An Expert's Guide to Cryostats

Author: Simon Mitchinson

andor.oxinst.com

What is a Cryostat?

A cryostat is a vacuum insulated sample environment that uses liquid cryogens, such as nitrogen or helium, or a mechanical cooler to cool the temperature of a sample. Base temperatures of 77 K (nitrogen) or <4 K (helium) can be reached depending on the boiling point of the cryogen used. Accurate temperature control can be achieved by combining the cooling power of the cryogen, or cooler, with a voltage applied across an electrical heater element.

There are lots of different types of cryostats and lots of things to consider when purchasing an optical cryostat system, which can seem overwhelming. For example, cryostats can have different shapes, different numbers of windows, different window types, different cooling regimes and can be better suited for specific experiments or samples. Samples can be held in different environments, either in an exchange gas or under vacuum.

In addition, cryostats can use different mechanisms for cooling, for example, using a liquid cryogen such as nitrogen or helium or a cryogen free system. Over the remainder of this article, we will expand on these points and answer questions to explain the different options available.

What are Cryostats used for?

Cryostats have a wide range of applications to cool liquid, powder, or solid samples for spectroscopy and microscopy experiments.

Microscopy Cryostat Applications	Spectroscopy Cryostat Applications
• Ultraviolet-Visible Spectroscopy	• THz spectroscopy
• Micro-Raman	• UV/Visible reflectivity & absorption
• Micro-FTIR	Photo/Electroluminescence
• Micro-photoluminescence	• Raman Scattering
• Fluorescence Spectroscopy	• Electrical transport measurements
	• Ultrafast spectroscopy

What are the types of cryostats?

- **Bath cryostats** contain enough liquid cryogen to allow for an adequate experimentation time, with no need to continuously refill from a storage Dewar.
- **Continuous flow cryostats** are cooled by a continuous flow of liquid, fed from an external transport Dewar.
- **Dry cryostats** or cryogen free cryostats use a mechanical cooler to cool the sample.

What is a liquid cryogen?

A liquid cryogen is a gas, cooled to below its boiling point and stored in a vacuum insulated transport vessel. They can be used to cool to extremely low temperatures, <4.2 K for Helium and 77 K for Nitrogen, for cooling experiments or storing samples. To work out the cost of running your cryostat with helium please use our helium cost calculator.

What's the difference between a nitrogen and helium cryostat?

Nitrogen and helium have different physical properties and as such the design of cryostats will vary accordingly.

Liquid nitrogen (LN2) will exist in an uninsulated vessel for some time; however, it will eventually boil away. It will also result in icing around the vessel as water vapour freezes. Nitrogen cryostats therefore have a vacuum space to reduce the boil-off and isolate the outer surfaces from the cold liquid. The boiling point of LN2 (77 K) is not cold enough to freeze the gases left in the vacuum space (cryopump) and so a charcoal sorption pump is fitted within the space to improve the vacuum.

Liquid Helium (LHe) is more difficult to store without losing much of the liquid due to radiated and conducted heat loads. For small cryostats it can be more efficient to store the liquid in an external transport Dewar and supply the helium through a “low loss” transfer line. In addition to the vacuum space between the sample and room temperature, LHe cryostats also have an intermediary radiation shield to intercept radiated heat loads from room temperature (300 K). When used for optical cryostats this requires a window in the shield in addition to the outer casing.

What's the difference between a wet cryostat and a dry cryostat?

A liquid cooled cryostat uses liquid cryogens, such as nitrogen or helium, to lower the temperature of a sample. These cryostats either hold the liquid within the unit (bath cryostats) or the liquid is fed through the cryostat from an external transport vessel (flow cryostats).

Dry or cryogen-free cryostats do not require any liquid cryogens at all. They use a mechanical cooler to lower the temperature of the sample space. These are typically GM coolers (Gifford-McMahon) but can also use PTR (Pulse Tube Refrigerator) coolers.

What are the advantages of dry cryostats over helium cryostats?

