INTRODUCTION TO LABORATORY CRYOGENICS

By

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PREFACE

This work is meant for scientists, students and laboratory personnel who have had little or no experience in cryogenics. It concentrates primarily on cryogenic systems that are commercially available for operation between 1.5 K and 300 K (room temperature). For those users who want to design and build their own equipment, or who are interested in a more detailed analysis of cryogenic systems, a number of excellent references are listed at the end of this booklet.

The first section discusses the vacuum requirements of laboratory dewars and variable temperature cryostats. This is followed by a section on liquid helium and liquid nitrogen dewars, and another section on variable temperature cryostats. The next section concentrates on superconducting magnets that are combined with variable temperature cryostats for laboratory experiments, requiring relatively large magnetic fields.

Section five describes closed cycle refrigerator cryostats that require no liquid cryogens, and the last section concentrates on experimental techniques, thermometry, and automatic temperature control. This last section also includes a few tables that are helpful in estimating heat loads on the cold stage and attached sample. More detailed information and experimental data are available from the references listed at the end of this booklet.

The figures (drawings) spread throughout sections 2 through 6, show a variety of dewar and cryostat designs, based primarily on our experience at Janis. These designs have evolved over a 40-year period, and represent a typical cross section of laboratory units that can be used for a wide variety of experiments. Many other configurations with differing designs are of course available, but were not included due to lack of space. The designs that are discussed in this booklet should, however, address most standard experimental requirements for work at low temperatures.

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1. VACUUM REQUIREMENTS

A liquid helium or nitrogen dewar usually consists of one or more reservoirs containing the cryogen. The cryogen reservoirs are surrounded by a jacket that is evacuated in order to reduce (or eliminate) the conductive heat load due to any gases inside this jacket. This conduction will cause condensation of moisture around the outside of the dewar, and more importantly, result in an unacceptable heat load on the cryogen reservoir(s). Thus, most dewars and transfer lines are supplied with an evacuation valve, located on the outer jacket, for evacuating this space.

Liquid nitrogen shielded, liquid helium dewars will usually have two interconnected vacuum jackets surrounding the helium and the nitrogen reservoir. In such dewars, the liquid helium reservoir will be surrounded by a liquid nitrogen reservoir, in order to reduce the radiational heat load into the helium reservoir. Since this heat load is proportional to the fourth power of the absolute temperature, a liquid nitrogen reservoir at 77 K will radiate significantly less than an equivalent surface at 300 K (room temperature). Other liquid helium dewars and transfer lines are supplied with multi layer insulation surrounding the cryogen reservoir (or inner line), and only one evacuated jacket. Multi layer insulation (MLI) or floating radiation shields reduce the radiational heat load into the liquid helium space, as the inner layers are cooled by the escaping helium vapor in a properly designed dewar. Liquid nitrogen dewars will contain only one vacuum jacket, and may also contain MLI for reducing the radiational heat load from room temperature on to the nitrogen reservoir.

The pressure in the vacuum jacket of dewars and transfer lines should be reduced to at least 10^{-4} torr, and preferably one or two orders of magnitude lower. At room temperature, a pressure of 10^{-4} torr results in a mean free path of approximately 100 cm, (Ref. 1, Ch. VII) much larger than the typical separation between the outer and inner walls of most research dewars. This is the region of molecular flow, where the dominant type of collision processes is between the molecules and the walls of the vacuum jacket(s). The heat load in a typical cryostat (from 77 K to 4.2 K) due to helium gas at a pressure of 10^{-5} torr is a few milliwatts (Ref. 2, Ch. 4). It is thus critical to reduce the pressure inside the vacuum jacket to the 10^{-5} or 10^{-6} torr region.

A variety of pumping stations are available for obtaining pressures of 10^{-5} to 10^{-6} torr. The most commonly used stations consist of a turbo molecular pump, backed by a rotary pump. You may also use a diffusion pump, backed by a rotary pump, with a liquid nitrogen cold trap to prevent oil vapors from back-streaming into the vacuum jacket. A 3" diffusion pump is generally sufficient for most laboratory dewars and cryostats. Turbo molecular pumping stations have a small advantage over diffusion pumping stations, since the turbo molecular pump can be activated simultaneously with the backing rotary pump. On the other hand, a diffusion pump should not be turned on until the back up rotary pump has reduced the pressure to about 5×10^{-2} torr, in order to prevent the oxidization of the oil in the diffusion pump. Lower speed (40 – 60 lit/sec) turbo molecular pumps are now available at the same price as the 3” diffusion pumps, and are quite adequate for use with smaller laboratory type cryostats. For very critical situations where oil vapors are to be completely avoided, one can use an oil free turbomolecular pumping system, where the turbo molecular pump is backed by an oil free diaphragm pump. These pumping stations typically have lower pumping speeds and ultimate vacuum levels.

Furthermore, the diaphragm pumps are not as rugged as the standard mechanical pumps and will this require replacement after a couple of years of use.

Rotary pumps are occasionally used (in line with a cold trap) to reduce the pressure to10^{-2} torr (single stage) or 10^{-3} torr (good double stage). Such pressures are not preferred for operation of liquid nitrogen dewars or cryostats that operate between 77 and 300 K. Occasionally, however, they will give satisfactory performance with liquid helium dewars, if the dewar evacuation valve is sealed just before the liquid helium is transferred into the dewar. This is because the liquid helium reservoir will cryopump the dewar vacuum and results in a pressure of 10^{-6} torr, which is quite adequate. If this set-up is used with some variable temperature cryostats, then once the cryostat is heated above 80 K, out gassing will occur, and could result in poor performance due to the deteriorating vacuum level.

Most pumps start out at a high pumping speed (usually quoted by the manufacturer) then as the pressure drops in the chamber, so does the speed of the pump. A quantity that is very useful in describing the system being evacuated is the throughput. This is defined as the quantity of gas (in pressure volume units) flowing per second through a specified cross section of the system [Q = d(PV)/dt]. For steady state flow in a pipe with no sources or sinks, the throughput of gas between two points is proportional to the pressure difference between those points (Ref. 3, Ch. 2). This may be expressed as:

$$Q = U(P_2 - P_1), \qquad 1.1$$

where U is the conductance of the section between the two points. When several sections of a pumping system are connected in series, then the combined conductance U_s is given by,

$$U_S^{-1} = U_1^{-1} + U_2^{-1} + U_3^{-1} \ldots$$

Now the speed (S) of a pump (for a particular gas) is defined as the ratio of the throughput at the pump entrance, to the pressure of that gas. Thus

$$S = Q/P \qquad 1.2$$

When one attaches a pump to the evacuation valve, through a specific pumping line, then the throughput of gas at the pump is the same as the throughput of gas leaving the vacuum jacket. Since the pressure at the pump is lower than that at the vacuum jacket (when dewar evacuation is in progress), this results in an effective pumping speed (S_d) at the dewar, where the pressure is P_d, obtained from the relation:

$$Q = SP = S_d P_d \qquad 1.3$$

When this is combined with 1.1, the resultant effective pumping speed at the dewar becomes

$$S_d = S \Big/ \left(1 + S \big/ U\right) \qquad 1.4$$

The conductance of a long tube (length L much greater than the radius r) for air at room temperature is approximately 100 r^3/L (Ref. 3, Ch. 1) liters/second (with r and L expressed in cm). If one were using a 1.5" diameter x 5' long evacuating line, this would result in 4.5 lit/sec conductance. Thus, using a 27 cubic foot/minute (cfm) pump (12.7 liters/second) at one end of this evacuating line, the effective pumping speed at the other end would drop to 7 cfm (3.3 lit/sec).

A variety of vacuum gauges can be used at different pressure ranges. A thermocouple gauge is often used with rotary pumps in the range of 1 atmosphere to 10^{-3} torr. For lower pressures (10^{-3} to 10^{-7} torr) an ionization or cold cathode gauge is commonly used. These gauges are usually located at the pump opening, and it is important to note that depending on the pumping line connecting it to the dewar, the pressure at the dewar can be significantly higher (at room temperature). Thus in the above example of the 5' line, using equation 1.3, one gets a pressure at the end of the line 3.8 times that of the pressure at the pump.

Once the vacuum jacket of the dewar is evacuated, and the dewar cooled (to LN_2 or LHe temperatures), the pressure in the cold region will drop. An approximate relation that holds under these low pressures is that the pressure will vary in proportion to the square root of the absolute temperature ($P/\sqrt{T}$ = constant) (Ref. 2, Ch. 4). It is therefore recommended that when liquid helium is transferred into a dewar, the dewar evacuation valve should be closed. If this is not done, the cryopumping that occurs due to the liquid helium cooled cold surfaces may pump back oil vapors (from the pump) into the vacuum jacket. This is especially true if the pumping station used does not have a liquid nitrogen cold trap. This will contaminate the vacuum jacket and result in a higher heat load, and poor dewar performance.

Very often, the design of metal dewars requires the use of thin walled (0.01" - 0.06" thick) stainless steel tubing for the helium (or nitrogen) reservoirs. These reservoirs are therefore not designed to withstand a pressure difference of one atmosphere on their exterior surface and will thus collapse. They are however, designed to withstand an atmosphere pressure difference on their inner surface. A potentially dangerous situation can occur if one evacuates the helium reservoir while the outer jacket is at atmospheric pressure. It is thus best to always maintain the vacuum jacket under vacuum, and preferably install a pressure gauge on this jacket to indicate the pressure inside the jacket. It is also good to avoid introducing helium gas inside the vacuum jacket since helium gas has a high thermal conductivity (about 12 times that of nitrogen gas), will not condense at 4.2 K, and is generally more difficult to remove than air or nitrogen. This is especially true in transfer lines and vapor shielded dewars that have MLI in their vacuum jackets.

2. LIQUID HELIUM AND NITROGEN DEWARS

As mentioned earlier, liquid helium and nitrogen dewars usually consist of one or more reservoirs, surrounded by a vacuum jacket, which isolates these reservoirs from room temperatures. Most dewars are made from stainless steel since it can be easily and permanently joined to similar or dissimilar (copper, brass, etc.) metals by welding (in an inert gas atmosphere) or silver soldering. Such joints can withstand many cycles between room temperature and helium (or nitrogen) temperature and still remain leak tight after years of use. Other dewars are manufactured from low thermally conducting epoxy-fiberglass necks and aluminum reservoirs.

A. Welded Dewars

The simplest dewars will have an all welded construction with direct access to the cryogen reservoir through the top of the dewar. These dewars are used for storage or for directly immersing the sample in the cryogen. Such dewars will have an evacuation valve for evacuating the space surrounding the cryogen reservoir, and also a safety pressure relief valve to protect the vacuum jacket should an internal leak develop. Such a leak will result in the cold cryogen entering the vacuum jacket, warming up after contact with the room temperature wall, and expanding. This could result in a high pressure build up in the vacuum jacket. A safety pressure relief valve set to open at 2 to 5 lb/in^2 (psi) above atmospheric pressure, will safely vent the leaking gas to the outside of the vacuum jacket and prevent any dangerous pressure build up.

A simple open neck, "bucket type" liquid nitrogen dewar is shown in Figure 2.1. The liquid nitrogen reservoir is a cylinder with diameter A, and the outer jacket is a concentric cylinder of diameter B. The space between A and B is evacuated through the evacuation valve, which should be a good bellows sealed valve. The liquid nitrogen cools the charcoal getter located in the vacuum jacket, and helps maintain a good vacuum over extended periods of operation. Such a dewar can be used to immerse a sample or any insert directly in liquid nitrogen.

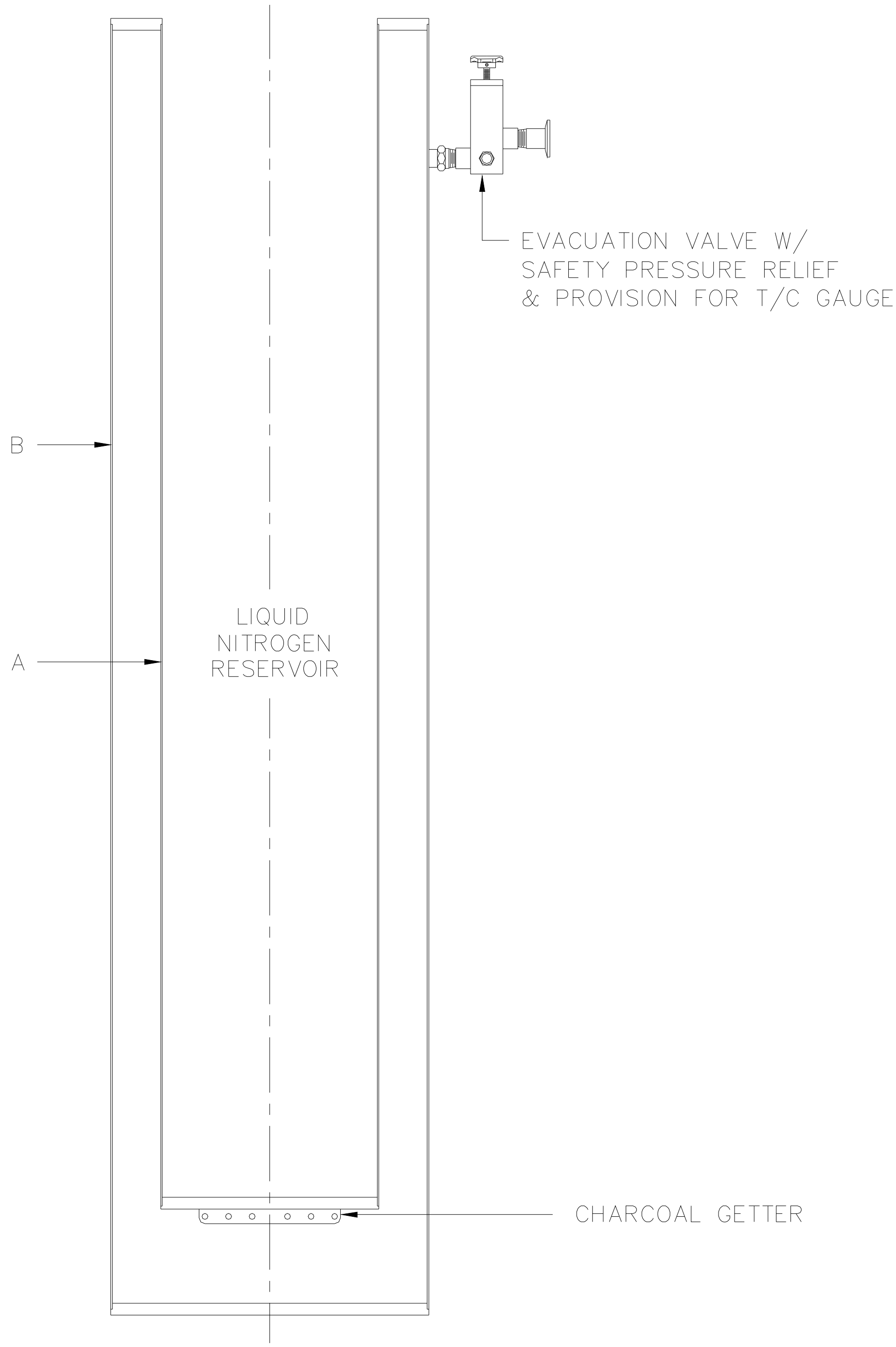

FIGURE 2.1 OPEN NECK LIQUID NITROGEN DEWAR

Figure 2.2 shows an open neck (bucket type) liquid helium dewar where the helium reservoir is a cylinder of diameter A. This is surrounded by a nitrogen reservoir (annular space between tubes B & C), which shields the surface of the helium reservoir from room temperature radiation. The space surrounding the helium reservoir (between A & B), and the space surrounding the nitrogen reservoir (between C & D) is evacuated through the evacuation valve on the outer jacket (diameter D). These two spaces are connected through a slot in the radiation shield, which is bolted to the bottom of the nitrogen reservoir. Thus both spaces are evacuated simultaneously through the evacuation valve. The radiation shield bolted to the bottom of the nitrogen reservoir is usually made from a good thermal conductor (aluminum or copper), while the rest of the dewar is made from stainless steel or a combination of aluminum and low thermal conducting epoxy fiberglass (in the neck region). For stainless steel dewars, tube A has a wall thickness of about 0.025" – 0.062" (for larger diameter necks), in order to reduce the conductive heat load into the liquid helium. For many stainless steel dewars, the thin walled helium reservoir should not be evacuated before ensuring that the main dewar is under vacuum.

When the nitrogen reservoir is filled with liquid nitrogen, the thermal anchor F and the region of the helium reservoir neck to which it is connected, will be cooled to approximately 80 K. This intercepts the conductive heat load into the helium reservoir from room temperature along the neck of the helium reservoir (top portion of tube A). Generally, the top of the helium reservoir is separated from the thermal anchor F by some distance to provide thermal isolation for the liquid helium (which is at ~4.2 K) and the 80 K temperature at F.

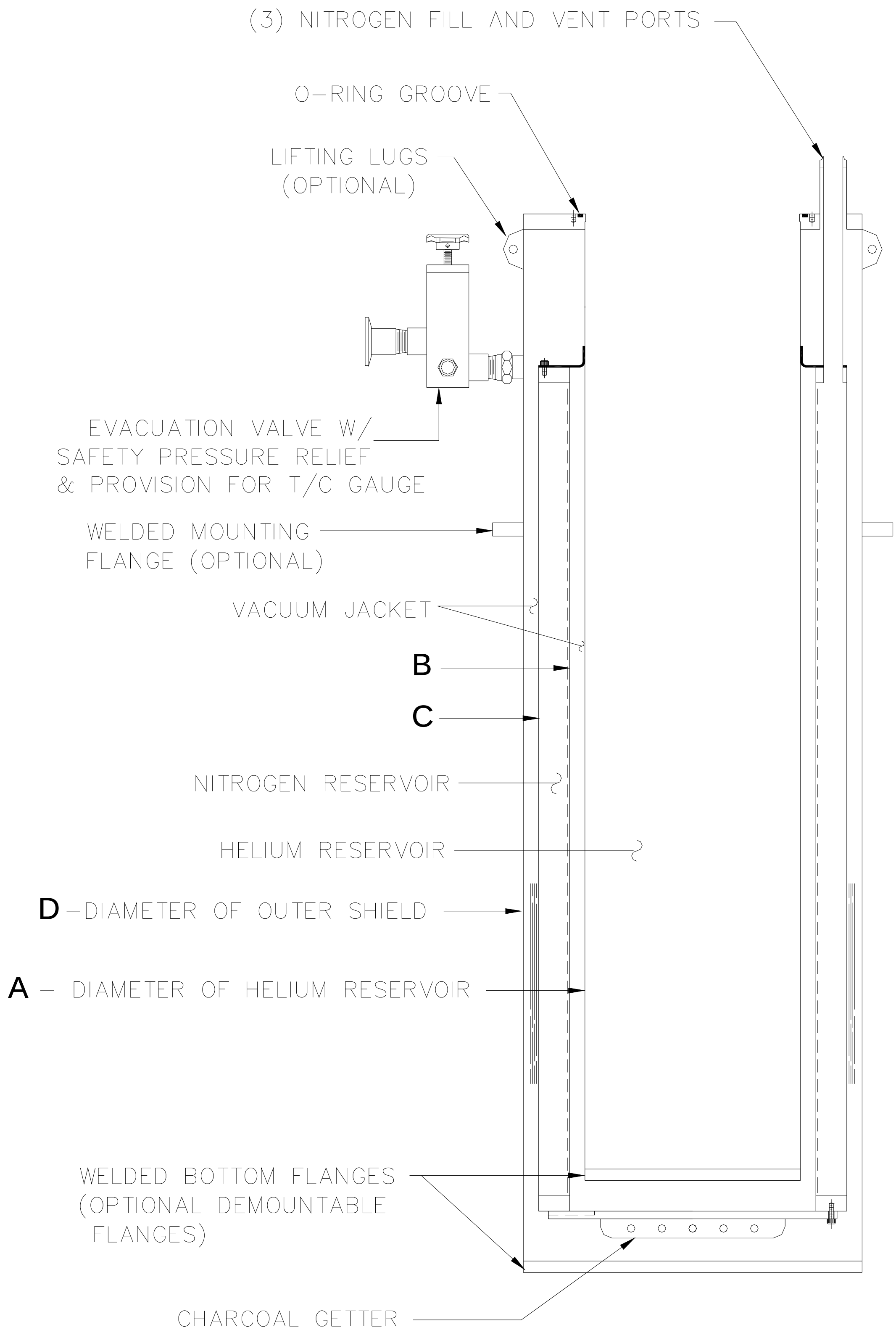

FIGURE 2.2 OPEN NECK NITROGEN SHIELD LIQUID HELIUM DEWAR

Other types of bucket dewars are also shown in Figures 2.3 through 2.5.

Figure 2.3 shows another type of bucket dewar, which is designed to operate with no liquid nitrogen shielding. These vapor shielded dewars are built with several radiation shields that are attached to the dewar neck, and are cooled by the escaping helium vapor. These shields are thermally isolated from each other and eventually float at lower and lower temperatures (starting from the outermost shield), with the inner most shield floating at a temperature that is close to 80 K or less. This reduces the radiational heat load into the helium reservoir to a value similar to the nitrogen shielded dewars, resulting in acceptable helium evaporation rates. These shields are also surrounded by multiple layers of insulation (MLI) made from very thin aluminized Mylar, commonly referred to as superinsulation. This further reduces the radiational heat load on the inner radiation shields, and ultimately on the helium reservoir. The diameter of the helium reservoir A determines the maximum size of the insert and the outer diameter B is typically 4” to 5” larger than A.

Figure 2.4 shows a vapor shielded “belly” type dewar where the helium reservoir has a much larger diameter than the neck that connects it to the room temperature flange. For aluminum dewars, this neck is made from special impregnated epoxy fiberglass tubes that are connected to the aluminum top flange and the top helium reservoir flange with special epoxies designed to offer a strong vacuum tight joint and support. This dewar also has a narrower “tail” which is particularly useful for immersing a superconducting magnet that needs to be always covered with liquid helium during operation. The smaller volume around the magnet reduces the amount of liquid helium that is needed to cool the magnet and maintain it in the liquid. The Belly diameter (and length) determines the helium capacity, while the neck diameter B and tail diameter C determine the maximum size of the insert that will fit inside the dewar. The outer diameter D is typically 5” larger than the belly.

Figure 2.5 shows an ultra low loss bucket dewar that includes a liquid nitrogen reservoir surrounded by MLI, plus an intermediate radiation shield between the nitrogen reservoir and the helium reservoir. This shield floats at a temperature between liquid nitrogen and liquid helium and significantly reduces the radiation heat load at the helium reservoir. The result is an ultra low loss dewar that typically reduces the helium consumption by a factor of two or more. The belly diameter (and length) determines the capacity of the helium reservoir, while the neck diameter B and tail diameter C determine the maximum size of the insert that will fit inside the dewar. The nitrogen reservoir is located in the upper section of the dewar, with an outer radiation shield (cooled by the nitrogen reservoir) surrounding the helium reservoir. A second radiation shield (cooled by the helium vapor that is venting through the neck) is built between the helium reservoir and the first radiation shield. The vacuum spaces surrounding the reservoirs and radiation shields are all interconnected, so the dewar requires only one single evacuation valve.

Any insert placed in the helium reservoir should contain several radiation baffles (usually made from copper) in the neck region, with a diameter 0.25" less than that of the neck. These baffles are cooled by the escaping helium vapor and thus intercept the radiational heat load from the room temperature top flange of the insert (which can be very large). The baffles also force the vapor to make more intimate contact with the helium reservoir neck, thus intercepting about 85% of the conductive heat load down the neck (when properly designed).

The inserts that are placed into this type of dewar could be a simple immersion type or a variable temperature cryostat. The former will usually place the sample in direct contact with the liquid helium at 4.2 K or lower. Lower temperatures are achieved by reducing the pressure on top of the helium through an appropriate pumping port. Variable temperature inserts (as will be discussed later) will place the sample in an environment where its temperature can be raised above 4.2 K (usually to room temperature or higher), or reduced below 4.2 K (to 1.5 K, 0.3 K or a few milli-K). These inserts should always be supported from thin walled stainless steel or epoxy fiberglass tubing, in order to reduce the conductive heat load into the helium reservoir. This is due to the fact that the latent heat of vaporization of liquid helium is extremely small (1 watt will boil off ~1.4 liters LHe/hr when absorbed by the liquid). Also any wiring that enters the helium reservoir should be as thin as possible, and (whenever possible) made from poor thermal conductors such as manganin, phosphor bronze, stainless steel, etc. In all cases one should either intercept some of the heat load with liquid nitrogen, which has a much higher heat of vaporization (1 watts boils off 22.4 milli-liters LN_2/hr), or make use of the tremendous cooling power of the boiling helium vapor between 4.2 K and 300 K (about 80 times the latent heat of vaporization of LHe at 4.2 K).

Inserts that contain superconducting solenoids which need to be immersed in liquid helium, are also placed in these "bucket type" dewars. Special counter-flow vapor cooled high current leads should be used with such inserts in order to intercept most of the heat generated in those leads, before it reaches the helium reservoir. These and other types of inserts will be discussed in Chapter 3.

