samples. The thermal radiation heat load from room temperature (300 K) surroundings is much too high to permit cooling of an unshielded LHe sample to the 1.5-3.5 K temperature range, and cooling to this range by conduction through a long thermal link for standoff is also not feasible. Extending the high pressure loading capabilities of the Z current drive to He EOS studies requires major modifications to the existing cryogenic target system design. The following sections will describe the physics requirements, conceptual design, and cryogenic engineering details of a LHe sample system suitable for use on Z.

II. LHe Physical Properties Affecting Cryogenic System Design

II.1. Basic physical properties of liquid ^{4}He

Liquid ^{4}He (LHe) has a number of unique properties which place critical constraints on the design of a cryogenic system. These properties will be discussed briefly in this section.

Because of the closed s-shell of ^{4}He, the binding forces between atoms are extremely weak, resulting in a very low normal boiling point (4.21 K) and critical temperature (5.20 K) compared to other liquids. It is the only substance without a triple point. Because of its large zero-point energy, ^{4}He remains liquid under its own vapor pressure even when cooled to absolute zero and must be compressed to at least 25.4 bar to become a solid.

The λ-line in the P-T phase diagram of ^{4}He [11, p. 59] separates the region where ^{4}He behaves as a dense classical gas, known as ^{4}He I, from the region where it undergoes a Bose-Einstein condensation and exhibits a number of interesting properties of a quantum liquid, termed ^{4}He II. Below the λ-transition temperature (T_λ = 2.176 K at saturated vapor pressure), very high thermal conductivity leads to the rapid cessation of boiling. Viscosity is very low, and entropy and specific heat decrease rapidly with temperature. The Bose-Einstein condensation of atoms into the ground state in ^{4}He II may be described in terms of a two fluid model [11, p.101; 12, p.56]. In this model, ^{4}He II behaves as a mixture of a normal fluid which carries all the entropy of the liquid and has normal viscosity, and a superfluid component with zero entropy and very low viscosity. The normal and superfluid components are completely mixed but do not interact. As the temperature is reduced below the λ-point, more atoms occupy the ground state. The specific heat, which depends only on particles in excited states, falls off rapidly. By the time the temperature of ^{4}He has been reduced to 1 K, the ^{4}He II liquid is almost completely superfluid.

The latent heat of evaporation of liquid ^{4}He is rather small (~90 J/mol) compared to other cryogenic liquids [13, p.18]. As a result, LHe baths have limited cooling power. All low temperature experiments relying on LHe cooling must be efficiently insulated against heat flow from the surroundings in the form of radiation, heat conduction through supports, and heat flow along instrumentation wires.

Temperatures below the normal boiling point can be achieved in a LHe bath by pumping on the vapor above the liquid [13, p.20; 14, p.63,154]. As atoms are pumped away from the vapor phase, the most energetic atoms leave the liquid to replenish the vapor and the liquid will cool. The vapor pressure and cooling fall off exponentially with decreasing temperature and pumping becomes rapidly less efficient. The practical limit where evaporation cooling by the biggest pump is balanced by external heat flow to the bath in a well-insulated system is about 1.3 K for ^{4}He.

The specific heat of liquid ^{4}He is many orders of magnitude higher than other materials such as stainless steel and copper that are used in cryogenic apparatus to contain the LHe [13, p.21]. Also, the latent heat of evaporation of LHe, while small compared to the latent heat of most materials, is large compared to the specific heat of those materials at low temperature. As a consequence, LHe can rapidly cool other materials by evaporation in response to a changing heat load. The thermal response time of any apparatus will therefore be determined by the size and thermal behavior of its refrigerating LHe bath.

The vanishing viscosity of liquid ^{4}He II in the superfluid state [15, p.167] also has important consequences for experiment design. Superfluid ^{4}He at $T < T_\lambda$ will be able to flow through tiny cracks in an apparatus that was leaktight to viscous ^{4}He I at $T > T_\lambda$ [13, p.26]. A sample holder containing superfluid ^{4}He II will have to be fabricated to a very high standard, with nonporous material and quality welding and brazing, to avoid "superleaks".

Another effect involves the formation of a thick He film on the vertical walls of a container of LHe as a result of adsorption of atoms from the vapor phase [13, p.26; 14, p.64]. A normal fluid layer with finite viscosity will remain stationary on the wall. However, a superfluid ^{4}He II film with vanishing viscosity can move up the walls of a He bath fill tube by frictionless flow until it reaches a hotter region and evaporates. This can result in increased evaporation rates from superfluid ^{4}He II baths. The superfluid ^{4}He film flow rate for clean glass at $T < 2$ K is 7×10^{-5} C ml/(s cm), where C[cm] is the circumference of the exit tube. Transport rates can be three times higher for clean metal and ten times higher for dirty surfaces. The superfluid ^{4}He film flow rate up a tube can be limited by adding a small diameter constriction just above the bulk liquid level.

II.2. Temperature and density stratification in ^{4}He I samples

The heat and fluid transport properties of ^{4}He have important implications for the design of a liquid He sample holder. For EOS measurements on most cryogenic liquids, it is desirable to produce a quiescent liquid sample, one whose temperature is sufficiently below the boiling point so there are no low density vapor bubbles rising through the liquid. Thermal conductivity within the liquid sample will generally be sufficient to eliminate thermal gradients and produce a sample with an initial density which is uniform and accurately known as a function of temperature and external pressure.

The situation for liquid ^{4}He is somewhat more complex. Because of the large specific heat and low thermal conductivity of ^{4}He I in its normal fluid state above T_λ, temperature stratification can occur when He I samples are prevented from boiling, as would be the case in our LHe sample cavity. But large temperature gradients can exist even in a boiling sample of ^{4}He I [P1, p.24], where heat transfer takes place primarily by

convection. The He I density is a strong function of temperature [15, Table 3.51], leading to the uncertainty in the initial sample density.

By contrast, superfluid ^{4}He II at $T < T_\lambda$ has very high or infinite thermal conductivity, depending on the rate of heat flow into the system. A bath of ^{4}He II is able to transport heat with extreme efficiency to eliminate thermal gradients and maintain thermal equilibrium throughout its bulk. The liquid does not boil in response to pumping or boundary heating because the formation and transport of vapor bubbles requires a thermal gradient in the bulk liquid. Heat is removed from the sample by evaporation of atoms from the surface, and no bubbles are formed as long as the He liquid remains in a superfluid state. Below the λ-point, not only is the sample density uniform throughout, but that density is essentially constant as a function of temperature [11, p.93]. Small temperature differences between the pumped He II bath and a separate He II sample cavity, resulting from thermal gradients in the sample holder body produced by any external heat load and Kapitza (thermal boundary) resistance of the He bath walls, will make a negligible contribution to the uncertainty in the initial LHe sample density.

Based on these arguments, it appears that a sample of liquid ^{4}He II cooled to about 1.5-1.8 K would best meet the initial condition requirements for shock compression measurements.

III. Cooling Concept: Continuously Operating Evaporation Refrigerator

Several options for generating LHe samples on Z are presented in the Appendix. From that discussion, it is apparent that continuously operating refrigerator systems have a clear advantage over periodically-filled systems because they do not interrupt the Z shot sequence and make the most efficient use of LHe.

To achieve a quiescent (non-boiling) liquid helium sample of known initial density, it is desirable to cool the liquid sample below the ^{4}He λ-point temperature (T_λ = 2.176 K at saturated vapor pressure) to the 1.5-1.8 K temperature range. The most practical way to generate a quiescent ^{4}He II sample at 1.5 K is to make the LHe sample holder a continuously operating, self-regulating ^{4}He evaporation refrigerator [13, p.101; 14, p.75; 16-22]. Such an apparatus has the advantage that it can maintain constant temperatures below the λ-point of ^{4}He for extended periods in the presence of a variable heat load and without the requirement of external control of needle valves or other moving parts. The schematic of a continuously operating ^{4}He evaporation refrigerator is shown in Fig. A6. The sample holder is surrounded by 80 K and 5 K radiation shields to minimize the external heat load. The sample is cooled by contact with a small (few cc) LHe evaporation chamber. This LHe bath is suspended in vacuum by a thin-walled pumping tube which runs through the main LHe reservoir. Temperatures below the ^{4}He normal boiling point of 4.2 K are achieved in a liquid helium bath by pumping on the vapor

above the liquid. When the evaporator is pumped, LHe is drawn from the main LHe reservoir through the flow impedance at a constant molar flow rate.

The function of the flow impedance is to maintain the pressure difference between the main reservoir at 760 Torr and 4.2 K and the evaporator chamber pumped to 3.6 Torr for cooling to 1.5 K. Sufficient LHe is evaporated in the impedance and bath to cool the remaining liquid to 1.5 K. If the impedance is too large, there will be insufficient refrigeration to balance the heat load and no LHe will accumulate in the LHe evaporation chamber. If the impedance is too small, liquid will rise in the pumping tube to a level where the heat loss to the external bath results in high LHe consumption and an increase in the minimum temperature that the sample holder can reach for a given pumping speed. For an optimum range of flow impedance values, it is possible to maintain a constant temperature, independent of the heat load on the evaporator, for an external heat load from zero up to some critical power. For zero external heat load, the evaporator and tube will fill to a level where the heat load through the tube from the main bath exactly balances the additional refrigeration provided by the latent heat of the LHe entering the evaporator at a fixed flow rate. As the external heat load on the evaporator is increased, the liquid level will drop until a new equilibrium is reached between the total heat load and the cooling power of the LHe flow. Eventually a critical power will be reached where the liquid level descends into the evaporator and heat loss to the external bath is negligible. With a further increase in external heat load above the critical power, it will no longer be possible to provide sufficient refrigeration to maintain a constant temperature with the given constant flow rate of LHe.