Dry or cryogen free cryostats do not require any liquid cryogens at all. Although a dry or cryogen free cryostats can cost more to purchase initially, they can have a lower cost of operation because they do not require liquid helium to run.

Alternatively, a helium cryostat will require a set up like that outlined in figure 1 below, with a cryogen storage Dewar, transfer tube, and a system for helium recovery.

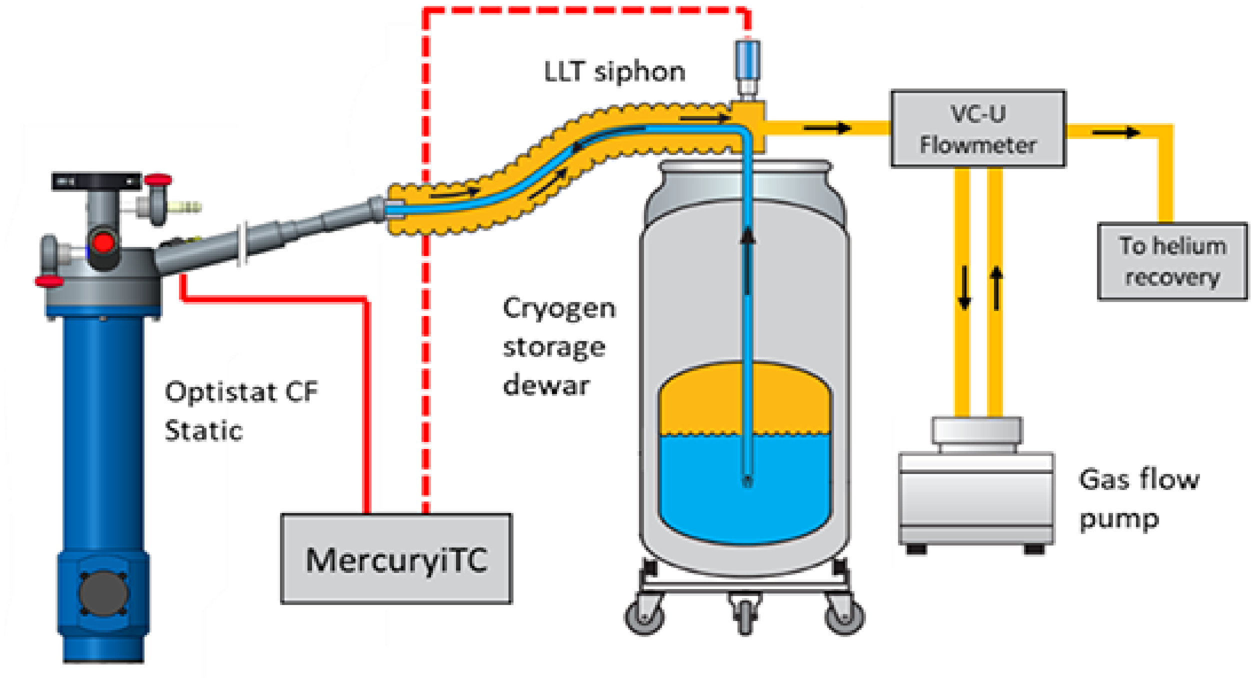


Figure 1: Helium cryostat experimental set-up.

When considering the running costs of a Dry system vs. a wet one, you should consider the infrastructure required to obtain, store and manage liquid gases. Although these costs do not apply to dry systems, there is an electrical supply cost associated with the compressor (3-6 kW) and the need to cool the compressor with either a water chiller or fan.

How much does a cryostat cost to run?

This will depend on the type of cryostat you have. For example, running a helium cryostat will cost more than a dry or cryogen free model over the lifetime of your experiment. To work out the cost of running your cryostat with helium please use our helium cost calculator.

How do you control the temperature of a cryostat?

Cryostats can typically be purchased with a temperature controller with a proportional-integral-derivative (PID) controller. For example, Andor's cryostats can be purchased with the Mercury iTC controller with one sensor/heater PID loop as standard. The controller works with the heat exchanger in the cryostat and heating/cooling is balanced via the voltage across the heater with the flow of the cryogen or coolant.