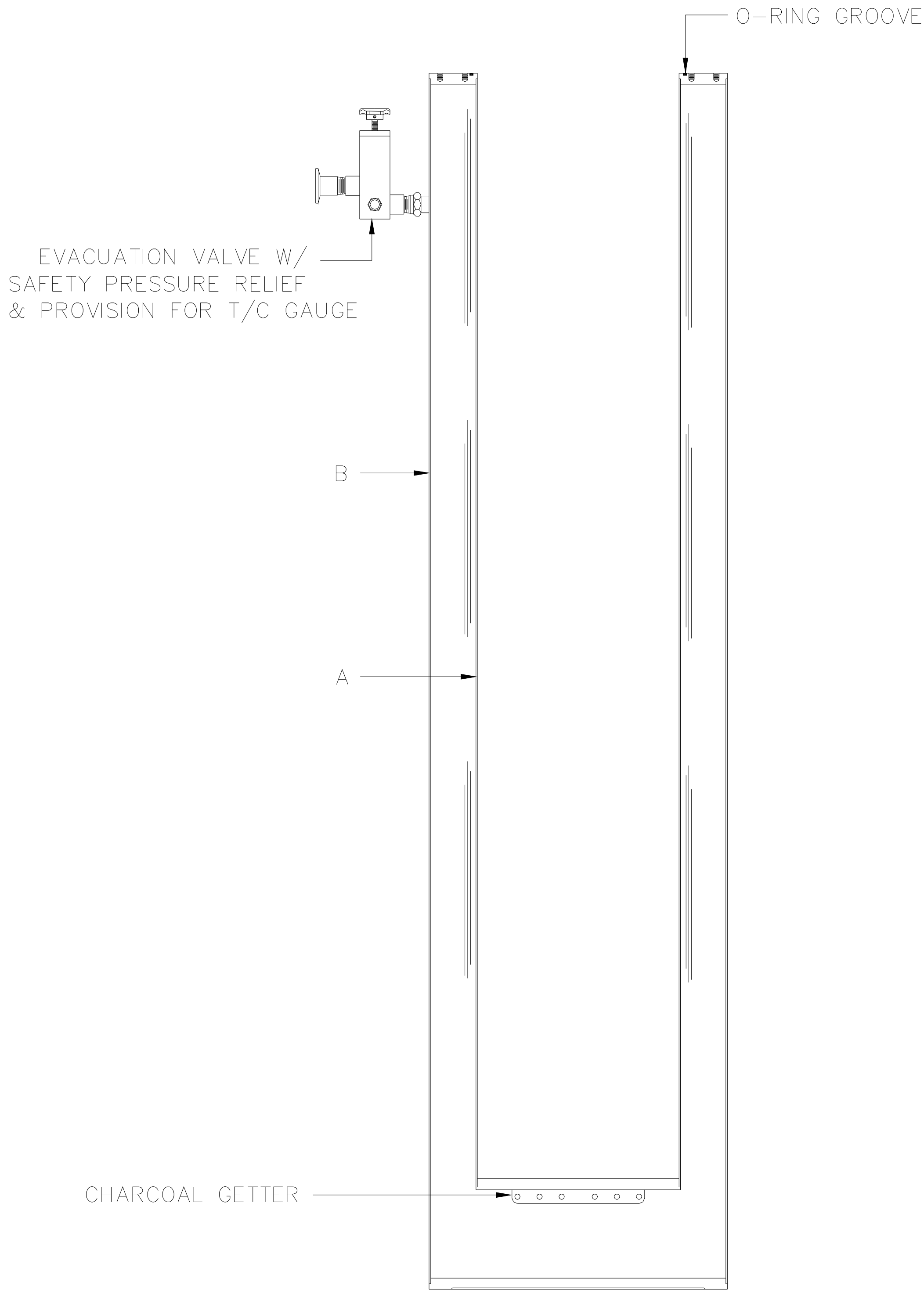

FIGURE 2.3 OPEN NECK SUPERINSULATED LIQUID HELIUM DEWAR

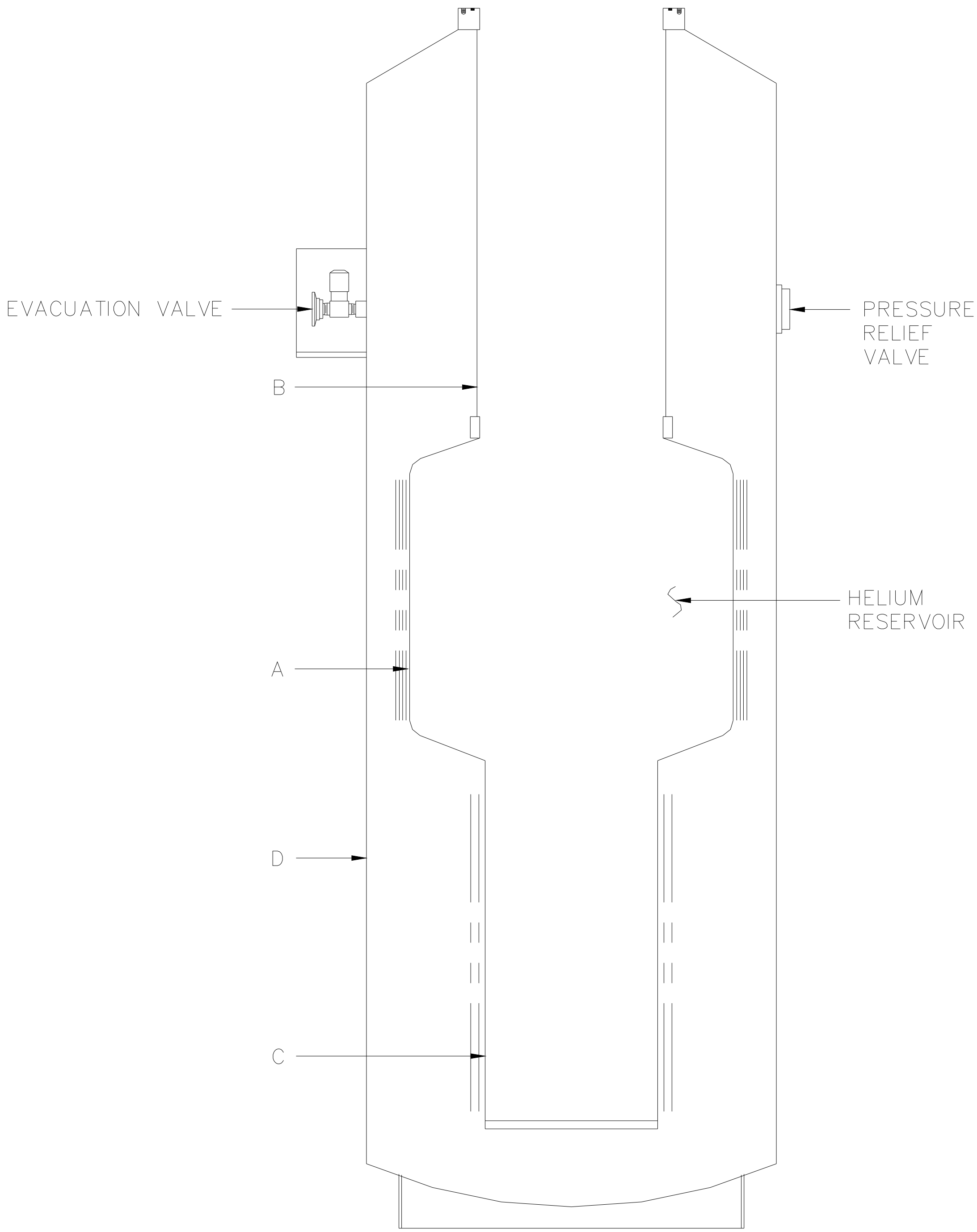

FIGURE 2.4 SUPERINSULATED LIQUID HELIUM BELLY DEWAR

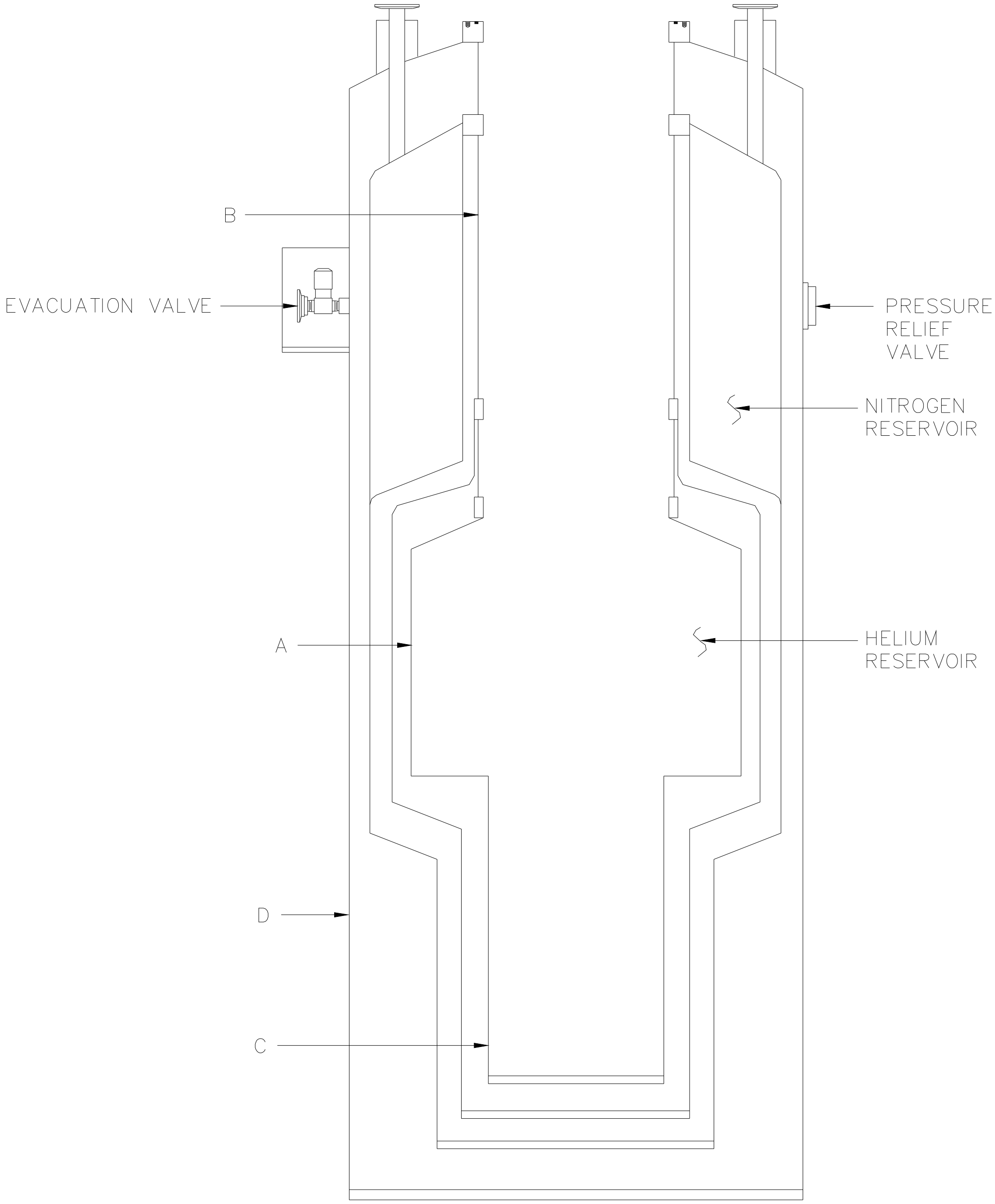

FIGURE 2.5 NITROGEN SHIELDED LOW LOSS HELIUM DEWAR

B. Detachable Tail Dewars

While some liquid helium and nitrogen dewars are made in an all welded design, others can be made with detachable bottom flanges for additional versatility. Figure 2.6 shows an example of such a helium dewar.

In this case, a crushed indium seal is used at the bottom of the helium reservoir and an O-ring seal is used at the bottom of the outer jacket. A radiation shield flange is mechanically attached to the bottom of the annular nitrogen reservoir. This arrangement offers access to the helium reservoir through the bottom of the dewar. It also allows various tail extensions to be welded to these flanges to give a variety of configurations.

The detachable tail dewar shown in figure 2.6 shows a nitrogen reservoir that is supported by three (fill/vent) tubes (F), which are thermally isolated from the top flange. The outer (vacuum) jacket is usually completely removable from the top flange. The neck and the helium fill and vent tubes support the helium reservoir from the top flange. A copper thermal anchor then links the top of the nitrogen reservoir to the helium reservoir neck and fill and vent tubes, in order to intercept the conductive heat load from room temperature. Both reservoirs are surrounded by a common vacuum.

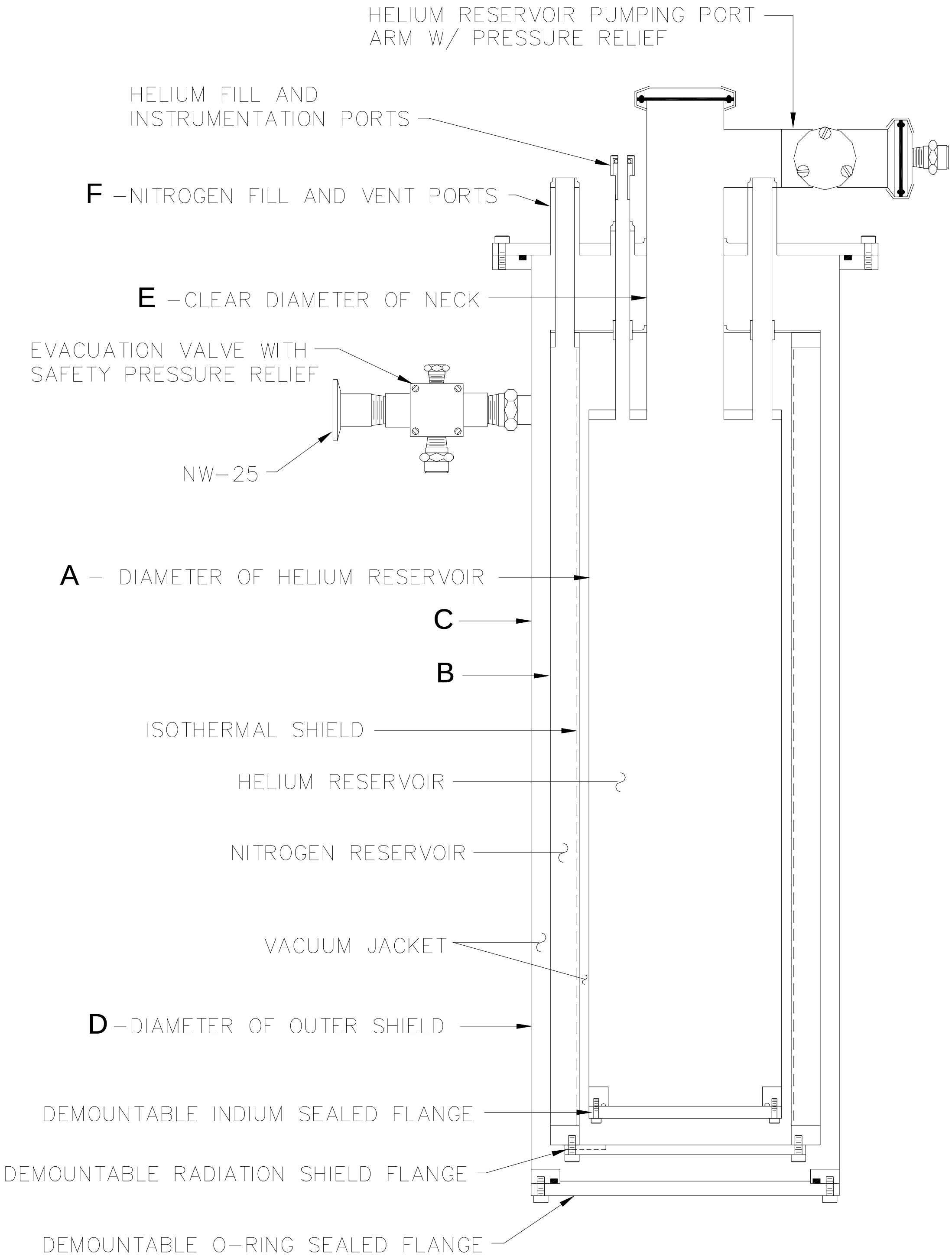

FIGURE 2.6 DETACHABLE TAIL LIQUID HELIUM DEWAR

C. Tail Extensions

As mentioned earlier, one of the main advantages of a detachable tail dewar is that a tail extension can be added to the dewar. These result in either constant or variable temperature cryostats that can be used for a large variety of scientific experiments at low temperatures. A few examples of such tail extensions will be discussed in what follows.

1. Immersion Tails

The simplest extension tail that can be added to a detachable tail dewar, is one that extends the helium reservoir directly into a narrower region (tail) below the dewar. Figure 2.7 shows such a set of tails, attached to the bottom of a detachable tail dewar. The helium tail (diameter A) is simply an extension of the helium reservoir, with liquid helium inside it. The (aluminum) radiation shield tail (diameter B) is welded to the radiation shield flange, which in turn is bolted to the bottom of the nitrogen reservoir, and is thus conductively cooled. The outer tail (diameter C) is welded to the bottom O-ring sealed flange, and now becomes an extension of the outer vacuum jacket. The main application of this configuration is to fit in a limited space such as the pole gap of an electro-magnet. The usual clearance required between the helium tail and outer tail diameters is a function of the length L, but is normally 1" (C-A = 1"). This clearance may be reduced to 0.5" (or less) with appropriate use of concentricity spacers between the tails, to avoid physical contact, which would result in a thermal short. The addition of such spacers between the tails will result in an additional heat load into the helium and/or nitrogen reservoir, which could result in a higher cryogen consumption. These spacers are added only when dictated by the geometrical configuration desired.

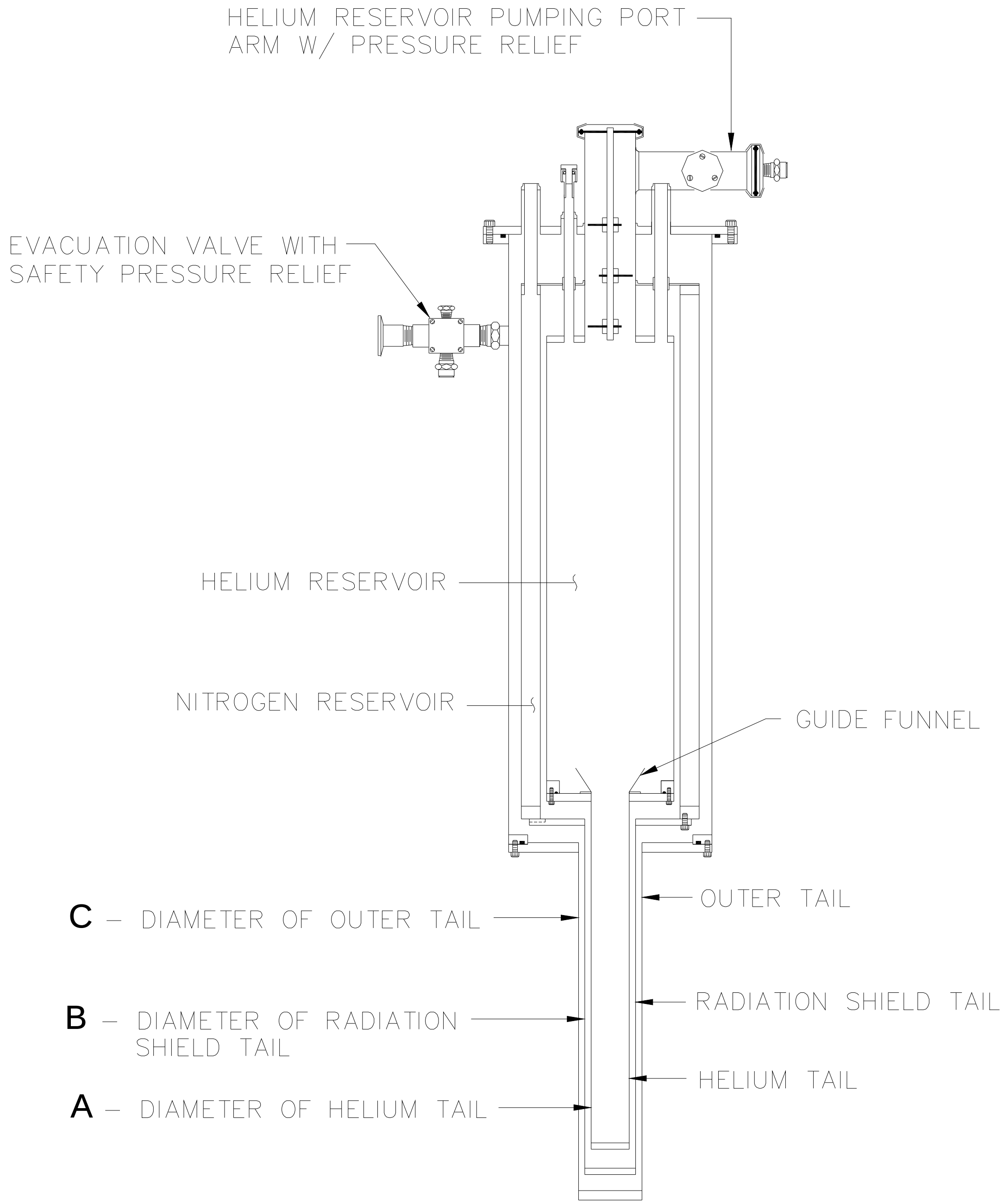

FIGURE 2.7 IMMERSION TUBULAR TAIL DEWAR

Figure 2.8 shows another example of immersion tails, but with windows, providing optical access to the sample immersed in liquid helium. The cylindrical helium tail (diameter A) now has an additional window block, located at a distance Y below the main dewar body. The window block is required since the inner windows are usually flat discs that need to be sealed (vacuum tight) at helium temperature. This seal is usually achieved using special epoxies that are guaranteed to seal the windows against the block and withstand the thermal cycling between helium and room temperatures. The windows may also be sealed via a crushed indium O-ring, for situations requiring special materials, large window openings or window replacements. The indium sealed windows require a larger window block and a correspondingly larger diameter radiation shield and outer tail.

An added bonus of such a tail extension is that optical access to the sample does not need to pass through the (double walled) nitrogen reservoir. Now, a simple opening in the radiation shield tail is all that is required, since there is vacuum on both sides of this tail. The hole in the radiation shield tail, which is required for optical access to the helium tail, also allows (300 K, IR) radiation from the outer tail, to fall directly onto the helium tail. This presents an additional heat load on the helium tail, which is estimated at 25-50 milliwatts/cm^2 opening in the radiation shield. In order to reduce this heat load, the size of the hole is always minimized, or else an additional window (cooled to the temperature of the radiation shield) is installed. Installation of radiation shield windows usually requires another window block at that tail, resulting in a larger outer tail, which is not always desirable. However, when such windows are properly installed, they can practically eliminate the radiational heat load through the radiation shield ports. One should also note that for some specific applications (such as Gamma rays or X-rays), thin film Mylar (or aluminized Mylar) windows can be wrapped directly onto the cylindrical tails and thus require no more room than non-optical (tubular) tails.

Windows on the outer tail are also installed on a window block with flat faces, and are usually O-ring sealed. A retainer usually holds the window in place before the dewar is evacuated. Once the vacuum jacket is evacuated, the windows are held against the O-ring by atmospheric pressure. With no radiation shield windows, typically the outer tail diameter will be 1.5" greater than the inner tail (C-A = 1.5"), and the distance between two outer windows (180° apart) will be 0.75" larger than the outer tail diameter (A). The clear view of the windows is designed to offer the required solid angle as measured from the center of the sample tube, consistent with the limitations dictated by the cryogenic seal of the inner window. The size and material type of windows used, are also a function of the transmission range (wavelength or frequency) required. A variety of window materials that will withstand thermal cycling between helium and room temperature are available to cover most regions of the electromagnetic spectrum. The dimensions of the outer tail and window block can sometimes be reduced by appropriate use of spacers between the various tails, and by epoxying the outer windows. This latter option reduces the distance between opposing windows since the O-ring and tapped holes for the window retainer will no longer be required.

The above two examples of tail extensions are designed for applications where the sample is immersed in the liquid cryogen. This is usually done by introducing the sample through the top of the dewar, using a long thin wall stainless steel tube to support it off the top of the neck. The temperature of the liquid cryogen (and the sample) may then be reduced below the normal boiling point, by reducing the pressure on top of the liquid. Using a typical 27 cfm roughing pump can usually reduce the temperature of a helium reservoir to below 2 K (at a pressure of 24 torr or less).

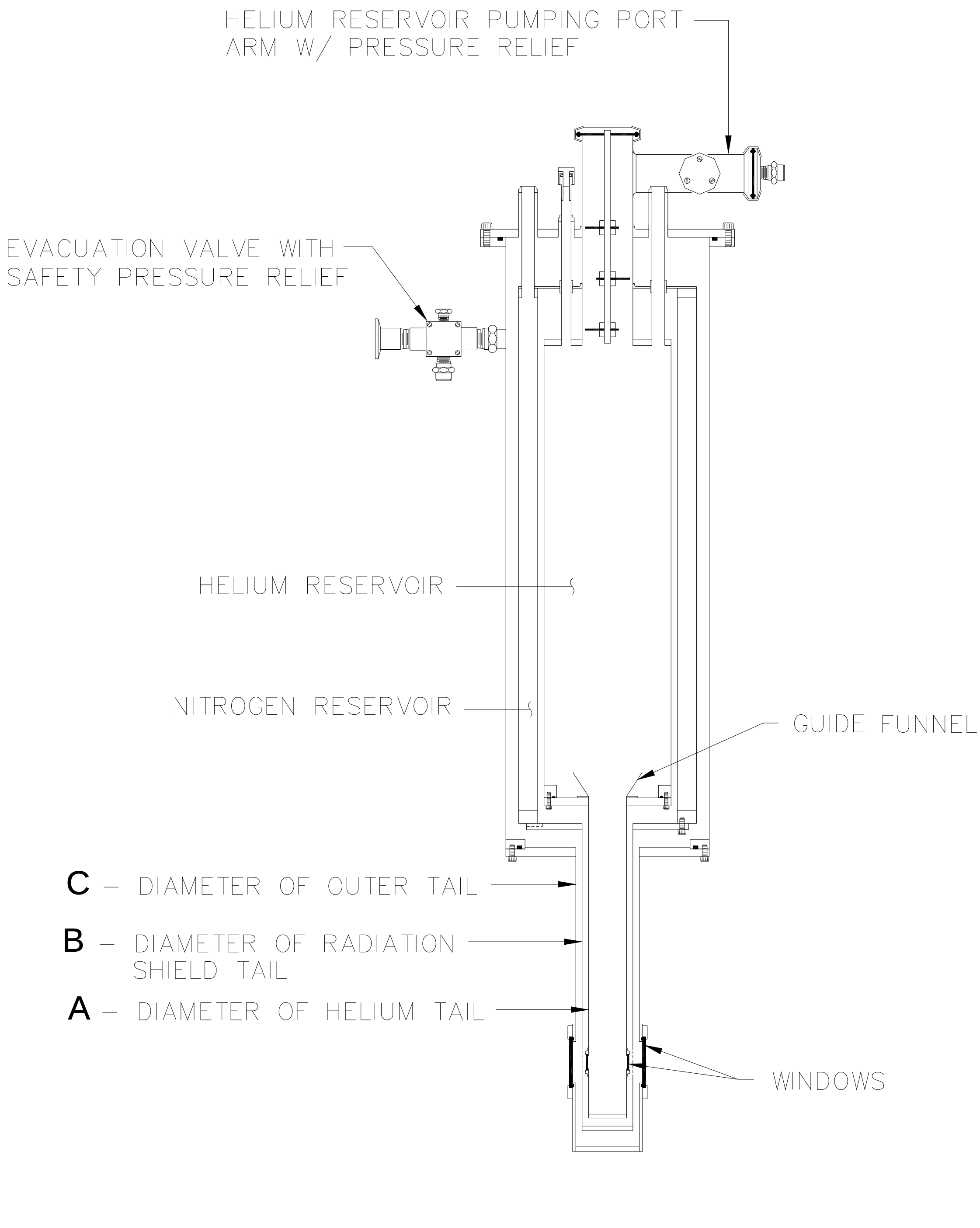

FIGURE 2.8 IMMERSION OPTICAL TAIL DEWAR

2. Tails for Sample in Vacuum

Some experiments require the sample to be cooled down while surrounded by vacuum. For such cases, one can locate the sample in the dewar vacuum, using a tail extension similar to the immersion tails, except that the sample is located below the bottom flange of the inner tail.

Figure 2.9 shows such a tail extension where the bottom of the helium tail is sealed by a copper flange. The face of the copper flange, which lies in vacuum, contains some dead tapped holes, and is located at a distance X above the window center line. This copper piece acts as a sample mount (usually called a cold finger) for attaching samples in vacuum. Since the cold finger is in contact with the liquid cryogen (above it), it remains at the temperature of the cryogen. Samples or sample holders attached to the cold finger must be thermally anchored very carefully in order to be cooled, since the cooling is primarily achieved by contact with the cold finger (in vacuum). Using pressure contacts with either an indium interface, good conductive epoxies or silicone grease usually does this. It is always best to install a thermometer at the sample, in order to measure its actual temperature, which may be several degrees above the temperature of the cold finger. A second radiation shield attached to the cold finger, and surrounding the sample, is usually quite helpful in reducing the radiational heat input into the sample.

The same configuration can be provided without outer windows, for experiments not requiring optical access to the sample. In such a case, the diameter of the outer tail will be about 1" larger than the diameter of the inner tail (C-A = 1"). When windows are supplied on the outer tail, the diameter of the outer tail does not change, and the window-to-window distance (for 180° opposing windows) will be about 0.75" larger than the outer tail diameter (C). Once again, the outer tail diameter and window-to-window distance may be reduced by appropriate use of concentricity spacers and epoxied windows.

The temperature of the liquid cryogen (and the sample mount) can be varied, in the same manner as the immersion tails, by reducing the pressure on top of the liquid in the inner reservoir. A radiation shield bolted to the sample mount (surrounding the sample) is again advisable, especially at lower temperatures, in order to help reduce the temperature differential between the sample and its holder.

Changing samples in such a configuration is significantly different from the immersion tail configuration. Whereas in the immersion case one can remove and introduce samples into the cryogen any time the cryogen is at atmospheric pressure, this cannot be done when the sample is in vacuum. Now, the dewar must be free of cryogens, and preferably at room temperature, before the sample is removed or installed since one needs to break the dewar vacuum. If the dewar is still cold, then air and moisture can condense (or freeze) on the vacuum walls of the dewar, and would need to be completely cleaned and dried prior to evacuating and re-cooling the dewar. For this reason too, it is undesirable to provide such a tail for a dewar with MLI, since re-evacuation can take a long time, especially if any moisture condenses on the MLI.