Using a continuous flow evaporation refrigerator, it should be no more difficult to generate a superfluid liquid ^{4}He II sample in the 1.5-1.8 K temperature range than a normal fluid liquid ^{4}He I sample in the 2.5 K < T < 3.5 K range. Both require the same radiation shielding for stable temperature operation. Rapid boiling could be suppressed in a normal liquid He I sample by over pressuring the gas supply, but this largely eliminates convection as a means to smooth temperature gradients that develop. This problem is avoided with a liquid He II sample where thermal equilibrium can be rapidly established and maintained, provided the heat load is not so large that it destroys the superfluid state.

The unique and challenging goal for this project is to use an extreme low temperature cryocell to condense pure LHe samples for the first time in the Z environment. The proposed method for cooling these LHe samples is to configure the LHe sample holder as a continuously operating, self-regulating evaporation refrigerator. Fig. 1 shows the detailed conceptual design for the full LHe cryogenic sample system with a continuously operating ^{4}He evaporation refrigerator built into the sample cryocell. This design has the basic components of the system in Fig. A6, but is configured to meet the unique requirements for LHe shock compression experiments on Z. The features of this detailed conceptual design will be discussed in the next section.

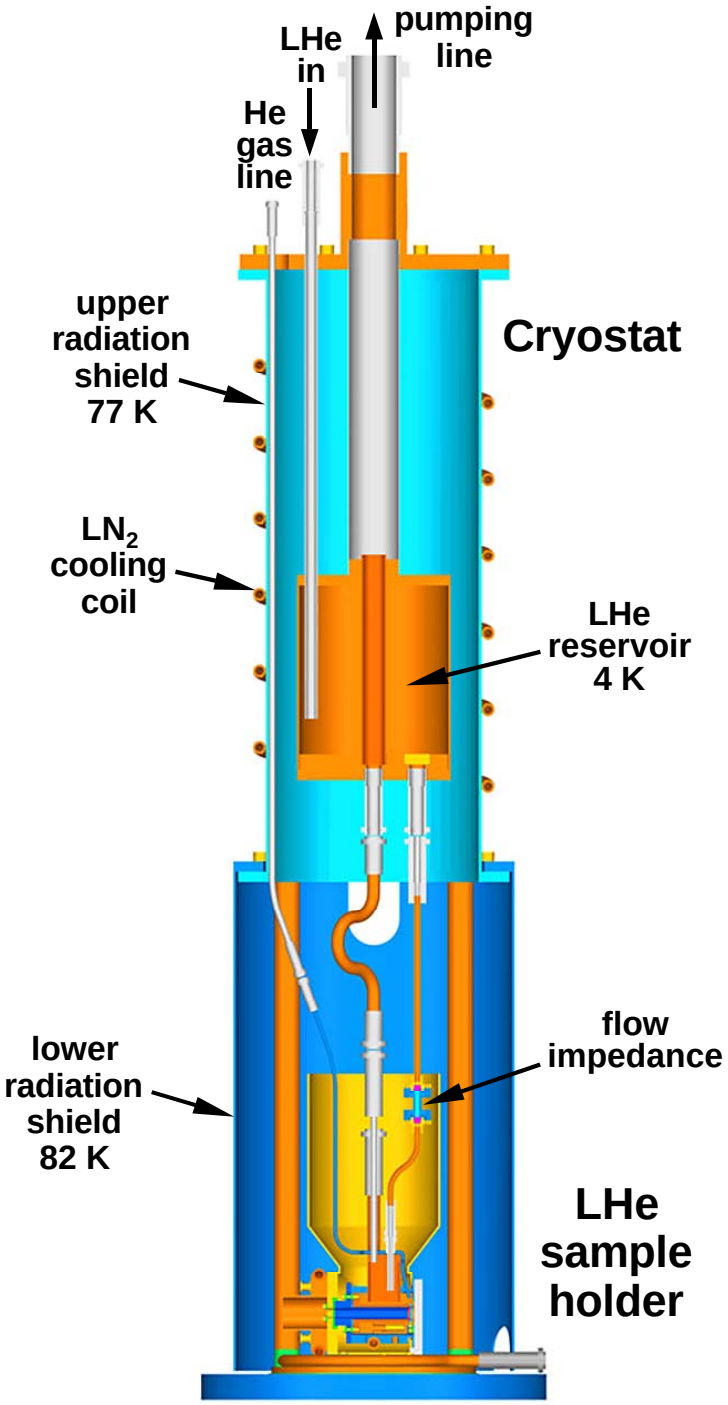

Figure 1. Conceptual design for the LHe cryogenic sample system with a continuously operating, self-regulating LHe evaporation refrigerator.

IV. Design Details: Cryostat

The design for the LHe cryogenic sample system cryostat is shown in Fig. 2. The cryostat assembly consists of a LHe reservoir suspended from a top flange by thin-wall stainless steel tubing for the LHe input, output, and vacuum pumping lines. The LHe reservoir in the cryostat is continuously fed from an external LHe Dewar and provides a large, stable LHe bath at approximately atmospheric pressure and 4.2 K from which LHe can be drawn through a flow impedance to the pumped LHe bath in the sample holder. The on-axis pumping line also exits through and is cooled by the LHe reservoir. The entire experiment is surrounded by LN_2-cooled radiation shields to reduce the heat load on interior LHe-cooled components. The upper radiation shield is supported by four copper posts insulated at their base, and is cooled by LN_2 flowing through a spiral copper cooling coil on the outside of the shield. The He gas line feeding the LHe sample holder is brazed to the inside of the upper radiation shield for cooling and support. The lower radiation shield is split into two halves that are suspended from and cooled by conduction to the upper radiation shield. Various cutouts are included in the lower radiation shield as exit points for LN_2 and LHe cooling lines emerging from inside the radiation shields. The current panel supporting the LHe sample holder is mounted on a base plate radiation shield that is cooled by LN_2 flow through a copper cooling coil brazed to the plate. This base shield completes the 80 K radiation shield enclosure of the LHe-cooled components. This entire cryogenic assembly will be immersed in the Z center section vacuum that is typically around $2x10^{-5}$ Torr. The additional cryopumping provided by the large-area LN_2-cooled radiation shields surrounding the sample holder assembly will provide much needed local improvement for the vacuum in the load area.

This cryostat assembly must be mounted close to the current source on the Z axis and cannot be adequately shielded from the destructive blast and on-axis jetting that results when the electrode panels forming the short circuit load, the sample holder body, and associated radiation shields are vaporized or reduced to molten metal and small shrapnel by the > 1.5 MJ of energy dissipated in the load region during a Z shot. As a result, the entire cryostat and sample holder assembly is likely to be destroyed on each shot, and the cost and complexity of the entire design must be aimed at a level appropriate for an expendable device. Wherever possible, cooling is performed by flowing liquid cryogens through a cooling coil rather than by filling a reservoir. Cooling coils are easier and less expensive to fabricate and assemble, occupy less space than enclosed containers, and can provide efficient localized cooling of modular components. But an adequate cryogen flow rate must be maintained through multiple cooling coils connected in series to minimize turbulent two-phase flow and avoid temperature fluctuations and vibrations in connecting flex lines.

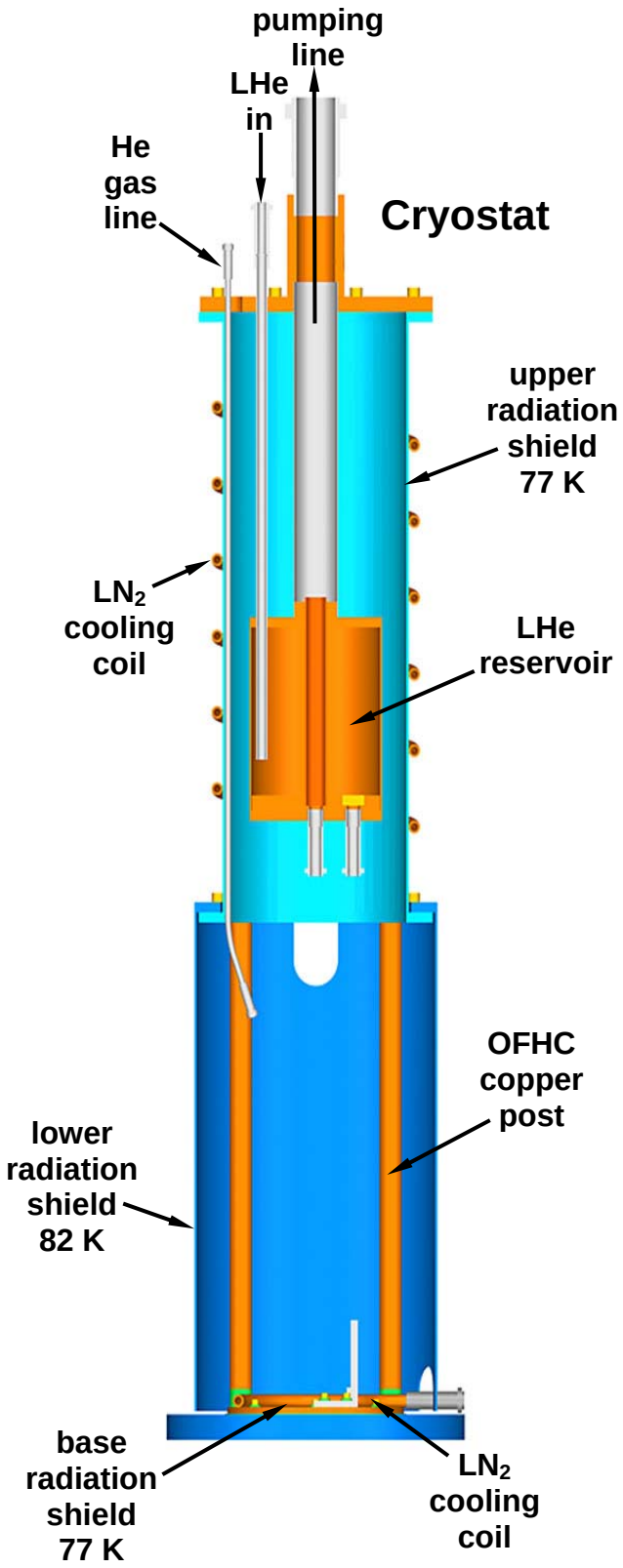

Figure 2. Cryostat assembly with radiation shields for LHe cryogenic sample holder.

