

Picking the Perfect Cryostat

If you want to probe the quantum behavior of a material and search for minute effects that are completely masked at room-temperature, or if you simply want to improve the properties of a device by cooling it below room temperature, you need to know cryogenics.

The most common cryogens into which you can dunk your sample are liquid nitrogen and liquid helium. Liquid nitrogen costs about half as much as gasoline, while the price of liquid helium is ten to fifteen times as much. A liter of liquid nitrogen absorbs about 60 times the amount of energy (heat) as a liter of liquid helium. If you do not need temperatures below 77 K, choose liquid nitrogen.

You could also start with a mechanical cooler, if you want to avoid the costs associated with consumable cryogenics. You will need a hefty down payment, but then you only need to worry about your electric bill and an occasional refurbishing charge (every 1,000-10,000 hours). This choice typically means you have to live with the vibrations associated with such a cooler, or try to eliminate them.

If you need to use a high-field superconducting magnet (5-20 Tesla), or have interests in Josephson junctions, quantum fluids or other very-low-temperature phenomena, go with liquid helium and a serious search for an even lower temperature system.

You will soon learn that thermal isolation is required to separate the cold experimental region from the room-temperature (and atmospheric pressure) environment of the laboratory. In simpler cases, a Styrofoam container or permanently evacuated jacket in a glass thermos may provide sufficient insulation. For more serious—and more rugged—applications, metal containers with surrounding vacuum jackets are preferable.

The conductive heat load resulting from gases that make contact with both the cold region and the room-temperature jacket produces undesirable moisture condensa-

tion around the outside of the jacket and results in an unacceptable heat load on the cryogen reservoir or refrigerator cold stage. At room temperature, a pressure of 10^{-4} torr results in a mean free path for the gas molecules of approximately 100 cm, much larger than the typical separation between the outer and inner walls of most cryostats.



A dilution refrigeration insert for operation below 10mK, with turbomolecular pumps

This is the region of molecular flow, where the dominant type of collision processes are between the molecules and the walls of the vacuum jacket, and the thermal conductivity of the gas is proportional to the pressure. A helium gas pressure of 10^{-5} torr will add a few milliwatt extra load on your helium

reservoir from a surrounding nitrogen jacket. You may decide it is critical to reduce the pressure inside the vacuum jacket to the 10^{-5} or 10^{-6} torr region.

Fortunately, a variety of pumping stations are available for obtaining these low pressures. Most use a diffusion pump or turbomolecular pump backed by a rotary pump. A liquid-nitrogen cold trap prevents oil vapors from backstreaming into the vacuum jacket. In some systems a “getter” (charcoal or a molecular sieve) is built into the vacuum jacket to maintain the vacuum when the pumping station is disconnected. If you are very meticulous (that is if your experiment requires it), you may want an ultra-high-vacuum (UHV) environment.

Dewars

The containers used for storing cryogenics are known as storage dewars, and experimental devices are known either as dewars or, particularly when they are capable of variable temperature operation, cryostats.

Liquid helium and liquid nitrogen dewars typically consist of one or more reservoirs surrounded by a vacuum jacket that isolates the reservoirs from room temperatures. Although some dewars are manufactured from glass or epoxy-fiberglass and aluminum, the most reliable ones are made from stainless steel, which is very rugged, has relatively low thermal conductivity and can be joined easily and permanently to similar or dissimilar metals by welding or silver soldering. These joints can withstand many cycles between room temperature and helium (or nitrogen) temperature and still remain leak-tight after years of use.

Particular applications can dictate the choice of materials from which dewars are constructed. Special dewars have been made for flight experiments (balloon, airplane, space shuttle or rocket), for operation in specific types of radiation environments (nuclear or high energy) and for magnetic or radio frequency shielding.

