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Abstract. Oxford Instruments has recently experienced an increasing demand for UHV sample environments for both its traditional 'wet' Kelvinox, and Triton cryofree™ range of dilution refrigerators. This demand is often accompanied by requests for particular sample handling mechanisms, usually designed to prevent the need to break the hard-won vacuum when changing samples, and other application-specific features to be integrated into the systems. We have recently installed systems for a wide range of applications including STM, magneto-optical trapping of atomic clouds, and for use on synchrotron beam lines. In this paper we review three such systems and their novel features that we have installed in laboratories in Europe and the USA in the last few years.

1. Introduction

Oxford Instruments has, for a number of years, produced cryostats for cooling samples in ultra-high vacuum (UHV) environments to temperatures of <350 mK and in magnetic fields up to 14 T [1][2]. These cryostats have also been configured to allow in vacuum sample changes by manipulating the whole cryostat insert into the customer's UHV sample preparation chamber. Once a new sample has been mounted onto the insert it can be withdrawn back into the magnetic field centre for cooling and measurement. The small diameter of such sorbtion pumped helium-3 inserts means that they lend themselves to such geometry; the whole insert can pass through the cold bore of the magnet without making extreme demands on the magnet design. Sample space diameters of 50 mm in 10 T have been achieved. In addition the magnet bore can also accommodate a double-walled baking-sock that allows hot gas to be forced around the UHV sample environment to bake the whole insert and samples to temperatures greater than 100 C.

The natural progression of this arrangement is to perform measurements on samples at ever lower temperatures [3]. The following three sections describe some of the technical detail of three such systems - all dilution refrigerators - for very different applications.

2. System 1 – 10 mK and 14 T for STM with insert manipulation for sample access

This system is the logical progression from the helium-3 pumped cryostats mentioned above. A vapour shielded helium cryostat, see figure 1, contains a conventional 14 T superconducting solenoid with a 102 mm cold bore. The dilution refrigerator insert has an inner vacuum can (IVC) which contains a 1 K pumped helium-4 pot and associated 1 K thermal shield, and the dilution unit. The IVC extends through the bore of the magnet and is ultimately open to a 336 mm OD CF flange in the bottom of the cryostat via two sets of spring-loaded thermal radiation flap baffles; one cooled by the main helium-4 bath at 4.2 K and the other thermally anchored to the outer cryostat shield at nominally 100 K. The CF flange interfaces directly with a UHV gate valve connected to the customer's sample preparation chamber (not shown). The UHV space extends from the IVC and customer chamber at the bottom of the insert up to the top of the insert via a baffled wiring access tube. At the top of the insert this breaks out into a UHV chamber containing multiple CF ports for customer, and system diagnostic, wiring access.

The UHV space can be pumped continuously from both the top wiring access chamber and from the customer's sample chamber at the bottom. Provision for a light bake out of the sample area has been made by the inclusion of a pair of heaters mounted on the 1 K shield at the location of the sample. Room temperature gas can be forced to flow through the magnet to keep it from heating too much during the bake-out procedure. The dilution unit, magnet, and the heaters themselves are monitored using platinum resistance thermometers.

During factory tests the UHV space was pumped only from a dummy test chamber fitted to the bottom of the cryostat. The bakeout heaters around the sample space were maintained at 100 C for several hours. During this time the magnet was supplied with a flow of room temperature helium gas at a flow of 3.5 litres per minute and its temperature reached only a few degrees above room temperature. The mixing chamber temperature reached 50 C within 5 h at which point the heaters were turned off. When the system had cooled back to room temperature, pressures of $<2\times10^{-5}$ mbar were observed at the (un-pumped) wiring access housing on top of the insert and $<2\times10^{-6}$ mbar at the dummy sample chamber. Once the main bath had been cooled below 4.2 K pressures of 2×10^{-8} mbar were routinely observed.

To enable the sample to be changed without the need for breaking the vacuum or warming to room temperature, the whole insert can be driven downwards so that the tail of the 1 K shield containing the sample penetrates into the customer’s UHV chamber via the two radiation flap baffles and the gate valve. The sample centre line protrudes 290 mm below the bottom CF flange of the cryostat. A window in the 1 K shield can be opened by a wobble stick in the customer's chamber and the sample changed. The movement of the insert is driven by a room temperature manipulator with a z-stroke of 650 mm. A stainless steel edge welded bellows on the manipulator keeps the main helium-4 bath sealed from the atmosphere. Inside the cryostat the top portion of the IVC is also made from bellows to take up the movement within the main helium-4 bath.