V. Design Details: LHe Sample Holder Assembly

The design of the LHe sample holder and associated radiation shielding is shown in Figs. 3-7. The LHe sample holder assembly consists of four main components: (1) an outer LHe–cooled 5 K radiation shield (Fig. 7); (2) an inner LHe-cooled 5 K radiation shield (Fig. 6); (3) the LHe sample holder body (cryocell) (Fig. 5); and (4) a diagnostic fiber optic probe array that is mounted in a cylindrical channel on the back of the inner radiation shield.

The LHe–cooled inner 5 K radiation shield (Fig. 6) is the backbone of the sample holder assembly, supporting the outer 5 K radiation shield, the LHe sample holder, and the fiber optic array (Figs. 4 and 5). The sample holder assembly is attached to the current-carrying load panel through the inner 5 K radiation shield which is thermally isolated by a nylon thermal break. The outer 5 K radiation shield (Fig. 7) surrounds the inner 5 K shield, extends upward in a funnel surrounding the lines going to the cryostat, and is split into two parts along the axis of the sample holder for mounting. Each of the 5 K radiation shields is directly cooled by LHe flowing in a single-turn cooling coil. The nested pair of 5 K shields together with the base of the LHe reservoir in the cryostat completely surround the LHe sample holder. The 5 K shields are essential for minimizing the heat load and providing a stable thermal environment in which the LHe sample holder can maintain a 1.5-1.8 K equilibrium temperature.

The LHe sample holder (Figs. 4 and 5) consists of a small, top-mounted LHe bath (evaporator) that controls the cooling of the sample holder body, and a small cavity at the front in which high purity He gas is condensed into a quiescent superfluid LHe sample for the experiment. The sample holder is operated as a continuously fed evaporation refrigerator. The sample holder LHe bath is pumped to about 3.6 Torr through a large diameter pumping line which runs through the main LHe reservoir in the cryostat and is extended to the LHe sample holder with a thin-wall stainless steel tube. LHe from the main cryostat LHe reservoir at 4.2 K and 760 Torr is drawn through a flow impedance (Figs. 3 and 8) where it is collected in the sample holder LHe bath at 1.5 K and 3.6 Torr. Sufficient LHe is evaporated during passage through the flow impedance to cool the remaining liquid to 1.5 K. High purity He gas at a fixed over pressure is delivered to the sample cavity through a capillary tube connected to an external gas line which is thermally tied to the upper radiation shield of the cryostat and to the sample holder evaporator. As long as the cryostat reservoir is at least partially filled with LHe at 4.2 K, a continuous flow of LHe at 1.5 K into the sample holder bath will be maintained. The temperature of the sample holder is controlled primarily by the pressure over the LHe in the sample holder bath, provided the flow impedance has the correct value. The minimum temperature that can be achieved will be determined by the pumping speed of the roughing pump and the external heat load on the sample holder. The net radiant heat flux to those parts of the sample holder which view only the 5 K shields is on the order of 10^{-8}–10^{-9} W/cm^2, depending on the emissivity of the surfaces. For any small areas which view the surrounding 80 K shields or the current panel (which will also be cooled to 80

K), the net heat flux is about 10^{-5} W/cm^2. So the parts of the sample holder enclosed by radiation shields are very effectively insulated. The main heat load involves the back of the LHe sample cavity, which directly views the fiber optic probe array at room temperature (300 K). The nylon holder for the fiber optic probes is epoxied into the inner 5 K shield and may also represent a significant conduction heat load for that shield. However, the fiber probe mount assembly is small in area and contributes less than 1 mW to the radiant heat load of the sample holder. The conduction heat load from the three cooled capillaries (gas, flow impedance, and pumping lines) entering the top of the sample holder body is more difficult to estimate but should be manageable. For the purpose of designing a flow impedance, it is reasonable to estimate that the required cooling power for the sample holder falls in the broad range of 10–100 mW.

A small constriction (0.5–1.0 mm) may be needed in the gas fill and pumping tubes to limit superfluid He flow. This should work well for the gas line feeding the ^{4}He II sample cavity. For the sample holder LHe bath, the fluctuation of the liquid level in the pumping line in response to changing heat loads is a integral part of the temperature stabilization process. A constriction located at the wrong position could interfere with temperature stability. A small constriction will also limit the pumping speed and the ultimate temperature of the sample holder. For these reasons, it is probably not advisable to include a small limiting aperture in the pumping line as a barrier to superfluid He II flow.

The critical element of this system is the flow impedance (Fig. 8). This must be optimized to provide an adequate flow rate to maintain the desired equilibrium temperature for a given range of external heat load. The required flow rate can be determined from the critical power per unit flow rate which is approximately 4.5 mW/10^{-4} mole/sec [13, 14, 16, 18, 19]. Several methods exist for constructing flow impedances. Packed-powder flow impedances can be constructed from off-the shelf grinding powder [23]. These are compact, rugged, and not easily plugged with dirt. Because the packed-powder flow impedance depends on the square of the grain size (rather than the fourth power of some spatially varying constriction that cannot be measured), impedances can be designed with fairly predictable properties. Impedances can also be fabricated from meter lengths of fine capillary tubing or from shorter lengths of capillary with a wire constriction inserted [17-22]. These devices require protective filters to avoid being plugged with dirt, and are less predictable in their impedance values.

The high purity He gas supplied to the sample cavity will be pre-cooled by having the gas line thermally anchored to the inner wall of the upper radiation shield for the cryostat (77 K) and to the top of the sample holder evaporator (1.5-1.8 K) before connecting to the He cavity in the sample holder cryocell. This places constraints on the initial He gas pressure in a closed system. Because we want to be able to detect the gas-to-liquid phase transition in the sample cavity by observing a pressure change in the gas line, it is desirable to operate at the highest allowed pressure. Operation at 10 psi He gas pressure will allow LHe condensation in the sample holder cavity and gas line at any temperature less than 3.8 K and result in a measurable pressure change at the He gas-to-liquid phase transition. The He II in the line will rise to a level where the evaporation refrigerator can no longer provide adequate cooling to maintain the sample temperature below the λ-

transition point. The lower section of copper capillary cooled by the sample holder to 1.5-1.8 K will contain superfluid He II while the upper section where T > 3.8 K will contain He gas at about 10 psi.

The effect of the fiber optic probe array behind the window in the sample holder must also be determined. The 300 K probe array in its nylon mount represents the most significant radiation heat load on the LHe sample holder (as well as a conduction and radiation heat load on the 5 K shield in which it is mounted). Further, when the lasers for the VISAR and active shock breakout diagnostics are turned on, energy is deposited directly in the LHe sample cell. The effects of these heat loads on the equilibrium cell temperature need to be evaluated.

Temperature measurements in the LHe sample holder cryocell, 5 K shields, and LN_2-cooled radiation shields at 80 K are performed with silicon diode temperature sensors with standard calibration curves extending down to 1.5 K. For more accurate temperature measurements in the range of 1-5 K, Ruthium Oxide (ROX) sensors are built into the LHe sample holder cryocell. There is limited space for mounting temperature sensors, particularly in the sample holder. The leads from each temperature sensor in the sample holder cryocell exit through small holes in the 5 K radiation shields and are thermally anchored to bobbins on the outer 5 K radiation shield back to minimize external heat flow to the sensor. Thermal conduction across interface boundaries can be a particular problem at T < 5 K. This could affect the temperature distribution in the cryocell 5 K shield and the LHe sample holder. Each is made up of two or more parts held together with small screws and clamps. Thermal resistance between two pieces in contact can be reduced by cleaning and polishing the surfaces, by increasing the contact pressure between surfaces, and by increasing the area of surface contact through the use of conductive epoxy or grease or a thin layer of indium. The boundary resistance between copper surfaces can be reduced by a factor 20 by gold plating the surfaces [24, 25].

In addition to a design for the full LHe cryogenic sample holder assembly to demonstrate condensation and containment of superfluid LHe samples (Figs. 3-5), we have also designed a simplified test cell for basic cooling demonstration and flow impedance optimization (Fig. 9). This multiple prototype approach is intended to simplify lab testing requirements and improve the focus of cryogenic testing. The basic test cell cryogenic system was the first prototype configuration to be fabricated, assembled, and instrumented for initial lab testing.