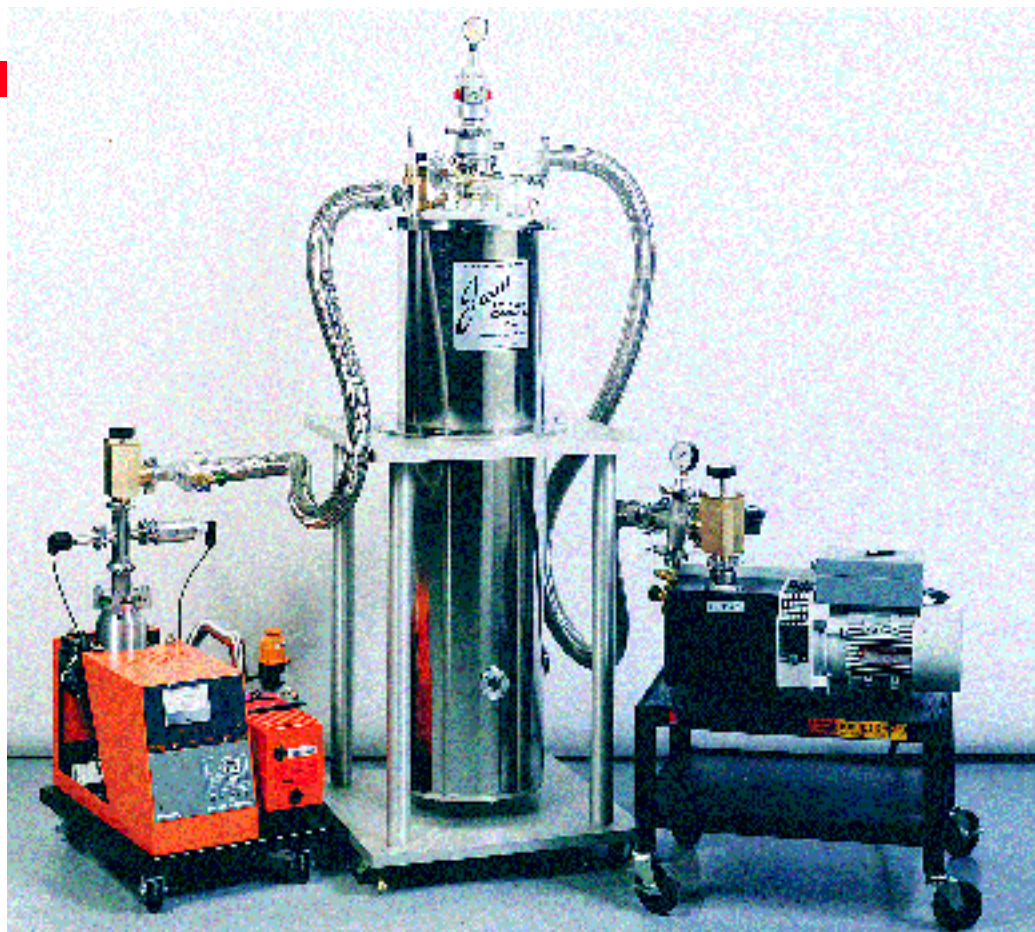
Immersion dewars

The simplest dewars have an all-welded construction with direct access to the cryogen reservoir through the top of the dewar. These simple dewars are for storage or for directly immersing the sample into the cryogen. They are equipped with both an evacuation valve for pumping out the space surrounding the cryogen reservoir and a safety pressure-relief valve to protect the vacuum jacket should an internal leak develop. Such a leak allows the cold cryogen to enter the vacuum jacket, then warm up and expand after contact with the room temperature wall, resulting in a high pressure buildup in the vacuum jacket. A pressure relief valve vents the leaking gas safely.

Liquid-nitrogen-shielded, liquid-helium dewars often have two interconnected vacuum jackets surrounding the helium and nitrogen reservoirs. In these dewars the liquid helium reservoir will be surrounded by a liquid nitrogen reservoir to reduce the radiational heat load on 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 multilayer insulation, which surrounds the cryogen reservoir (or inner line), and only one vacuum jacket. The floating radiation shields of this insulation reduce the radiational heat load into the liquid helium space, as the inner layers are cooled by the escaping helium vapor. Liquid nitrogen dewars contain only one vacuum jacket and may also contain multilayer insulation for reducing the ambient radiational heat-load onto the liquid nitrogen reservoir.

For measurements made at the normal boiling point of the liquid one can simply dip the sample into the reservoir. Reducing the pressure over the cryogen with an appropriate mechanical pump can produce a modest reduction in temperature, lowering liquid helium to approximately 1 K and nitrogen to 60 K. The insert supporting the



sample being cooled is made from a low-thermal-conducting material (stainless steel or epoxy fiber) and fitted with appropriate radiation baffles to reduce cryogen consumption.

Alternatively, one may place the sample in an evacuated chamber, where it makes no direct contact with the cryogen. Thermal linkage between the sample and the cryogen may then be established (for instance, through an exchange gas introduced into the vacuum chamber), and heaters and thermometers attached to the sample holder to vary its temperature. More sophisticated variable-temperature inserts channel the cryogen from the main reservoir into a vacuum-jacketed insert, allowing a wider range of temperature variation with more efficient operation. Special thermometry and automatic temperature controllers are readily available for controlling the temperature of the sample to various degrees of accuracy. The sample also may be located in vacuum, as required by the test being performed.