One example of such dewars are the so-called detector cooling dewars that are used for cooling a sample to a fixed temperature using either liquid nitrogen or liquid helium. Figure 2.10 shows an example of a liquid nitrogen dewar with a cold plate that is cooled by direct contact with liquid nitrogen. Access is typically through an O-ring sealed bottom flange or tail section, with specialized designs that can allow the dewar to be rotated by 90° or 180° for horizontal or inverted operation. The temperature of the liquid nitrogen may be reduced by reducing the pressure on top of the nitrogen (or helium) reservoir, with special internal heat exchangers that allow the dewar to work even with frozen nitrogen (approximately 63 K to 50 K). The size of the dewar will depend on the required hold time or on the required size of the cold plate. Electrical feedthrough access is typically located on the top flange so that the wiring to the cold plate region remains intact when the bottom section is removed to access the cold plate. Ultra High Vacuum (UHV) compatible units will have all metal seals and will generally have electro-polished stainless steel reservoirs and vacuum jackets and gold plated copper cold plates or cold fingers.

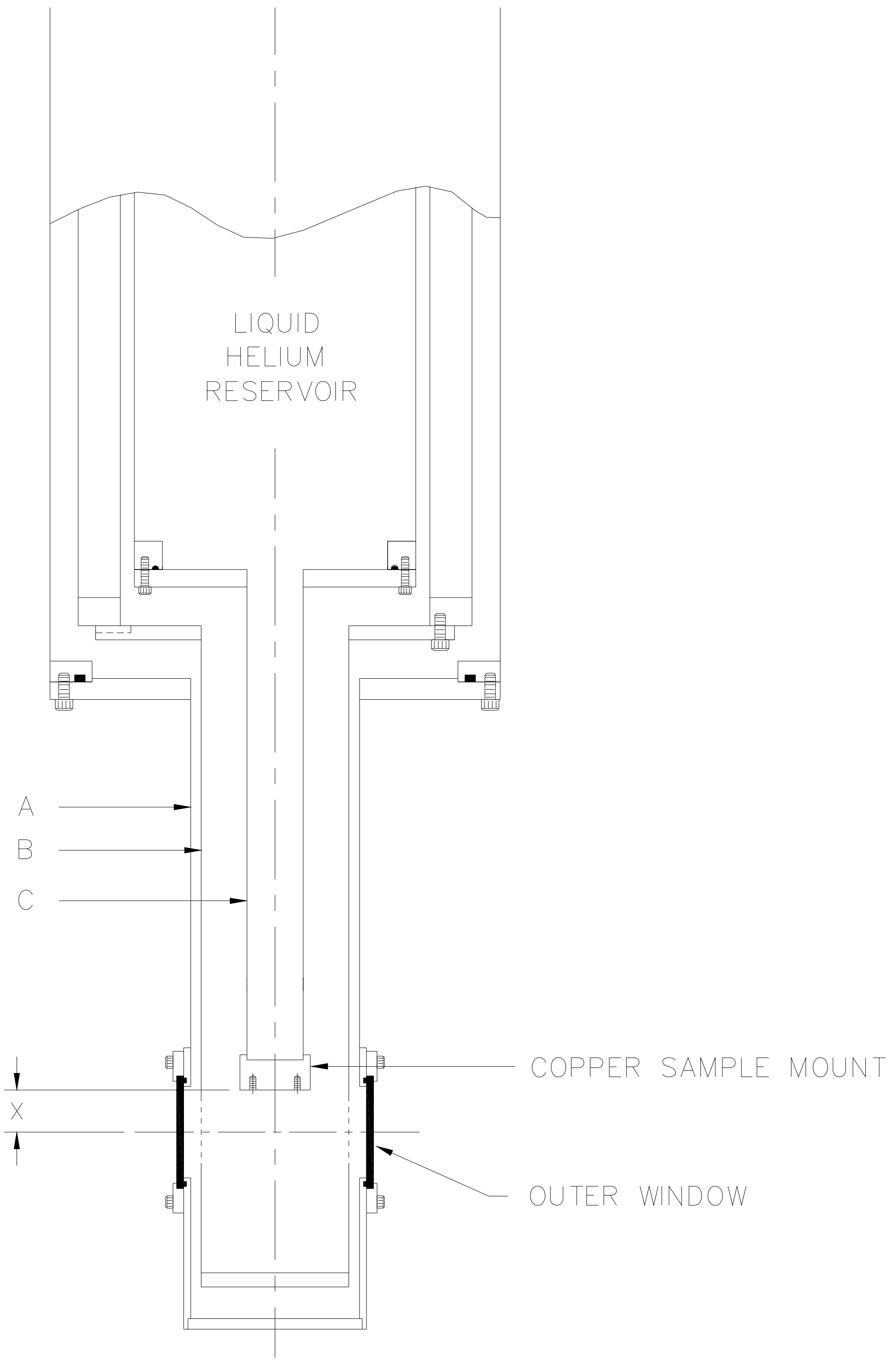

FIGURE 2.9 COLD FINGER / SAMPLE IN VACUUM

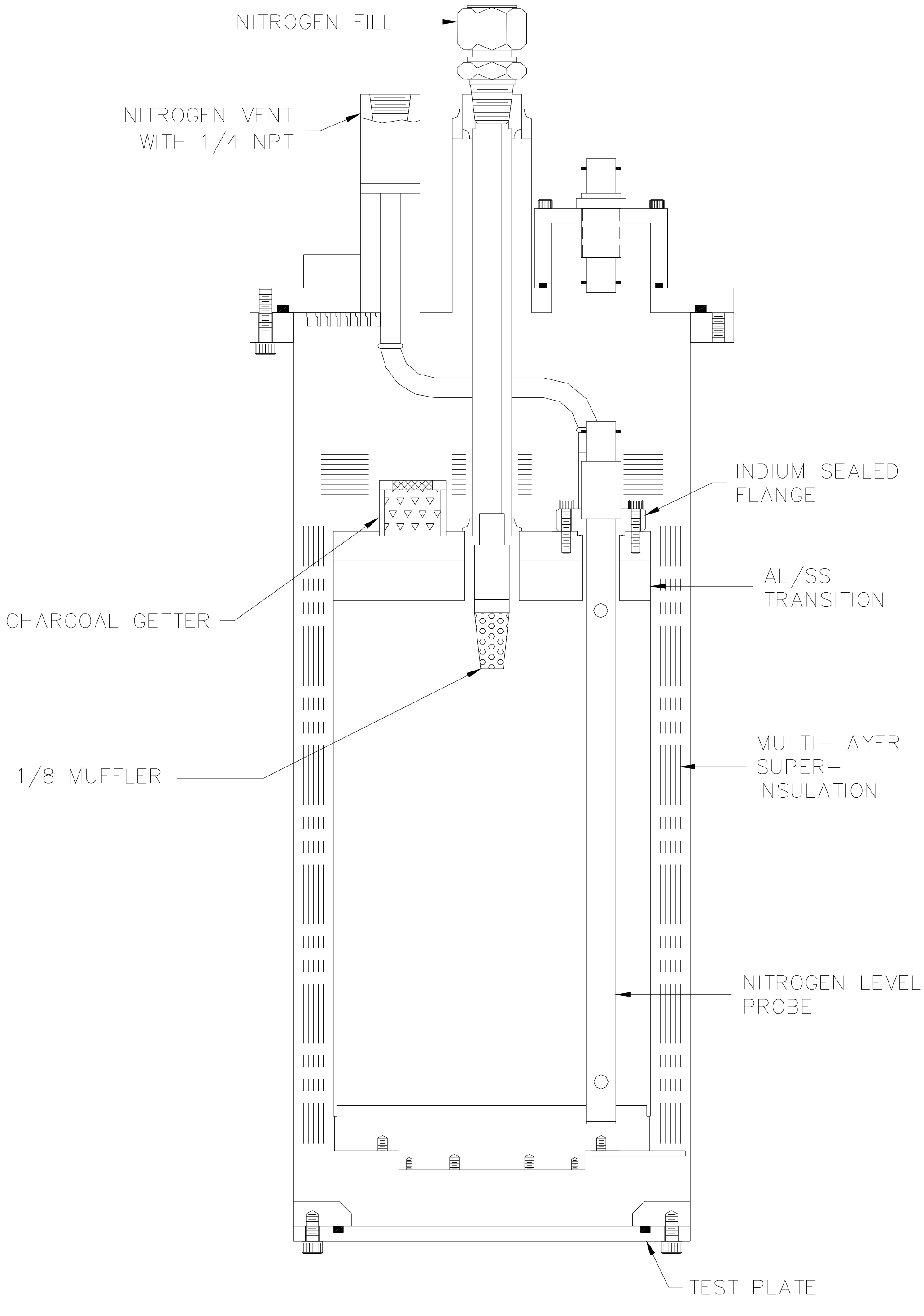

FIGURE 2.10 LIQUID NITROGEN DETECTOR DEWAR

3. VARIABLE TEMPERATURE CRYOSTATS

The simple helium (or nitrogen) dewars discussed previously, allow cooling an apparatus to the temperature of the boiling cryogen, either by direct immersion or by attachment to a cold finger in contact with the cryogen. Simply reducing the pressure on top of the cryogen can reduce the temperature below the normal boiling point of the cryogen. If the temperature needs to be varied above that of the cryogen, then one usually needs an external heat input into the sample holder. If the sample holder is in good thermal contact with the liquid cryogen, then any heat added to the sample holder will quickly be transmitted into the cryogen, and ends up boiling away the cryogen without significantly raising the temperature of the sample. Thus, the first requirement for obtaining higher temperatures is the ability to thermally isolate the sample from the cryogen. If this is done after the sample is cooled, then any heat added to the sample holder will increase its temperature above that of the cryogen, while a temperature gradient develops across the isolating element. In a well designed system, little heat is required to raise the temperature of the sample well above the temperature of the cryogen, without significantly increasing the cryogen consumption rate.

Another method of obtaining temperature variation above the normal boiling point, is to channel a small flow of the cryogen into the sample region and vaporize the cryogen with an external heat source and raise its temperature to the desired level. This method works very well with liquids that have a low heat of vaporization (e.g., helium), while the sample is placed in the path of the flowing heated vapor. When liquids having large heats of vaporization (e.g., nitrogen) are used, it becomes difficult to vaporize the liquid and increase its temperature. In such a case one can either work with flowing cold vapor which can be easily heated, or else separate the sample from the flowing liquid and then add heat to the sample.

Several kinds of variable temperature cryostats will be described in what follows, falling under the general categories discussed above for temperature variation. These cryostats are designed for use with liquid helium or liquid nitrogen, since these are the two most commonly available cryogens. These cryostats can usually be used with other cryogens (liquids air, oxygen, argon, neon, hydrogen) with certain modifications dictated by safe handling of the gas involved.

A. Thermal Impedance Cryostats

A simple method for obtaining variable temperatures for samples in vacuum configurations, is to modify the cold finger configuration of Figure 2.7, by inserting a tight fitting, thermally insulating cylinder into the inner tail. This arrangement is shown in Figure 3.1.

The insulating cylinder presents a thermal impedance (TI), which isolates the copper sample mount from the cryogen above it. A heater is also installed at the sample mount for raising its temperature. Initially, the sample mount will be at the same temperature as the cryogen. When heat is introduced through the heater, any cryogen in contact with the sample mount evaporates, resulting in a vapor bubble on top of the sample mount. This, in conjunction with the insulating cylinder, forms the thermal impedance necessary to isolate the sample mount from the liquid cryogen. The temperature of the sample mount can now be increased above that of the cryogen, by increasing the current through the heater. This insert works well with both liquid helium

(4.2 - 77 K) and liquid nitrogen (77 - 300 K / 500 K). For temperatures below the normal boiling point of the cryogen, the insulating cylinder is removed (from the top of the dewar), and the pressure on top of the cryogen is reduced. This insert forms the basis for the Janis SSVT system for temperature variation.

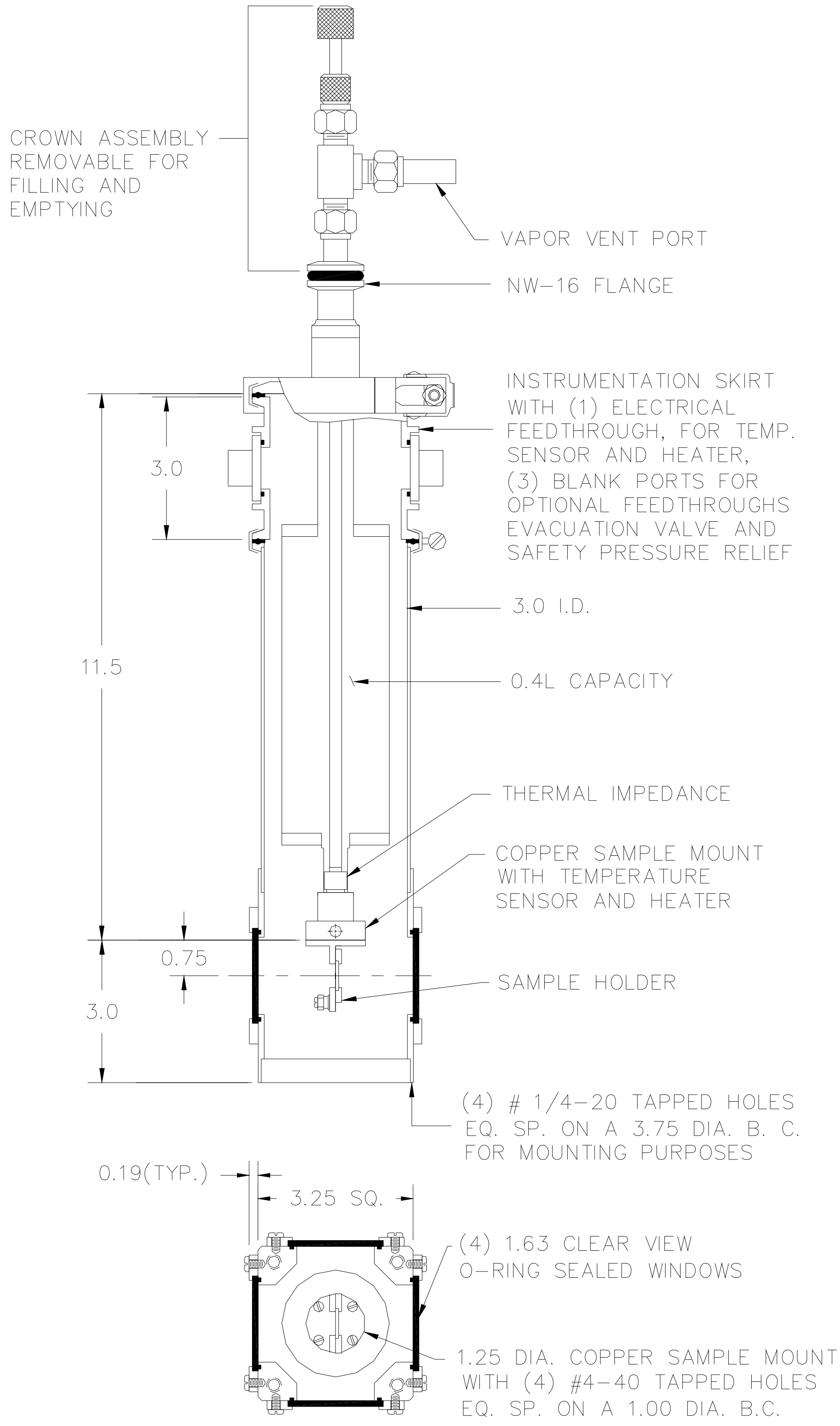

FIGURE 3.1 THERMAL IMPEDANCE CRYOSTAT

B. Exchange Gas Cryostats

Another method for varying the temperature of the sample mount, is by indirectly connecting it to the cooling source through a gas (usually helium) at sub-atmospheric pressure. As the pressure of the gas is varied between 760 torr and 10^{-4} torr, the thermal link between the cooling source and the sample mount will change by several orders of magnitude. This method is thus used effectively for making variable temperature cryostats in a variety of configurations, with the sample either in contact with the exchange gas, or on a cold finger in vacuum (with the cold finger contacting the exchange gas). Such cryostat inserts can be introduced into the cryogen reservoir of an open neck dewar, built into a detachable tail dewar or incorporated into a closed cycle refrigerator (discussed later). The exchange gas mechanism forms the basis for the Janis VT system for temperature variation. A few examples of such systems will be discussed in what follows.

1. Exchange Gas Insert for Open Neck Dewars

Figure 3.2 shows an insert that is introduced into the helium reservoir of an open neck dewar (only the liquid helium reservoir is shown). The insert consists of the stainless steel sample tube A, with its lower portion made from copper, to present an isothermal surface around the sample. The sample mount is supported by a thin walled stainless steel tube from the top of the cryostat (sample positioner). The sample tube is evacuated through the exchange gas valve prior to cooldown, then back filled with helium gas. A more sophisticated 3 valve assembly can be installed (instead of the sample exchange gas valve) in order to facilitate the evacuation and backfilling procedure. A pressure gauge in the same region monitors the pressure of the helium exchange gas in the sample tube.

After cooldown, the pressure of the exchange gas is usually adjusted to about 1 to 100 mtorr, depending on the heat load and the lowest temperature desired. Higher pressures result in good thermal linkage between the sample and the cryogen, which is desirable at low temperatures. The heater located at the sample mount, is used to raise the temperature of the sample that is thermally anchored to the sample mount. A temperature sensor is usually installed in the same region to monitor the temperature of the sample. For operation above 77 K, liquid nitrogen can replace the liquid helium in the main reservoir. In general, lower exchange gas pressures should be used at higher temperatures (77 K with liquid helium, or 300 K with liquid nitrogen) in order to decrease the thermal link between the sample and the cryogen. This will result in less heat being needed to raise the temperature of the sample, and therefore less heat absorbed by the reservoir, resulting in a lower cryogen evaporation rate.

Temperatures below the cryogen boiling point may be obtained by reducing the pressure on the main reservoir, while increasing the exchange gas pressure. In some cases, one may have to transfer liquid into the sample tube (or condense liquid by over pressurizing the sample tube), in order to reach 4.2 K or 77 K with liquid helium or nitrogen, respectively. Increasing the exchange gas pressure does have a disadvantage in that it also increases the conductive heat load from room temperature into the main reservoir. In general, this method of temperature variation works quite well, and results in excellent temperature stability. Its main disadvantage is that the heat introduced at the sample is absorbed by the liquid cryogen, and evaporates a certain amount as determined by the latent heat of the cryogen. Thus, no use is made of the enthalpy or cooling power of the cold helium (or nitrogen) vapor as it warms up to room temperature. Also, the insert shown in Figure 3.2 requires good thermal anchoring between the sample and its holder, since the sample is cooled by the exchange gas, but is heated through its contact with the sample mount. A more complicated (two wall) insert can be set up with heating introduced to the lower (copper) section of the sample tube, in order to heat the exchange gas itself. The exchange gas then heats both sample and sample mount. Such an arrangement eliminates the thermal interface between the sample and the sample mount, and results in easier determination of the sample's temperature.

The top loading design of this cryostat allows sample interchange while the dewar is cold. This is accomplished by bringing the pressure in the sample tube up to atmospheric, then quickly removing the sample mount and covering the top entrance of the sample tube to prevent any air or moisture from entering it.

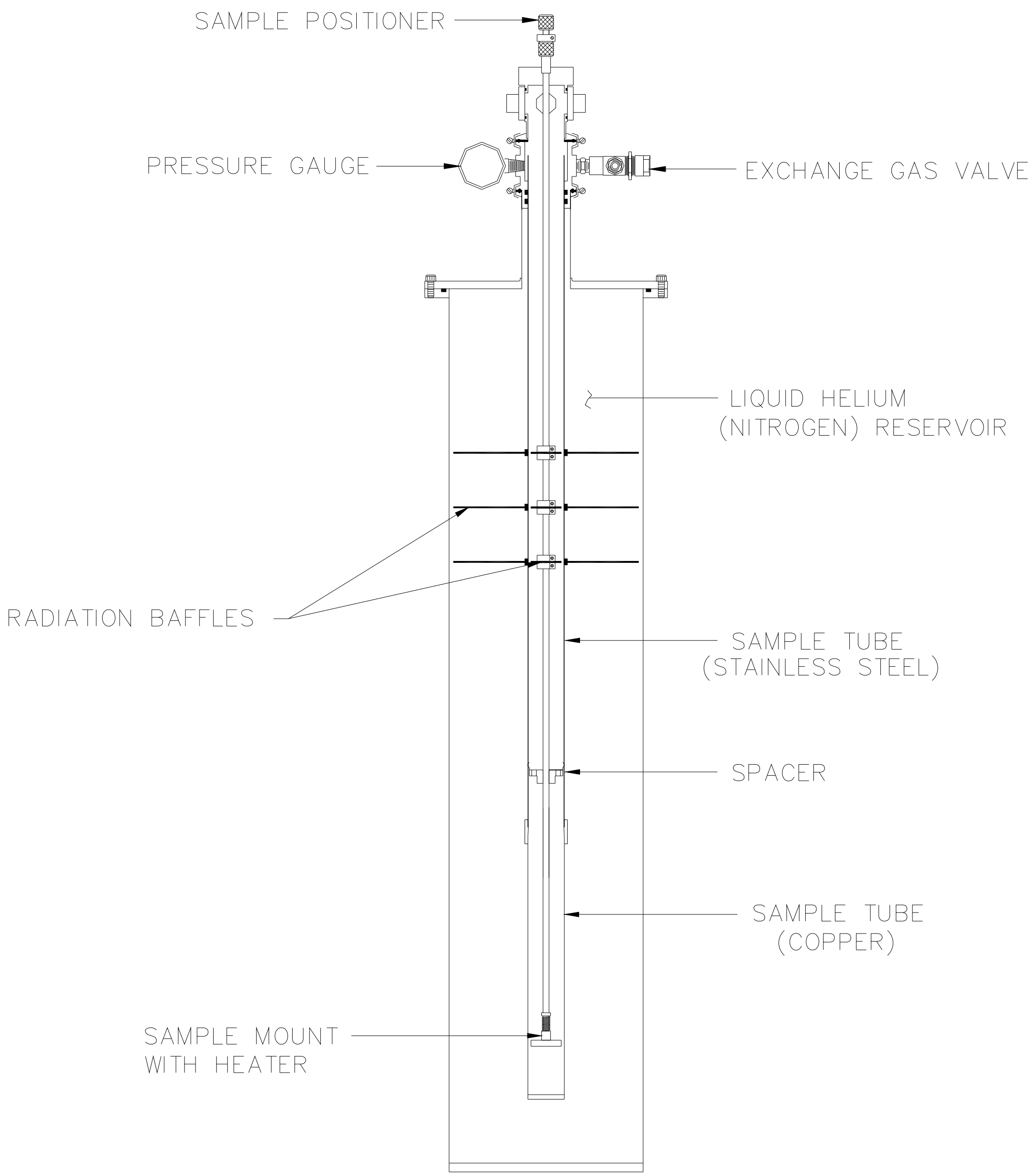

FIGURE 3.2 EXCHANGE GAS INSERT

2. Exchange Gas Inserts for Detachable Tail Dewars

Using the same concept for isolating and connecting the sample to the cooling source, through an exchange gas in a detachable tail dewar, one obtains several useful configurations of variable temperature cryostats. Two such examples are discussed in what follows, showing configurations that place the sample inside the exchange gas column or on a cold finger (in vacuum). Both examples show optical access to the sample, but if no optical access is required, the tail insert can be simplified by eliminating the windows. Non-optical inserts are obviously more compact, and the tail's dimensions can generally be optimized for tight fitting experiments (such as fitting in the pole gap of an electromagnet).

i. Sample in Exchange Gas

Figure 3.3 shows a detachable tail dewar with an exchange gas tail insert, placing the sample in contact with the exchange gas and providing optical access to the sample. The tails are attached to the bottom flanges of the dewar, with the innermost tail permanently attached to the helium reservoir bottom flange. This tail becomes the sample tube, which extends to the top of the cryostat. It contains the exchange gas, with the sample loaded through the top of the cryostat. The lower portion of the sample tube is usually made from copper, part of which lies inside the helium reservoir, while the lowest part is surrounded by the dewar vacuum. This configuration maintains the lower portion close to liquid helium temperature (or liquid nitrogen, in nitrogen dewars), and the sample is located in this region. The upper part of the sample tube is made from stainless steel to reduce the conductive heat load from room temperature. The pressure of the exchange gas determines the thermal link between the sample and the cryogen, in exactly the same manner as the previous cryostat insert. A radiation shield and an outer tail with windows surround the innermost tail, in the same manner as the immersion tail units (Figure 2.6). Installation of radiation shield windows will reduce the heat load on the sample tube, and thus reduce the helium consumption and the lowest temperature that can be reached at the sample. The sample can also be immersed in liquid helium if the liquid is transferred directly into the sample tube or condensed by over pressurizing helium gas in the sample tube. The latter option boils off a significant amount of helium from the main reservoir since the warm helium gas is cooled and condensed by dumping this energy into the liquid helium in the main reservoir.

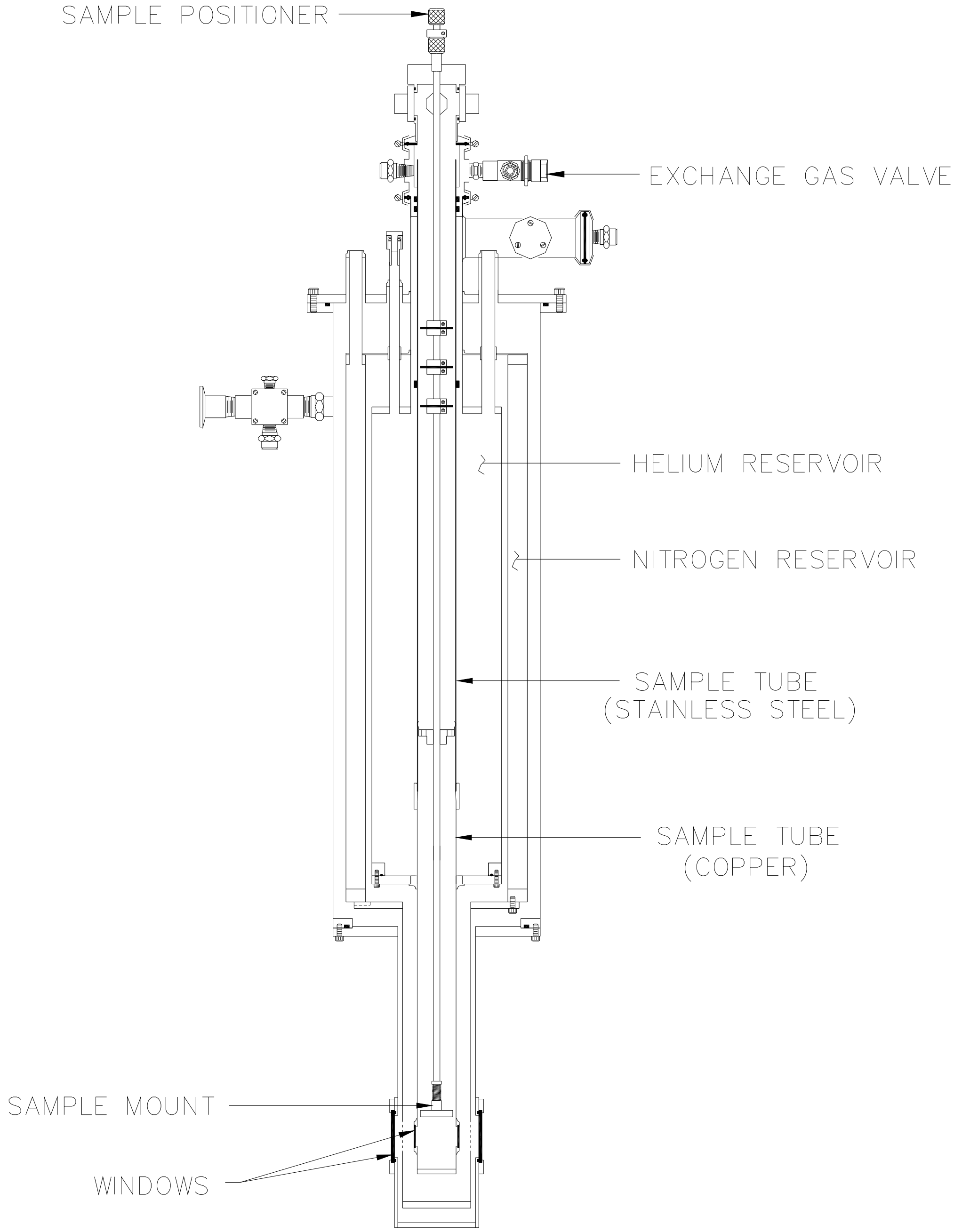

FIGURE 3.3 OPTICAL EXCHANGE GAS CRYOSTAT

ii. Sample in Vacuum

Figure 3.4 shows a set of insert tails, which provides a cold finger for attaching a sample in (the dewar) vacuum. The cold finger is isolated from the helium reservoir by a thin wall stainless steel tube, which also acts as the exchange gas space. The exchange gas inside this tube forms the (variable) thermal link between the cryogen and the cold finger. A (copper) heat exchanger is connected to the cold finger to increase the link between the exchange gas and the cold finger, and thus increase the cooling power at the lowest temperature. A heater, located at the cold finger, is used to raise and control the temperature of a sample attached to the sample mount.

