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Safety interlock and vent system to alleviate potentially dangerous ice blockage of top-loading cryostat sample sticks

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A combined solution is presented for minimizing the safety hazards associated with closed cycle cryostats described by Swainson & Cranswick [*J. Appl. Cryst.* (2010), **43**, 206–210]. The initial solution is to install a vent tube with one open end deep inside the sample space and a pressure relief valve at the top. This solution works for either a cryogen or a cryogen-free (closed cycle) system. The second approach, which can be combined with the first and is applicable to cryogen-free cryostats, involves electrically interlocking the closed cycle refrigerator compressor to the sample space, so that the system cannot be cooled in the presence of a leak path to air.

1. Introduction

Top-loading cryogenic systems are built to work at low temperatures while affording the ability to insert new samples or apparatus *via* a top-loading sample stick. The ability to change the sample stick in these systems without warming up the entire cryostat provides a great deal of convenience, flexibility and time saving. As reported by Swainson & Cranswick (2010) (hereafter SC) there are inherent risks and safety implications when operating a top-loading cryostat. The authors have first-hand experience of the destruction caused by the pressure that arises when heating a sample space in the presence of an ice blockage with trapped liquids/solids: a top-loading sample stick that buckled under the forces exerted on its radiation baffles from the trapped liquid/solid becoming gaseous is shown in Fig. 1. Additional scenarios described by SC and occurrences related by neutron scattering user facility colleagues are rocket-like ejection of unsecured sample sticks or sample sticks acting as flails if secured by cables.

The mechanism by which blockages occur in top-loading cryostats is common to both cryogen and cryogen-free versions. Air enters the system through a leak or opening in the sample space. This moisture-laden air encounters cold regions of the sample space and the water vapour in the air condenses, forming ice. At the same time, other components of the air, primarily nitrogen and oxygen, condense onto yet colder regions of the sample space found near the bottom where the sample is located. Since the sample space of the cryostat is operating at cryogenic temperatures, cryo-pumping on a persistent leak and condensation of the incoming air can lead to the accumulation of a considerable amount of water (freezing point 273 K), nitrogen (boiling point 77 K) and oxygen (boiling point 90 K). The construction details of top-loading cryostats provide the remaining ingredients that lead to the evolution of a dangerous condition. With an ice blockage above, heating of the sample space leads to evaporation of the cryogenic liquids below, with a resulting increase in volume by a factor of 700–1000 (at atmospheric pressure). Within the fixed volume of the sample space, pressures build dramatically and lead to the dangers discussed by SC.

The top-loading cryostats used for neutron scattering generally operate in a temperature range of 4–300 K and up to 700 K for cryogen-free cryo-furnace systems. Samples used in a top-loading cryostat are mounted on the end of a hollow thin-walled aluminium or stainless steel tube, the sample stick, which typically incorporates a multi-use O-ring seal system located at the top of the sample stick used for height adjustment. Failure of this particular seal is among the most common ways that leaks into the evacuated sample space occur. This is a result of the inability to replace or perform any maintenance on the O-ring itself, which is inherent in the design of most top-loading sample sticks. The sample space is typically fitted with a pressure relief valve (0.1–0.3 bar; 1 bar $\simeq 10^5$ Pa), located near the top of the sample stick, and a manual valve system to evacuate and backfill the sample space with thermal exchange gas (omitted for clarity; Fig. 2). These two valves along with vacuum fittings and hermetic wiring feedthroughs are also a common source for leak paths, owing to either malfunctions [*e.g.* valve(s) may not re-seat correctly after purging], worn components or user error.