One of the advantages of using a good, high-quality controller is that it will consistently monitor the temperature, the cryogen flow, and the voltage. It will slowly lower the voltage and the flow so that over time the cryostat becomes more efficient, consuming the minimum amount of cryogen.

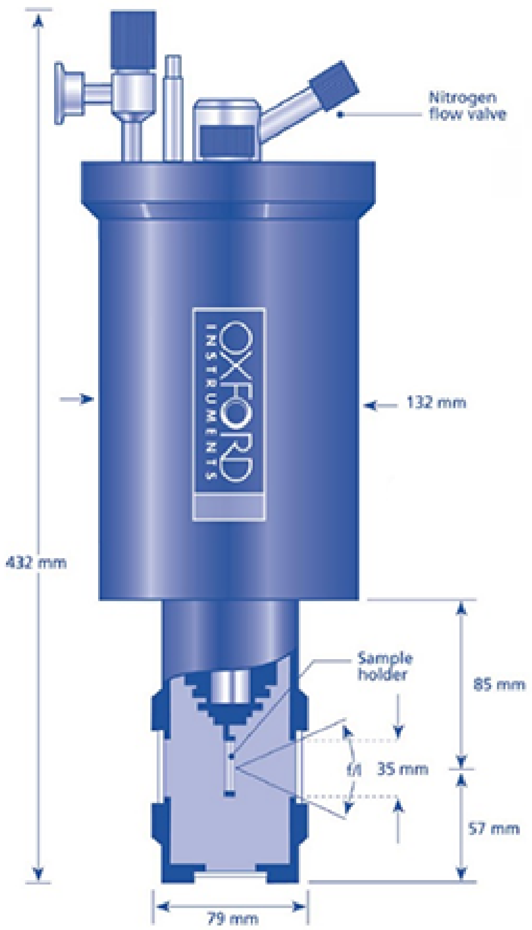
How to choose between a cryostat with the sample in an exchange gas or in vacuum?

Cryostats can be designed to mount your samples in an exchange gas or within the isolation vacuum. Different types of samples are better suited to different environments. For example, liquid samples, or powders, can be held in a cuvette and cuvette holder and then loaded into a sample tube filled with an exchange gas (typically a small amount of helium gas). It would be impractical to load liquid samples into a vacuum space. Advantageously in the exchange gas the sample can be changed very quickly by simply lifting it out. It only takes a couple of minutes. The presence of the gas also aids in uniformly cooling the sample.

One downside to having a sample tube with an exchange gas is the need for an additional window between the sample and the vacuum space, as shown in figure 3 below. There are three windows between your sample and the outside world for a helium cryostat with a sample in exchange gas. First there is the sample tube with an inner window, then the radiation shield with the middle window and then the outer vacuum casing with the outer window. Note there is no radiation shield on nitrogen cryostats, so only two windows are required.

For sample-in-vacuum systems it takes a little longer to change the sample as the whole cryostat must be warmed to room temperature first. Liquid samples cannot be used, but there are less windows in the beam path. To cool powdered samples in a vacuum cryostat, they can first be compressed into pellets before mounting.

Sample Type	Sample Exchange in Gas	Sample in Vacuum
Powder	Yes	(As compressed pellets)
Liquad	Yes	No
Solid	Yes	Yes