Exchange gas cryostats work quite well with either liquid helium or liquid nitrogen dewars. In a helium dewar, the helium reservoir may be filled with liquid nitrogen whenever temperatures above liquid nitrogen are required. Helium gas is usually used as the exchange gas with either liquid helium or liquid nitrogen, because of its high thermal conductivity, but one could use nitrogen gas when liquid nitrogen is used as the cryogen in the main reservoir.

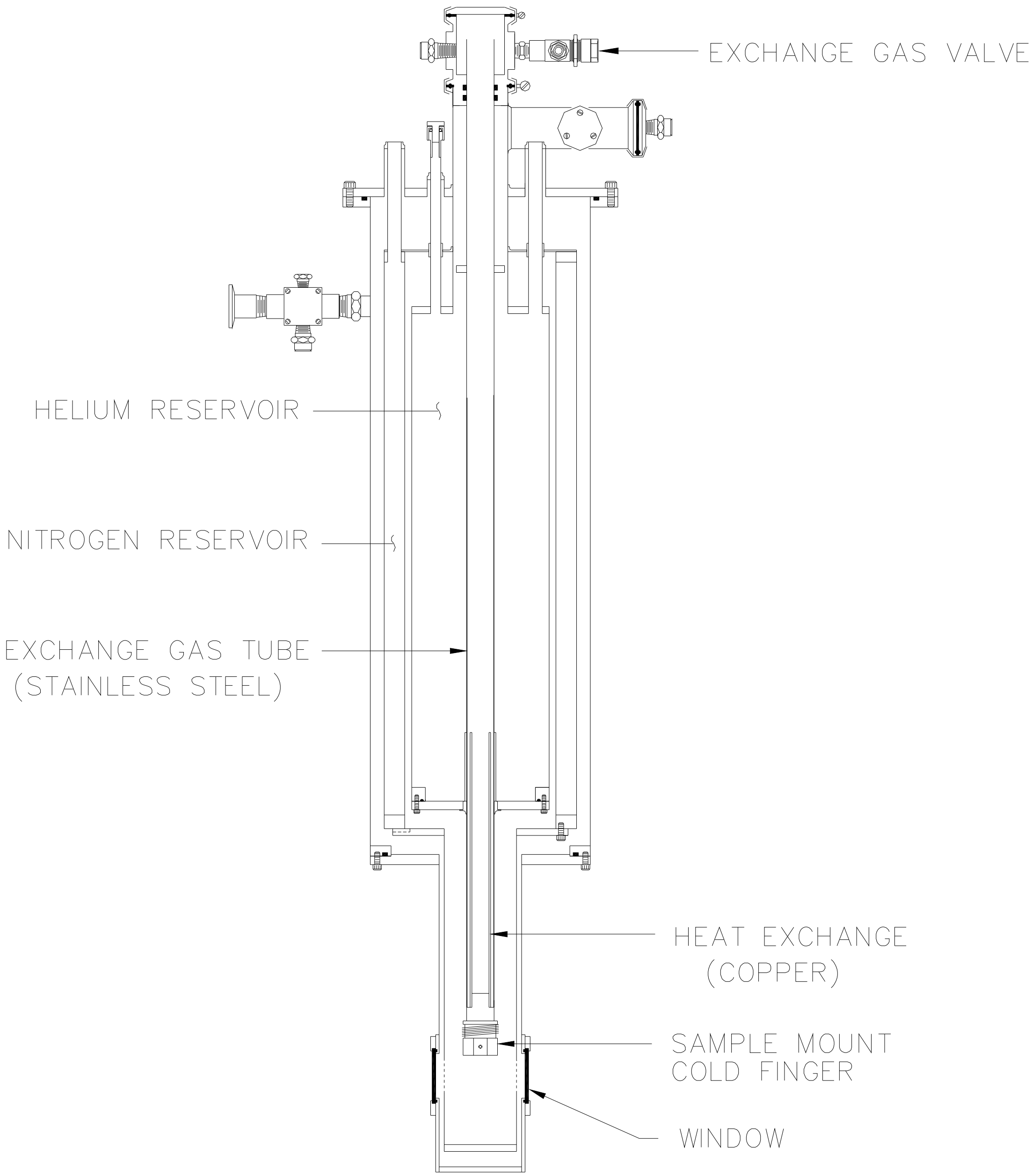

FIGURE 3.4 SAMPLE IN VACUUM EXCHANGE GAS CRYOSTAT

C. Continuous Flow Cryostats

As discussed above, temperature variation can be obtained by channeling a small flow of cryogen continuously to the sample region, and controlling its temperature. The cryogen may be contained in the main reservoir of the dewar (such as the Janis SuperVariTemp cryostats), or could be continuously transferred from a storage dewar (such as the Janis SuperTran cryostats). Since the cryogen has to be vaporized before raising its temperature, these cryostats are usually designed to operate with liquid helium. This is especially the case when the sample is located in the flowing vapor. In some instances the same cryostat may also be used with liquid nitrogen, with or without minor modifications. Several examples of such cryostats will be discussed in what follows.

1. Flow Cryostats with Detachable Tail Dewars

Figure 3.5 shows a detachable tail dewar with a cryostat which channels liquid helium from the main reservoir, to a sample chamber which is isolated from the helium reservoir. This is done through a helium valve, located at the bottom of the helium reservoir, connected to a heat exchanger/vaporizer at the bottom of the sample tube via a small capillary tube. The capillary tube and lower portion of the sample tube are located in the dewar vacuum, and surrounded by the radiation shield and outer tails. The isolation tube diameter is usually 0.25" larger than the sample tube diameter, with the dewar vacuum extending to the region between those two tubes. The helium valve is usually opened slightly to allow a small flow of liquid helium from the helium reservoir into the heat exchanger at the bottom of the sample tube. At that point, heat may be added to vaporize the liquid helium, and raise its temperature (usually up to 300 K) in order to change the temperature of the sample attached at the sample mount. The warm helium vapor flows up the sample tube and escapes out of a vent port at the top of the cryostat. Since the sample tube is isolated from the helium reservoir, the warm vapor flowing in the sample tube does not heat up the helium reservoir and cause additional helium boil off from the reservoir. Furthermore, the temperature of the helium vapor can also be reduced by reducing the pressure at the sample tube, without affecting the main reservoir. Thus, flowing vapor at 1.8 K is easily obtained with a medium size (typically 10-17 ft^3/min or 5 – 9 lit/sec) mechanical pump. The sample tube can also be quickly filled with liquid helium by opening the needle valve, and if desired, the pressure can be reduced to reduce the temperature of the liquid helium (to 1.5 K or less). The helium reservoir is usually slightly pressurized (to about 1 - 2 psig) by sealing all entrances to the reservoir except one that contains a pressure relief valve set at 2 psig. This arrangement provides a constant pressure gradient to drive the liquid helium through the capillary tube into the sample tube.

One of the major advantages of this cryostat is that the sample need not be heated by a heater at the sample mount, and it does not have to be thermally anchored to its mount. This is due to the fact that the cooling (and heating) of the sample is achieved by controlling the temperature of the flowing helium vapor, which makes direct contact with the sample. Attaching a temperature sensor close to the sample will now give a very accurate measure of the sample's temperature, since both sample and sensor are being cooled (or heated) by the same flowing vapor, at the same location. Thus one usually installs a control sensor at the vaporizer, and a second sensor at the sample mount for precise measurement of the sample's temperature. Such an arrangement is quite easily handled by most commercially available automatic temperature controllers. A typical temperature sweep between 4.2 K and room temperature takes about 30 to 45 minutes, as long as the mass of the heat exchanger remains reasonable small.

Another advantage of this system is that operation below 4.2 K is done without have to reduce the pressure on the main reservoir, which usually evaporates about 40% of the liquid helium by the time it is cooled to 2.2 K. This is particularly important if a superconducting magnet is located in the helium reservoir. Furthermore, throttling the valve and reducing the pressure in the sample tube, results in a stream of 1.8 K helium flowing past the sample. This mode presents the least amount of interference in optical experiments where the presence of superfluid (or normal) helium cannot be tolerated.

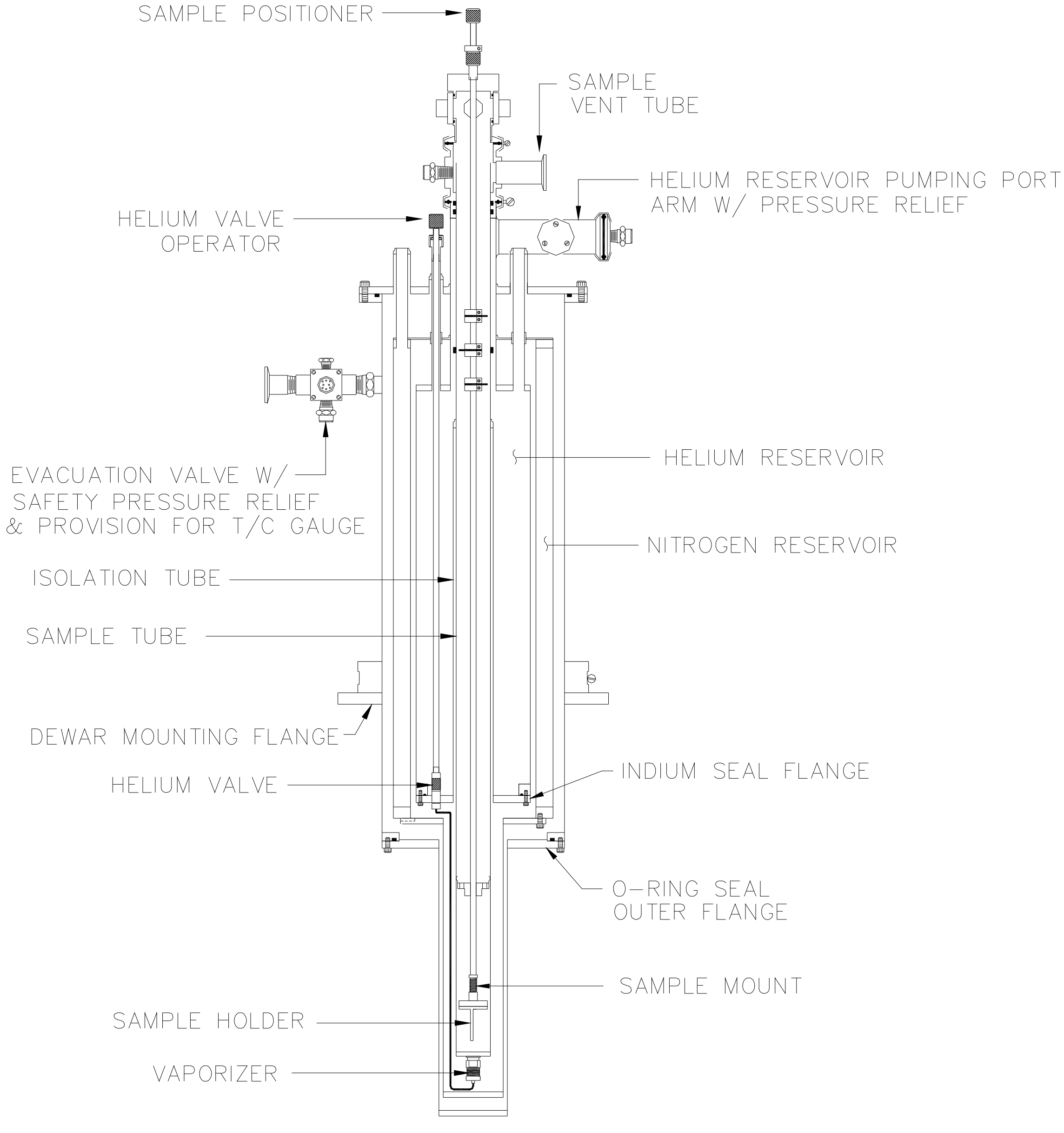

FIGURE 3.5 FLOW CRYOSTAT WITH DETACHABLE TAIL DEWAR

This variable temperature system is the basis for the Janis SuperVariTemp cryostat with sample in flowing helium vapor. Optical access to the sample is provided by adding outer and inner windows to the tail region, with the final configuration resembling the tail region of Figure 3.3. This system is also capable of dissipating large amounts of heat that may be generated at the sample (due to laser beams or other sources), since the helium valve and pressure gradient can be adjusted to provide a large flow (a few liters of liquid helium/hr) into the sample tube.

The sample holder is top loaded into the sample tube and its position adjusted by the sample positioner at the top of the cryostat. A simple 0.25" tube passing through an O-ring compression seal allows rotation as well as translation of the sample mount about the dewar axis. Above 4.2 K, with the pressure in the sample tube at 1 atmosphere, changing samples is simple, since the flow of helium (coming in through the bottom of the sample tube) prevents air from easily entering the sample chamber.

By modifying the design of the heat exchanger (vaporizer) in this insert one can also provide two useful configurations. The first one offers <u>bottom</u> <u>optical</u> <u>access</u> by making a toroidal vaporizer, and adding windows at the bottom of the outer and radiation shield tails. One slight disadvantage of this arrangement is that the inner bottom window tends to get dirty from any foreign material that enters the sample tube, and settles at the bottom. The other modification of the heat exchanger is to machine it with a flat mounting surface (with dead tapped holes) so that it acts as a cold finger for attaching samples in vacuum. The capillary tube can now enter through the side (as opposed to the bottom) of the heat exchanger, into the venting tube (what used to be the sample tube) in order not to interfere with sample mounting surface of the cold finger. The sample is located in the dewar vacuum, and can only be replaced by breaking the dewar vacuum, with the dewar at room temperature.

The temperature stability (at the sample) in the range between 4.2 K and 25 K is usually better than ±0.1 K, using an appropriate automatic temperature controller. The stability is inherently limited due to the two phase mixture (liquid and vapor) of helium flowing past the sample. Should a better stability be required, a simple exchange gas insert, with a bottom isothermal section (made from copper) can be inserted through the top of the cryostat, inside the sample tube. This insert now becomes the new sample tube, and results in a much better temperature stability at the sample. Such an insert is also very useful for equipment that may be disturbed by any gas flowing across the sample, such as Faraday Balance experiments. With the sample located in a static exchange gas, any disturbance due to flowing helium vapor is now eliminated. It is also very useful when heating the sample above 80 K since it can reduce the radiational heat load into the main reservoir and the overall helium consumption of the cryostat.

Whereas the design of the vaporizer allows vaporization of a reasonable flow (50 to 500 cc/hr) of liquid helium entering the sample tube, attempting to do this with liquid nitrogen is usually unsuccessful. This is due to the relatively large heat of vaporization of liquid nitrogen. When liquid nitrogen must be used, a modified design for the heat exchanger and bottom of the sample tube can be used to improve the heat exchange between the heater and the liquid nitrogen. Such a design can then be used for operating the system above liquid nitrogen temperature, using a limited flow of liquid nitrogen into the heat exchanger.

2. Flow Cryostat Inserts for Open Neck Dewars

Using the concept for the cryostat described above (C-1), a variable temperature cryostat insert can be made for use with open neck research or storage dewars. Such a cryostat would have its own independent vacuum jacket to isolate the sample tube from the main reservoir, and a helium valve to draw the liquid into the sample tube in a controlled manner. Such inserts are very useful for introduction into the bore of a superconducting magnet located in a research dewar (Figure 3.6). The magnet has its own support structure, and the insert can be removed without disturbing the magnet.

The insert itself may also be used to support the magnet and eliminate the independent magnet support. The insert's isolation (bore) tube diameter is determined by the magnet, with the sample tube's diameter approximately 0.5" smaller than the isolation tube. The space between the sample and isolation tubes is evacuated, and the capillary tube carrying the helium from the main reservoir into the sample chamber lies in that vacuum space.

Access to the capillary tube and vaporizer (with its associated heater and control sensor) is through a demountable indium (or solder) seal. The helium reservoir should be pressurized (with a simple pressure relief valve) in order to maintain the slight over-pressure needed to drive the helium through the needle valve, capillary tube, and heat exchanger into the sample tube. The most convenient place for such a valve is at the (common) exhaust of any vapor cooled high current magnet leads, since this maintains the necessary flow of cold helium vapor through the leads.

Temperature variation is done in precisely the same manner as the detachable tail insert cryostat, and covers the range of about 1.8 K to 300 K in flowing vapor, or down to 1.5 K in superfluid helium. Once again, operation below 4.2 K is achieved without pumping on the main reservoir, which contains the superconducting coil.

For systems that need to operate with liquid helium, but for log periods of time above 80 K, one can include an additional exchange gas type insert into the sample tube. This provides a smaller sample tube in a static gas environment, which allows heating the sample at the sample mount to temperatures above 80 K, without increasing the helium consumption for the main reservoir. Thus when operating at the higher temperature range, one can shift the temperature control to the heater at the sample mount, while maintaining the temperature of the vaporizer between 5 K and 30 K. This usually requires the exchange gas pressure to be close to 1 milltorr, and reduces the amount of heat needed to raise the temperature of the sample mount. Such systems can be operated up to higher temperatures (up to 700 K – 800 K) if one uses an appropriate sensor (such as a type E thermocouple) and removes the typical Cernox sensor that is used in the temperature range between 1.5 K and 300 K. This would also require a special heater at the sample mount to withstand (and reach) the higher temperatures. The magnet and the main helium reservoir will not sense the high temperature of the sample mount and can very often operate with the same helium consumption from that reservoir.

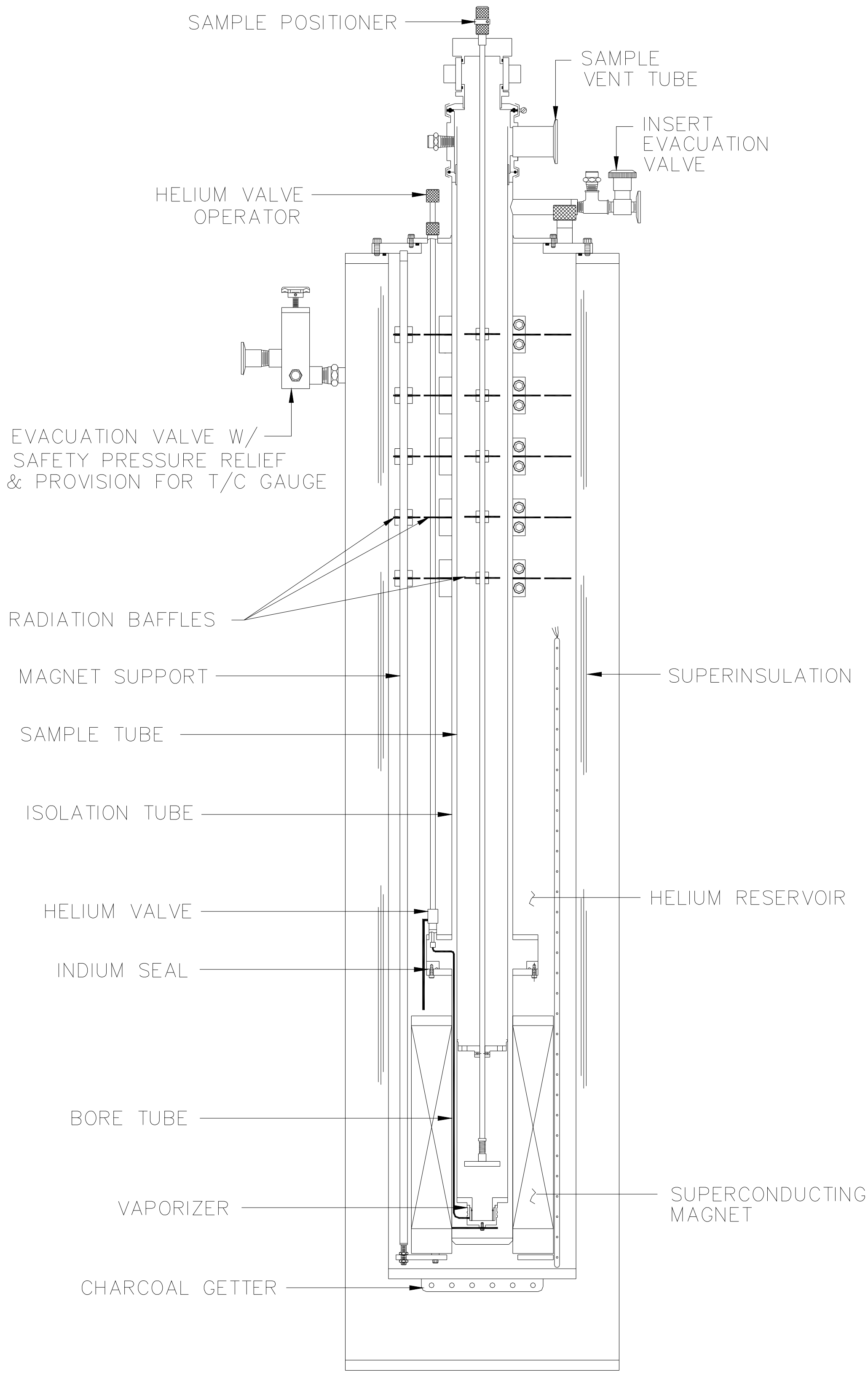

FIGURE 3.6 FLOW CRYOSTAT INSERT FOR SUPERCONDUCTING MAGNET

3. Continuous Transfer Cryostats

In many cases it is preferable to eliminate the research dewar with its own helium (and/or nitrogen) reservoir, because of lack of space or a desire for a more portable cryostat. For such applications, an efficient vacuum insulated transfer line, with a flexible section, can be used to carry the cryogen from a storage dewar to a small cryostat. Figure 3.7 shows an example of such a transfer line, with a flow control valve located at the bottom of the storage dewar leg. This arrangement is preferable to an "in line" valve since it presents a lower heat input into the flowing cryogen.

The outer line has a flexible section (5-10 ft. or 1.5 to 3 meters long) to provide more maneuverability, while the inner line may be made from 0.063 – 0.13" or 1.6 to 3 mm diameter stainless steel tubing and is surrounded by MLI. Properly designed and located spacers keep the inner line concentric within the outer flexible line. The storage dewar leg is usually made to match typical storage dewars, with a 0.5" or 12 mm diameter and a 40"- 60" or 1.0 – 1.5 meter length. The entire line is vacuum insulated with an evacuation valve available for maintaining a good vacuum (10^{-5} torr), plus a safety pressure relief valve to guard against cold internal leaks. With careful design and manufacturing techniques, the heat load on the inner line can be reduced to less than 300 milliwatts for a typical unit (6 ft. or 183 cm flexible section, 0.063" or 1.6 mm inner line, 48" or 122 cm storage dewar leg and 20" or 51 cm cryostat leg). This line can be used to continuously transfer the cryogen into the cryostat, so it is important to have it as efficient as possible.

Two types of configurations are used with this transfer line. One unit provides a cold finger with the sample in vacuum, while the other unit places the sample in the path of flowing vapor – as discussed below.

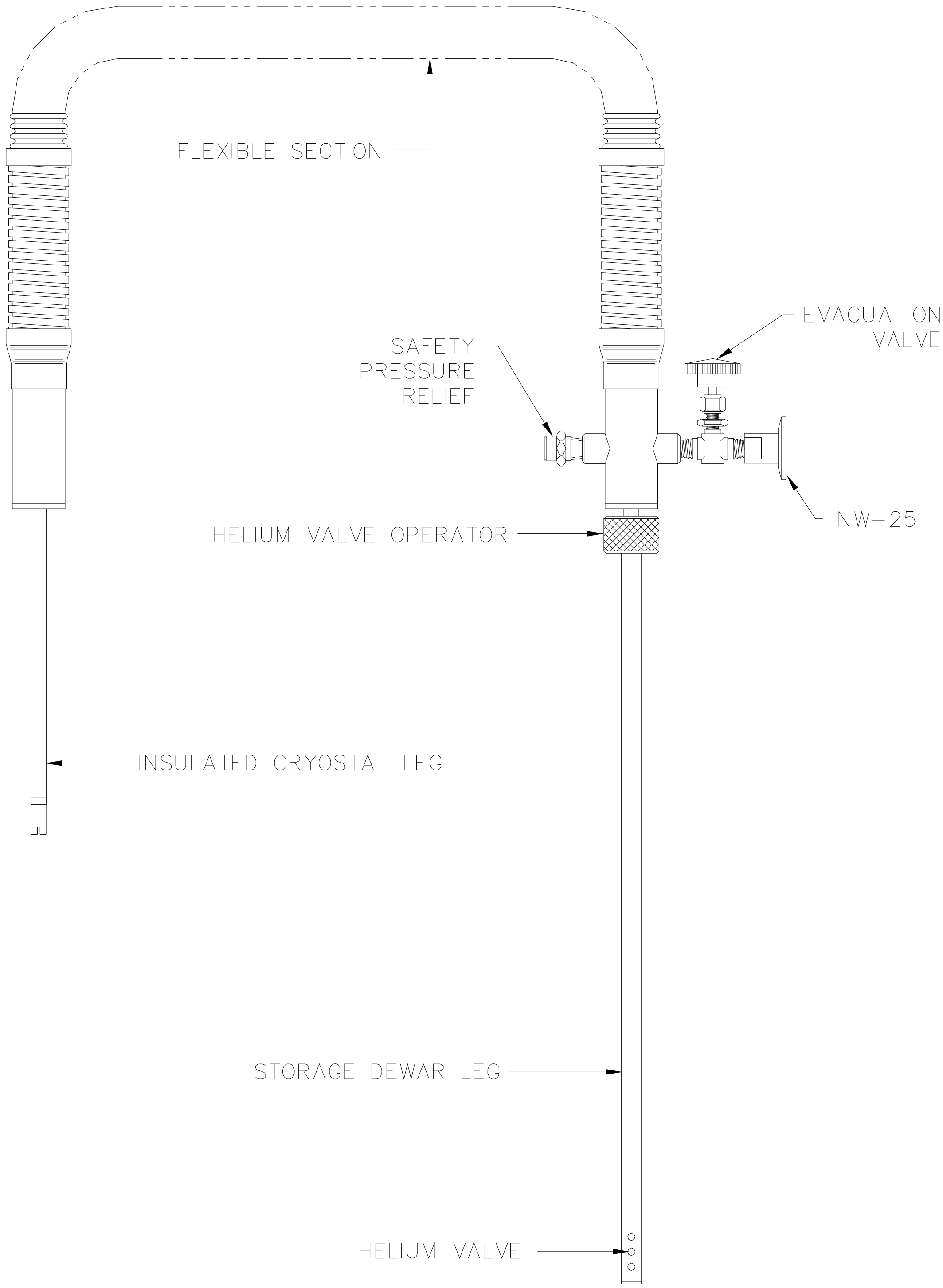

FIGURE 3.7 FLEXIBLE TRANSFER LINE FOR CONTINUOUS TRANSFER

i. Sample in Vacuum

Figure 3.8 shows a simple cold finger cryostat that mates with the transfer line shown in Figure 3.7. The leg of the transfer line fits inside an O-ring compression seal at the top of the cryostat, and delivers the cryogen to the inside of the cold finger. The cryogen exits through a concentric stainless steel tube (isolation tube) and cools a thermal anchor to which a radiation shield is attached. The cryogen eventually exits at a vent port away from the detachable seal for the vacuum jacket.