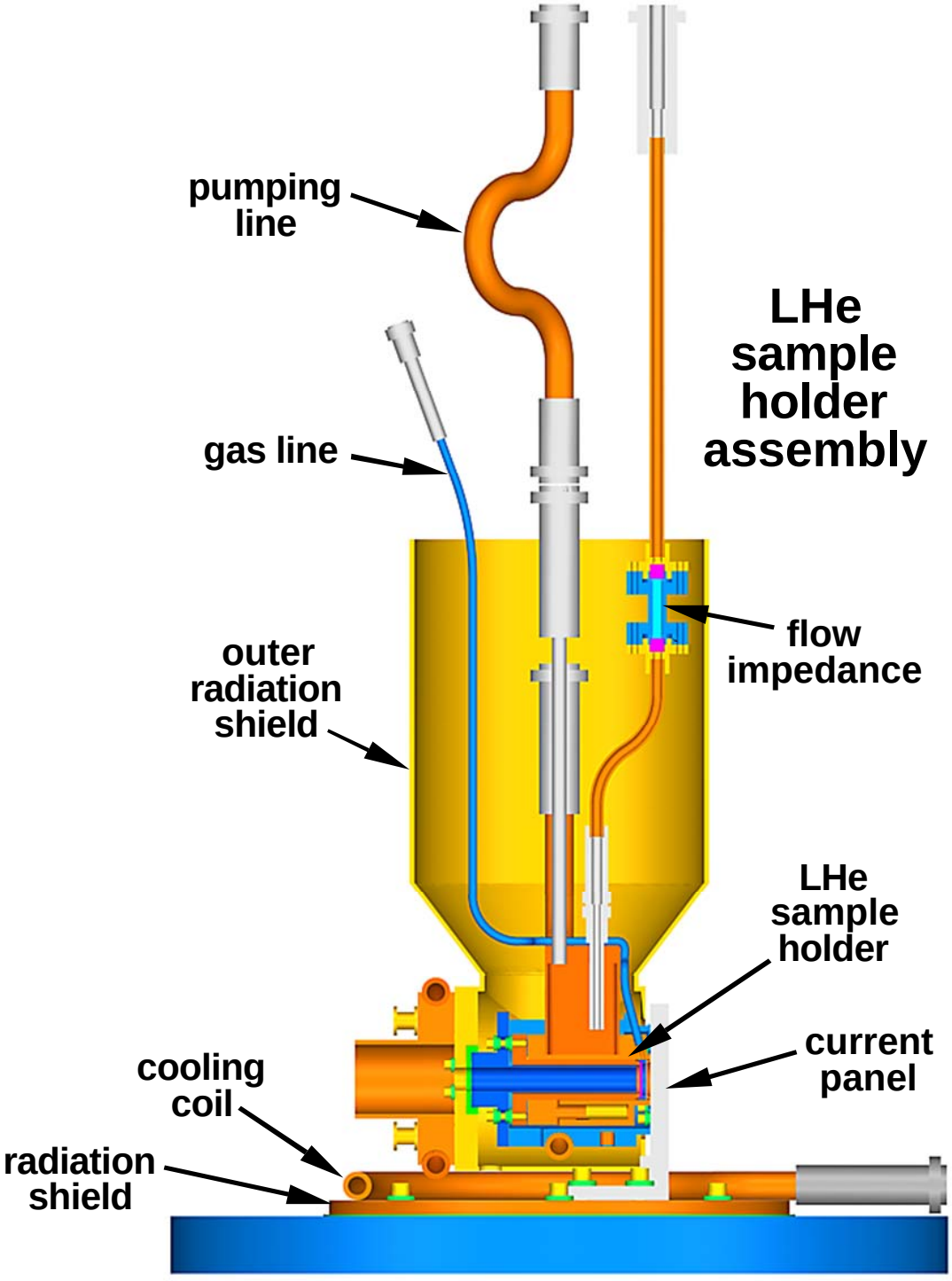

Figure 3. LHe sample holder cryocell assembly below cryostat.

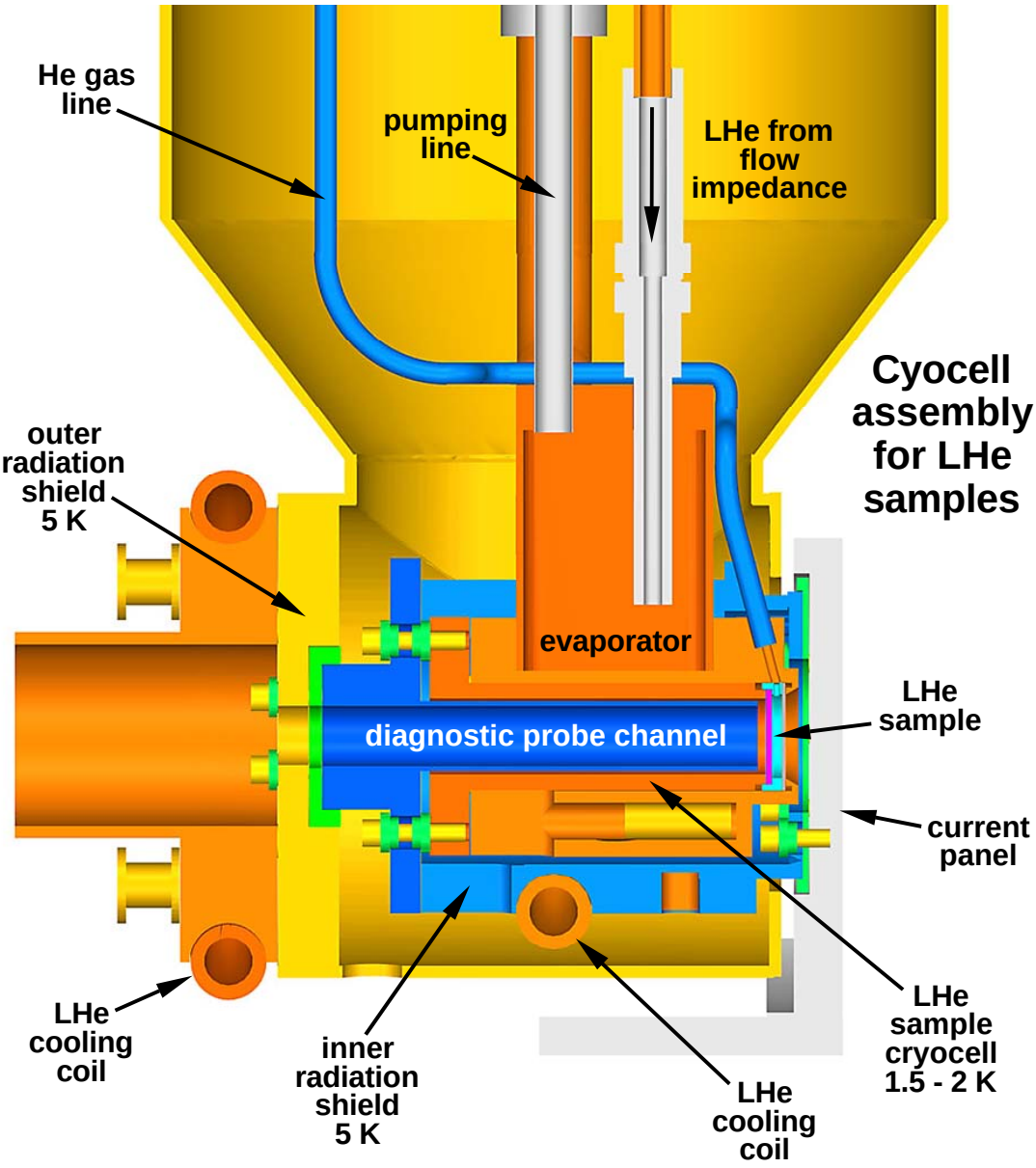

Figure 4. LHe sample holder cryocell with radiation shields.

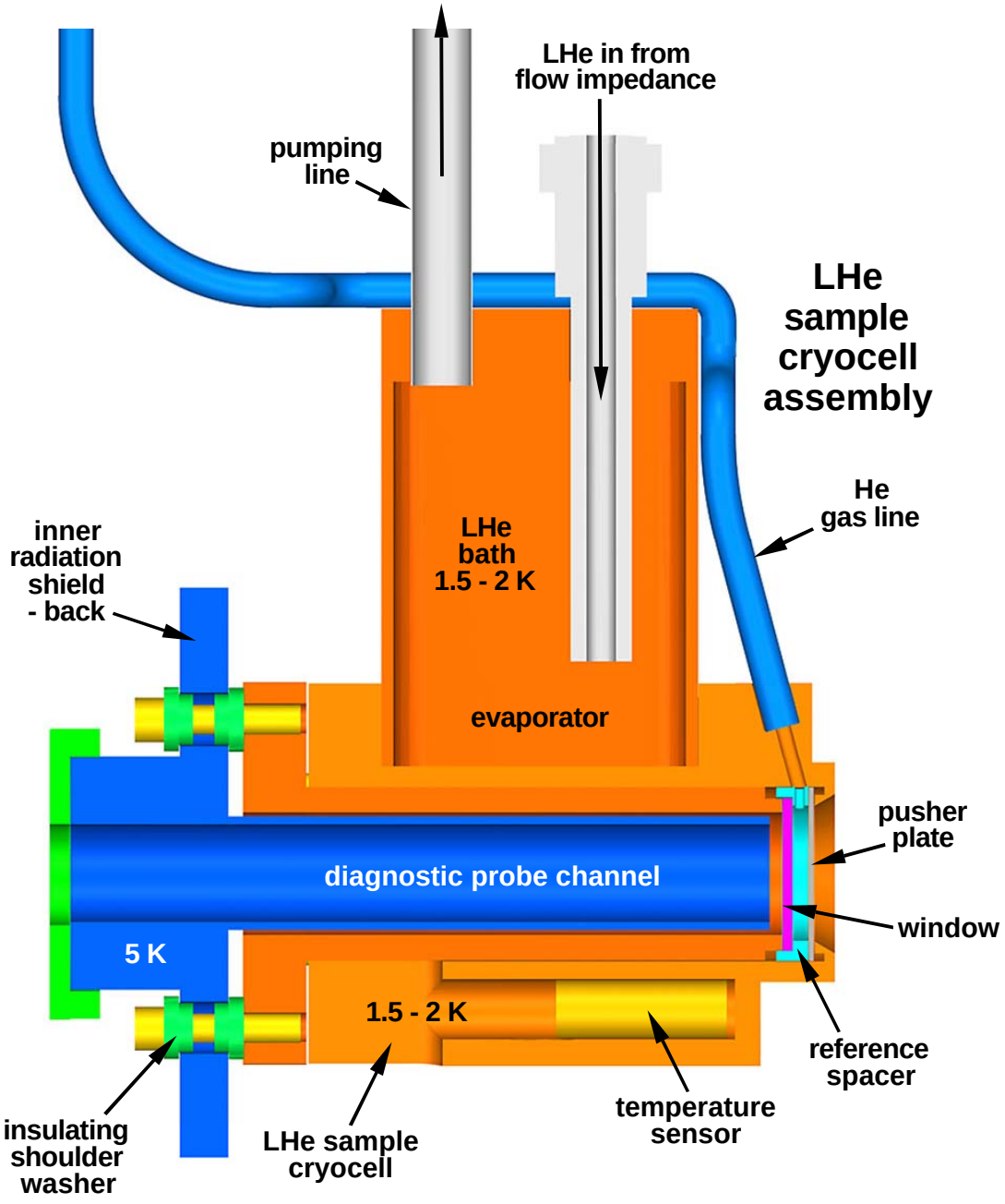

Figure 5. Detail of LHe sample holder cryocell mounted on inner radiation shield back.