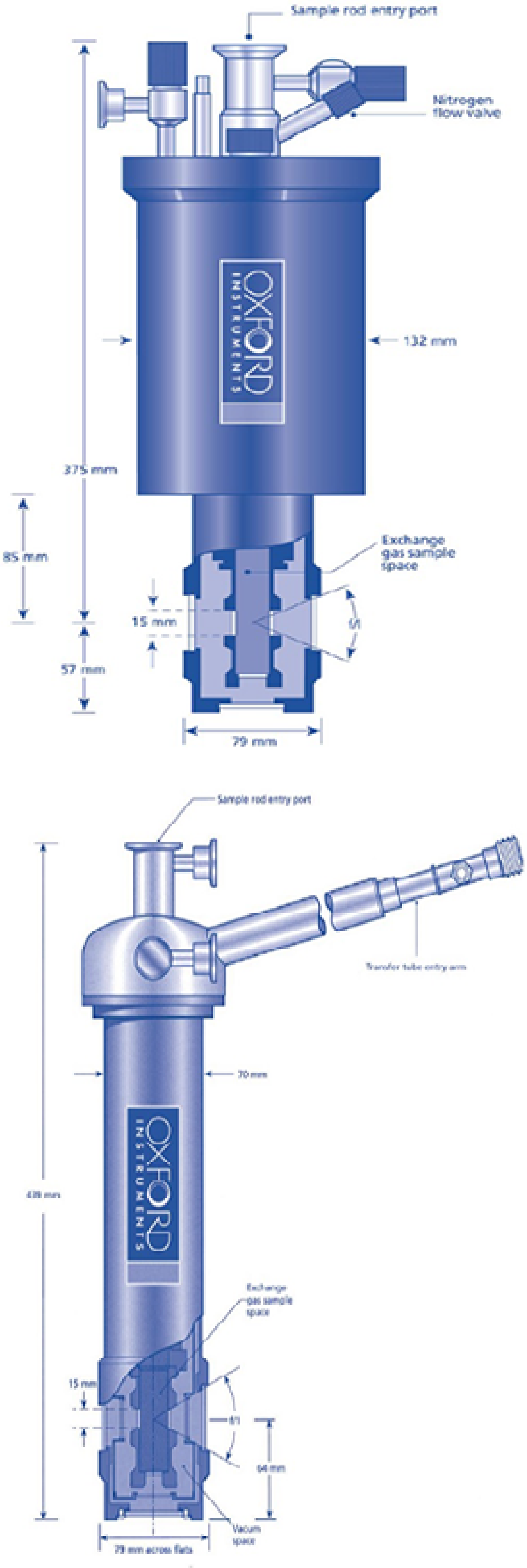


Figure 2: Top: sample in vacuum (1 window). Middle: Schematic of nitrogen cryostat with sample in exchange gas (2 windows). Bottom: Schematic of helium cryostat with sample in exchange gas (3 windows).

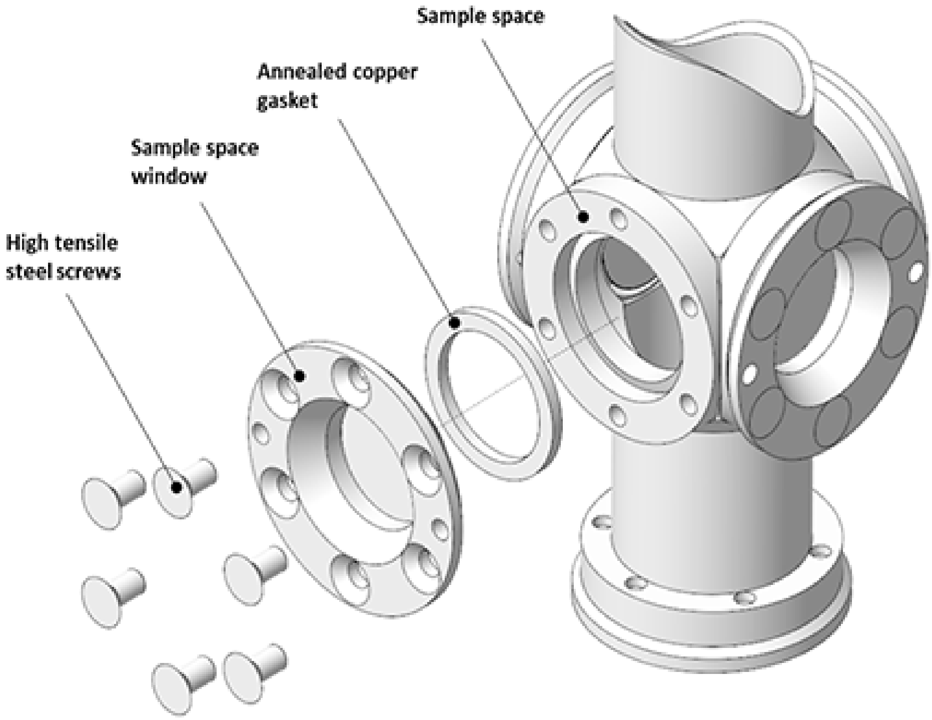
What is a cryostat window?