The outer (vacuum) jacket is usually sealed by a quick removable elastomer seal to allow easy access to the sample holder. The radiation shield, which is cooled by the escaping vapor, may be bolted to the thermal anchor, and is required for use with liquid helium. The cryostat can also be used with liquid nitrogen, as long as the flow control valve can be adjusted to significantly reduce the flow. A heater located at the cold finger will allow temperature variation above 4.2 K (liquid helium) or 77 K (liquid nitrogen). Optical access to the sample is easily achieved by adding windows to the vacuum jacket, and holes (or windows) to the radiation shield. Evacuation and pressure relief valves, as well as vacuum tight electrical feedthroughs are usually located above the joint of the removable vacuum jacket, in order to provide the necessary vacuum and wiring access to the cold finger.

Although such cryostats may be operated in any orientation, the vertical position requires the least amount of cryogen at any specific temperature. Temperatures above 4.2 K can be obtained by reducing the liquid helium flow, or by adding heat at the cold finger wound heater. Temperatures below 4.2 K are obtained by either throttling the flow control valve and pumping at the vent port, or by allowing helium to collect on top of the cold finger and reducing the pressure (at the vent port) with the flow valve closed. The latter mode usually results in the lower temperature (approximately 1.4 K). This system forms the basis for the Janis SuperTran-B cryostat.

ii. Sample in Vapor

Figure 3.9 shows a cryostat that can be used with the same transfer line. The transfer line fits (horizontally) into a mating (female) bayonet connection at the top of the cryostat, and the cryogen is channeled through a capillary tube into a heat exchanger/vaporizer at the bottom of the sample tube. The cryostat shown in Figure 3.9 provides optical access through inner (cold sealed) windows and outer (room temperature) windows. The cold vapor exits out of a vent (or pumping) port at the top of the cryostat, and in the process cools a thermal anchor and the radiation shield attached to that anchor. The system operates in a similar manner to the flow cryostat with detachable tail dewars (Janis SuperVariTemp) with the cryogen now continuously transferred from a storage dewar.

The system was originally designed for use with liquid helium as the cryogen, but a newer version can also be used with liquid nitrogen. Operation below 4.2 K (down to 1.8 K) requires filling the sample chamber with liquid helium, closing the flow control valve and reducing the pressure through the sample tube vent port. The flow valve can also be throttled while pumping at the sample tube vent port to operate continuously in a flowing vapor mode at approximately 3 K.

The liquid helium consumption of this system will generally be higher than that of the cold finger system (SuperTran-B) while using the same transfer line. This is probably due to the fact that the helium has to travel through a capillary tube (usually about 12" long), which undergoes at least two 90° bends, with associated heat loads due to radiation and flow turbulence. This usually results in adding approximately 600 milliwatts into the flowing cryogen, resulting in a total system consumption of about 1.5 liter/hr at 4.2 K, but dropping below 0.5 liter/hr above 20 K. With its quick cooldown, its portability and ease of use, this can be a more desirable cryostat to use than a regular dewar in some applications. This system forms the basis for the Janis SuperTran-VP cryostat.

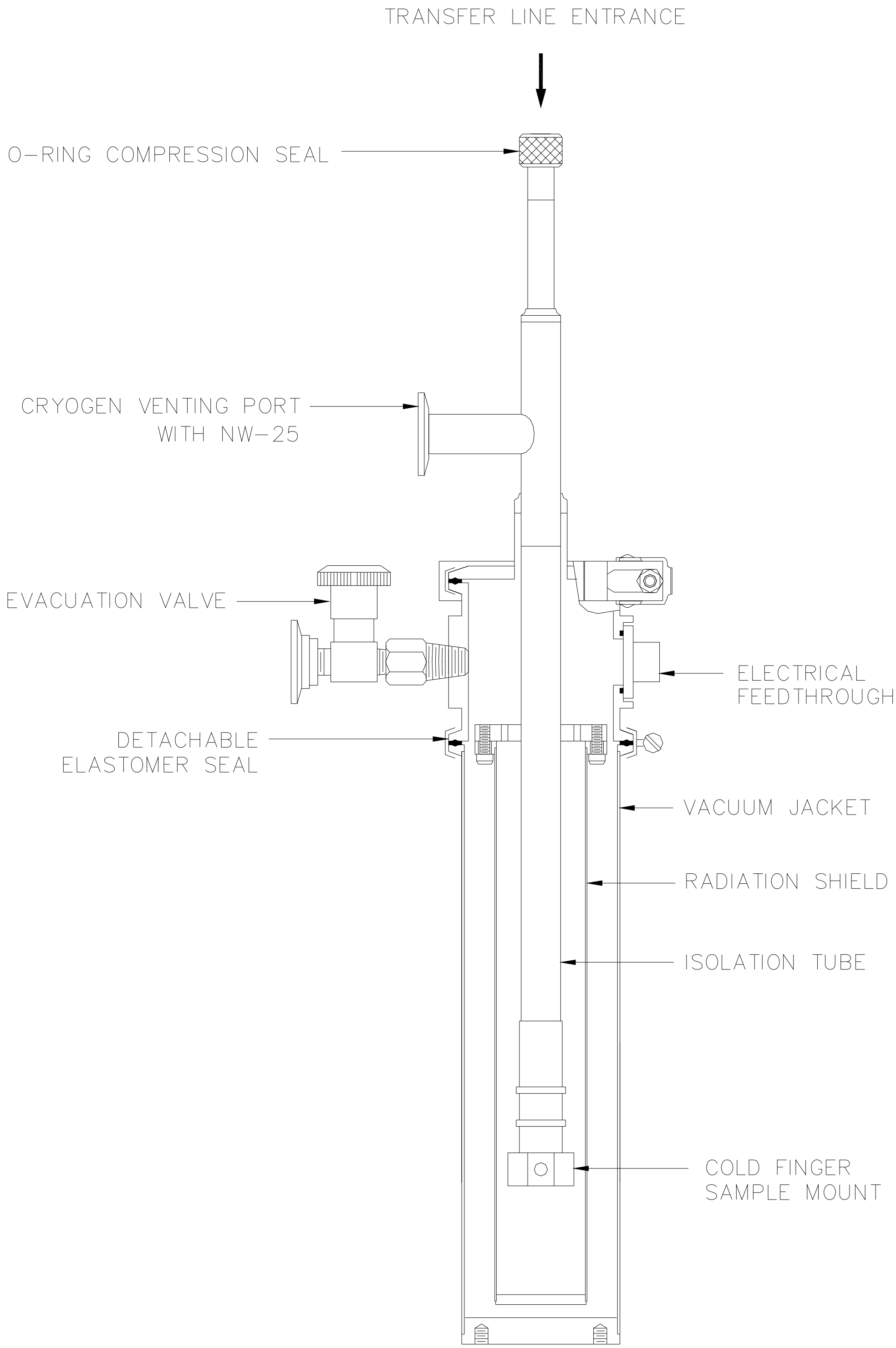

FIGURE 3.8 CONTINUOUS TRANSFER COLD FINGER CRYOSTAT

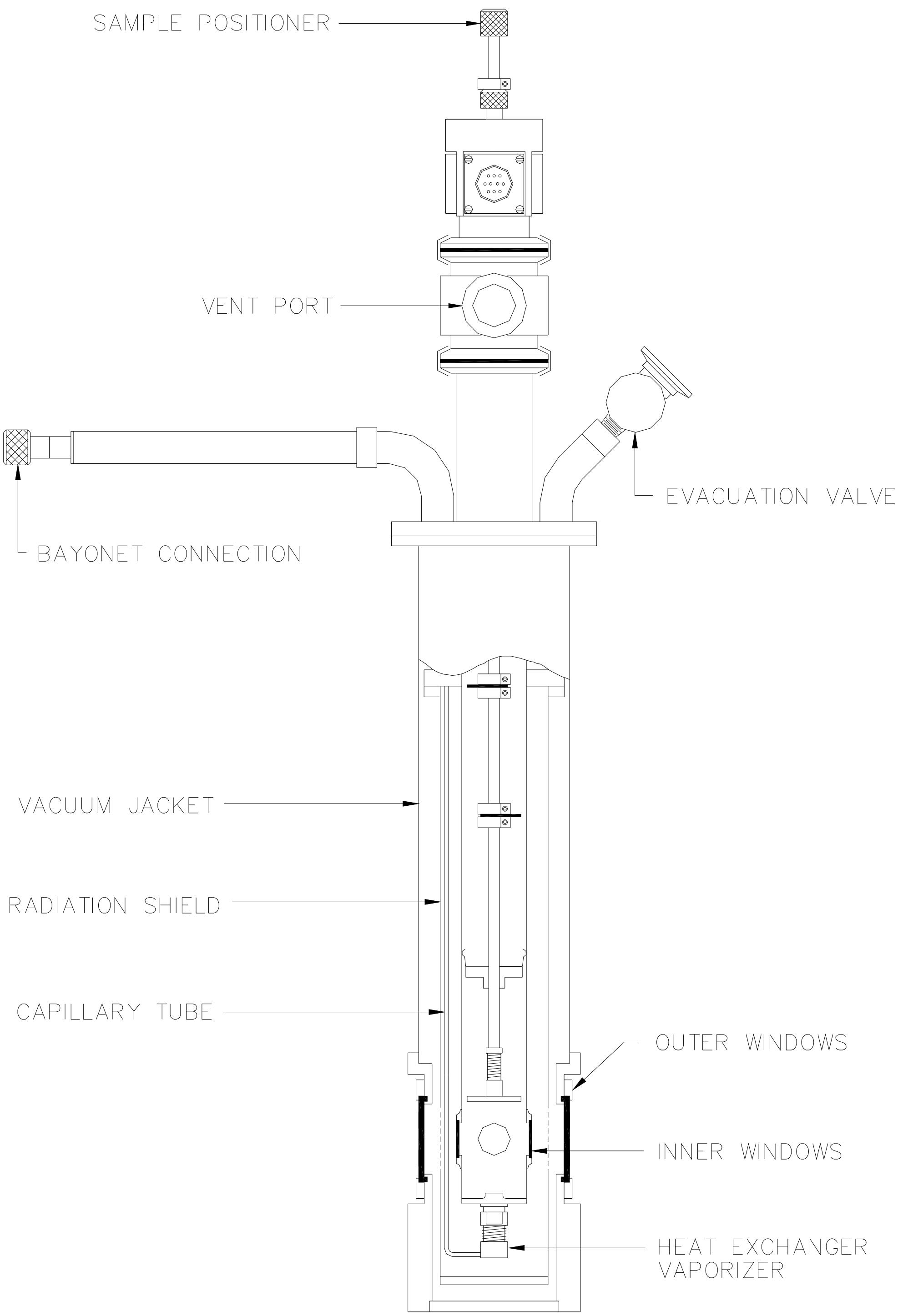

FIGURE 3.9 CONTINUOUS TRANSFER SAMPLE IN VAPOR CRYOSTAT

iii. Special Microscopy Systems

Special systems have been developed for microscopy experiments, which require a very small distance between a microscope objective and the sample. These systems are carefully designed to offer rigid stable support for the sample as well as effective de-coupling from the incoming and outgoing cryogen lines. This results in a system with excellent spatial stability with a typical drift of 2 nm / min. These cryostats typically have a short height to fit under standard microscope objectives, and a stable base to support the cryostat rigidly under the microscope. Figure 3.10 shows an example of such a cryostat, with separate helium entry and vent lines. This cryostat operates with the same type of high efficiency transfer lines that are used for the other continuous transfer cryostat.

Access to the sample is usually through the top cover of the vacuum jacket. This cover will have a small window that is located very close to the sample (typically between 3 and 10 mm). These cryostats usually have a bottom window to allow transmission measurements and are also supplied with extended top vacuum jackets and radiation shields for insertion into the bore of a superconducting magnet, an electromagnet or permanent magnets. They are also supplied with X-Y-Z nano-positioning stages inside the cryostat for precise maneuvering of the cold finger and attached sample.

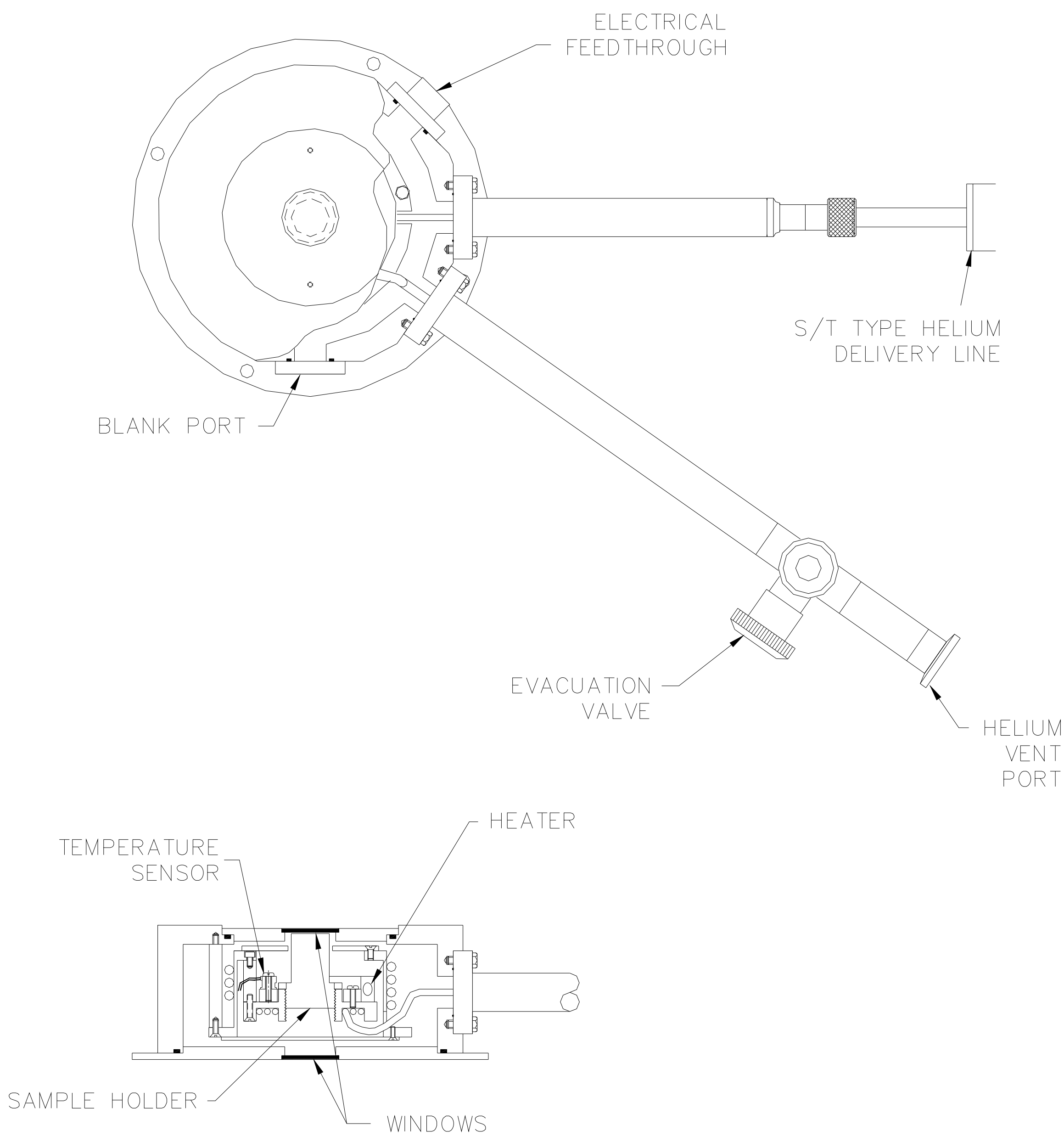

FIGURE 3.10 CONTINUOUS TRANSFER MICROSCOPY CRYOSTAT

4. SUPERCONDUCTING MAGNET SYSTEMS

The improvement in the manufacturing and production techniques of superconducting wires and coils within the past 40 years, has made them readily available to most research laboratories. The most common materials that are commercially available for superconducting magnets are the type II superconductors NbTi and Nb_3Sn, with the latter having the higher critical field. Since these materials lose their superconductive properties above a certain critical temperature (along with an associated critical field and current density), the magnets wound from these materials are usually immersed in liquid helium during operation. For this purpose, a variety of liquid helium research dewars and cryostats have been designed to contain these magnets and offer temperature variation with optical and/or physical access to the high field region. An example of one such cryostat was shown in Figure 3.6, where the magnet was supported from the top flange, and an independent variable temperature insert providing temperature variation in the high field region. The magnet support system contains radiation baffles (as does the variable temperature insert) to prevent room temperature radiation from reaching the liquid helium through the top of the reservoir. The baffles are cooled by the helium vapor boiling out of the main reservoir, and they also force the vapor to interact more intimately with the neck of the helium reservoir. This further reduces the conductive heat load down the neck, as was mentioned earlier.

For magnetic fields of 5-9 Tesla (at 4.2 K), twisted multifilament NbTi (embedded in copper) wires are used to eliminate flux jumping and associated heat generation. Typically these magnets run at currents that range between 50 and 100 Amperes for laboratory magnets (up to about 4" bore). These same magnets may also reach fields of up to 11 or 12 Tesla when operated at pumped helium temperatures (2.2 K), where the values of the critical current and magnetic field are larger. Nb_3Sn magnets are used for the higher fields (12 to 20 Tesla), but these magnets tend to be significantly more expensive. With such (relatively) large currents, special vapor cooled leads are usually used in the neck region, with a good portion of the escaping helium vapor forced to pass through these leads. This flow reduces the temperature, the resistance, and the Joule heating generated due to the large current passing through these leads. In addition, the helium vapor also intercepts the conductive heat load coming from room temperature down the leads. The vapor cooled leads are then connected (either permanently or through a detachable joint) to low resistivity copper and superconducting cables which run to the magnet at the bottom of the helium reservoir. Detachable joints are used when one wants the option of eliminating the conductive heat load down the magnet leads, when there is no electric current passing through the leads. More recently other designs for vapor cooled leads have been developed for currents under 100 A, which offer heat loads that are comparable to the detachable leads, and thus in many cases they are preferable to use. This is due to the fact that the detachable joints introduce a significant electrical resistance at the joint, which generates additional Joule heating when current is passing through the leads.

In many instances it is desirable to run a superconducting magnet in a closed (superconducting) loop. This is done through a persistent current switch, which consists of a small length of superconducting wire shorting the magnet terminals, with a small heater wire wrapped around it. The heater is capable of raising the temperature of the switch, and thus forcing it into its normal (resistive) state. This enables the power supply to send current through the (superconducting) magnet for charging it up to the rated field (or any lower field). At that point the persistent switch heater may be turned off to place the magnet in the persistent mode, while the current in the leads, which comes from the power supply to the magnet, may be reduced to zero. This eliminates any Joule heating in the leads and leaves a very stable current (and magnetic field) in the magnet. Typical decay rates of commercially available magnets are about 10 to 25 ppm/hr. When the field needs to be changed, the power supply can be turned back on, in order to re-establish the same current through the leads, and finally the persistent switch heater is turned back on. The power supply now regains control of the magnet, and the necessary changes in the current can be made. The persistent switch heater usually generates about 150 milliwatts, which results in an additional helium boil-off of 210 cc/hr.

Magnet power supplies are usually provided with a sweep generator to charge the magnet at various rates, and have special protection (usually diodes) to protect against voltage mismatch with the magnet as may be encountered when the magnet is switched out of its persistent mode, or if the magnet should accidentally turn "normal" and discharge. Most magnet power supplies are also designed with an energy absorbing circuit, which allows relatively quick discharges for magnets with large inductance.

In addition to the high current leads for the magnet, the top flange should also contain an electrical feedthrough for wiring the persistent switch heater and voltage taps that run directly to the coil terminals in the helium reservoir. The voltage taps are used to monitor the precise voltage across the magnet, required to charge the magnet at a specific rate. This is determined by the relation.

$$E = L\frac{dI}{dt}$$

where E is the applied voltage at the magnet, L the inductance of the coil and dI/dt the rate of change of the current passing through the coil. The inductance and the charging voltage are usually specified for each individual coil, resulting in a maximum charging rate which should not be exceeded, since it may result in generating too much heat in the coil, which can force it into its normal (resistive) state. This would result in a magnet quench, where all the magnetic stored energy (½ LI^2) will dissipate as Joule heating in the magnet, and absorbed by the liquid helium around the magnet. Other causes of magnet quench would be operating the magnet above its rated current, or allowing the liquid helium level to drop below the magnet, to a level that warms the magnet above its critical temperature. A liquid helium level sensor should always be installed in the helium reservoir to monitor the level of the liquid helium, and make sure it remains at a safe level.

Typical laboratory magnets have inductances of 10 to 20 Henries and operate at maximum currents of 50 to 100 Amperes. The stored energy for these magnets thus ranges between a few thousand to a 100 thousand Joules, which would need to be dissipated very rapidly (few seconds) in case of a magnet quench. The magnets are thus designed to absorb this energy, and the dewar should have a quench relief port to vent the large amount of helium gas that is generated as the magnet dumps this heat into the liquid helium reservoir. The magnet should have protective diodes across its terminals (or a protective resistor circuit) to protect it during such a quench.

The higher field magnets (12 to 17 Tesla) are usually made in a hybrid design, where two or more sections are connected in series (NbTi in the lower field outer region and Nb_3Sn in the high field inner region), and each section would have its own protective circuit. In addition, since the quench can result in very large generated voltages at the windings, special devices are used to reduce the voltage surges within the windings.

The magnet design also takes into account the forces that are generated at the windings when the magnet is at full field. In straight solenoids, these forces generally result in added tension along the windings, which can easily support the stress. In magnets with "bucking" coils (to reduce the field at a specified location near the center of the magnet) or gradient coils, the structure is designed to support the repulsive forces between the coils, so no wire movement (which may result in a magnet quench) occurs. In split or Helmholtz coils that are required for optical access or radial access to the high field region, the former (or bobbin) holding the coils should also be designed to withstand the attractive force between the two sections, without allowing any significant deflection. Thus, a well designed system should always consider the magnet and cryostat as one unit to suit specific experiments, along with any required automatic control for the temperature, magnetic field or physical measurement in question.

The cryostat should always allow access from the top flange to the bottom of the helium reservoir (below the magnet), either directly or through an initial helium fill line. This allows liquid nitrogen pre-cool, (and removal of all the nitrogen from the helium reservoir) and also allows delivering the liquid helium to the lowest point in the helium reservoir during initial helium transfer. This is very critical since it allows full use of the cooling power of the helium vapor to cool the magnet down from 77 K to 4.2 K. With the helium transfer starting at a slow steady rate, which does not allow liquid helium accumulation before the magnet cools down to 4.2 K, the cooldown usually requires 0.4 liters per kilogram of magnet. A much faster helium transfer could fill the helium reservoir quickly, only to result in a very high evaporation rate which can empty the helium reservoir in the next 30 minutes as the magnet gets cooled using only the low heat of vaporization of liquid helium. Installing a thermometer at the magnet, or monitoring the resistance of the coil as it is being cooled, are helpful for inexperienced users. Any helium refills should deliver the new liquid at a level higher than the level of the existing liquid (above the magnet), in order not to risk evaporating any of the existing liquid (by inserting a warm transfer line leg or initially delivering a mixture of warm gas and liquid). A few examples of typical superconducting magnet cryostat systems will be described in what follows.

A. Variable Temperature Top Loading Systems

Figure 4.1 shows an open neck vapor shielded liquid helium research dewar with a variable temperature insert which supports a superconducting solenoid at its bottom. The insert draws helium from the main (dewar) reservoir, and channels it to the sample tube through the vaporizer at the bottom of the sample tube. This is the same flow cryostat design described earlier, where the flowing helium vapor can be controlled in temperature between 1.8 and 300 K. The evacuated isolation tube encloses the sample tube, and prevents the warm helium vapor traveling up the sample tube from heating up the helium in the main reservoir. It also allows operation below 4.2 K, by simply pumping at the sample tube (as described in Section 3).

This cryostat is very similar to the one shown in Figure 3.6, except that the magnet is now supported from the bottom of the insert, thus eliminating the need for an independent magnet support. A helium level sensor monitors the level to make sure it covers the magnet during operation. Vapor cooled high current leads are usually used with copper/superconducting cables to carry the current to the coil. The magnet may also have a persistent current switch and some thermometer to monitor its cooldown. The diameter of the helium reservoir should be large enough to allow a transfer line leg to fit between the magnet and the wall of the helium reservoir. This allows efficient cooldown from 77 K and also allows easy removal of liquid nitrogen from the helium reservoir after cooldown from room temperature.

The top flange should contain helium fill and vent ports plus a quench relief port (in case of accidental magnet quench), along with an electrical feedthrough to wire the level sensor, magnet voltage taps and any persistent switch heater or magnet thermometer included in the system. The high current magnet vapor cooled leads may either be of the older type counterflow leads with the helium vapor escaping through the inside of the leads, or of the newer type leads (for currents of 100 A or less) which do not require internal helium vapor flow. The first type should typically have their exhaust joined into a single chamber with a pressure relief valve. The newer type will not require such a chamber and the helium can vent out through the standard large pressure relief / quench relief valves that are installed on such magnet systems. These relief valves also serve the purpose of maintaining a small over pressure in the helium reservoir, which is useful when driving liquid helium through the needle valve and capillary tube of any SuperVariTemp insert that in the helium reservoir.

If the magnet is designed to offer a higher magnetic field at 2.2 K, it is convenient to add a Lambda plate between the helium valve of the variable temperature insert and the top of the magnet. Such a refrigerator will have its own helium valve and pumping line, which allows it to cool the liquid helium below its location, to 2.2 K. In such a case it is useful to have a field independent thermometer installed at the magnet to monitor its temperature during operation of the Lambda plate. When the magnet reaches 2.2 K, the current through it can then be increased to its higher value in order to provide a higher field. The liquid helium in the upper section of the reservoir stays at atmospheric pressure, and regular helium transfer and operation of the variable temperature cryostat is maintained.