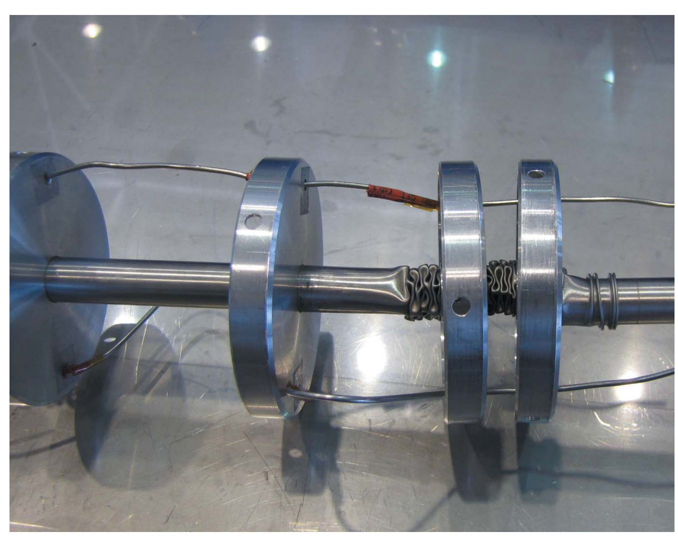

Figure 1
Example of damaged top-loading sample stick.

One of the design elements in these low-temperature cryostats is a thermal radiation shield that protects the colder-temperature sections from room-temperature thermal radiation. In a cryogen cryostat, this outer radiation shield is cooled by a liquid nitrogen reservoir (Fig. 2). In a cryogen-free cryostat, this outer radiation shield is cooled by the first stage of a two-stage cryo-cooler (Fig. 3). In both of these cases, the radiation shield is thermally anchored to the sample space toward the top of the cryostat. The radiation baffles on the sample stick form near-complete seals at intervals along the sample space. By design, one or more of these radiation baffles is in the vicinity of this thermally anchored section of the sample space. As a result, a relatively thin layer of ice can form around the baffles and completely seal off the lower section of the sample space. Even in such cases as when the baffles are not so tightly fitted, ice accumulation can be relatively rapid. In at least one case, staggered semi-circular radiation baffles in a 50 mm-diameter sample space allowed an ice blockage that developed overnight and sealed off the remaining open area.

2. Design description

2.1. Pressure relief system – a vent tube with PRV on top

NIST personnel devised and tested an improved venting system that greatly reduces the likelihood of a catastrophic build-up of pressure due to an ice blockage within a cryostat (Fig. 4). The easily implemented retrofit solution modifies existing top-loading sample sticks to incorporate a continuous vent tube that extends past the region where ice forms, with a safety pressure relief valve (PRV) at the top to release excess pressure. This venting design can be incorporated into new designs easily by utilizing the sample stick shaft as the vent tube. For reasons described in detail below, the most important characteristic of this vent tube is that it is continuous from the PRV to a point below the likely formation of an ice blockage.

Inspection of the representative cryostat schematics shows that air leaking into the sample space of a cryogen cryostat will first encounter a cold condensation region where the liquid nitrogen reservoir is coupled to the sample space (Fig. 2), or equivalently where the first stage is coupled for the case of a cryogen-free system (Fig. 3). We conjecture that practically all of the easily condensable gases/vapours will accumulate in this region because of the reduced convection owing to the influence of radiation baffles. Examination of multiple near-miss incidents with both cryogen and cryogen-free systems confirms that the only evidence of icing is found along this narrow region at the upper part of the sample stick. Therefore, a vent line that allows pressure from below the blockage to be relieved at the top of the cryostat should alleviate all but the most egregious icing conditions. The concern is whether condensable air can find its way to the vent tube and block this exit path simultaneously with forming the main blockage.