The fridge components inside the UHV/IVC cannot be pre-cooled from room temperature using the traditional exchange gas method as the vacuum is shared by room temperature parts of the cryostat. Instead a mechanical heat switch has been developed to thermally short the inside of the 1 K shield to the dilution refrigerator's mixing chamber. Once the main bath is full of liquid helium the dilution unit and 1 K pot are still at around 200 K and cooling slowly. With the heat switch closed cold liquid is drawn from the main bath through the pot which eventually cools below 4.2 K. The dilution unit follows and the helium-3/4 mixture can then be condensed as normal. From the time that the needle valve was opened it took 20 h to cool the mixing chamber to <4 K. The heat switch is a copper lozenge operated by a drive rod that extends into the wiring access chamber at the top of the insert and is terminated with a UHV rotary feed through.

The 1 K pot itself has an open volume of one litre allowing a single-shot lifetime of 12 h with the dilution refrigerator circulating at base temperature. Separate inlet and outlet ports have been provided on the pot’s needle valve so that a fixed impedance can be installed in parallel to the (closed) needle valve. Both features give the option of eliminating the noise traditionally associated with the needle valve design.

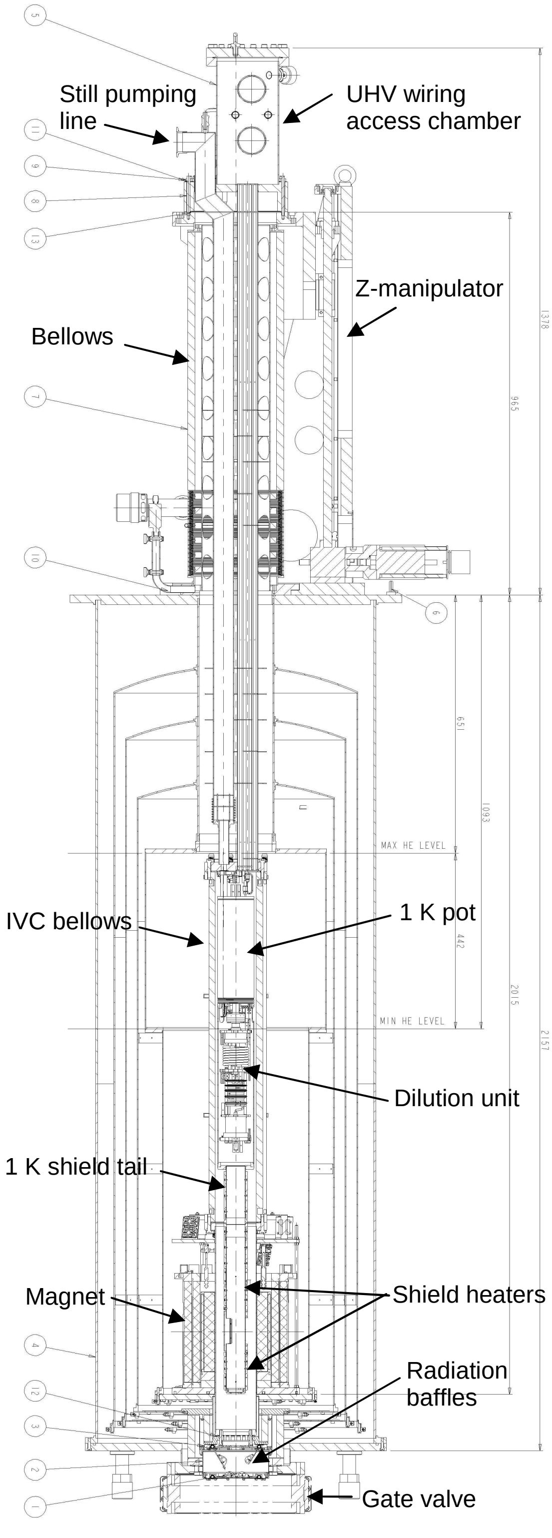

Figure 1. The complete system shown in the 'measurement' position with the insert raised.

Figure 2. The UHV compatible dilution unit. The pre-cool heat switch strike-plate can be seen at the bottom left of the image.

The dilution refrigerator unit, see figure 2, has a conventional Oxford Instruments design with several key modifications. All trapped volumes are vented either by including appropriate extra vent holes or by the use of vented fasteners. All the parts that would normally be soldered on assembly have been vacuum brazed. The only exceptions to this are the terminations of the continuous heat exchangers (hard soldered) and the assembly of the discrete sintered-silver heater exchangers (soft soldered). Other non-UHV compatible materials have been changed; for example nylon has been replaced by PEEK.

During factory tests the dilution refrigerator performed with a base temperature of 8.1 mK and the cooling power was measured to be 400 μW at 98 mK with a helium-3 flow rate of 1 mmol/s.

3. System 2 – 10 mK and 7/1/1 T vector rotate magnet for synchrotron beam line with bottom loading sample access

The second system we describe here was a hybrid 'wet' magnet and cryostat containing a cryofree Triton DR200-10 dilution refrigerator insert in its own separate UHV space. The magnet was a 7/1/1 T vector rotate manufactured directly onto a UHV-clean bore tubes giving detector/beam access on all four of its horizontal entries, see figure 3.

