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DESIGN NOTE

Construction of a gravity-fed circulating liquid nitrogen dewar for experiments in high vacuum

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Abstract
The construction of a high-vacuum gravity-fed liquid nitrogen dewar is detailed. The dewar allows the liquid nitrogen to be directed to internal parts of the chamber using standard fittings and stainless steel tubing. By using the chamber vacuum as the primary insulation for the dewar, a lifetime in excess of 12 h prior to the need for refilling is found.

Keywords: cryogenic pumping, liquid nitrogen dewar, vacuum cryogenic

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1. Introduction

Many experiments which are carried out in high-vacuum systems require a liquid nitrogen cooled region to be incorporated inside the chamber. As an example, atomic physics experiments which use an effusive source of atoms from a metal vapour oven often require a cold trap upon which the atoms can be efficiently deposited following emission. This is necessary to reduce the background vapour pressure inside the chamber to an acceptable level. Examples of the use of liquid nitrogen cooled cold traps can be found in electron impact and laser experiments on mercury [1], sodium [2–4], rubidium [5] and calcium [6]. In these experiments the cold trap is often constructed from a copper cold finger attached to a liquid nitrogen dewar. The cold finger then extends from the dewar into the vacuum chamber. In these experiments radiation heating and other losses often result in a temperature at the end of the cold finger which can be as much as 200 °C higher than at the dewar [7]. The trapping region therefore acts far less efficiently than could be achieved if it were held at liquid nitrogen temperatures.

Another important use of liquid nitrogen inside a vacuum chamber is in surface studies, where a cleaned surface often needs to be cooled to a controlled temperature by the application of both liquid nitrogen and resistive heating. In these experiments, the liquid nitrogen is usually pumped through the wall of the vacuum chamber via specially constructed manipulation flanges[1]. In this case, there is significant heating loss at the pump connected to the external dewar, and at the flange interface.

In this note the design of a new type of dewar is reported which acts as a reservoir of liquid nitrogen attached to the vacuum chamber. Under realistic operating conditions, as used in atomic physics experiments, the liquid nitrogen is found to last in excess of 12 h prior to the requirement for refilling. This design allows the liquid to be gravity fed to the region which is to be cooled using standard Swagelok fittings[2] and stainless steel tubing. This has an advantage over other systems since the cooled region is held at liquid nitrogen temperatures (around −200 °C) for as long as the dewar contains liquid. For atomic physics experiments, this provides a highly efficient cold region which acts as a trap for any metal vapour. Since the dewar is incorporated as part of the vacuum chamber, the

[1] Part Number ZLN, Vacuum Generators, Hastings, Sussex, UK.
[2] Swagelok Company, Solon, OH 44139, USA.

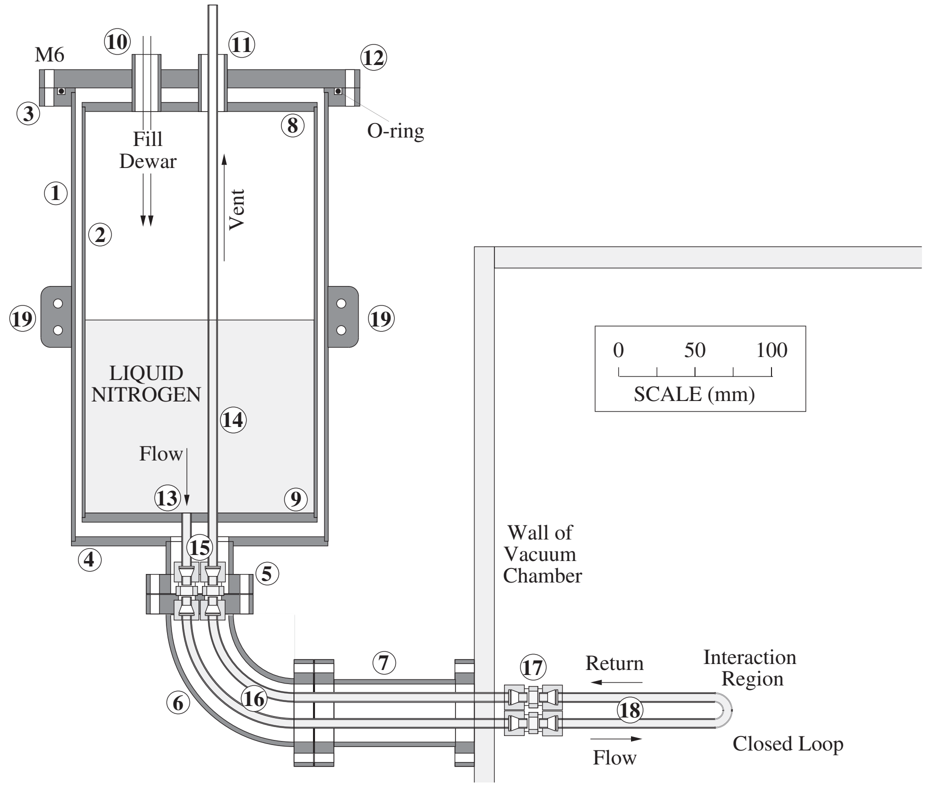

Figure 1. The gravity-fed liquid nitrogen dewar. For details see text. Full CAD drawings are available by linking to the electronic version of this note.

dewar also acts as a vacuum pump for water vapour inside the system, thereby improving the final chamber pressure.