Two possible avenues exist for the safety vent tube to form an ice blockage. Both the opening of the vent tube and the thermally anchored region of the vent tube may become blocked. However, any

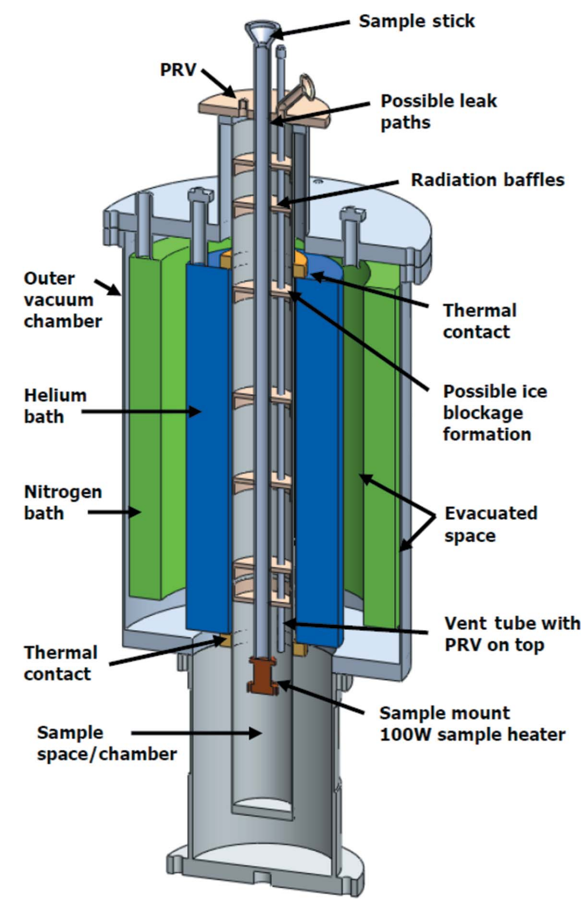

Figure 2
Schematic of a top-loading cryogen cryostat.

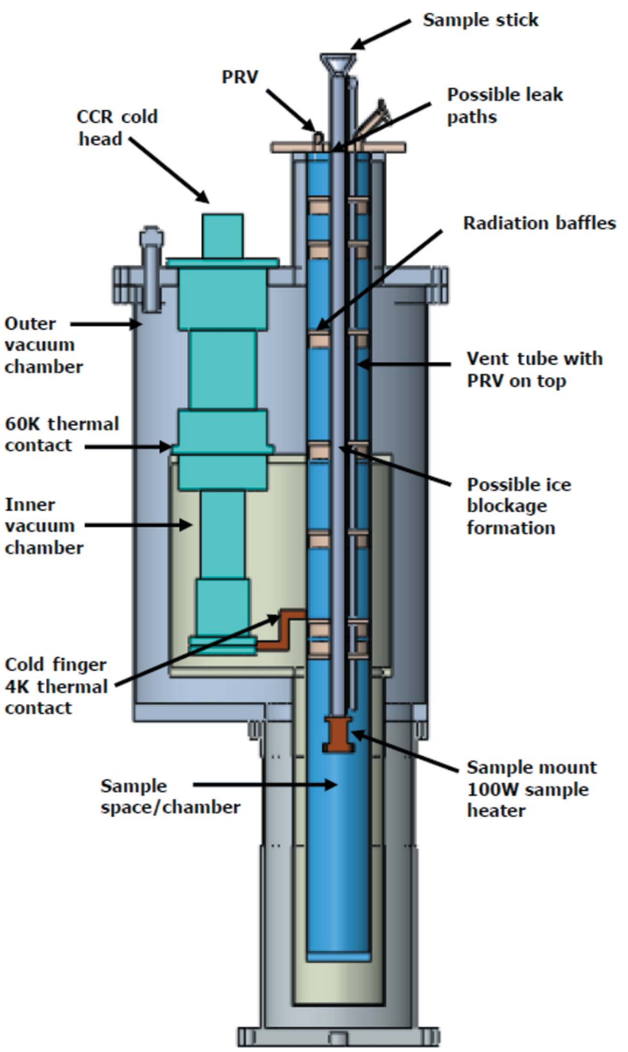

Figure 3
Schematic of a top-loading cryogen-free cryostat.

condensable matter that passes the thermally sunk section at the top will encounter a huge surface area from the walls of the sample space compared to the area represented by the opening of the vent tube. The open end of the vent tube will not accumulate any condensable matter preferentially, so only if the entire sample space is coated by a layer of ice will the end of the vent tube become blocked. Likewise, it is very unlikely that warm air could travel from the top of the sample space, past the cold section and into the vent tube, and finally condense inside the vent tube again at the cold section on the way up.