The window on a cryostat forms a 'transparent' barrier between the sample environment and either the vacuum space or outside environment. Every window type has its own transmissive properties and is 'transparent' to a unique portion of the light spectrum, so it is important to check that the window type is suitable for your experiment.

Why are there multiple window options for a cryostat?

Different window options are required for different experimental needs. For example, some researchers may need a window with high transmission in the far infrared, or across the spectrum. Choosing a window will depend on your individual experimental requirements and the budget available. Material options can include, Spectrosil B or WF, crystalline quartz, sapphire, polythene, KRS-5, zinc selenide, calcium fluoride and Mylar.

Andor offer a range of window options for both our Optistat and Microstat ranges, that cover a wide range of wavelengths and materials. Please see our Optistat and Microstat specifications for more information. Inner, middle and outer window fittings are shown in Figure 3.



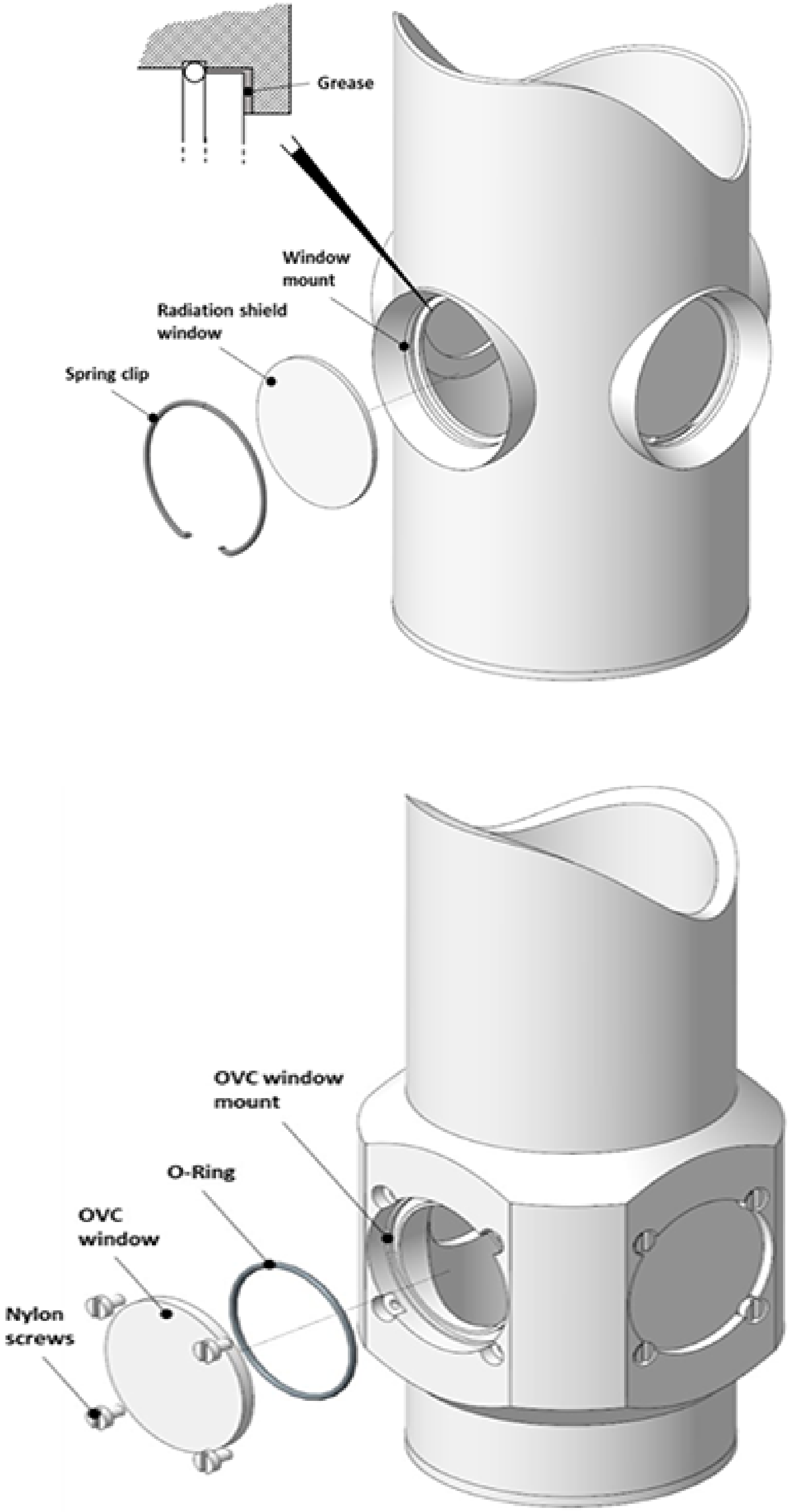


Figure 3: Top: Inner window mounts. Middle: Middle - radiation shield - window mounts. Bottom: Outer window mounts

Are Optistat windows interchangeable?

Yes, for Andor's optical cryostats the windows are interchangeable and can be changed by our customers themselves. However, this will depend on the manufacturer of your cryostat so ensure to check with your supplier.

How much does a cryostat cost?

Cryostat costs can vary depending on the type and methods used for cooling and the windows used. Costs can vary from £5-6 K GBP for simple nitrogen bath cryostats to £15 - 20 K for helium flow cryostats. Cryogen free cooler systems can cost upwards of £30 - 60 K depending on design and performance.