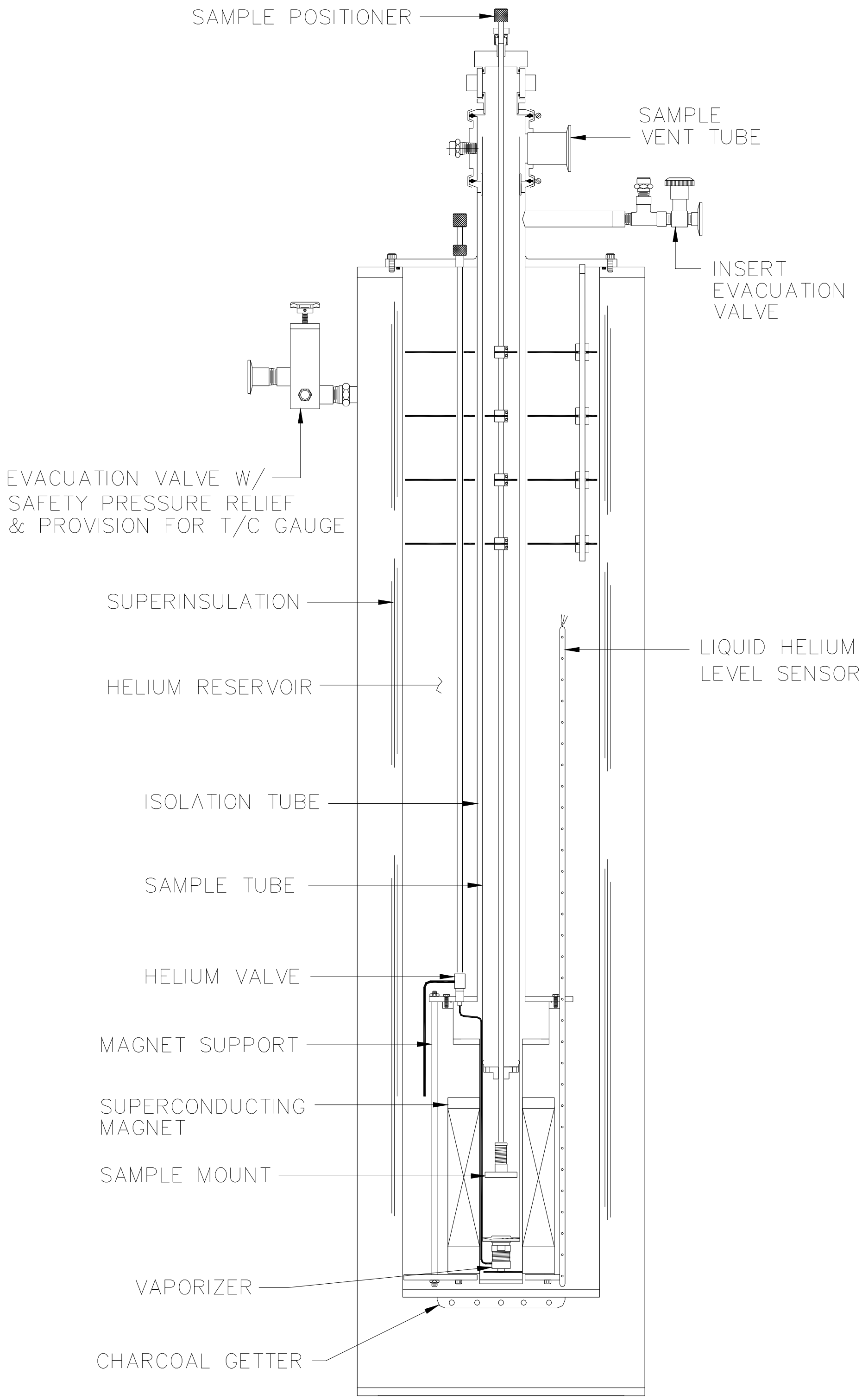

FIGURE 4.1 VARIABLE TEMPERATURE MAGNET SUPPORT CRYOSTAT

B. Optical Access Straight Solenoid Systems

When optical access is required into the high field region of a variable temperature cryostat, the easiest way is to use a detachable tail dewar with a bottom window and a straight solenoid. An example of such a cryostat is shown in Figure 4.2, with the sample mount removed. The heat exchanger (vaporizer) at the bottom of the sample tube has a toroidal shape with a window at its center for bottom optical access into the sample tube. Helium is channeled from the main reservoir through a capillary tube and needle valve, controlled from the top of the dewar. In many cases a detachable joint is made at the bottom of the vapor cooled leads in order to reduce the conductive heat load into the helium reservoir, when no current is passing through these leads. Again as in the previous variable temperature inserts, the sample tube is separated from the helium reservoir by a vacuum (in the isolation tube), in order to maintain low helium consumption as the temperature increases to room temperature. This configuration forms the basis for the Janis OptiMag System.

A simpler exchange gas insert may also be used for this configuration (as well as the configuration described in Section 4.B). Such an exchange gas insert would provide a sample tube (in direct contact with the liquid helium), which fits directly into the bore of the magnet. The sample would be mounted on a copper sample mount in the exchange gas, with a heater for raising its temperature. For a given magnet bore, this offers a slightly (0.25") larger sample space than the previous insert since no isolation tube surrounds the sample tube. Since the magnet must always be immersed in liquid helium (NbTi or Nb_3Sn coils), one has to reduce the exchange gas pressure for operation at temperature above 77 K, in order to maintain a low consumption of liquid helium. Furthermore, the sample should be in very good contact with the sample mount since it is heated indirectly through the sample mount heater. Finally, operation below 4.2 K requires pumping on the entire helium reservoir (containing the magnet), or the inclusion of a Lambda plate.

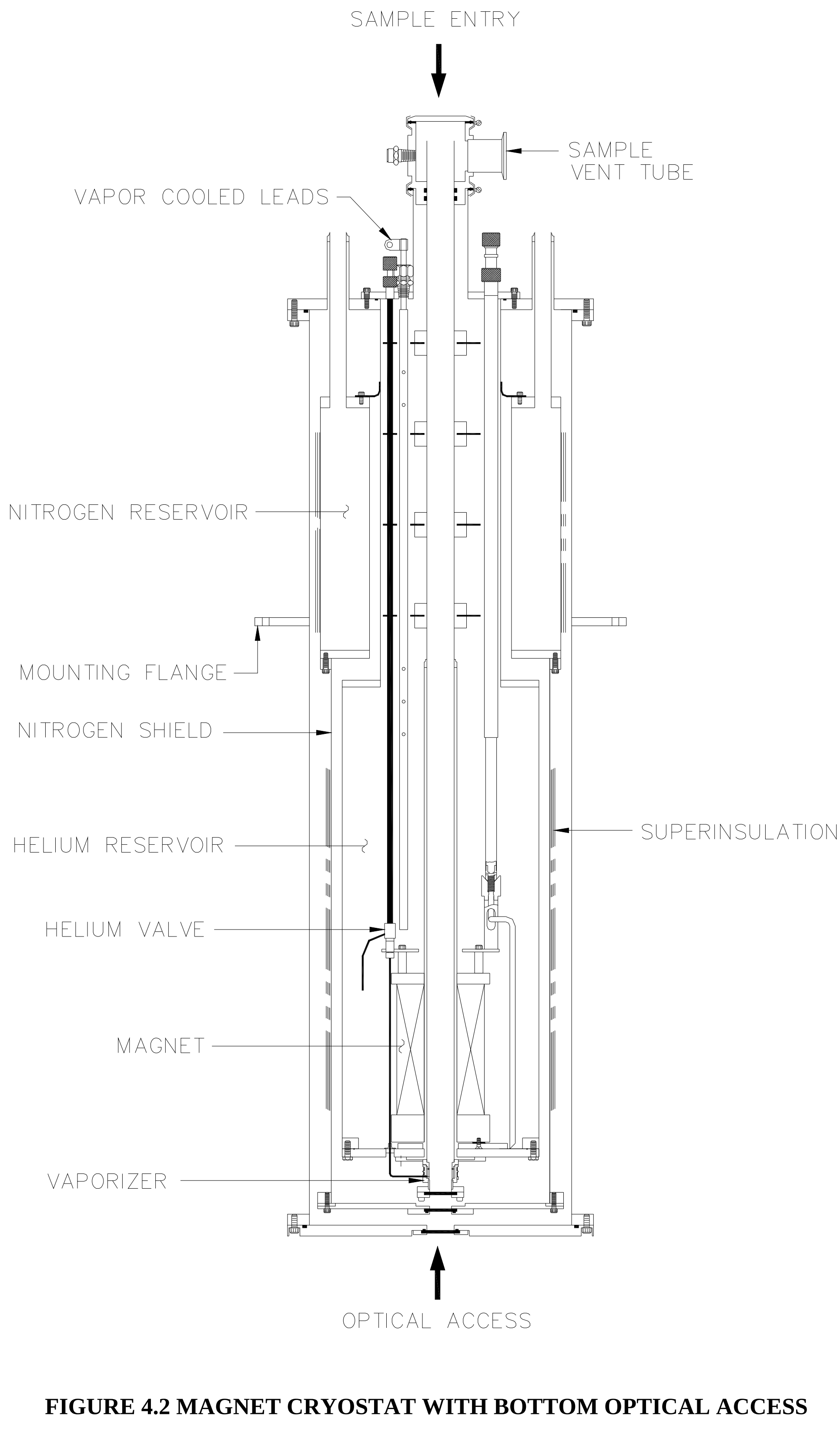

FIGURE 4.2 MAGNET CRYOSTAT WITH BOTTOM OPTICAL ACCESS

C. Optical Access Split Solenoid Systems

Experiments requiring optical access along two or three directions into the high field region of a variable temperature cryostat, are best done with a split or Helmholtz type coil. The variable temperature cryostat provides horizontal windows (looking through a vacuum) into the sample tube as is shown in Figure 4.3. Optical access along the vertical direction can also be supplied (as in 4.B above) if desired. The magnetic field direction can be either horizontal or vertical, with the horizontal configuration slightly preferable since light can then be directed either along or perpendicular to the magnetic field. Once again helium is channeled through a needle valve and capillary tube into the vaporizer at the bottom of the sample tube. The sample tube is surrounded by vacuum inside the isolation tube, which is connected to the vacuum bobbin on which the split coil is wound. An initial helium fill adapter sends a tube to the bottom of the helium reservoir for efficient cooldown of the magnet from 77 K to 4.2 K, and for removal of liquid nitrogen from the helium reservoir after cooldown from room temperature to 77 K. This adapter mates with an initial transfer line guide tube, which is removed after the helium transfer to reduce the heat load into the helium reservoir.

Radiation shield windows are usually installed in order to reduce or eliminate the radiational heat load to the helium reservoir from the large openings in the radiation shield. These openings are usually 1" to 2" in diameter to maintain a large solid angle for the scattered or incident beams. As an alternative to the radiation shield windows, one can also use a re-entrant radiation shield that fits into the horizontal bores of the magnet, and ends in a flange with a small opening close to the sample tube. Such a set up will offer low radiation heat load due to the small hole, while still maintaining a reasonable solid angle access to the sample since the hole is closer to the sample. The sample is top loaded into the sample tube, and is cooled (or heated) by the helium vapor whose temperature is controlled at the vaporizer. The sample holder (not shown in Figure 4.3) can be attached on a simple 0.25" diameter stainless steel tube passing through an O-ring compression seal at the top flange. such an arrangement allows easy rotation and/or translation of the sample along the axis of the sample tube. The helium vapor exits out of a port at the top of the sample tube, and temperatures below 4.2 K are attained by reducing the pressure in the sample tube while the sample tube is filled with liquid helium, or while the needle valve is throttled (as in 3.B). This system gives an operating temperature range from below 2 K up to room temperature, and is the basis of the Janis SuperOptiMag cryostats.

The windows at the sample tube may be strain relief mounted for experiments requiring polarized light. Since the sample tube and window block has to fit between the two split coils (in the radial ports), this usually requires a larger separation between the coils. In general this leads to a bigger magnet and a larger helium reservoir for the same size sample chamber.

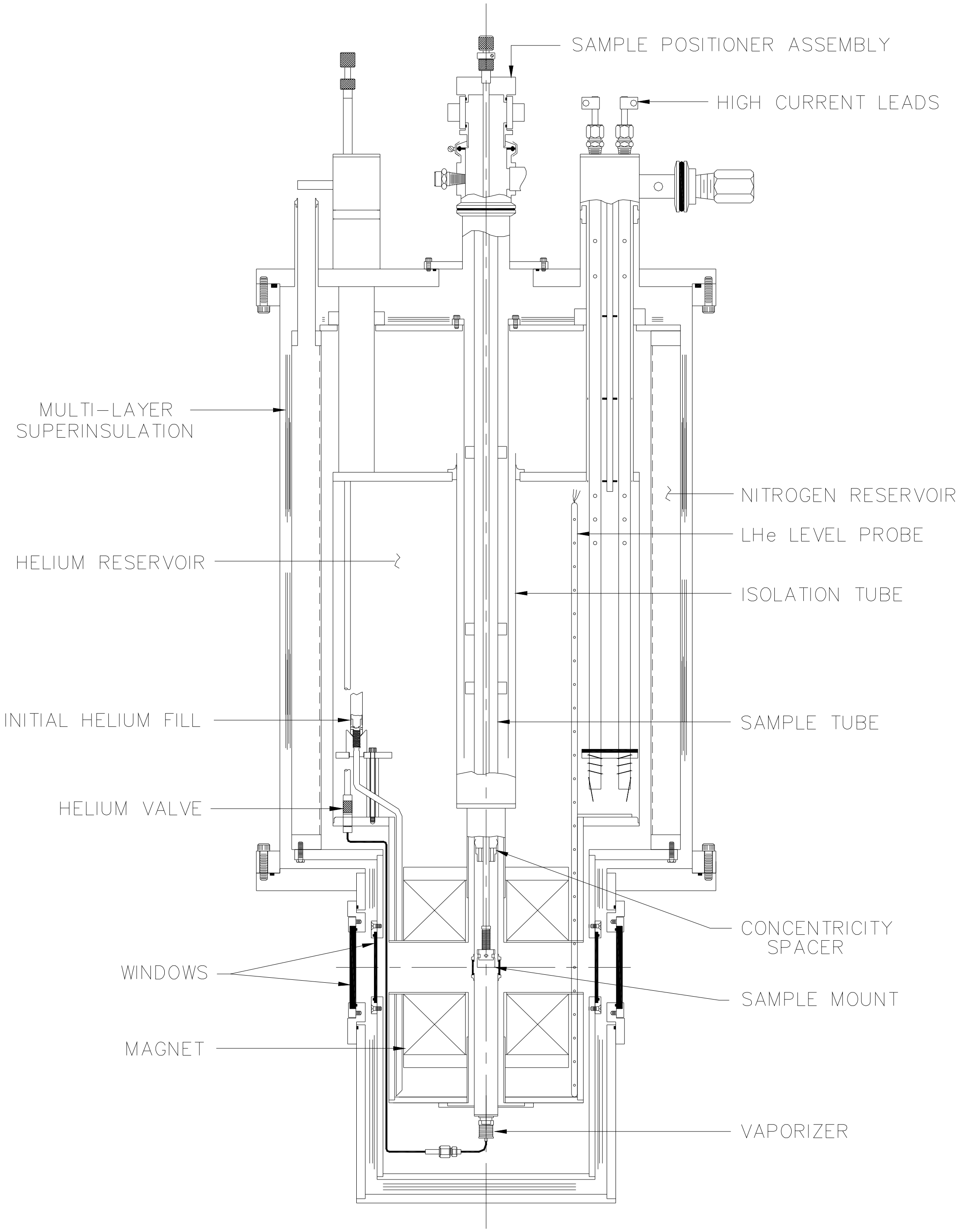

FIGURE 4.3 MAGNET CRYOSTAT FOR MAGNETO-OPTICAL EXPERIMENTS

D. Magnet Systems for Special Applications

Many applications require a large magnetic field for experiments conducted at room temperature. In such cases a superconducting coil is installed in a helium dewar with a room temperature bore tube, surrounded by one or two radiation shield bore tubes, which in turn is surrounded by the helium bore tube through the bore of the superconducting solenoid. The magnetic field may be either in a vertical or horizontal direction, with the dewar designed accordingly. The simplest configuration is the vertical room temperature bore in an open neck dewar that allows the magnet to be top loaded into the dewar. Since the outermost bore tube in these dewars runs directly from room temperature into the helium reservoir, this usually results in a higher liquid helium boil-off rate. A more efficient design, which offers a vertical room temperature bore, contains the magnet inside a sealed helium reservoir where the helium bore tube is welded to the top and bottom flanges of the helium reservoir. The dewar is then more difficult to assemble and the magnet is trapped inside the helium reservoir.

Inexpensive 10 K closed cycle refrigerators can be incorporated into the design of magnet dewars with room temperature bores. In such cases, the helium reservoir is surrounded by two radiation shields, which are cooled by the two stages of the refrigerator. Detachable vapor cooled leads are built into these dewars in order to disconnect the leads after the magnet is charged, and allowed to operate in the persistent current mode. This generally reduces the helium evaporation rate anywhere from 5 cc/hr to 50 cc/hr depending on the size of the dewar and the refrigeration capacity of the closed cycle refrigerator used. This arrangement is only used when the application requires extended operation (about 30 days) in a constant magnetic field.

Other specialized applications require very low temperatures (0.3 K) in a magnetic field. A compact self contained He^3 insert is usually used for these applications, designed either to fit directly into the helium reservoir containing the magnet, or into an existing variable temperature insert. Since He^3 inserts require a pumped He^4 bath to condense the He^3 gas, such an insert will contain its own He^4 bath or else make use of the helium bath offered by the variable temperature insert. This eliminates the necessity for reducing the pressure on the helium reservoir containing the magnet. A charcoal pump built into these inserts offers a convenient method of reaching 0.3 K at the He^3 bath without the complication of a special gas handling system for top loading sample in LHe^3or continuously operating He^3 inserts. A cold finger arrangement (sample in vacuum) or a top loaded (sample in liquid He-3) can be incorporated with the superconducting magnet to provide a high field environment at very low temperatures.

To achieve temperatures below 0.3 K combined with a magnetic field, a dilution refrigerator can be combined with a superconducting. The dilution refrigerator will typically have an external gas handling system for operation in the temperature range between 10 mK and 200 mK, depending on the required heat load and sample access.

5. CLOSED CYCLE REFRIGERATOR SYSTEMS

Where handling of liquid nitrogen and liquid helium is not desired or when obtaining these liquids is difficult, a mechanical (closed cycle) refrigerator can be used to cool down the samples. The most common (commercially available) refrigerators make use of a two-stage Gifford-McMahon (GM) or the newer pulse tube (PT) coolers using high purity (99.999%) helium gas as the working fluid (Ref. 2, Ch. 2). In these units, the cold head is separated from a compressor by a couple of flexible high pressure tubes, which circulate the compressed helium into and out of the cold head. This enables easy handling of the cold head which weighs about 8 kg to15 Kg while the heavy (approximately 65 kg 180 Kg) compressor is stationed in a fixed location nearby (about 3 m – 20 m). The cold head for the GM coolers contains the displacers (pistons) that are used to compress and expand the helium gas inside the two stages of the cold head, along with the regenerators (reverse flow heat exchangers) that are critical for cooling the incoming gas and resulting in the ultimate cooling of the second stage. These regenerators are usually made from materials that have a poor thermal conductivity, high specific heat, and a large surface area to enable them to absorb the heat from the incoming warm helium gas, and give up heat to the colder exiting helium gas. The PT coolers have the advantage of not having any displacers or other moving parts, and are thus less likely to require maintenance for such moving part.

The compressors are typically charged to high pressures (220 psi – 300 psi) and are connected to the cold heads through stainless steel flexible lines and special Aeroquip fittings that allow quick attachment and detachment of the lines, without permitting any air into the high purity helium working gas. The GM coolers have inlet and exhaust valves in the cold head that are actuated by a rotary mechanism and the displacers may be driven either pneumatically or by the synchronous motor that drives the valves. The PT coolers that have no displacers in the cold head are supplied with a rotary valve and motor that may either be located at the top of the cold head or may be separated from it by a semi-flexible line located about 60 – 100 cm away from the cold head. The motor can be optionally electrically isolated from the cold head to reduce the electrical noise to the any cryostat attached to the cold head. The motors themselves have to be located in a low magnetic field (500 Gauss or less) in order not to slow the movement of the motor and reduce the cooling power. The cold head itself should also be located in a relatively low magnetic field (1000 Gauss or less) in order not to risk affecting the performance of the regenerators and reducing the cooling power of these coolers

The frequency of the motor and displacer movement in the GM coolers is about 2 Hz to 3 Hz, and varies with the frequency of the available ac signal (usually 50 or 60 Hz). The lower frequency (50 Hz) typically results in a lower refrigeration capacity at the first stage but does not affect the cooling capacity at the second stage, except for the 10 K coolers. In PT coolers the motor frequency is approximately 1 Hz to 2 Hz and the cooling power is not affected by the frequency of the AC signal. The motor and associated piston (displacer) movement in the GM cold heads, results in a certain level of vibration being transmitted to the cold finger and outer body of the cold head. The lack of the moving parts in the PT coolers results in less vibrations transmitted to the outer body of the cold head, but the cold stages will still have some vibrations that are associated with the pressure variation during the cooling cycle. These vibrations (approximately 4 μ) are typically lower that the associated vibrations of a GM cooler by a factor of 2 – 4. Fortunately, these vibrations are quite tolerable for many experiments at low temperatures, and special vibration isolation techniques are available for reducing these vibrations to much lower levels. These techniques typically involve using flexible copper braids or an exchange gas mechanism coupled with rigid sample mounting, resulting in less cooling power and higher base temperature at the sample.

In general the two stage GM coolers are available in two categories. The smaller units have a base temperature of 8 K – 10 K with a typical cooling capacity of 2 watts refrigeration capacity at 20 K, or 2 watts at 10 K, up to 9 watts at 20 K (or higher). This cooling capacity is quite satisfactory for a majority of experiments that are conducted in a typical low temperature laboratory. The second category of GM coolers reach temperatures of less than 4.2 K with various cooling powers that range between 0.1 watts up to 1.5 watts (as of this date) at 4.2 K, with base temperatures that reach below 3 K. These units also require very little maintenance to either the cold head or compressor. Typically one needs to replace a filter (activated charcoal adsorber) in the compressor after 9,000-10,000 hours of use. The filter removes all traces of oil vapors from the helium gas returning to the cold head, and if not replaced could result in some oil vapors contaminating the cold head and freezing there. In some systems, replacement of the displacers and associated gasket seals is also recommended after this period of operation. An elapsed-time meter at the compressor keeps track of the total operating time.

The two stage PT coolers are also available in two categories (10 K and 4.2 K) with the latter category much more in demand. The 4.2 K PT coolers are also available in a variety of cooling powers ranging between 0.25 watts up to 1.5 watts (as of this date). The PT cold heads require maintenance every 20,000 with potentially even longer periods (up to possibly 30,000 hours), but there is currently not enough data on these relatively new coolers to guarantee the longer period. One final thing to note is that the pulse tube cold head cannot operate in an inverted position as does the GM cold head. The pulse tube cold head may be tipped approximately 15 to 30 degrees from the vertical access before it starts to lose some of its cooling power.

The compressor also contains a heat exchanger, which is used to dissipate the heat generated when the helium gas is compressed. Both water cooled and air cooled systems are available, with the air cooled one usually slightly more expensive. Safety protection switches are installed at the compressor to turn it off if the temperature gets too high. This could be due to too high water temperatures, or too low a throughput of cooling water, or too high an ambient temperature. Other protective switches turn the compressor off if the pressure is too high or too low, and a safety pressure relief valve protects against system overcharge. The compressor and cold head must be cleaned (using 99.999% pure helium gas), and the system re-charged if it gets contaminated (with air, water vapor or oil), or if the pressure drops too low (usually below 30 psi). In general, the power requirements for the various refrigerators vary between approximately 2 KW for the 10 K systems up to approximately 8 KW for the 4 K systems, with AC power input raging from 220 VAC 1-phase (for 10 K systems) up to 400 VAC or 470 VAC 3-phase options for the 4 K systems.

The construction of the cold head lends itself naturally to a cryostat with a cold finger configuration. Indeed these refrigerators are also used (with appropriate baffles and condensing fin arrays) as cryopumps in vacuum chambers requiring clean environments. When used as variable temperature cryostats, two general categories are obtained, with the sample in vacuum, or sample in an exchange gas column cooled by the two stages of the refrigerator. These two categories are described in what follows.

A. Cold Finger Cryostats

Figure 5.1 shows the cold head of a (10 K) closed cycle refrigerator with mechanically driven pistons, to which is attached a vacuum jacket and adapter, containing electrical feedthroughs and an evacuation valve and a safety pressure relief valve (not seen in the drawing).

The vacuum jacket surrounds the cold head, consisting of the second stage of the refrigerator, which acts as a cold finger/sample mount, and the first stage to which is attached a radiation shield surrounding the cold finger. This arrangement provides a temperature of 8 K to10 K at the cold finger, while the radiation shield is cooled to 60 - 80 K by direct contact with the first stage. The Figure shows the cold finger pointing upwards, and so it is only appropriate for GM type coolers, since as mentioned earlier, pulse tube coolers will not operate in this orientation. A heater attached at the cold finger will allow temperature variation up to room temperature. Should higher temperatures be required, a special insulating stage can be added at the second stage. This is required since the cold head is usually not designed to withstand temperatures much higher than room temperature. In this case, it is advisable to have a protection circuit to prevent the second stage from reaching these higher temperatures, and causing damage to the cold head.

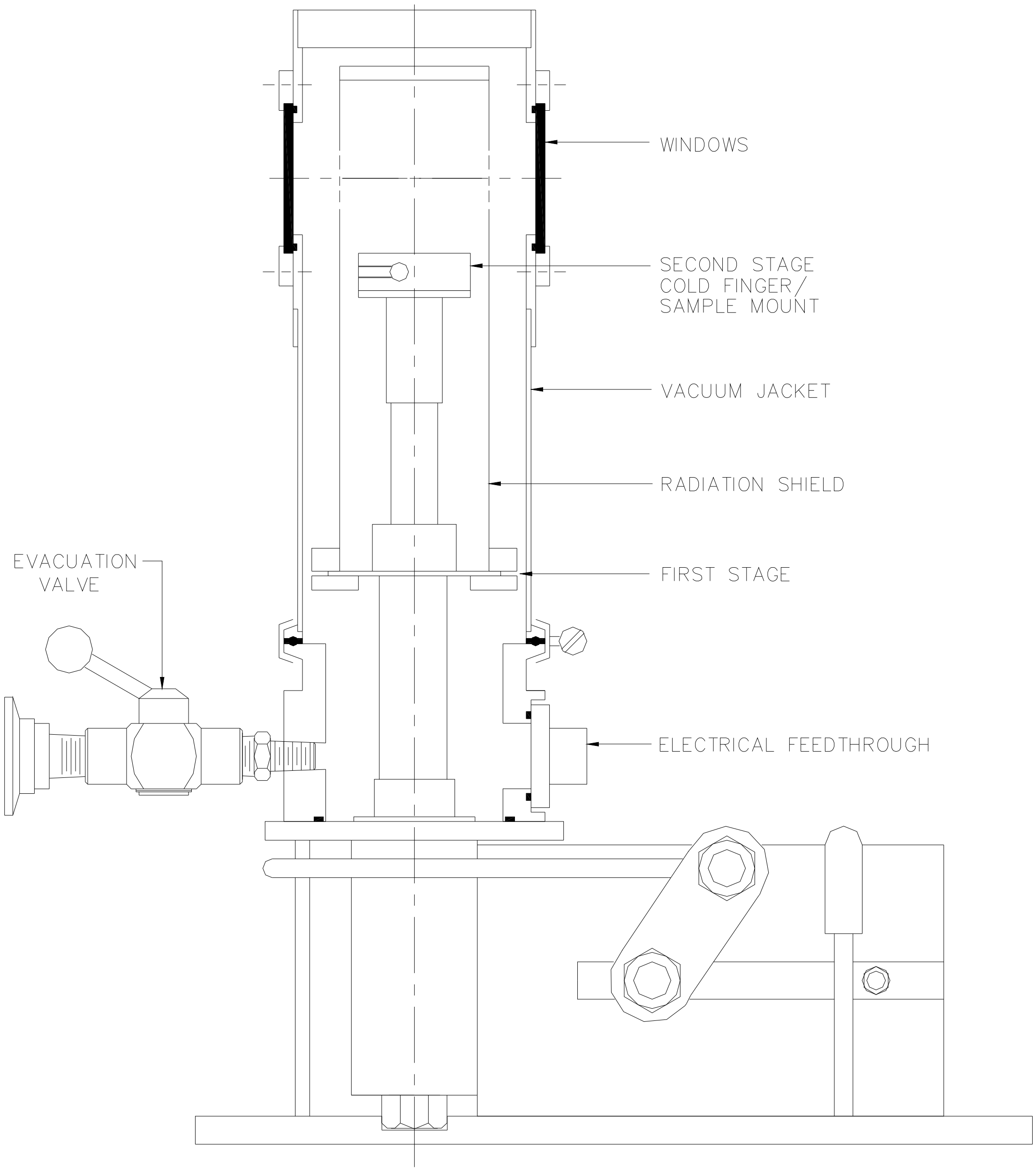

FIGURE 5.1 CLOSED CYCLE REFRIGERATOR CRYOSTAT

Figure 5.2 shows a PT cold head in a cold finger configuration where the cold head itself is oriented in an upright position (which is require for PT coolers). The configurations shown in Figure 5.1 & 5.2 include a simple arrangement providing optical access to the sample mount. These windows can be eliminated when no optical access is required, and the radiation shield and vacuum jacket can be manufactured closer to the cold finger if a more compact configuration is needed. For very tight spaces, the cold finger can be extended, using a long thin OFHC copper extension to the cold finger. The radiation shield and vacuum jacket can then also be narrowed down to fit inside narrow spaces (such as a pole gap of an electro-magnet). The lowest temperature attained at the end of such a sample mount may be one (or more) degree higher than the temperature of the cold finger, depending on the details of the set-up in question.