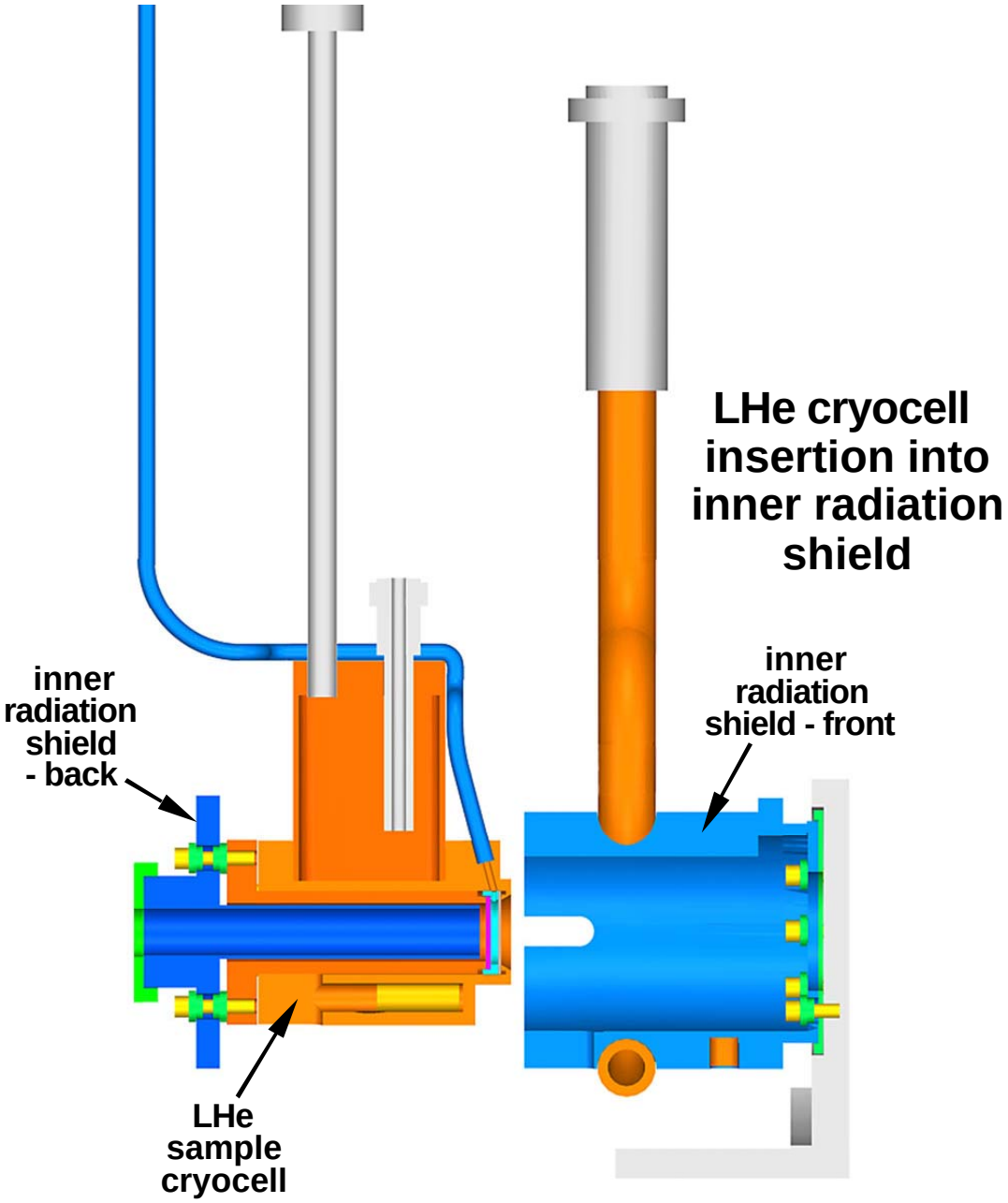

Figure 6. Cryocell assembly ready for insertion into front section of inner radiation shield.

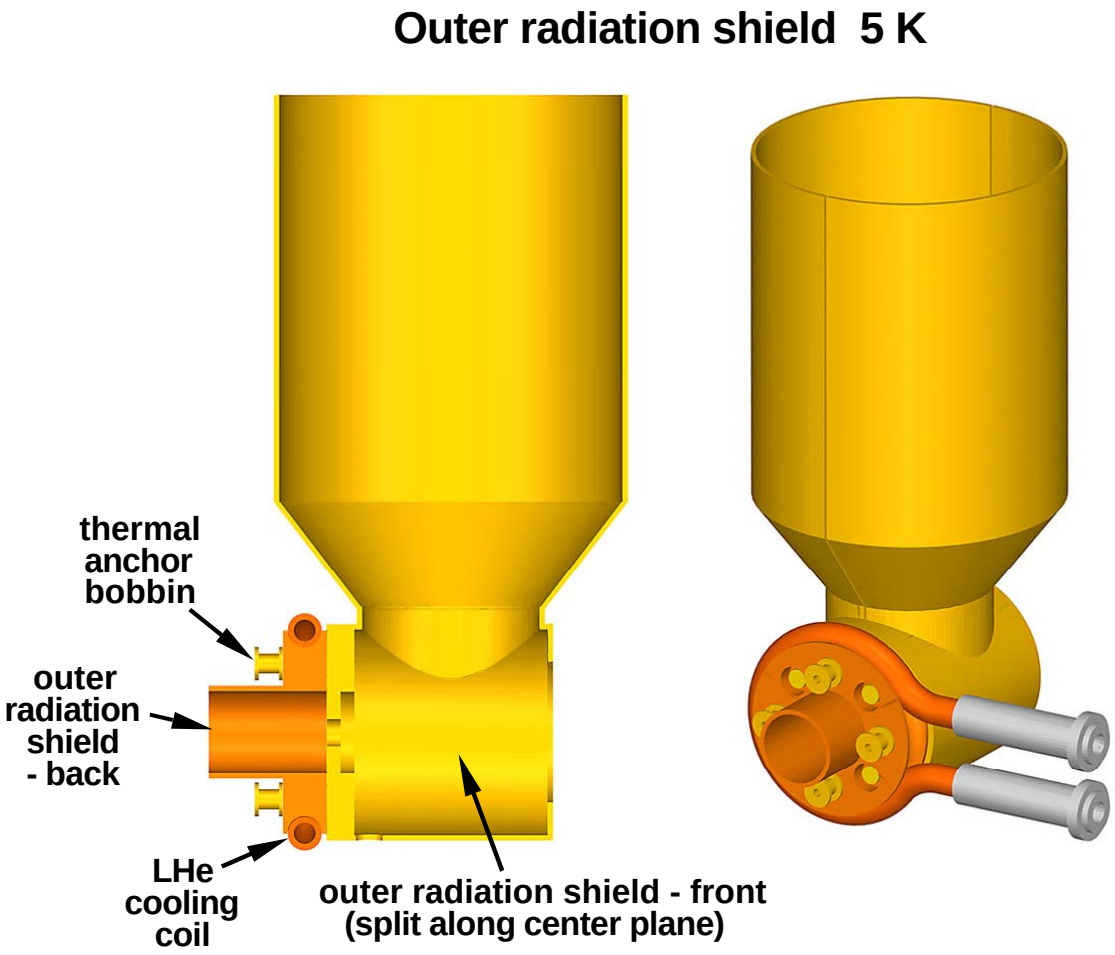

Figure 7. Outer radiation shield – Left: cross section; Right: full assembly.