Why are dry cryostats more expensive to buy than helium cryostats?

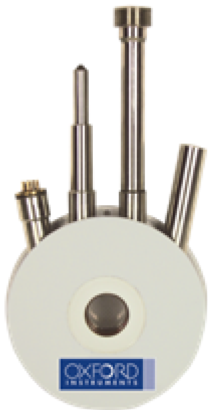
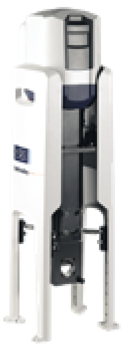
All cryostats have costs associated with the mechanical build, materials and control electronics. Dry (cryogen free) systems have the additional cost of the GM cooler and compressor. This can often be the largest cost of a new cryostat.

What is the difference between Andor's Optistat and Microstat ranges?

The Optistat range is suitable for spectroscopy whereas the Microstat range is suitable for microscopy, an overview of the product range is shown in the table below.

Our Optistat range for spectroscopy includes nitrogen cryostats (DN-V or DN-X) liquid helium-flow cryostats (CF-V or CF-X), and two cryogen-free, or Cryofree® systems. These are all designed for general spectroscopy. They all have the same optical tail design forming a cube with five windows to a sample position. Four radial and one on the bottom. This design allows for good all-round optical access to either excite your sample with light or measure the light that is emitted.

Our Microstat range has a nitrogen (N), and two helium-flow cryostats (He and HiRes). They work in the same way as the Optistat range; they are just a different shape and size. These are designed to get your microscope objective as close to your sample as possible. So, with these systems there is a single window in the top and one in the bottom for transmission, allowing a close-up of the sample within a couple of millimetres. All samples in the Microstat range are held under vacuum.