A typical system will reach its lowest temperature in about one hour. This will be affected by the size of the sample holder and sample that are attached to the cold finger. Some units exhibit a small (0.1 - 0.5 K) temperature fluctuation at the lowest temperatures achieved by the cold finger. This is related to the displacer motion (and associated thermodynamic cooling cycle) inside the second stage, and may be improved by using an automatic temperature controller and thermometer to control the temperature slightly higher than the lowest attained temperature. It can also be minimized or reduced to a few milli-K by adding specially designed stages to the cold finger.

As with any other cold finger (sample in vacuum) cryostat, one should not assume that the sample is exactly at the same temperature as the cold finger. For this purpose it may be useful to attach a thermometer to the sample, in order to obtain a more accurate measurement of the temperature of the sample.

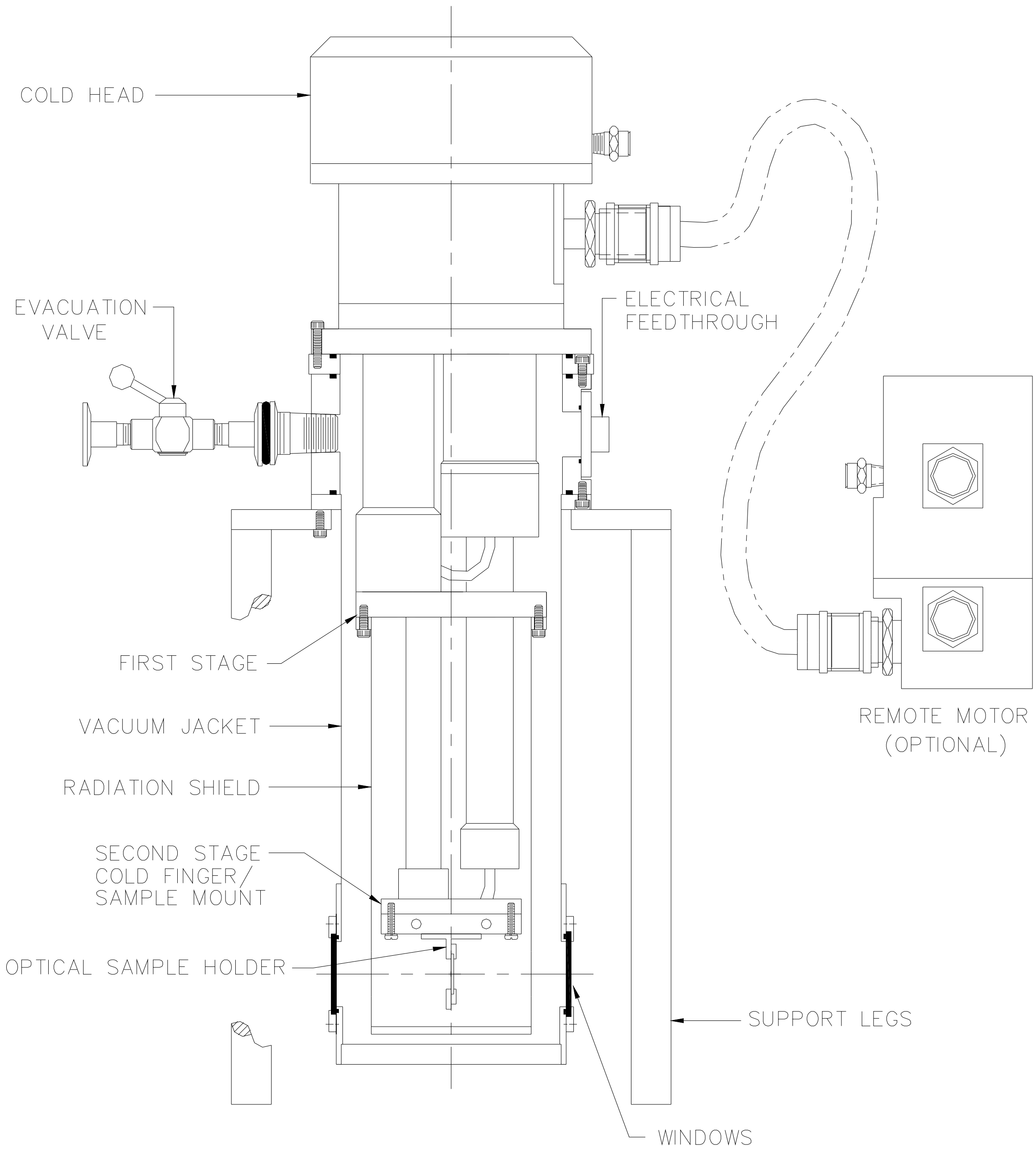

FIGURE 5.2 PULSE TUBE COOLER CRYOSTAT

B. Exchange Gas Cryostats

Figure 5.3 shows the cold head of a 7 K or 10 K GM closed cycle refrigerator, with pneumatically driven displacers, attached to an exchange gas sample chamber surrounded by a radiation shield and vacuum jacket. The lower portion of the sample tube is formed from an isothermal copper surface that is cooled by direct contact with the second stage of the refrigerator. The radiation shield is made from aluminum or copper, and is cooled by direct contact with the first stage. It surrounds the main length of the sample tube, and is anchored to a point at the upper portion of the sample tube, to intercept the conductive heat load coming down the sample tube from room temperature.

The sample is attached on a copper sample mount, which is supported by a long thin walled stainless steel tube with baffles, which emerges at the top of the cryostat. Electrical access to the sample area is located at the top of the sample positioner, while electrical access to the second stage (inside the vacuum jacket) is located near the bottom of the vacuum jacket. Heaters and control thermometers may be attached to the second stage of the refrigerator and/or to the sample mount. The temperature may be controlled at either of these two points, with control at the second stage being a little slower because of the large mass attached to that stage. With about half an atmosphere of helium exchange gas in the sample tube, the sample and its holder can be cooled and/or heated indirectly through the second stage (and its heater). Controlling the temperature at the sample mount is quicker because of its smaller mass, but it requires good thermal anchoring of the sample to the sample mount, to ensure that the sample is at the same temperature as its mount, especially when it is heated.

The pressure inside the exchange gas tube is not as critical as in the case of a liquid helium dewar, where one has to worry about the helium consumption. A temperature of about 10 K is easily achieved at the sample in a properly designed system. This arrangement is desirable since it permits quick sample interchange (through this top-loading configuration), without having to heat up the entire cold head and breaking its vacuum, as would be required in the cold finger configuration. One only needs to bring the exchange pressure up to atmospheric pressure, in order to prevent air from entering the sample tube while changing samples. The penalty one pays for this arrangement is the higher ultimate temperature (about 3 to 5 K higher), and the added cost of the exchange gas system.

Another advantage of this configuration is that one can decouple the sample holder from the vibrations generated by the drive mechanism for the displacers and valves inside the cold head. This is done by adding a vibration isolation bellow and a support flange between the sample positioner and the top of the sample tube. The flange can be rigidly bolted to a stationary stage, which supports the sample rod and the bellows. The sample rod and holder, along with the radiation baffles should make no contact with the walls of the sample tube, and should be supported vertically from the stationary stage.

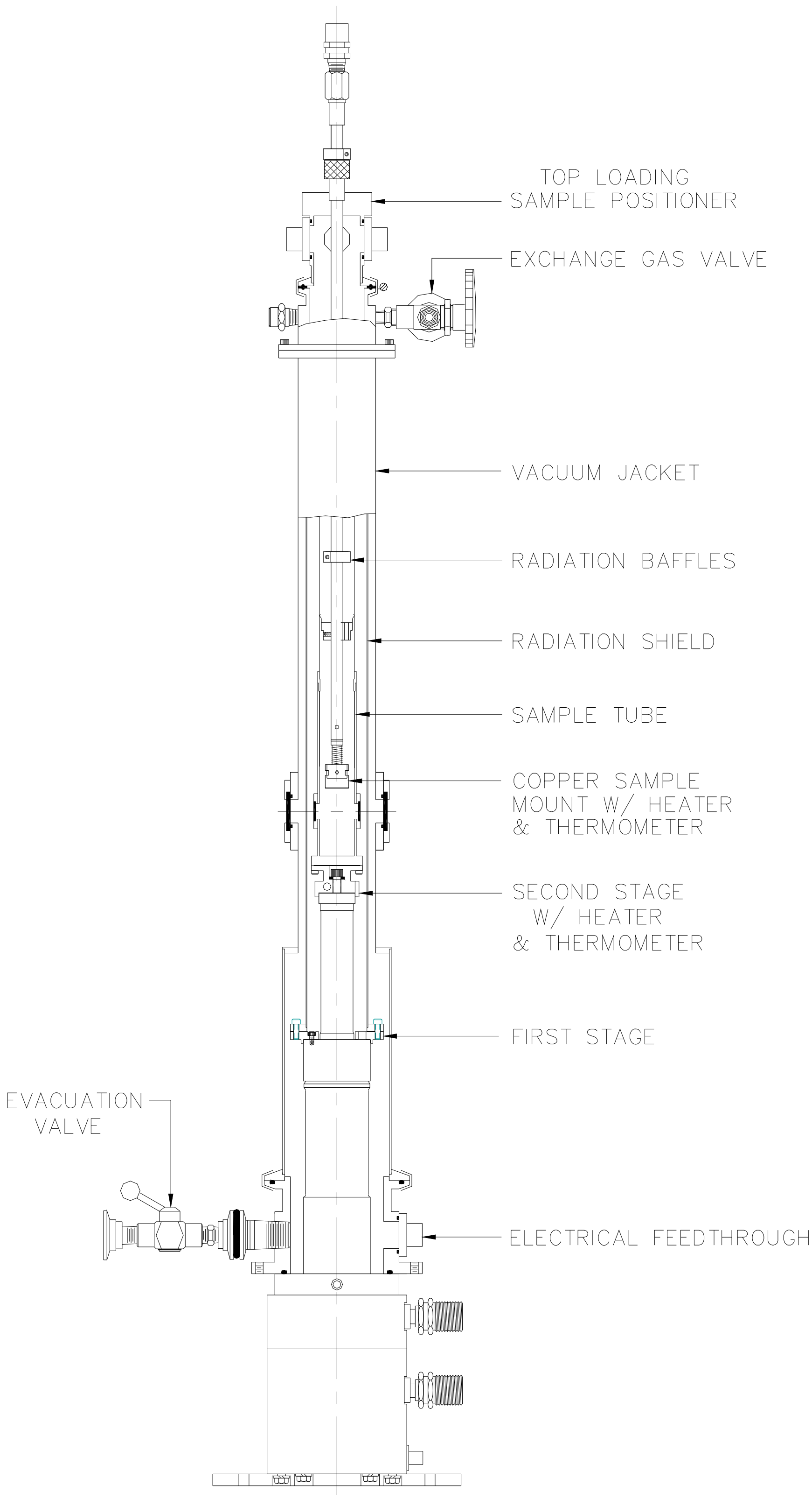

FIGURE 5.3 EXCHANGE GAS REFRIGERATOR CRYOSTAT

Figure 5.4 shows another configuration for an exchange gas cryostat that maintains the cold head in an upright position. This geometry can be much more convenient for the end user, all be it a little more expensive to manufacture. In this specific example, a PT type cold head is used, and the two stages of the cold head are thermally anchored but mechanically decoupled from the exchange gas sample tube where the sample rod is inserted. The decoupling is done with specially designed OFHC copper flexible thermal links that have relatively large thermal conductance to ensure that the sample reaches a low enough temperature (typically 4.5 K or less in a 4 K cooler).

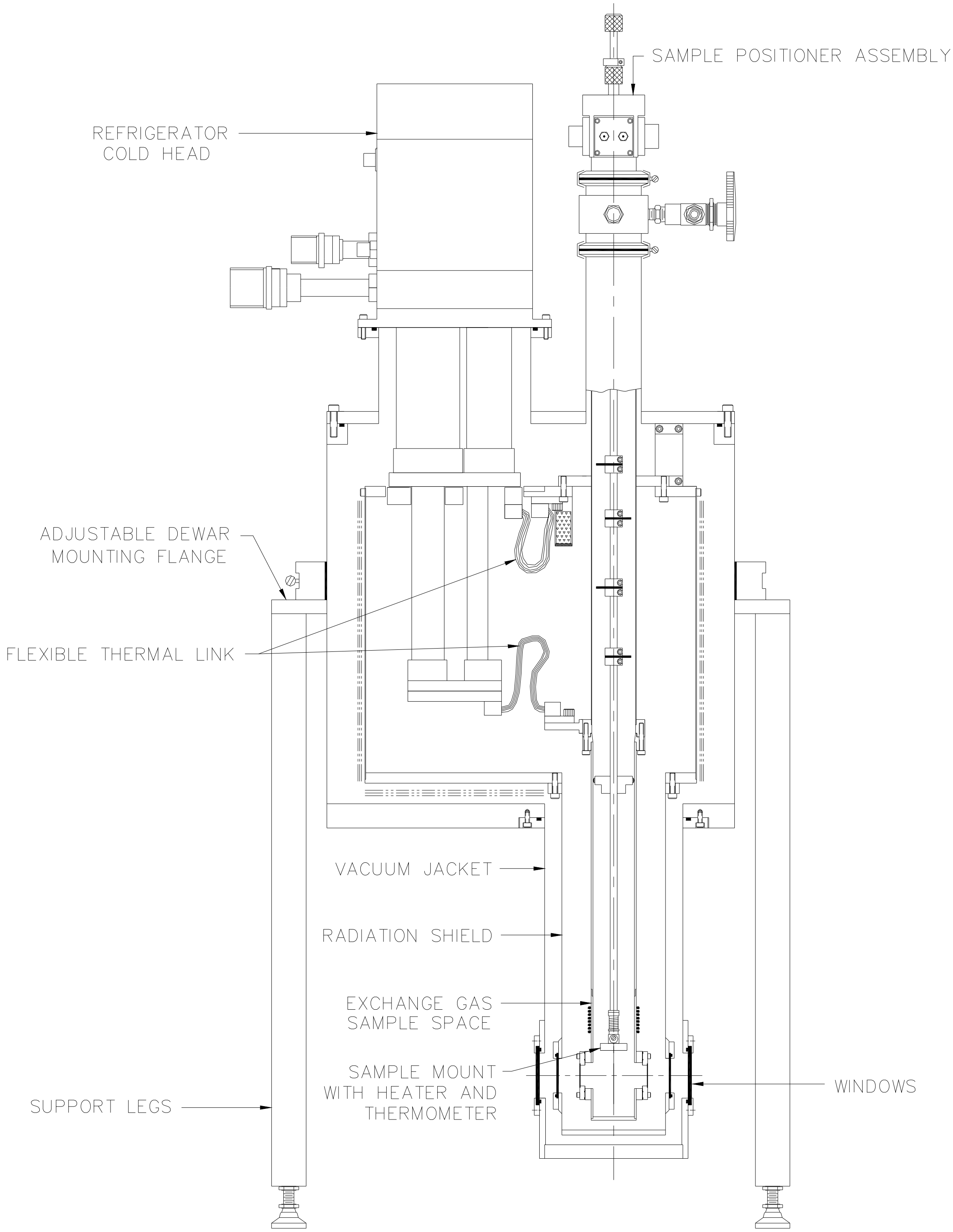

FIGURE 5.4 VIBRATION ISOLATED SAMPLE IN EXCHANGE GAS

C. <u>Vibration</u> <u>Isolated</u> <u>Cryostats</u>

With the increasing interest in cryogen free systems based on mechanical coolers, there is also the desire to eliminate the vibrations that are associated with these systems. Two previous examples have been mentioned earlier which locate the sample in an exchange gas environment. Figure 5.5 shows an example of such a cryostat where a new cold finger is offered that is mechanically decoupled from the body of the main cold head. In this case the thermal link to the new cold finger (and surrounding radiation shield and vacuum jacket) is made through helium exchange gas. The cryostat will require rigid support on a vibration isolated table and the cold head itself will need to be independently supported. A flexible bellows links the cold head to the cryostat, while isolating the cryostat from the vibrations of the cold. This design can also be modified to offer a microscopy type cryostat where the cold finger is located very close to the outer window. The cooling power of the cold head is significantly reduced in these cryostats and the lowest temperature can be several degrees higher than the base temperature of the cold head.

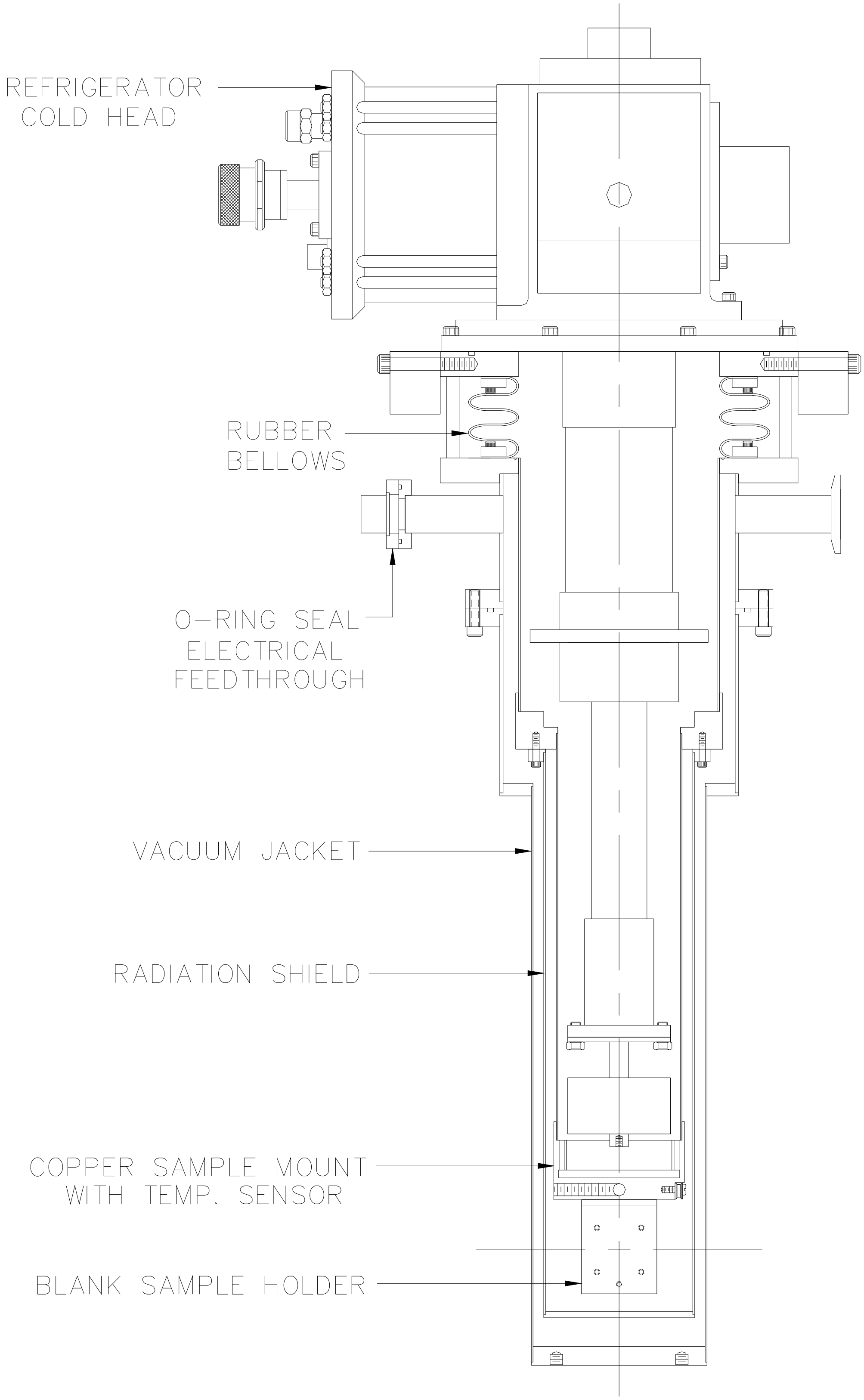

FIGURE 5.5 VIBRATION ISOLATED G-M COOLER

6. EXPERIMENTAL TECHNIQUES AND DATA

The final configuration for a variable temperature cryostat, will generally involve appropriate thermometry, automatic temperature control, sample attachment and system cooldown. What follows will concentrate on commercially available instrumentation, and general considerations for ensuring efficient cooldown and the necessary anchoring techniques to achieve the desired temperature at the sample.

A. Cooldown of Helium Cryostats and Samples

The enthalpy (total heat) of the common materials used for constructing cryostats (stainless steel, copper, aluminum...) drops rapidly with decreasing temperature. This, coupled with the low cost of liquid nitrogen and its high heat of vaporization (relative to helium), makes it the natural choice to cool the dewar from room temperature down to 77 K. Once this is done, the liquid nitrogen can be removed (from the helium reservoir) by inserting a tube, which reaches the bottom of the reservoir, and blowing the nitrogen out through this tube by over pressurizing the reservoir. As mentioned earlier, it is very important to remove all liquid nitrogen from the helium reservoir prior to transferring liquid helium, since the heat capacity of solid nitrogen is quite large (an order of magnitude greater than for copper). Even a couple of inches of liquid nitrogen remaining in the bottom of the helium reservoir, will require large amounts of liquid helium to cool them to 4.2 K and allow helium to accumulate. In simple reservoirs, the cooldown to 77 K will take less than an hour. If, however, there is a large mass (e.g. 4 K radiation shield, or large sample) attached to the reservoir, this process may take several hours, and is best monitored by a thermometer attached at the end of this mass.

Once all the liquid nitrogen is removed, liquid helium can be transferred using an appropriate helium transfer line. The vacuum jacket of the transfer line should be periodically evacuated, and the insulated storage dewar leg should extend practically to the bottom of the storage dewar. The delivery end can be either fully insulated, or have a short extension tip (few inches), and should deliver the helium to the lowest point in the helium reservoir. The helium transfer must start at a very low rate, and be maintained at that rate until the entire contents of the helium reservoir (including any attached mass) is cooled below 20 K. This ensures that one is making full use of the enthalpy of the cold helium gas, as it rises and escapes out of the top of the cryostat, to cool the reservoir and its contents. A poor helium transfer, which does not make full use of this enthalpy, can use 10 times more liquid to cool the dewar from 77 to 4.2 K. A poor (fast) helium transfer can also result in quick filling of the helium reservoir, even while most of the reservoir itself is at a temperature above 20 K. However, this usually results in the liquid helium quickly boiling off within the next hour, as it cools the rest of the reservoir to 4.2 K.

Any sample being cooled, should preferably be cooled at the same time as the helium reservoir. When the sample is located inside the reservoir, it gets cooled (along with the reservoir) by direct contact with the helium vapor. If however, the sample is on a cold finger in vacuum, it should be thermally anchored extremely well to the cold finger. This will produce faster cooldown times, and a lower ultimate temperature at the sample. If possible, a thermometer should be placed at the sample (or its holder) to get a better idea of the actual temperature of the sample. This thermometer would be in addition to any existing thermometer at the cold finger itself.

When two pieces are in contact (in vacuum) a thermal (boundary) resistance will always develop across the surface interface. This usually results in a temperature difference between the two pieces, especially if one is cooled by contact with a cryogen (or a cooling gas), while the other is only being cooled through the surface contact with the first piece. The temperature difference that develops is proportional to the thermal resistance and to the heat load on the second piece. The thermal resistance can be decreased by increasing the contact pressure between the two pieces, and by employing conductive epoxies or greases. In some applications, a thin layer of indium is used to increase the surface contact.

The thermal resistance between two components pressed together is usually difficult to measure, since it depends on the condition of the surfaces in contact. In general, the surfaces should be very clean and polished, if possible. Typical values for boundary resistances of copper-to-copper surfaces with a load of 100 lbs. are about 100 K/watt at 4.2 K, and 3 K/watt at 77 K. Equivalent resistances for stainless steel are 185 K/watt at 4.2 K and 3.8 K/watt at 77 K. Gold plating copper surfaces can decrease their boundary resistance by 20 times at 4.2 K (see Ref.1, Ch. VII & Ref. 2, Ch. 4).

When a sample is attached on a cold finger, it is necessary to establish good thermal contact between the two, and also reduce the heat load into the sample as much as possible. Thus, any wires running to the sample, should be wrapped around the cold finger and thermally anchored to it (using a conductive epoxy), prior to reaching the sample. Surrounding it with a radiation shield that is anchored directly to the cold finger can also reduce the radiational heat load on the sample. All these precautions are taken to ensure that the temperature difference between the sample and the cold finger are minimized.

B. Thermometry and Temperature Control

There are many types of commercially available temperature sensors that can be used at low temperatures. The ones that are most commonly used are silicon diodes, Cernox resistors, ruthenium oxide resistors, platinum resistors, Gallium (and Gallium-Aluminum) Arsenide diodes, and the less commonly used germanium resistors, carbon glass resistors and rhodium-iron resistors. These thermometers tend to retain their characteristics with repeated thermal cycling, and thus can be reliably calibrated. Silicon, gallium arsenide and gallium-aluminum arsenide diodes are usually activated with a constant current source, and the voltages developed vary (in a reproducible manner) with temperature. Platinum and rhodium-iron resistors are also activated with a constant current source and their resistance varies with temperature. Cernox, ruthenium oxide, germanium and carbon-glass resistors must be activated with a variable current source since their resistances vary over several orders of magnitude as the temperature changes. Thermocouples consisting of a pair of dissimilar metal wires, soldered or welded together are also useful when a small sensor is required. They generate an emf, which varies with temperature and is thus used as a basis for measuring the temperature. In general, they are not as sensitive as other available sensors at 4.2 K (typical sensitivity for a gold-iron vs. chromel thermocouple is 10 microvolt/ K), and small differences in the wire composition can cause significant deviations from the standard curves. The discussion will thus be limited to the diode and resistance thermometers listed above.

1. Thermometer Installation

Before discussing the various types of cryogenic thermometers, it is important to note one common characteristic of most such sensors. Even though manufacturers make every effort to establish a good thermal link between the body of the thermometer and the internal temperature-sensing element, 70 to 80% of the actual temperature sensing is through the electrical leads. While this does not present any difficulties if the thermometer is in liquid helium or flowing vapor (where both the leads and the body are immersed in the vapor), it can be a source of error when the thermometer is attached to a cold finger in vacuum. The leads running down from room temperature to the cold end of a helium cryostat, in vacuum, can easily carry enough heat to result in a thermometer reading of 10 K above the real temperature of the surface to which it is attached. It is thus extremely important to remove any "Teflon spaghetti" covering the sensor leads as they exit from the body, and wrap them tightly around the cold finger and thermally anchor them with a loaded epoxy (such as Stycast 2850). This of course has to be done with no electrical shorts between the leads and the cold finger.

The leads going up to room temperature should preferably be manganin or phosphor bronze -- i.e. some alloy of low thermal conductivity. Typically, 32 gauge wires should be used, and they should be spiraled as they travel to the room temperature end in order to increase their effective length and reduce the amount of heat they carry to the cold end. It is also important to anchor these leads to an intermediate cold stage (at 80 K), in order to intercept the large heat load from room temperature. A quick calculation (using the tables at the end of this chapter) may be helpful to ensure that the cryostat has enough cooling power to handle the heat load due to these leads. This becomes much more important below 4.2 K, where the heat capacities are very small, and a small heat load can easily result in a relatively large rise in temperature, or erroneous temperature reading.

i. Silicon Diodes

Silicon diodes have become the most common type of thermometers used in the temperature range of 1.5 K to 300 K (or 500 K). When activated with a 10-microampere constant current source, they offer a voltage of about 1.7 volts at low temperatures, dropping down to about 0.5 volts at room temperature. Their sensitivity ranges between approximately 25 mV/ K below 20 K to 2.3 mV/ K above 70 K. Because they are activated with a constant current, they are the obvious choice for use with an automatic temperature controller. Their main disadvantage is that their voltage changes significantly in a magnetic field, below 77 K, and thus they cannot be used for temperature measurement or control even with a low magnetic field of 1 Tesla.