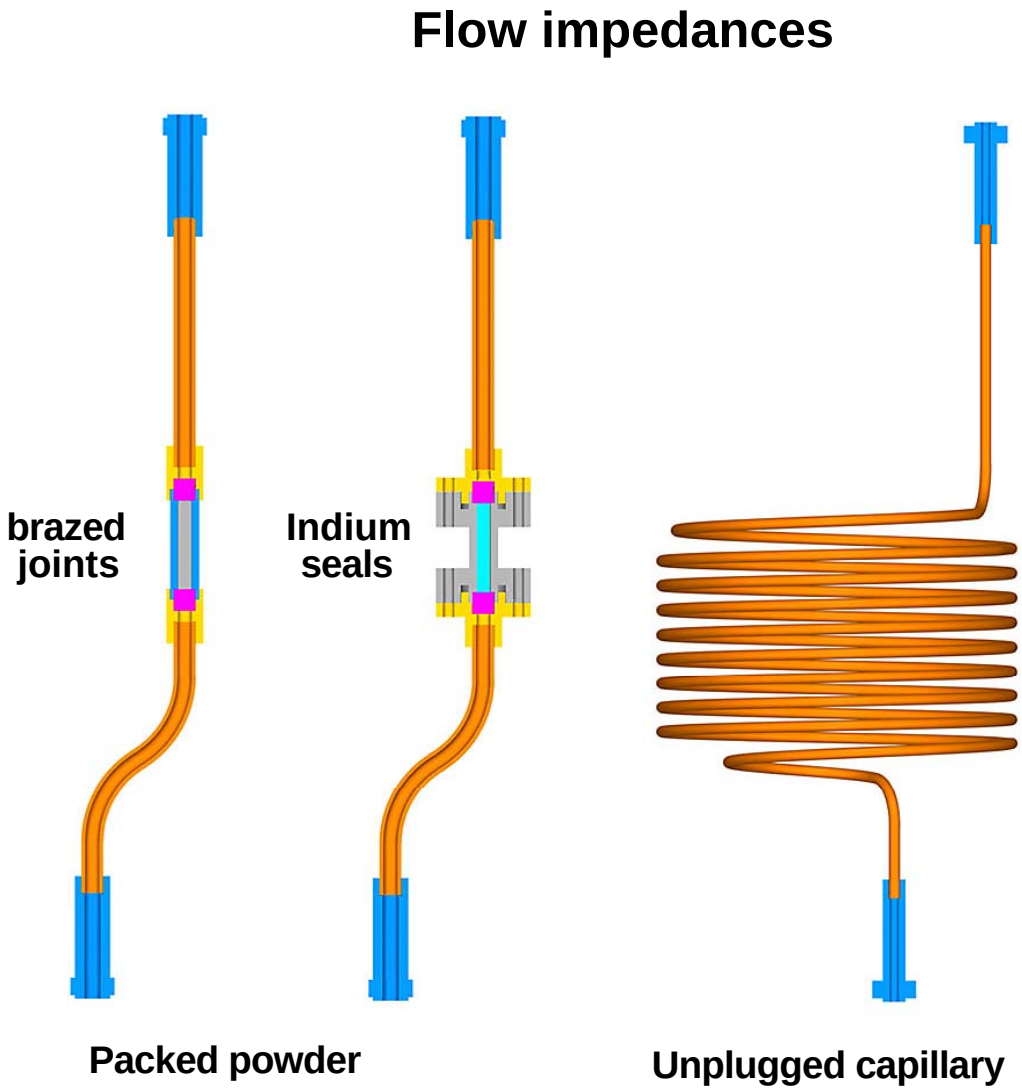

Figure 8. Flow impedance designs.

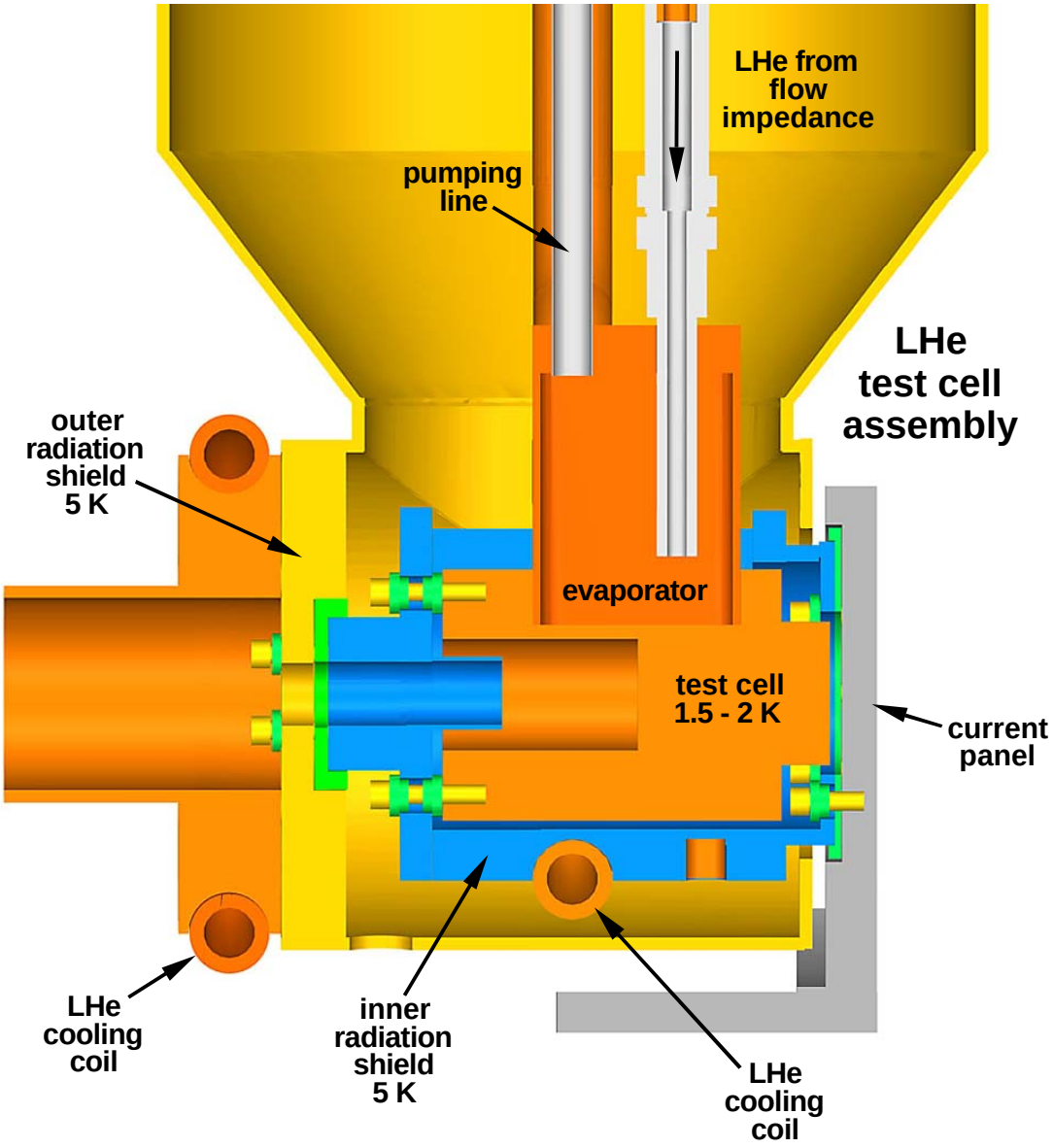

Figure 9. Detail of basic test cell assembly.

VI. Test Results

A basic cryogenic test cell system prototype (Fig. 9) has been fabricated, assembled, and instrumented for initial lab testing (Figs. 12 and 13). The test cell configuration is a simplified version of the LHe sample holder where the focus is on cooling a block of copper for flow impedance development and for demonstration and optimization of cooling with a pumped LHe evaporation refrigerator. At the time that this is being written, only preliminary tests have been performed to characterize the behavior of the radiation shields in the LHe cryogenic system.

Fig. 10 shows the cooling history for each of the radiation shields vs elapsed time after the cryogen flow was started. All radiation shields cooled rapidly to their expected minimum temperature values with no leaks. The bottom of the lower radiation shield, cooled by conduction from the upper radiation shield, reached 81 K in 40 minutes after the LN_2 flow was started. The test cell mounting panel on the cryostat base, cooled directly by a LN_2 cooling loop, reached 78 K in less than 20 minutes. It is expected that the upper radiation shield (and cryostat top flange) was cooled on a similar trajectory to the cryostat base by its cooling coil in series with the cryostat base loop.

The nested outer and inner 5 K shields on the test cell assembly, each cooled directly by LHe flow through a single-turn copper cooling loop, reached 6 K in 30 minutes with temperature fluctuations of several hundred mK. After 100 minutes, temperatures reached the LHe boiling point range of 4.3-4.5 K, with temperature fluctuations of 10 mK (Fig. 11), as the system approached thermal equilibrium and the LHe flow rate through the LHe reservoir and two shield cooling loops in series was reduced and stabilized.

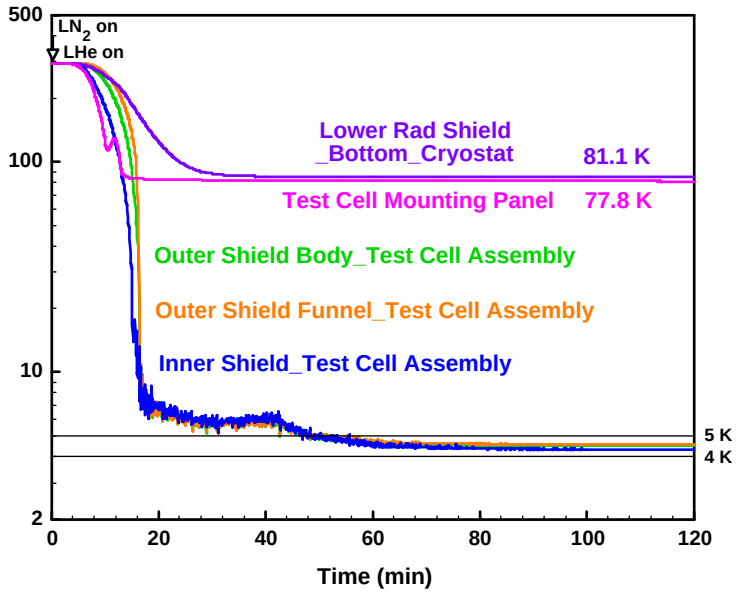

Figure 10. Cooling history for test cell cryogenic system radiation shields.