More sophisticated dewars are made with detachable bottom flanges that include an O-ring-sealed room-temperature flange and

A superconducting magnet system for magneto-optical experiments, with attached pumping station for dewar evacuation.

an indium-sealed helium-reservoir flange. This design allows the user to attach a number of different “tails” to the same dewar for various applications. Immersion tails and tails with the sample in vacuum are used for constant-temperature operation. For variable-temperature applications, one can choose between thermal impedance tails, exchange gas tails and continuous flow tails. Optical access to the sample is offered in a variety of window materials, sealing techniques and transmission ranges.

Magnets

Immersion and detachable-tail cryostats are also designed to accommodate superconducting magnets, which produce magnetic fields from 1 to 20 Tesla, in a laboratory-size dewar. NbTi coils produce fields up to 9 Tesla. Higher-field magnets use multifilamentary, Nb₃Sn inner coils, surrounded

by outer NbTi coils. The coils are available with simple immersion systems, room temperature bore systems and variable-temperature systems. Split coils that allow optical access to the sample are also available for a variety of magneto-optical experiments. A lot of effort has been focused recently on developing high-temperature superconduct-

ing coils, but these are still largely in the development stage, limited to lower fields.

As with most cryogenic systems, the final product is dictated by its application. Standard magnet systems are now available for transport and Hall effect measurements, nuclear magnetic resonance studies, Mössbauer spectroscopy, magneto-optical stud-

ies and much more. Other systems are designed for specialized applications. While most of these systems offer temperature variation between 1.4 K and room temperature, ultra-low-temperature systems that reach a few milliKelvin or lower are also available. He³ cryostats offer temperatures down to 0.3K, by condensing the He³ gas in contact with a pumped He⁴ reservoir (or 1-K “pot”), then reducing the pressure on top of the He³ reservoir. An internal charcoal sorption pump or an external (sealed) pumping system is used to contain the precious He³ gas.

Dilution refrigerators use the unusual properties of He³-He⁴ mixtures below 0.85 K (where phase separation occurs) to reach temperatures as low as 1.9 mK. This is done by removing He³ gas from the He⁴-rich phase of the mixture at about 0.6-0.7 K, and continuously re-cooling and re-condensing this gas into the He³-rich phase in the mixing chamber. This process forces the He³ to “evaporate” from the He³-rich phase into the He³-dilute phase, thereby cooling the mixing chamber to a few mK. The ultimate temperature and cooling power of such systems is a function of the heat exchangers, gas circulation and overall thermal isolation of the cryostat.

Continuous transfer

In many cases it is preferable to eliminate the research dewar due to a lack of space or a desire for a more portable cryostat. For these applications, an efficient vacuum-insulated transfer line with a flexible section carries the cryogen from a storage dewar to a small cryostat. A flow control valve is usually built into the transfer line to govern the cryogen flow to the cold end. The cryostat itself generally has its own vacuum jacket and sample chamber and, as in other cryostats, allows sample placement in a vacuum (or UHV), in a static gas or in a flowing vapor. Optical access to the sample is provided in much the same manner as in the cryostats that are based on detachable tails.

Most of the continuous transfer systems are provided with quick-loading sample

holders and with electrical and optical access to the experimental cold end. In general, dewar-type cryostats have a lower consumption rate, but require an initial charge of cryogen (typically about 5 liters of liquid helium), making them more efficient for longer experiments (8 hours or more). Continuous transfer cryostats, however, use relatively less cryogen in shorter experiments (about 4 hours or less).

While it is possible for some laboratories to design and make their own cryostats, it is usually easier and less costly (especially without the free labor of a graduate student), to seek out the appropriate commercially available system. With established designs or specialized innovative designs, the industrial physicist has much from which to choose.

Further reading

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CRYOSTAT SUPPLIERS

The following is a partial list of cryostat manufacturers. For a more complete listing, refer to the *Physics Today Buyers' Guide*:

Abbess Instruments, Inc.
American Magnetics, Inc.
APD Cryogenics, Inc.
Applied Engineering Technologies Ltd.
Balzers
CRYO Industries of America, Inc.
Cryomagnetics, Inc.
Cryomech, Inc.
CVI, Inc.
George Associates
Janis Research Co. Inc.
Kelvin International Corp.
Oxford Instruments, Inc.
Pope Scientific, Inc.
Quantum Design, Inc.
RMC, Inc.