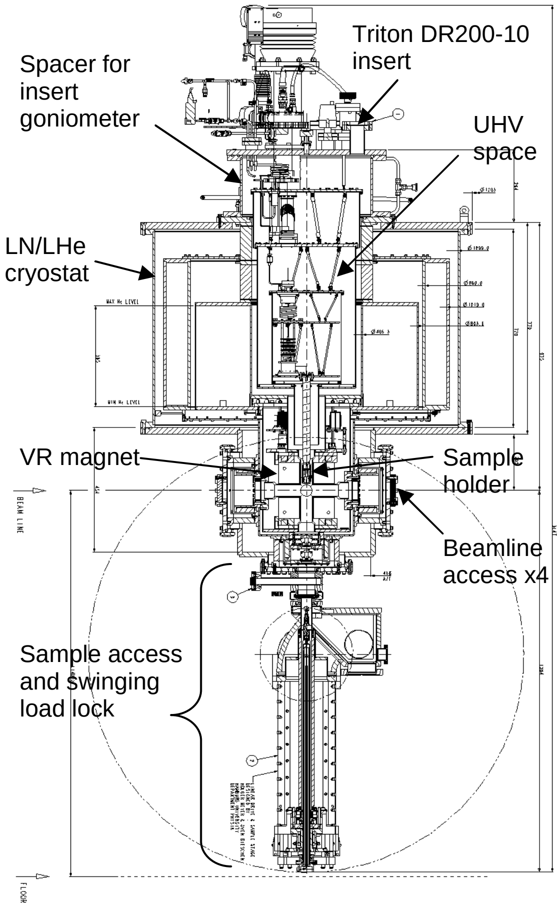

Figure 3. The system as configured for beam line and sample change access. The swinging sample change enables access with limited ground clearance.

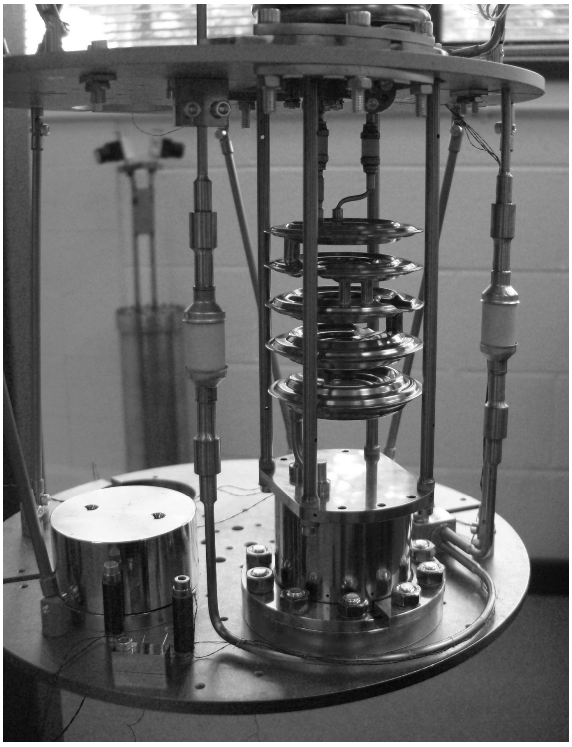

Figure 4. The lower half of the dilution unit showing the detail of the ceramic electrical isolation elements in the pre-cool gas lines and the unit's heat exchangers.

The UHV space was sealed against the 'dirty' vacuum of the cryostat by stainless steel edge welded bellows on the cryostat neck and the five access ports of the magnet. The upper vertical access of the magnet bore contained a cold finger extending from the dilution refrigerator's mixing chamber and terminated in a left-hand threaded sample mounting point. The lower vertical access allowed a customer-supplied sample loading stick to move samples in and out of the sample mount from below the cryostat without breaking the UHV space's integrity or disturbing the instrument from the beam line. The sample mount also contained a set of spring loaded electrical contacts to automatically connect measurement lines to the sample when it was loaded.

The Triton insert itself incorporated a specially modified Cryomech 1 W pulse tube refrigerator (PTR) mounted using a CF flange at the insert top plate. The PTR construction was vacuum brazed including the PT2 stage helium-3 heat exchanger which was incorporated into the PTR at time of manufacture. Where possible the materials of the insert were replaced with more UHV compatible substitutes. For example the brass continuous heat exchanger tube on the dilution refrigerator was replaced with stainless steel. All other ports on the insert top plate were of the standard CF flange type. An experimental requirement to measure the electrical current from the sample via the mixing chamber required that it, and the sample holder, were electrically isolated from the rest of the system, see figure 4. This was achieved by incorporating insulating supports and inserting ceramic breaks in the dilution refrigerator's heat exchangers and the insert's gas pre-cool circuit. A 64 W heater was mounted on the mixing chamber so that a light bake of it and the sample could be performed before cooling the system below room temperature. A pressure of 1.0×10^{-8} mbar was recorded with no bake out and with the insert and cryostat cold. The dilution refrigerator achieved a 10 mK base temperature and a cooling power of 400 μW at 103 mK.