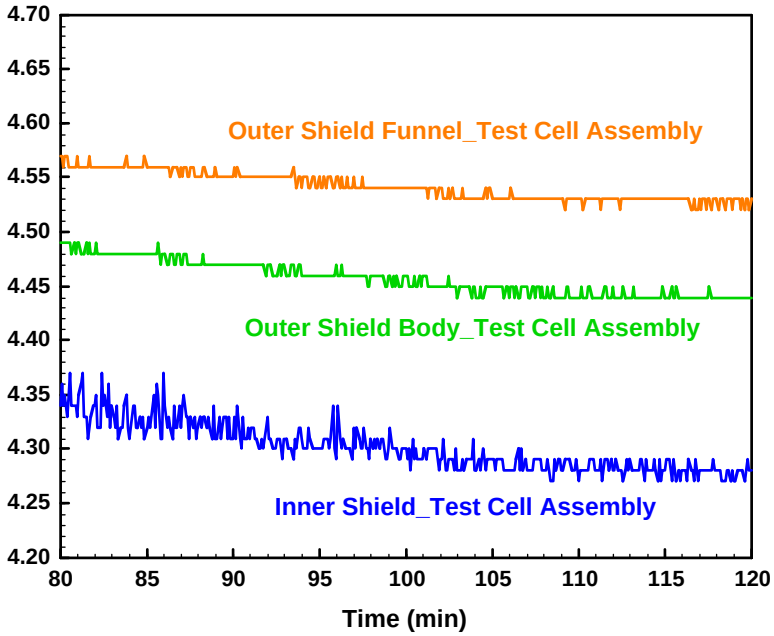

Figure 11. Temperature fluctuations in test cell assembly radiation shields after final approach to thermal equilibrium.

VII. Accomplishments and Future Work

The key R&D accomplishments of this LDRD include:

(1) development of a conceptual design for an expendable cryogenic system of reasonable cost and complexity to condense large area superfluid LHe samples at 1.5-1.8 K for dynamic high pressure He experiments on Z;

(2) development of a detailed final design and drawing package for a *Basic Test Cell* cryogenic system prototype configured for flow impedance development and cooling optimization;

(3) development of a detailed final design and drawing package for a *Liquid Sample Holder* cryogenic system prototype configured for demonstration and optimization of LHe sample condensation and containment;

(4) development and fabrication of flow impedance components having both packed powder and coiled capillary structures and designed to operate over a wide range of flow impedance values and cooling power;

(5) fabrication, assembly, and instrumentation of a prototype system for the *Basic Test Cell* configuration; fabrication of the components for *Liquid Sample Holder* prototype assemblies; lab testing for demonstration and optimization of Basic Test Cell cooling is

currently in progress.

Photos of Test Cell configuration prototype system assemblies are shown in Figs. 12 and 13.

Future work, after the formal conclusion of this one-year LDRD project, required to arrive at the point where LHe shock compression measurements can be performed on Z includes the following activities:

(1) Test LHe sample holder system prototype designs in the Cryogenics Development Lab, and modify to optimize cooling and containment of LHe. This lab testing should continue through Q1 and Q2 of FY11 as part of the normal cryogenics lab testing program.

(2) Adapt the extreme low temperature cryocell prototype design to the specific configurations required for LHe EOS measurements on the Z current drive, in collaboration with the Dynamic Material Properties group (FY11 Q3-Q4).

(3) Add cryopumping capacity in the Z load region, through LN_2-cooled panels on the experiment blast shield, to provide improved vacuum conditions and reduced heat loading for reliable operation of the extreme low temperature cryocell on Z. This feature would also improve our ability to perform cryogenic experiments on other materials of interest at higher temperatures.

(4) Perform dry run cooling tests on Z to demonstrate proper cryogenic system assembly and operation in the Z environment before committing to shots on the Z schedule.

Figure 12. Left: Basic test cell system with radiation shields in place and removed; Right: Cryostat removed from upper radiation shield.

Figure 13. Views of basic test cell assembly on mounting plate.

VIII. Impact and Significance

The result of a successful conclusion to this LDRD will be a new cryogenic capability for condensing LHe samples, an operating cryogenic system with an extreme low temperature cyocell that, with some additional effort, can be adapted directly to LHe dynamic measurements on the Z current drive. The successful development of this cryogenic capability for Z will produce a legacy of S&T (Science and Technology) advances in the form of fundamental dynamic studies of He properties at high pressure. Implementation of this capability on Z will impact DOE strategic needs in nuclear weapons stewardship by enabling very accurate EOS measurements of material relevant to the Nuclear Weapons program and will enhance the capacity of the Z facility for world class basic research of extreme interest to both theoretical physicists and astrophysicists. NW and STE SMUs will benefit in an expected time frame of 2 - 5 years.

The dynamic compression capability developed on Z over the last decade has become a mature platform, enabling material dynamic experiments in the Mbar pressure regime with unprecedented accuracy. The development of a cryogenic capability for the measurement of LHe properties at high pressure on Z has been a goal of the Center 01600 Dynamic Material Properties group (01646) for several years. Use of this new cryogenic capability for the study of He properties at high pressure will provide additional widespread visibility for the Z dynamic materials capability in areas of both programmatic and basic science interest. Features of the improved cryogenic technology in this system targeted on He will also enable or enhance the ability to perform fundamental science studies on other materials of interest.

IX. Summary

This final report on SNL LDRD Project 141536 summarizes progress made toward the development of a cryogenic capability to generate LHe samples for high accuracy equation-of-state (EOS) measurements on the SNL/NM Z machine current drive. We have developed a conceptual design for a cryogenic LHe sample system, with an extreme low temperature cryocell for condensing superfluid He II samples at 1.5-1.8 K. This LHe sample holder is effectively packaged in a very compact, modular geometry meeting the requirements for shock physics measurements on Z. This cryogenic system design adapts the basic elements of a continuously operating, self-regulating ^{4}He evaporation refrigerator capable of operating at 1.5 K to the constraints of Z shock compression experiments.

This should be considered a prototype design where details of certain modular components, such as the flow impedances, may change as problems become apparent during lab testing. Separate prototype configurations were developed to address different problems: (1) a simplified test cell for basic cooling demonstration and flow impedance optimization; and (2) a full LHe cryogenic sample holder assembly to demonstrate condensation and containment of superfluid LHe samples. This approach appears to simplify lab testing requirements and improve the focus of cryogenic testing. Modular

component designs were implemented to reduce complexity, simplifying assembly and allowing troubleshooting and optimization of individual components during prototype testing. Design features were also introduced allowing simplified fabrication procedures to reduce costs to an acceptable level for these expendable assemblies. Prototypes have been fabricated, assembled, and instrumented for initial testing, and some preliminary testing has been performed.

In addition to providing a unique capability for performing EOS measurements on LHe at high pressure, this project addresses a larger issue of developing cryogenic expertise on Z for future dynamic material properties and ICF target experiments. Development of a LHe cryogenic capability represents a parallel opportunity to advance cryogenic radiation shielding and vacuum enhancement solutions on Z which are directly applicable to cryogenic studies on other materials of interest.

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Appendix: Options for generating liquid He samples for Z experiments

There are several options for generating quiescent LHe samples for shock physics measurements on Z. For each option discussed below, a schematic diagram is presented showing the basic elements in the cryogenic system. An actual cryogenic design for Z would be more complex. The main point here is that options involving continuous filling of the sample holder bath have a major advantage over the periodic-fill options because their operation is relatively simple and will not interrupt the Z shot sequence.

Option 1: Continuously-filled single-stage LHe evaporation refrigerator operated at 4.2 K

The simplest option (Fig. A1) is to directly fill the sample holder LHe bath by connecting it to the cryostat LHe reservoir through a large area transfer line. The cryostat LHe reservoir is continuously filled from an external LHe Dewar. In this arrangement, the sample holder LHe bath and the cryostat LHe reservoir are integrated to form a single continuously-filled evaporation refrigerator operating at 4.2 K. This simplifies the required transfer lines and radiation shielding compared to other schemes. Boiling of the LHe sample in a separate cavity can be suppressed by setting the sample gas pressure above the saturated He vapor pressure at 4.2 K. A sample with low thermal conductivity and high specific heat will be generated that may be stratified in temperature and density. There will be no heat transfer from convection due to boiling and the temperature distribution in the sample will reflect any temperature gradient around the cavity. LHe density is a strong function of temperature at 4.2 K. Also, the LHe density at 4.2 K is about 0.126 g/cm^3 compared to 0.145 g/cm^3 at 1.5 K, so the LHe sample will support a higher pressure for a given drive at 1.5 K. If the initial LHe density distribution is defined accurately enough to make this arrangement a viable option, then this configuration represents an attractive choice because of its relative simplicity of construction and operation compared to the other methods that will be described.

Option 2: Periodically-filled single-stage LHe evaporation refrigerator operated at 2.5-3.5 K

Higher initial He sample density could be achieved by cooling the sample holder to a temperature lower than 4.2 K. Using a similar configuration to Option 1 (Fig. A2), the cryostat reservoir and sample holder bath are periodically filled with LHe at atmospheric pressure and then sealed off and pumped to a lower pressure to cool the entire LHe system to 2.5-3.5 K. To get to 3.0 K, about 25% of the LHe must be boiled off to cool the remaining liquid, which will then boil off at a rate determined by the external heat load. This approach would require careful monitoring of the LHe level, periodic refilling of the cryostat LHe reservoir during the Z shot sequence, and is practical only for generating He I samples. To cool the sample holder to 1.5 K in this arrangement would

require boiling off more than 55% of the LHe in the large cryostat reservoir, in addition to the normal evaporation loss from the external heat load, each time the cryostat reservoir was filled. This procedure would require frequent refilling.