The assertion that the vent tube would not become blocked internally under expected use conditions was tested. To do so, the cryostat had to be prepared into a normally dangerous condition. A sample stick was manufactured that provided two vent tubes (Fig. 4). A generic sample space is shown with the prototype vent tube system installed on the sample stick. The vent tube on the left-hand side is open at the bottom, while the one on the right is outfitted with a cryogenic burst disc. They have identical 1.4 bar PRVs at the top (shown), as well as pressure gauges and fittings to allow addition/removal of gas (omitted for clarity). The burst disc was used to keep the interior of the second vent tube clear, in case the above argument was incorrect. A burst disc with a rupture pressure slightly above 1.4 bar was desired. Aluminium foil was sandwiched into a commercial compression seal to form the burst disc. While at room temperature this foil would burst at approximately 1 bar, when cooled in liquid nitrogen the burst pressure increased to approximately 2.8 bar, which was adequate for this test. A burst disc was not desired in the final version because a burst disc, if necessary, would have to be inspected before each use of the sample stick. These inspections, and possible repairs, are not advisable in a user facility.

Fig. 4(*a*) shows the system after sufficient air has entered to form a pool of liquid in the bottom of the sample space and a complete ice blockage around the baffles near the top of the cryostat. A user might notice a problem at this stage and turn on the heater thinking it will melt the blockage. This boils the liquid pooled at the bottom, but since the bottom is now isolated from the top of the sample space, a dangerous over-pressure condition is created. A pressure relief valve on the cryostat (not shown) would relieve this pressure in the absence of a blockage, but with a blockage present as in this case, such a PRV would not deploy. The pressure is vented through either vent tube as shown in Fig. 4(*b*).

The cryostat was cooled to base temperature and moist air was allowed to cryo-pump into the sample space freely. Ice built up over a matter of hours and blocked the sample space by sealing the edges around the radiation baffles. We judged that a complete blockage had been formed when helium introduced through the vent tube did not raise the pressure at the top of the sample space. The bottom of the sample space was then heated to 90 K. Pressure gauges on the two vent tubes and the top of the sample space were monitored. The pressure at the top of the sample space, which is the only gauge that is normally visible to a user, remained low owing to the cold sample space and cryo-pumping. The pressure in the vent tube gradually increased until the 1.4 bar relief valve released the pressure. The pressure in the vent tube with the burst disc also increased, but only because of the warming of the sample space. This test was conducted twice more with the same results. Several attempts resulted in weak blockages that initially held as the sample space was heated, but leaked before the pressure built to the vent tube relief valve pressure. In these cases, the standard PRV at the top of the sample space vented the excess pressure.

SC describe the blockage as probably coming from solid nitrogen. This scenario is not as likely as ice from water vapour in the air. In the case of a cryogen cryostat with a liquid nitrogen bath connected to the upper thermal contact, a solid nitrogen blockage could only occur further down where the sample space can reach lower temperatures. As stated above, our examination of multiple sample stick near-miss incidents does not support icing at the lower end of the sample stick. Water vapour, on the other hand, is frozen directly by the action of the liquid nitrogen bath (or first stage of a two-stage cryo-cooler for a closed cycle refrigerator system). Additionally, solid nitrogen has only a fraction of the strength of ice at these temperatures. While the specifics of the blockage geometry would be required for an accurate model, the relative strengths of the constituent materials should be indicative of their capabilities for forming a shear-resistant seal in this tubular geometry. At a temperature of 55 K, Young's modulus of solid nitrogen is 170 MPa (Norris *et al.*, 1997). At the similar temperature of 77 K, Young's modulus of ice is 10 000 MPa (Parameswaran & Jones, 1975). Solid nitrogen has only about 2% of the strength of ice. This strength argument, however, does not rule out the possibility that both materials may be capable of sealing to pressures that lead to damage like that shown in Fig. 1.