4. System 3 – 10 mK and multiple optical access to sample for atomic/optical experiments

This system was designed to be a UHV compatible version of the cryofree dilution refrigerator, the Triton DR200-10, combined with some specific modifications to allow optical access to the system's two sample spaces for optical experiments on atomic systems. The Triton insert, see figure 5, for this system was based on the one described in System 2 above but without the experimental requirement of electrically isolating the mixing chamber.

The rest of the OVC of the system completed the common UHV space in the cryostat. The OVC was of an all-welded stainless steel construction manufactured in three sections and joined using copper wire-seal flanges. The lowermost section was fitted with ten DN40CF ports arranged in a radial pattern for experimental access and a single DN160CF port at the bottom to allow pumping access.

The two PTR radiation shields, see figure 6, surrounding the insert were also fitted with corresponding access ports and all of the ports were fitted with Spectrosil quartz optical windows for some of the system tests. There are effectively two sample spaces on the cryostat. The first is the space (240 mm in diameter and 240 mm long) inside the dilution refrigerator's still temperature radiation shield with direct access to the mixing chamber. This space also had two further DN40CF optical access ports through the OVC and the cryostat shields. The second is a 4 K isothermal plate at the level of the lower optical access ports to mount and cool the optical components of the experiment. This was suspended on copper thermal links direct from the PT2 plate.

The system was designed to be bakeable to 50 C (by heating the outside of the OVC only) as per the system described in section 1 above. The limit on this baking temperature is the few indium seals present on the system's dilution refrigerator. Pressures of 2.5×10^{-6} mbar and 1.7×10^{-8} mbar were recorded at room temperature and 10 K respectively with no bake out of the system performed. The dilution refrigerator reached 9 mK with all windows blanked and 32 mK with all windows open. The maximum cooling power at 100 mK was 240 μW.

Acknowledgements

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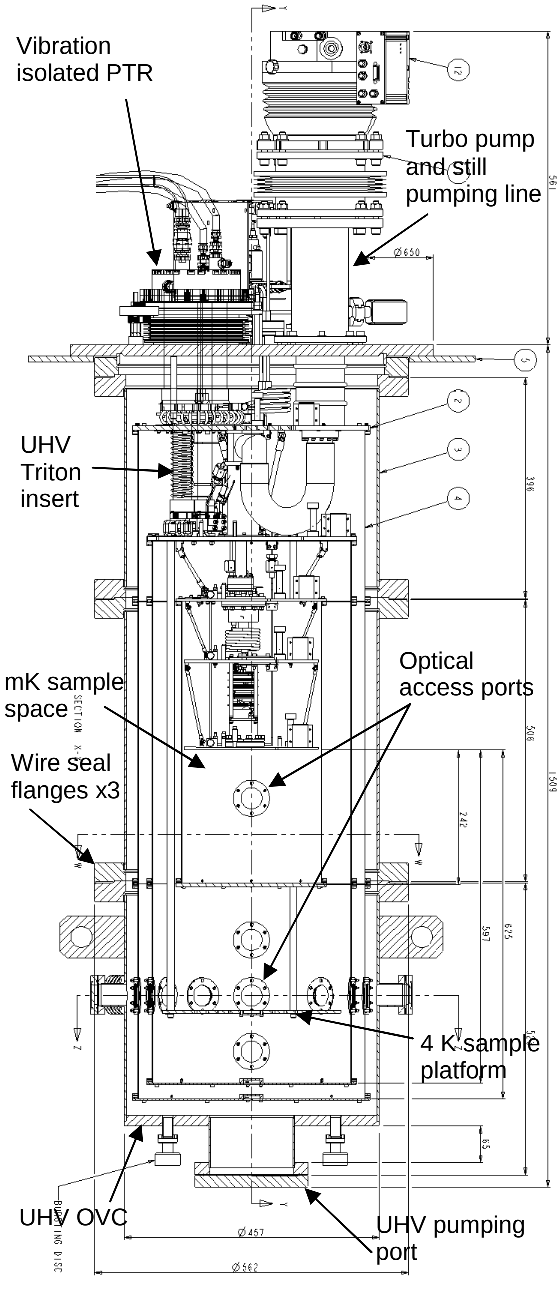

Figure 5. General assembly of the system.

Figure 6. The system with the OVC removed showing some of the optical ports in the PT1 shield.

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