Option 3: Two-stage LHe evaporation refrigerator at 1.5 K with periodic filling of the sample holder bath using He gas

In this configuration (Fig. A3), the continuously-filled cryostat LHe reservoir cools a pumping line which connects to the sample holder LHe bath (evaporation refrigerator). The pumping line tube is periodically filled with He exchange gas from an external supply at slightly above atmospheric pressure. LHe condenses at 4.2 K to fill the sample holder bath and the pumping line tube well up into the cryostat. Then the tube is sealed and pumped to 3.5 Torr, evaporating about 55% of the liquid He, and cooling the sample holder to 1.5 K. The condensed LHe in the sample holder bath and sample cavity undergo a transition to superfluid He II. While this method can be used to cool a small sample to 1.5 K, the LHe level in the pumping line must be monitored and the sample holder bath may require frequent refilling during the Z shot sequence.

Option 4: Two-stage LHe evaporation refrigerator at 1.5 K with periodic filling of the sample holder bath through a needle valve

This method is similar to Option 3 except the evaporation refrigerator is periodically filled from the cryostat LHe reservoir through a remotely-operated mechanical needle valve (Fig. A4). In addition to the problems of LHe level monitoring and periodic refilling of the system, a remote control system would be required to operate the needle valve in the Z center section.

Option 5: Two-stage LHe evaporation refrigerator at 1.5 K with continuous filling of the sample holder bath through a needle valve

In this method, the pumped He sample holder bath at 1.5 K is continuously refilled from a bath at atmospheric pressure and 4.2 K through a carefully adjusted needle valve (Fig. A5). The effectiveness of such a system depends critically on the quality of the needle valve and on the degree of its thermal isolation from the main bath at 4.2 K. Again, remote control operation of the needle valve guided by LHe level monitoring of the sample holder bath is required to make this system work on Z.

Option 6: Two-stage LHe evaporation refrigerator at 1.5 K with continuous filling of the sample holder bath through a passive flow impedance

In this arrangement, LHe from the cryostat reservoir at about 4.2 K and 1 atmosphere is drawn continuously through a passive flow impedance into the sample holder liquid He II bath at 1.5 K and 3.6 Torr (Fig. A6). The sample holder temperature is controlled by the

pressure over the bath and limited by pumping speed. The main problem is fabricating a compact flow impedance unit with the correct impedance value to provide adequate refrigeration and self-stabilizing temperature regulation for the existing heat load. If the flow impedance is too large, the sample holder He bath will run dry, and if the flow impedance is too small, the desired operating temperature of 1.5 K will not be reached. This appears to be the best option for cooling superfluid He II samples on Z because it is continuously operating and self-stabilizing. To maintain a given constant temperature with the evaporation refrigerator, it is only necessary that the cryostat LHe reservoir be at least partially full and that the required constant reduced pressure be maintained over the sample holder LHe bath.

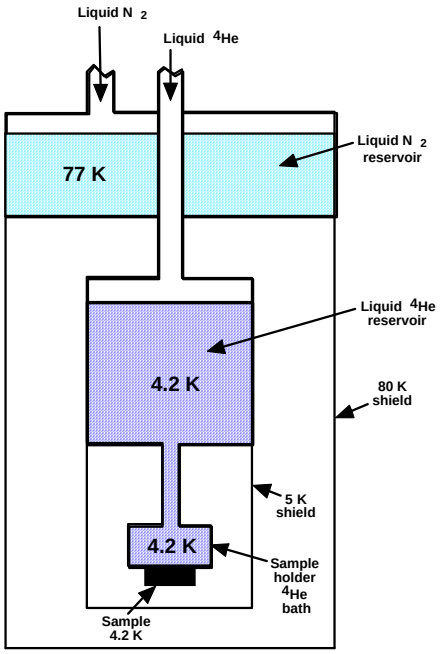

Fig. A1. Schematic of continuously-filled single stage liquid He evaporation refrigerator at 4.2 K

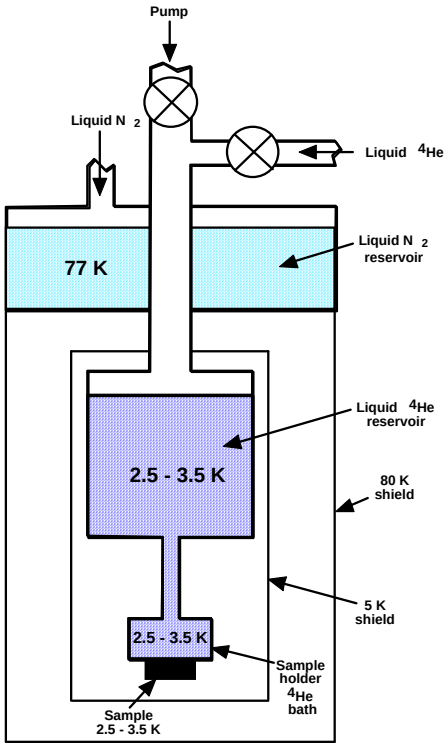

Fig. A2. Schematic of periodically-filled single stage liquid He evaporation refrigerator at 2.5 - 3.5 K

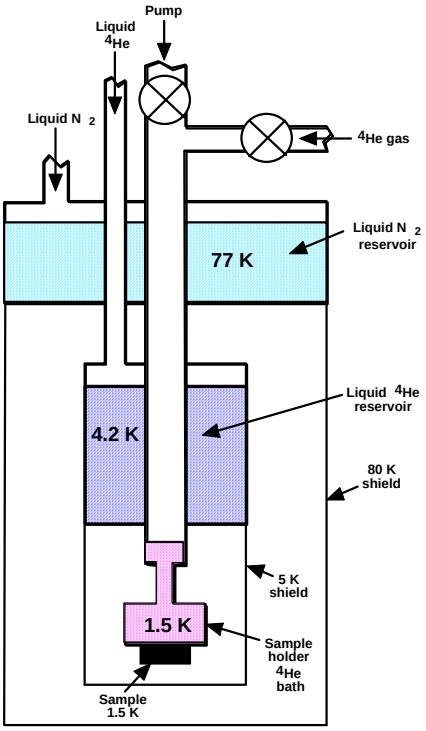

Fig. A3. Schematic of two-stage liquid He evaporation refrigerator at 1.5 K with sample holder bath periodically-filled using ^{4}He exchange gas

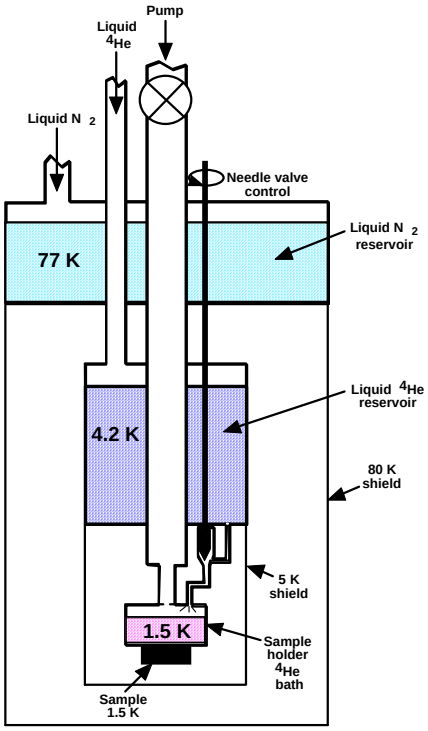

Fig. A4. Schematic of two-stage liquid He evaporation refrigerator at 1.5 K with sample holder bath periodically filled through needle valve

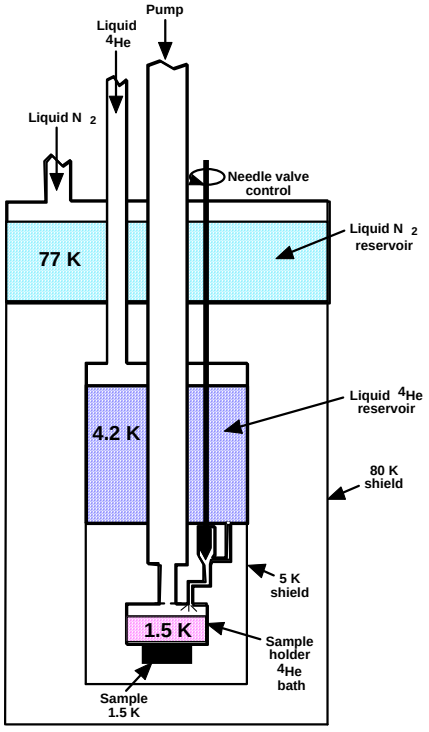

Fig. A5. Schematic of two-stage liquid He evaporation refrigerator at 1.5 K with sample holder bath continuously filled through needle valve

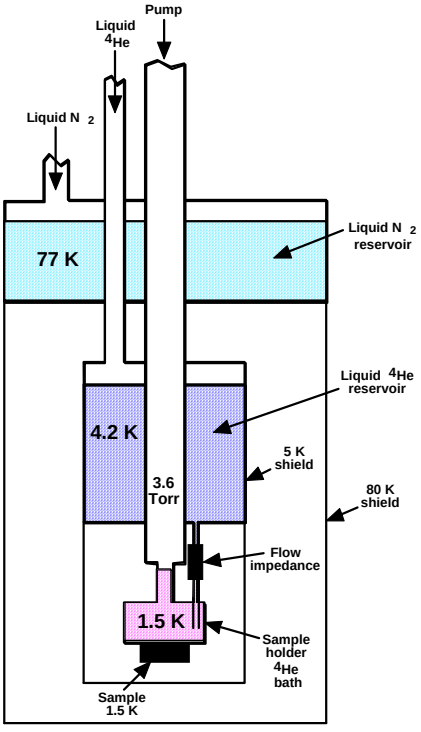

Fig. A6. Schematic of two-stage liquid He evaporation refrigerator at 1.5 K with sample holder bath continuously filled through flow impedance

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