The design detailed here may also be incorporated into surface science experiments, since the dewar can be positioned directly above the manipulation flange. Liquid nitrogen from the dewar is then fed via tubing to the surface which is to be cooled. Losses occurring in the methods described above are minimized in this method of delivery. The vacuum chamber does not need to be permanently connected to bulky external dewars, which may limit accessibility to the apparatus.

2. Design of the dewar

Figure 1 shows the design of the dewar, which is constructed of two concentric cylindrical chambers. The outer cylinder acts as the external wall to the vacuum chamber, whereas the inner enclosure acts as the containment vessel for the liquid nitrogen.

The vacuum cylinder (1) is constructed from 316-grade stainless steel tube which was obtained from commercial stock. The top flange (3) welded to this cylinder is of diameter 210 mm and thickness 12 mm. This 316LN steel flange has a groove with square cross section machined into its upper surface to allow seating of a Viton O-ring gasket. Eight M6 tapped-through holes are distributed uniformly around this flange.

The lower flange (4) welded to the vacuum cylinder has a diameter of 168 mm and is of thickness 6 mm. This flange has a stepped hole bored into the centre to allow a standard 70 mm CF35 tubulated Conflat flange (5) to be welded to this flange. The flange incorporates a knife edge for high vacuum sealing using a copper gasket, and is commercially available from most manufacturers.

A 90° curved elbow (6) incorporating rotatable CF35 Conflat flanges is attached to tubulated flange (5). This elbow is available commercially from various manufacturers (see for example[3,4,5]). The elbow allows the dewar to be secured to the vertical face of the vacuum chamber via a straight Conflat connector (7) as shown in figure 1. Straight connectors are also available from the same manufacturers.

The inner enclosure of the dewar is also constructed of 316-grade stainless steel. This is a more complex design to the outer chamber. The inner cylinder (2) is constructed of commercially available tubing, and was selected to have an outer diameter of 154 mm with a wall thickness of 2 mm.

316-grade stainless steel flanges (8) and (9) of diameter 154 mm and thickness 6 mm are welded to the top and bottom of the cylinder to complete the enclosure of the liquid nitrogen. Two stainless steel venting tubes (10), (11) are welded to the top plate (8) as shown in figure 1. These venting tubes are also welded to flange (12), to allow the enclosure to be filled with liquid nitrogen through venting port (10) and to let nitrogen vapour escape from the enclosure. The venting tubes protrude 10 mm beyond the top flange to reduce the effects of icing.

The top flange (12) of the enclosure is constructed from 12 mm 316LN steel and has a diameter of 210 mm. This flange has eight M6 clearance holes equally spaced around the flange to match the associated tapped holes in flange (3). The bottom face of flange (12) is machined flat, and locates onto the top flange of the outer cylinder, the O-ring inserted between these two flanges providing a vacuum seal.

[3] Vacuum Generators, Maunsell Road, Hastings, Sussex TN 38 9NN, UK.
[4] Caburn-MDC Ltd, The Old Dairy, Glynde, East Sussex BN 8 6SJ, UK.
[5] Kurt J Lesker, 1515 Worthington Ave, Clairton, PA 15025-2700, USA.

In the bottom plate (9) of the inner enclosure two 6.35 mm (1/4″) stainless steel tubes of wall thickness 0.7 mm are welded. These tubes span the centre line of the enclosure and are a distance of 17.2 mm apart to allow swaged fittings to be connected (13), (14). The tubes protrude from the bottom of plate (9) by a distance of 41.2 mm. Tube (13) terminates at the inner surface of plate (9) inside the enclosure, while tube (14) passes up through the enclosure and out of the vacuum chamber via venting port (11). This is used to vent nitrogen gas from the tubing internal to the vacuum chamber.

Swagelok unions (15) are connected to the lower ends of tubes (13) and (14) so that additional tubing (16) can be fitted inside the Conflat elbow. The spacing between tubes (13) and (14) allows these unions to be attached without touching the inner wall of tubulated flange (5). A second set of Swagelok unions (17) is connected to curved tubes (16). This allows additional internal tubing (18) to be connected to the feed of the dewar (13), directed to the interaction region, and then brought back to connect to venting pipe (14) as a continuous enclosed loop. The tubing (16) is carefully bent to pass around the 90° elbow so that the tube does not touch any internal surface, thereby ensuring excellent thermal insulation. It is necessary to use a curved elbow (6) to allow the Swagelok unions and tubing to be assembled.