	Spectroscopy (Optistat range)		Microscopy (Microstat Range)	
	Sample in vacuum	Sample in exchange gas	Sample in vacuum with standard vibration	Sample in vacuum with low vibration
Liquid Nitrogen				
Liquid Helium				
Cryogen Free				

Common acronyms used with cryostats

PID controller	Proportional-integral-derivative controller
OVC	Outer vacuum casing
LN	Liquid Nitrogen
LHe	Liquid Helium
LLT	Low Loss Transfer
ITC	Intelligent Temperature Controller

How to Use a Cryostat

A cryostat is a vacuum-insulated vessel with a sample environment that can be cooled to cryogenic (very low) temperatures, typically measured in Kelvin. Cryostats can use liquid cryogens, such as Nitrogen or Helium, or a mechanical cooler to achieve low temperatures. Samples are mounted either on a Copper heat exchanger or placed in a sample tube filled with cooled gas (typically Helium). The outer vacuum casing protects the sample. There is often a second, inner radiation shield to further insulate the sample from radiated heat, helping to maintain the lowest temperatures.

Cooling is achieved by either flowing liquid cryogens through the copper heat exchanger or by mechanical contact with a mechanical cooler such as a Gifford McMahon (GM) cooler.

The temperature is controlled by balancing the cooling effect of the flowing cryogen with a voltage across a heater and by monitoring the temperature of the sample with a sensor.

You can vary the voltage across the heater and then increase or decrease the flow of cryogen to achieve a balance and maintain temperature stability. This is usually automated with the use of an electronic temperature controller. Temperature controllers use a simple PID loop to achieve temperature stability and manage temperature ramping, minimising temperature overshoot and maximising the efficient use of the cryogens.

The two major cryogens used are liquid Nitrogen, which is cheap, easy to source and gives you a base temperature of 77K, or liquid Helium, which has a boiling point of 4.2 degrees Kelvin or 4.2 degrees above absolute zero.

By using vacuum pumps to reduce the pressure over the liquid, you can decrease the temperature further to about 1.5 degrees Kelvin for liquid Helium. Cryostat designs also exist that use 3He or a mixture of 3He and 4He to achieve temperatures as low as 10 milliKelvin.

Liquid Nitrogen Bath Cryostats

These types of systems are straightforward and simple to use. First, we need to evacuate the isolation space – the space isolating the sample from the outside world. The air is pumped out using a turbo pump to reach a base pressure of 10-4 to 10-6 mbar. Once the vacuum space has evacuated, the cryostat can be cooled, usually by pouring liquid Nitrogen into the bath using a funnel. Once cold you can connect the temperature controller and set the temperature for your sample space. The Nitrogen flow is gravity-fed to the heat exchanger using a thumbwheel connected to a needle valve. Opening the valve will create more flow and cool faster. Closing the valve down will reduce the flow to maintain set temperatures. The set temperature and stability is maintained by the temperature controller, which varies the voltage across the heater according to the sensor reading at the sample position.

Liquid Helium Systems are a Little More Complicated

With liquid Helium systems, again the vacuum space must first be evacuated using a turbo pump or similar. Helium is transferred to the cryostat from a storage vessel (dewar) using a vacuum insulated transfer line. A small flow pump is used to draw the helium through the system. Once the "spent" helium gas has passed through the cryostat it is usually vented to a helium recovery system where it can be cleaned, compressed and re-liquified for re-use.

Helium flow can be controlled either manually with a needle valve or, in some cases, the temperature controller can vary the flow using a stepper motor connected to the needle valve. Again, voltage is varied by the controller to maintain stable set temperatures. The use of automated gas flow can vastly improve the cost and efficiency of the cryostat by minimising the volume of helium required to maintain the target temperature.

Health and Safety Considerations

There are important health and safety considerations when using liquid cryogens. Firstly, cryogens cause burns. You will never see liquid Helium in the atmosphere because it can't exist as a liquid above 4 Kelvin. You can see it inside a cryostat with glass windows, but outside of the cryostat it will never be in liquid form. It's very, very cold. Anything that has been cooled by liquid Helium will be cold enough to cause burns as skin sticks to the cold surface. Serious burns can occur faster than your ability to react to contact.

Oxygen and air near cold surfaces can become Oxygen-enriched as Oxygen liquefies in contact with anything at those temperatures. This can pose a fire hazard, especially in the presence of oil or grease on nearby surfaces.