The vent tube system as described above works well over a wide temperature range. However, when implemented in a top-loading closed cycle refrigerator with a base temperature of approximately 4 K, thermoacoustic oscillations developed in the vent tube (see Taconis *et al.*, 1949). These oscillations transfer heat to the cold region of the cryostat, limiting its ability to reach base temperature. A ballast volume was added next to the pressure relief valve to prevent these Taconis oscillations. To date, this system has been adopted by three neutron scattering user facilities in the United States and has been incorporated into the standard designs for top-loading cryostats from at least three US manufacturers.

2.2. Safety interlock system for cryogen-free cryostats

The solution developed at NIST provides a high degree of probability that any pressure build-up can be safely vented. Even before a safety condition develops, however, the incoming air from a faulty

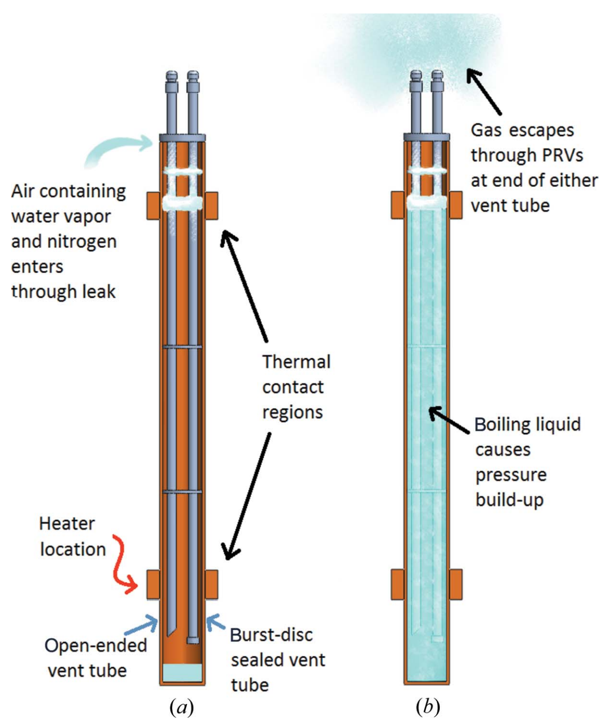

Figure 4
NIST prototype vent tube system – operation schematic.

setup contaminates the neutron scattering signal leading to loss of useful data and beamtime. At the Bragg Institute the decision was made to develop a safety interlock system that, when combined with the vent tube design, would provide a complete engineering control. Essentially, the safety interlock system will not allow the user to operate a closed cycle cryostat when there is a vacuum leak present.

The logic behind this system is to interlock the closed cycle refrigerator (CCR) compressor to the sample space with a digital capacitance vacuum gauge. The CCR compressor will automatically turn on only if the sample space, starting from atmospheric pressure, has been pumped down to a pressure of 2×10^{-2} mbar or lower (gauge head located on cryostat), to ensure that the system does not have a vacuum leak. Once this initial condition is met, the CCR compressor will automatically turn on and continue to operate until the pressure within the sample space exceeds 950 mbar, the digital vacuum gauge fails, the CCR compressor is remotely turned off through the temperature controller's relay control or the compressor unit is locally shut off by the user. The low- and high-pressure boundary conditions are flexible enough to allow the user to flood the sample space with He exchange gas up to a value that corresponds to a particular experiment and cryostat.

To change the sample within the cryostat the users will be required to flood the sample space with He gas overpressure, at which point the pressure will rise above 950 mbar and the CCR compressor will automatically be shut off by the safety interlock system. After the CCR compressor has been shut off the user may proceed with pulling out and replacing the sample stick whilst the sample space is still being flooded with an overpressure of He gas. Once the new sample stick is secured in place the user can initiate the cooling cycle by beginning to pump down on the sample space. The safety interlock system will only allow the CCR compressor to turn on again once the sample space is pumped down to satisfy the prerequisite vacuum condition, which indicates a leak-free system.