Finally, two support lugs (19) are welded to the outside wall of the dewar. These lugs allow the assembled dewar to be supported separately from the connection to the main vacuum chamber, which is necessary since the weight of the full dewar exceeds 15 kg.

3. Construction of the dewar

It is important to assemble the dewar in the correct order so that the welds are accessible throughout the construction process. Tubes (13) and (14) are welded to the top face of flange (9) so that the welds are on the non-vacuum side. Similarly, vent ports (10) and (11) are welded onto the lower non-vacuum side of flange (8). The inner enclosure cylinder (2) is then welded to flanges (8) and (9). Vent ports (10) and (11) are finally welded to the outside top surface of flange (12), thereby completing the internal enclosure assembly.

For the outside chamber, flange (5) is initially welded to the top surface of lower flange (4). Flange (3) is welded to cylinder (1) on the top surface, and this flange is then machined flat. Lower flange (4) is then welded to cylinder (1), completing assembly of the outer chamber. All parts of the dewar are then cleaned to high-vacuum specifications.

4. Assembly of the dewar

To assemble the dewar, Swagelok unions (15) are first connected to pipes (13) and (14). To eliminate leakage of nitrogen into the vacuum chamber it is important to use new, ultrasonically cleaned Swagelok fittings and to tighten these correctly. The inner enclosure is then lowered into the outer chamber and the flanges are clamped together, the O-ring between flanges (3) and (12) provides the vacuum seal.

The Swagelok unions (15) protruding from flange (5) are then connected to tubes (16) which pass through the elbow (6) and straight connector (7). Again, these fittings should be cleaned and tightened correctly. A copper gasket and the 90° elbow is then passed along tubes (16) and the elbow fixed to flange (5). The straight connector (7) is then attached to the 90° elbow, and Swagelok unions (17) are connected to tubes (16) which protrude through the wall of the vacuum chamber. The straight connector (7) is clamped to the vacuum chamber and the support lugs are connected. Additional tubing (18) is finally connected to Swagelok unions (17), allowing liquid nitrogen to pass to the region to be cooled.

The design of the dewar allows easy assembly and disassembly from the vacuum system. This is necessary since if air inadvertently leaks into the chamber while the dewar is full, water vapour will freeze onto the walls of the inner cylinder producing significant quantities of ice. If this occurs, it is essential to disassemble the dewar and dry all components. This can be facilitated either by baking the assembly or by heating individual components. Since the dewar is constructed of stainless steel and the seal is made of Viton, the dewar can be baked at a temperature between 150 and 200 °C until clean and dry.

5. Experimental testing

The liquid nitrogen dewar has been tested in a number of different atomic and molecular physics experiments in our laboratory, and has been found to operate very efficiently. The temperature of the cooled interaction region inside the vacuum chamber was monitored directly using a thermocouple.

After initially filling the dewar with liquid nitrogen the temperature of the interaction region was seen to decrease rapidly as the liquid was fed by gravity through the 6.35 mm tubing. Considerable gas emission through vent tube (14) was initially observed, and at times a small stream of liquid nitrogen would be emitted as the tubing inside the chamber cooled. It is necessary to prevent this expelled liquid from causing damage, and this can be facilitated by attaching a filter to the vent tube to catch and vaporize this liquid.

Approximately 2 min after filling the dewar, the tubing inside the vacuum chamber completely fills with liquid nitrogen and the emission of nitrogen gas from tube (14) reduces significantly. The temperature of the interaction region is then measured to be around −200 °C. The interaction region maintains this temperature throughout the lifetime of the dewar, which has been found to be over 12 h when operating with an effusive atomic source, and with the vacuum chamber being held at room temperature [8]. This lifetime is reduced when an oven is used as an atomic beam source, but the temperature at the interaction region remains at −200 °C, providing a very efficient cold trapping region for deposition of vapour.

It should finally be noted that it is important not to attach vent tube (14) to the external liquid nitrogen dewar to fill the vacuum dewar, as occurred in our laboratory recently. In this configuration gas is prevented from escaping through this vent, and as the liquid boils off inside the chamber, the resulting vapour can only escape into the dewar which is filled with liquid. This causes localized warming of the tube inside the chamber. Atoms and molecules frozen onto the tube then revaporize into the chamber for short periods of time until the nitrogen gas vents through the dewar and the tubing recools. This is observed as a sudden increase in background pressure, followed by a slow decrease back to normal operating pressures. Such effects can severely disrupt experiments that are being conducted. By filling the dewar correctly through vent pipe (10) this problem is eliminated.

6. Conclusions

A new self-contained gravity-fed dewar is described which allows liquid nitrogen to be directed to any region inside the vacuum chamber. The dewar is constructed using high-vacuum techniques, can operate at pressures down to 10^{-8} Torr, and has been demonstrated to have a lifetime in excess of 12 h. The design allows easy assembly and disassembly by adopting Swagelok and Conflat fittings throughout. This provides a significant improvement over many other designs currently adopted.

Detailed CAD drawings are available as pdf files by linking to the electronic version of this note.

Acknowledgments

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