Asphyxiation hazards: If you are using liquid cryogens in a laboratory or room, storage dewars or cryostats will slowly boil-off Nitrogen or Helium gas. One litre of liquid Nitrogen will evolve into 690 litres of gas at room temperature. Over time this has the potential to displace the air in the laboratory resulting in a lack of Oxygen and danger of death. It is imperative to ensure good ventilation, especially in enclosed spaces such as optics labs. The use of oxygen depletion monitors is also highly recommended. These can be fitted into the laboratory and/or carried by all staff who enter.

Can You Damage the Sample?

It depends what the sample is. Thermal shock is a risk for delicate samples. It is important to consider how quickly the sample will be cooled down and warmed up again. There is the presence of Helium, which, though inert, may affect your sample if it is prepared in another gas, or a vacuum. Then there is moisture. If you take a cold sample out before it has thoroughly warmed up, water will condense on the sample, which could damage it.

What Type of Experiments are Performed with Cryostats?

We can study the electrical and physical properties of materials that change with temperature. We might want to cool the environment down so that our sample signal can be dominant – increasing the signal to noise ratio. We may also wish to observe electrical transition states at different temperatures.

We can use cryostats for infrared spectroscopy for polymer research, inorganic chemistry, medicinal chemistry and solid state and semiconductor physics, for example. Raman spectroscopy is used to analyse organisms and tissue and UV-Vis spectroscopy in analytical chemistry.

It is easier to detect photoluminescence and fluorescence at low temperatures. Photoreflectance is used to look at the electronic structure of semiconductors. Andor's cryostats are used in a huge range of applications around the world.

What is Cryostat Sectioning?

This is when people take biological samples and slice them into very thin sections, analyze them and do assays. You would use large cryogenic fridges to store biological samples or liquid nitrogen vessels. This has nothing to do with our optical cryostats!

How Do You Cool a Liquid Sample?

For liquid samples we supply systems with cuvette holders which can hold stoppered cuvettes. They can be put into cryostats that use an exchange gas sample environment. Exchange gas cryostats have a sample tube that is filled with a small volume of Helium gas. This gas is cooled by the cryostat. By inserting samples into the gas filled sample tube you can cool liquids, powders or solids. Cold gas cryostats are also good for cooling samples with poor thermal properties, bathing them completely in a cold gas.

How Do You Cool a Powdered Sample?

On standard sample holders, even in vacuum sample spaces, it is possible to compress powders into pellets under high pressure and then mount those pellets into a sample holder. Alternatively, bulk powder samples can be held in cuvettes, as with liquid samples.

Things to be aware of are:

- Good health and safety.
- Good PPE.
- Good preparation – have everything ready before you bring the cryogen to the cryostat particularly with Helium systems.
- Be aware and careful. There are other risks such as having glass windows under vacuum. It is possible to be clumsy and break a window so you lose your vacuum. The liquid cryogens in the system will boil very quickly and the problems will snowball. You need to be aware and careful.

Where Do Andor's Cryostats Excel?

Andor's product range excels in its very efficient use of cryogens. Our helium cryostats achieve low temperatures with a minimal use of Helium. Our low loss transfer tube technology uses the spent gas to precool the radiation shields in our transfer lines. This improves transfer efficiency and reduces helium losses. This means that the cost of ownership of our cryostats is very low. The initial outlay can be a little more, but over one or two years the cost of operation can typically be half that of a competitor's system. In the medium and long term the real cost of ownership of our systems can be considerably better.

Our Optistat Specification Sheets

Full details of our cryostat range can be found on our specification sheets, available on our website. We have a range of cryostats using Nitrogen, Helium or GM coolers.

An example is OptistatDryTM which is designed for ease of use with free-space optics. You can very precisely align your cryostat and sample position, changing samples in and out numerous times without moving the cryostat or disrupting the alignment of your experiment. Changing samples with minimal impact on optical alignment can be time saving and maximise efficiency.

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