These thermometers are usually provided in a four-wire configuration, which eliminates the effect of the potential drop across the wire leads connected to the sensor. When these leads are thin copper wire (32 gauge/0.008" diam.), the potential drop along a ten-foot length, with a 10 microamp current source is negligible compared to the sensor voltage – so a two-wire arrangement will be quite sufficient. Even if 32 gauge phosphor bronze wires are used (resistance of 1 ohm/ft), the potential drop for a 10 ft length wire will still be about 1000 times smaller than the sensor voltage.

One major advantage of silicon diode sensors is that they are usually available imbedded in a small (about 0.3" diam.) copper disk with its leads anchored to that disk. This configuration allows quick connection of the sensor to a cold finger surface (in vacuum), by simply bolting the sensor with a # 4-40 or M-3 screw. No other careful anchoring of the leads is required.

Finally, the silicon diodes are now commercially produced with temperature characteristics that deviate no more than 0.5 K -1 K (below 100 K) from a "standard" curve. This curve is usually stored on a PROM in the temperature controller, which can then be used with any of these diodes. If better accuracy is needed, the diode can be calibrated, and its calibration stored in the controller.

ii. Gallium Arsenide Diodes

GaAs and GaAlAs diodes are also excited with a constant current (10 or 100 micro-Amps), but they cannot be mass-produced with temperature characteristics that conform to any standard curve. They can still be calibrated and used with a simple (constant current) temperature controller. Their only advantage over Si diodes is that they can be used in low magnetic fields (1 to 2 Tesla) since their voltage does not vary significantly with the applied field.

iii. Platinum Resistors

Platinum resistance thermometers are generally used between 60 K and 500 K where the resistance increases at a fairly constant rate of about 0.4 ohms/ K. They are occasionally used down to about 20 K, but their sensitivity drops to about 0.08 ohm/ K. Their ceramic outer case, allows these sensors to be used up to 600 C, however it is difficult to find a technique that will thermally anchor the thermometer and its leads at the lower temperatures, and also withstand the higher temperatures.

Platinum thermometers are usually activated with a constant current, and the typical 100 ohm (at 0 °C) sensor will follow a standard curve down to 60 K to within a couple of degrees. Those thermometers are also useful (above 30 K) in large magnetic fields (14 Tesla or higher) due to their small field induced change in resistance.

iv. Cernox Resistors

These are thin film resistors that were develop around the end of 1994 and are used extensively in low temperature (down to 1.4 K– 4.2 K) and high magnetic fields (up to 19 Tesla) applications, and have largely replaced the older carbon glass resistors and to some extent, the older germanium resistors (that are not suitable for use in a magnetic field). They have excellent sensitivity at 4.2 K (approximately 1100 ohm/K) but their sensitivity drops by one order of magnitude at 10 K and by more than two orders of magnitude at 77 K. Special types have been developed for use down to He-3 or lower temperatures, but their magnetic field dependence makes them less useful in that temperature range (ruthenium oxide becomes the better choice).

These resistors can also not be made to follow a standard curve and thus will require individual calibration for each sensor for accurate temperature measurement. They are commercially available in the same configuration as Si diode sensors (see above), which makes then easier to install in a cryostat. They can also be calibrated and used up to 420 K, although as mentioned earlier, their sensitivity drops significantly (down to -0.09 ohm/K). They are supplied with a four-wire configuration and will require variable current excitation to maintain reasonable accuracy and to prevent self-heating at the lowest temperatures (below 4.5 K).

v. Ruthenium Oxide Resistors

These are thick film thermometers that are useful for use in a high magnetic field and specifically useful for use at very low temperatures (between approximately 25 mK and 20 K). They can also be produced commercially, with temperature characteristics that deviate no more than 10 mK at 50 mK to 0.6 K at 20 K from a "standard" curve. This curve is usually stored in the temperature controller, which can then be used with any of these sensors. If better accuracy is needed, the sensor can be calibrated, and its calibration stored in the controller. Their resistance increases significantly at the lower temperatures thus requiring a variable current source and current reversal and averaging to obtain accurate measurements and avoid self-heating.

vi. Germanium Resistors

These thermometers are always supplied in a four lead configuration, labeled for current and voltage. Installation of these sensors requires careful thermal anchoring of these leads as discussed earlier. The resistance of these thermometers decreases with increasing temperature, up to 100 K, where the curve starts slowly turning -- thus limiting the usefulness of these sensors to 100 K or less. One class of sensors is commercially available for use between 1.5 K and 100 K, while another class is used for lower temperature ranges (6 K to 0.3 K or 0.05 K). Their resistance changes by several orders of magnitude, thus requiring a variable current source. When measured properly at the appropriate current, and with current reversal to eliminate thermal emf's, they can be a very precise thermometer because of their excellent reproducibility. Special temperature controllers have been developed for these thermometers, which provide the variable excitation current necessary for these sensors, and they tend to be a little more expensive than the equivalent controllers for Si diodes or platinum resistors. The resistance of these sensors varies significantly with any applied magnetic field, and thus they cannot be used as thermometers in such applications.

vii. Carbon Glass Resistors

These thermometers are primarily used in high magnetic fields due to the small change in resistance with applied field. They are supplied in a four lead configuration, similar to the Ge resistors, and they also require careful anchoring of the leads for accurate temperature measurement. Their good temperature reproducibility and the fact that they have a small (orientation independent) magneto-resistance, has made them the best choice of thermometry in any magnetic field. $SrTiO_2$ capacitor sensors exhibit smaller (relative) magnetic field dependence, but they suffer from poor reproducibility with temperature cycling. The advent of

Cernox resistance thermometers had cut down on the use of the older carbon glass as a result of the better sensitivity of Cernox sensors over the useful temperature range.

viii. Rhodium-Iron Resistors

These thermometers offer a monotonically increasing resistance with temperature, with a typical sensitivity of 0.17 ohm/ K (between 100 K and 300 K), dropping down to about 0.08 ohm/ K at about 25 K. They are activated with a constant current source, and may be used between 1.5 K and 300 K (or up to 800 K). Their resistance varies linearly with temperature between 100 K and 300 K, and they will follow a standard curve to within a couple of degrees in this temperature range. They are useful in small magnetic fields (1 Tesla or less) since the typical change in resistance at 4.2 K in a 1 Tesla field is equivalent to about 0.08 K.

ix. Thermocouple Temperature Sensors

Thermocouples are generally made from two wires of different materials, which generate a potential difference between the two wires when the two ends of the wires are at different temperatures. Typically two ends are spot welded together and held at the cold region of the cryostat, and then travel (preferably uninterrupted) to a hermetically sealed feedthrough at room temperature. In general, thermocouples do not offer very good temperature accuracy below 40 K and are primarily used when the system is designed to operate at higher temperatures (approximately 800 K). One very useful thermocouple is the type E (Ni-Cr alloy and Cu-Ni alloy), which is useful in the entire temperature range (40 K to 800 K) since it is available in a configuration that can be thermally anchored reasonably well. While Pt resistance thermometers may offer better temperature accuracy, they are extremely different to thermally anchor in this entire temperature range. Other useful thermocouples include type K (Ni-Cr & N-Al alloy) and gold-iron thermocouples (useful at lower temperatures). In general, thermocouples are not a good choice of thermometry at cryogenic temperatures, and are used due to their practical ability to cover a wide temperature range, albeit with accuracies of a few degrees in the entire range.

2. Automatic Temperature Controllers

Commercially available temperature controllers designed to work with variable temperature cryostats in the range of 1.5 K to 300 K (up to 800 K) generally fall into two categories. The simpler ones use a constant current source designed to work with Si diodes, Pt resistance thermometers, Rh-Fe resistance thermometers and GaAs or GaAlAs diodes. The first two sensors (Si and Pt) can be obtained with "standard characteristic calibrations" that are stored in the controller, resulting in a temperature reading that is accurate within a few degrees (or better), depending on the temperature range. Such configurations are adequate for many experiments that do not require very precise knowledge of the temperature. For applications where a more accurate measurement of the temperature is required, the sensors are calibrated, and the calibration is stored in a PROM in the temperature controller. The controller then uses an appropriate interpolation scheme for the sensor in question, and provides an accurate readout (to better than 0.1 K) of the temperature. Most of these controllers have inputs for two to four sensors, which makes them very convenient to use for controlling the temperature with one sensor, and simultaneously reading (or displaying) the temperature at other points in the cryostat.

Thus, a very common configuration that is used includes a "standard" sensor for temperature control and a calibrated sensor for precise measurement of the temperature of the sample. Where a "standard" sensor is not available (e.g. GaAs diode), both sensors would have to be calibrated, and the calibration stored in the temperature controller.

Digital temperature controllers have become the norm for use in most laboratories, although a few analog ones may still be available. The digital controllers usually display the temperature directly in degrees Kelvin (or some other units), with options for displaying the voltage or resistance. Some of these controllers are also available in configurations that allow them to be used with thermocouples. The more sophisticated controllers have special circuits that allow them to be used with any of the sensors discussed above. In particular, they can be used with Cernox, ruthenium oxide, germanium and carbon glass resistance thermometers. These circuits can change the excitation current for the sensor as well as reverse the current and make average measurements that cancel thermal emf's, as the temperature and resistance of the sensor changes. The accuracy of the resistance measurements can reach ±0.01%, or better depending on the range involved.

The majority of these temperature controllers offer three term (PID) control. These controllers have three functions, know as Gain (Proportional), Reset (Integral) and Rate (Derivative), which can be set by the user. In addition, they have two or more heater output ranges, which determine the maximum amount of heat that the controller can send to the heater inside a cryostat. This output usually varies between 0.1 watts and 50 watts, with the higher outputs needed for larger systems at room temperature or above. The controllers always have a set point (voltage, resistance or temperature desired) that is compared with the actual sensor signal (voltage, resistance or temperature). The difference (deviation) between these two signals is usually amplified (via a deviation amplifier), and if the set point temperature is higher than the sensor's temperature, the controller will send heat into the heater in the cryostat. This heat counteracts the cooling power of the cryostat at the desired temperature. The heat output of the controller is proportional to the amplified deviation signal (difference between the set point and temperature sensor), within a certain range, called the proportional band region. Thus, once the top of this range is reached, the heater output is sending full power. The Gain setting changes the deviation amplifier gain, and determines the width of the proportional band (band width increases with lower Gain). A combination of sensor sensitivity and Gain setting determines the heater output from the controller, as a function of deviation (in degrees Kelvin) from the set point.

At the lower temperatures, where the heat capacities are small, a low Gain setting is required. This lower setting sends a smaller amount of heat for a specific temperature deviation. As the temperature increases, and the heat capacity of the cryostat increases, a higher Gain setting will be required. This higher setting sends a larger amount of heat for the same temperature deviation. If the Gain setting is too high, it can cause the temperature to overshoot the set point and develop temperature oscillations in the system.

The Gain (Proportional) circuit should result in the controller giving out zero power when the deviation from the set point is zero. At temperatures above the lowest temperature that can be achieved by the cryostat, a certain fixed level of heater output is required to balance the cooling power of the cryostat. This is usually achieved by an integrator circuit, which senses the steady state offset signal (in the proportional band) and "Resets" the power to a slightly higher level, in small steps, proportional to the offset. Once the offset goes to zero, the reset action stops (adding power) and maintains the power output at the correct value to balance the cooling power of the cryostat. As the temperature increases, the thermal time constant of the cryostat cold head also increases, since it is proportional to the ratio of the heat capacity divided by the thermal conductivity (this ratio is called the diffusivity) of the cold head. As this happens, the Reset setting also needs to be increased.

Most controllers also have a differentiator circuit that provides a signal proportional to the rate of temperature change, which gets subtracted from the proportional output signal. This reduces the effective amplifier gain, which drives the controller heat output, thus slowing down the rate at which the temperature rises. This allows more time for the cold head to stabilize, and prevents temperature overshoot. The "Rate" setting is more useful for controlling the temperature of large blocks, which have large heat capacities and longer relaxation times.

More recently many temperature controllers are available with a auto-tuning option that selects the appropriate PID values for a specific cryostat. While this option can be useful in some systems, it does not always result in the optimal set of PID values that give the most stable temperature control, especially at liquid helium temperatures.

Having a choice of two or more maximum output power settings for the temperature controller is useful since controlling the temperature of most laboratory cryostats requires low power outputs (less than a watt). If the maximum power output setting is large (say 25 watts), this may result in a temperature overshoot at the lower temperatures (1.5 to 20 K).

Finally, it is important to emphasize once more, the good thermal anchoring of the control sensor (and the heater) to the block whose temperature is being controlled. The sensor and heater should also be as close to each other as possible. This is important in order to reduce the time lag between the sensing of the increase in temperature and the actual time when the heat was introduced into the block. If this time lag is too long, it will cause large oscillations in the temperature of the block. An extreme example would be attempting to control with a sensor located at the sample holder of a SuperVariTemp cryostat, with the heat introduced into the vaporizer. This, of course, should always be avoided.

C. Useful Cryogenic Data and References

This section contains some useful information to help in estimating the heat load into the cold region of a cryostat, or estimating the amount of cryogen that is required for cooldown. It also lists several references from which more detailed information may be obtained.

1. Thermal Conductivities and Conductive Heat Loads

The heat flow Q_i through a solid of cross-section A, under a temperature gradient dT/dx is given by:

$$Q_i = kA\frac{dT}{dx}$$

where k is the (temperature dependent) thermal conductivity of the material. If the two ends of a long section of this material (of length l) are held at temperatures T_1 and T_2, then the heat flowing from the warm to the cold end will be:

$$Q_i = \frac{A}{l}\int_{T_1}^{T_2} k(T)dT = \frac{A}{l}k_{av}\left(T_2 - T_1\right)$$

where k_{av} is the mean heat conductivity between the two temperatures, and is defined as:

$$k_{av} = \left(T_2 - T_1\right)^{-1}\int_{T_1}^{T_2} k\left(T\right)dT$$

Table 6.1 (below) lists the mean conductivity of various materials that are most commonly used at low temperatures, at various temperatures T_2 and T_1. These values can be used to estimate the conductive heat loads down tubes, wires, supports, etc., without requiring detailed knowledge of the thermal conductivity as a function of temperature.

Average Thermal Conductivity in W/cm- K							
T_2-T_1 (k)	300-77	300-20	300-4	77-20	77-4	20-4	4-2
Copper (Electro-plate T-P)	4.1	5.4	5.7	9.7	9.8	10	4
Stainless Steel	0.123	0.109	0.103	0.055	0.045	0.009	0.002
Constantan Wire	0.22	0.21	0.2	0.16	0.14	0.04	0.006
Aluminum	2.0	3.0	3.0	2.0	2.0	0.8	0.3
Brass	0.81	0.70	0.67	0.31	0.26	0.08	0.02
G-10-CR	4.5×10^{-3}	4.5×10^{-3}	4.0×10^{-3}	3.9×10^{-3}	2.3×10^{-3}	1.3×10^{-3}	4.5×10^{-4}

TABLE 6.1 AVERAGE THERMAL CONDUCTIVITY IN W/cm- K (Ref. 1, Ch. VII, Ref. 4)

2. Radiational Heat Loads

The radiational heat load from a warm surface at temperature T_2 to a cooler surface at temperature T_1 may be expressed as

$$Q_r = S\left(T_2^4 - T_1^4\right)/V$$

where S is the Stefan-Boltzmann constant (5.67×10^{-8} W/m^2-K^4) and V (view factor) defined by (Ref. 5, Ch. 8 and Ref. 6, Ch. 7),

$$V = (1-e_2)/e_2 A_2 + F_{21}/A_2 + (1-e_1)/e_1 A_1$$

e_1 and e_2 are the emissivities of the two surfaces, A_1 and A_2 their areas, and F_{21} is the configuration factor which is determined by the relative geometry of the two surfaces.

For cylindrical metal dewars, two useful configuration factors to have are those for concentric cylinders and for parallel discs. For long (compared to the radius) concentric cylinders, and for two parallel discs of radii r_1 and r_2, separated by a distance h, the view factor in both cases will be a number of the order of 1. Thus, the view factor for both configurations can be approximated by

$$V = 1/e_2 A_2 + (1 - e_1)/e_1 A_1$$

Combining this with reported values of emissivities as listed below, quick estimates can be made for the radiational heat loads from room temperature, nitrogen temperature or helium temperature surfaces.

Experimental Values of Emissivities

Radiation From:	300 K to 77 K	77 K to 4.2 K
Al – anodized	0.78	0.67
Al – oxidized	0.5	
Al – as found	0.12	
Al – mech. polished	0.1	0.06
Al – electro polished	0.075	0.036
Cu – as found	0.12	0.06
Cu – mech. polished	0.06	0.023
Stainless Steel – as found	0.34	0.12
Stainless Steel – mech. polished	0.12	0.03
Stainless Steel – electro-polished	0.1	0.065
Stainless Steel – as found with Al foil	0.056	0.011

TABLE 6.2 EXPERIMENTAL VALUES OF EMISSIVITIES (Ref. 7)

3. Thermal Expansion

When a cryostat is cooled down to helium temperatures, the various components will contract, and it is useful to know the relative contraction for the various materials that are typically used in such cryostats. Most of the contraction usually occurs when the cryostat has been cooled to liquid nitrogen temperatures. Thus most leaks that occur, when a cryostat is cooled, will show up at liquid nitrogen temperature. Knowledge of the relative contraction is also helpful in designing joints or mechanical contacts, thermal contacts or standoffs at low temperature.

Thermal Expansion Data

Material	Percentage Change in Length (room temperature to 4.2 K)
Aluminum	0.414
Copper	0.326
304 Stainless Steel	0.296
Brass (70 cu, 30 Zn)	0.369
Brass (65 Cu, 35 Zn)	0.384
G-10CR (warp)	0.241
G-10CR (normal)	0.706
Nylon	1.39
Teflon	2.14
Fused Silicon	0.001
Sapphire (Z-axis)	0.007
Sapphire (X-Y)	0.006

TABLE 6.3 THERMAL EXPANSION DATA (Ref. 2, Appendix B & Ref. 8, Ch. 3)

4. Experimental Data for Cooldown

The following lists information about common cryogens and metals used in commercial cryostats that is helpful in estimating the amount of cryogen required for cooldown to helium temperatures.

Experimental Data for Helium and Nitrogen

	Normal Boiling Point	Liquid Density at Boiling Point (g/cm^3)	Heat of Vaporization ($Joule/cm^3$)	Volume of Gas at STP from 1 liquid liter
He^3	3.2 K	0.06	0.48	449 liters
He^4	4.2 K	0.13	2.6	740 liters
N_2	77.4 K	0.81	160	682 liters

TABLE 6.4 EXPERIMENTAL DATA FOR HELIUM AND NITROGEN (Ref. 9, Ch. 8)

Densities of S/S, Cu and Al

Stainless Steel	7.86 g/cc
Copper	8.96 g/cc
Aluminum	2.70 g/cc

TABLE 6.5 DENSITIES OF S/S, Cu AND Al (Ref. 10, Appendix H)

Enthalpy of S/S, Cu & Al

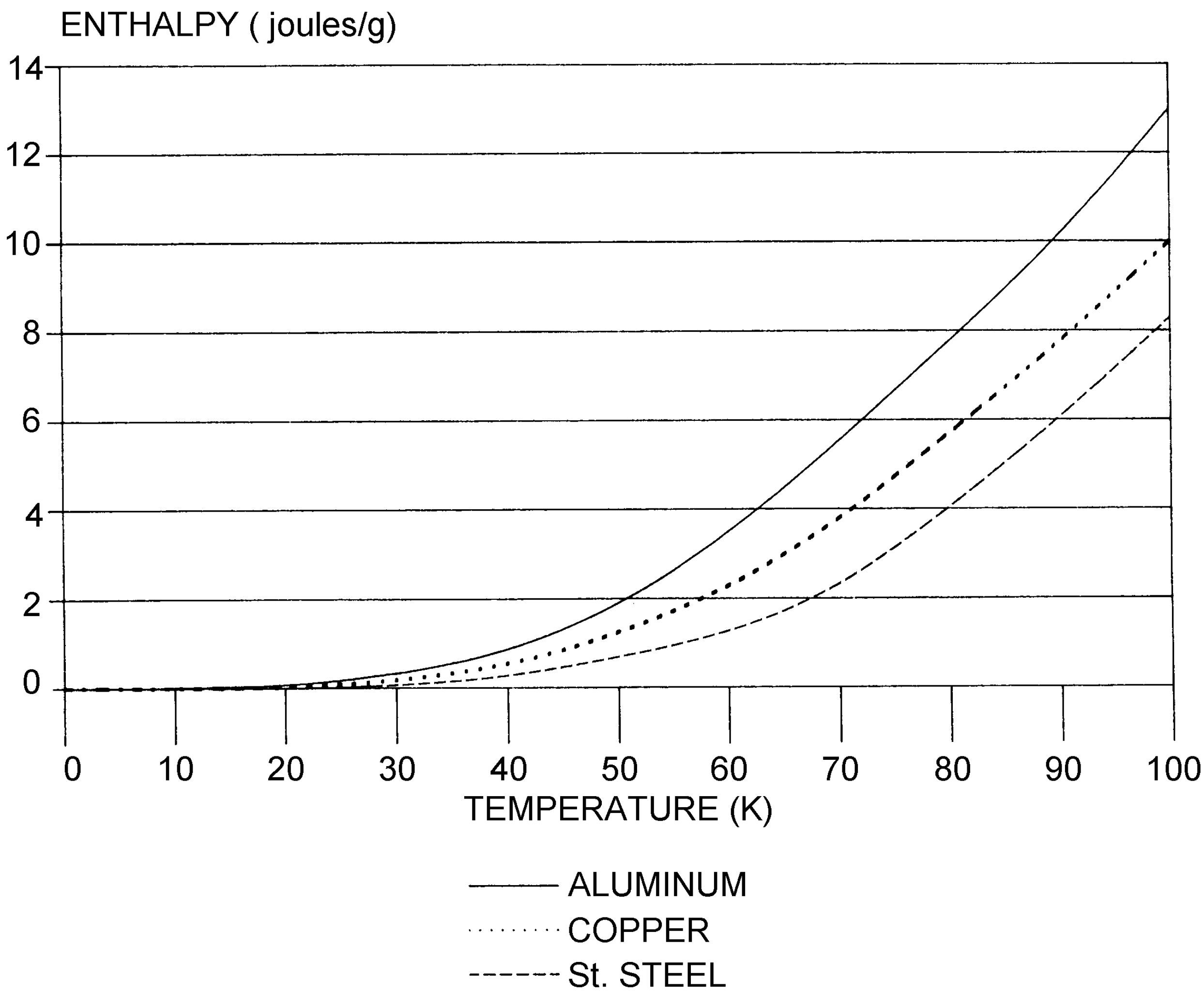

FIGURE 6.6 ENTHALPY OF S/S, Cu & Al (Ref. 9, Ch. 8)

Amount of Helium to Cool Common Metals
Litres of Liquid Helium per Kilogram of Metal

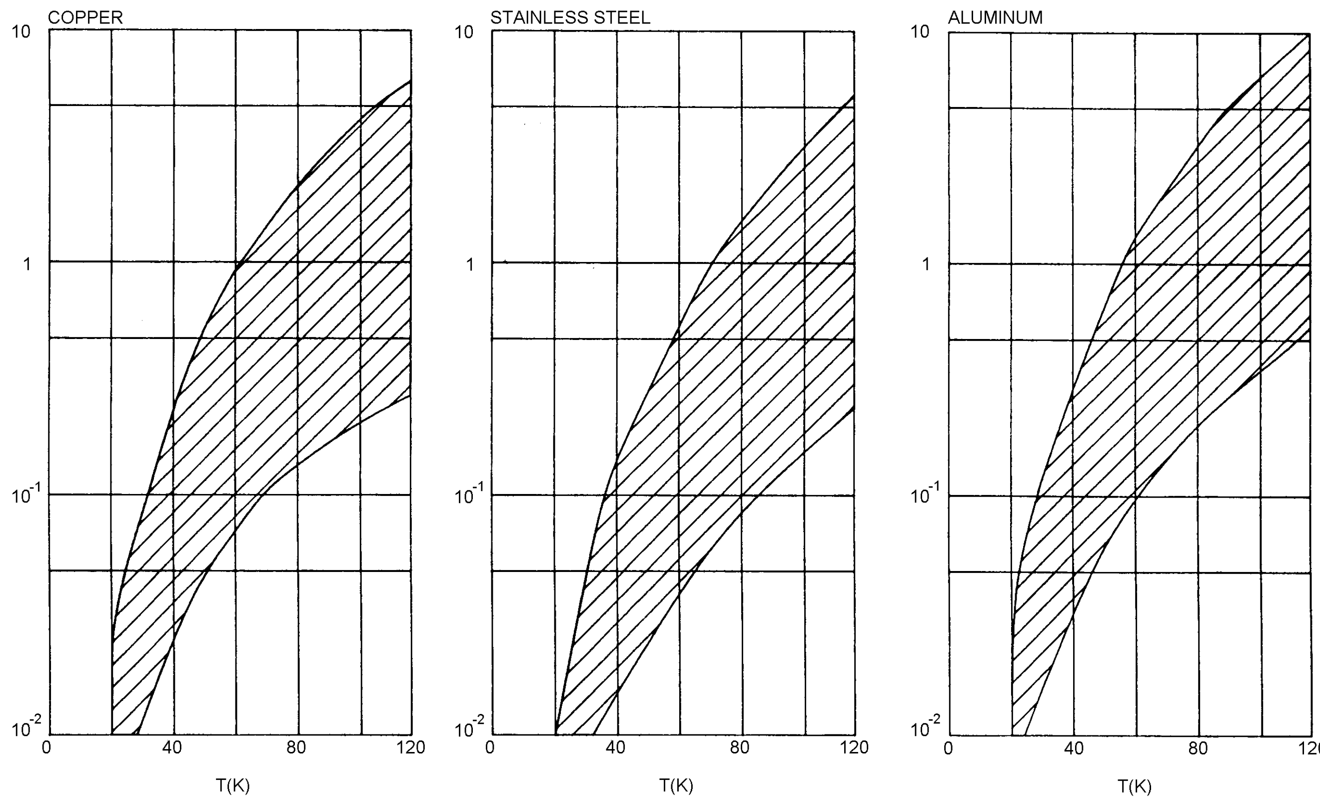

FIGURE 6.7 AMOUNT OF HELIUM TO COOL COMMON METALS (Ref. 9, Ch. 3)

REFERENCES AND SUGGESTED READING LIST

1. G. K. White, Heat Transfer, (Oxford University Press, London, 1968).

2. G. K. White & P. J. Meeson, Experimental Techniques in Low Temperature Physics. 4th Ed. (Oxford University Press, New York, 2002).

3. G. W. Green, The Design and Construction of Small Vacuum Systems, (Chapman and Hall, London, 1986).

4. J. R. Benzinger, Properties of Cryogenic Grade Laminates, Electrical/Electronics Insulation Conference, Boston, (1979).

5 J. P. Holman, Heat Transfer, (McGraw-Hill, New York, 1981).

6. R. Siegel and J. R. Howel, Thermal Radiation Heat Transfer, (McGraw-Hill, New York, 1981).

7. W. Obert, J. R. Coupland, D. D. Hammond, T. Cook and K. Harwood, Advances in Cryogenic Engineering, Vol. 27, p. 293 (1982).

8. Materials at Low Temperatures, R. P. Reed and A. F. Clark, Eds., (American Society for Metals, Metals Park, OH, 1983).

9. A.C. Rose-Innes, Low Temperature Laboratory Techniques, (English Universities Press, London, 1973).

10. J. M. Gere & S. P. Timoshenko, Mechanics of Materials, 3rd Ed., (PWS-KENT, Boston, 1990).

11. M. N. Wilson, Superconducting Magnets, (Oxford University Press, London, 1983).

12. Experimental Techniques in Condensed Matter Physics, R. C. Richardson and E. N. Smith, Eds., (Addison-Wesley, New York, 1988).

13. R. B. Scott, Cryogenic Engineering, (Met-Chem Research Inc., Boulder, 1989).

14. F. Pobell, Matter & Methods at Low Temperatures, 3rd Ed. (Springer, Berlin, 1992).

15. T. M. Flynn, Cryogenic Engineering, (Dekker, New York 1997).

16. J. W. Ekin, Experimental Techniques for Low Temperature Measurements, (Oxford University Press, New York, 2006).