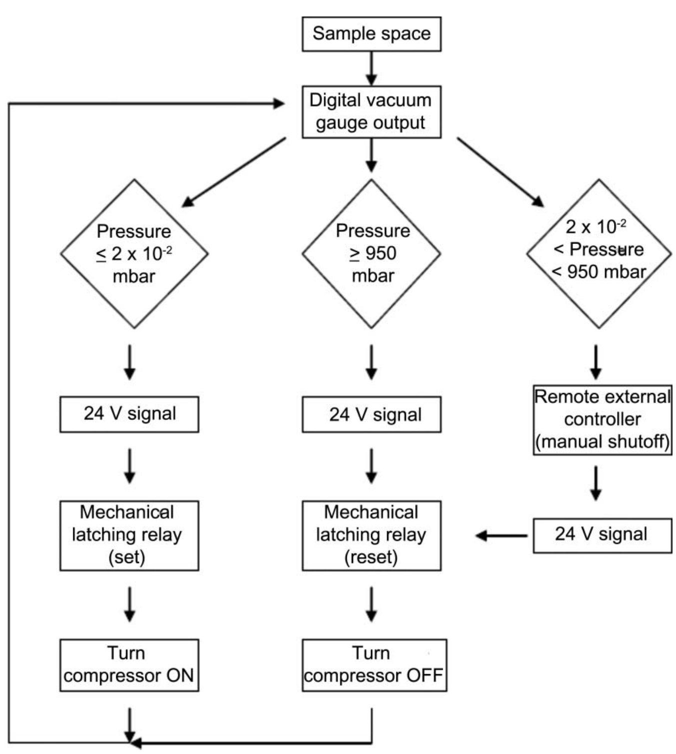

Figure 5
BI safety interlock system – logic diagram.

The safety interlock system can be effectively used for both top- and bottom-loading cryogen-free cryostats. Note that while bottom-loading cryostats do not present the same safety concerns, this approach will mitigate leaks that can affect instrument performance and negatively impact the quality of neutron scattering data. The required components are a mechanical latching relay, a temperature controller, a capacitance vacuum gauge, a digital measurement controller and a small-capacity roughing vacuum pump. These components are connected such that there is no need for the user to interact manually with the CCR compressor controls, and no software control is required (Fig. 5). Note that use of a small-capacity vacuum pump is important to ensure that it will not be able to pump to a low enough pressure on a system with a small leak.

The system also provides some protection for cryo-furnace equipment when operating at temperatures above 300 K. Damage to cold head components typically occurs if the cold head temperature rises above room temperature (see for example Sumitomo Heavy Industries, 2003). Therefore, when operating at temperatures higher than room temperature the heat transfer to the cold head should be minimized by pulling a vacuum to remove the exchange gas from the sample space and the CCR compressor should remain in operation to provide active cooling to the cold head. The safety interlock system restricts the user from remotely shutting off the CCR compressor while the sample space is under vacuum. This ensures that when in the high-temperature operational regime, the cold head continues to be cooled by the CCR compressor. If required the compressor can be locally shut off at any time.

The penalty to be paid for ensuring a good vacuum is that the time to change samples increases. The fastest and riskiest way to change samples involves no check for a good vacuum seal and does not require a systematic pump down of the sample space prior to recommencing cooling. Initial testing of the safety interlock system revealed that the time required to change from sample A at a temperature <50 K to sample B at a temperature <50 K increased by approximately 5–10 min, which is considered to be a reasonable delay in return for improved safety and reliability, particularly when samples may remain in the cryostat for several hours or days.

3. Conclusion

To date there have been several months of real-time operational usage of these safety systems on both cryogen and cryogen-free cryostats. The user feedback has been positive and there have been no incidents arising from ice blockages of the sample space. For either a cryogen or a cryogen-free cryostat system the two solutions mentioned in this paper can be easily and cheaply incorporated into existing and future sample environment equipment.

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