UHV compatible 3He – 4He dilution refrigerators for STM in high magnetic field

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Two new Janis dilution refrigerators for scanning tunnelling microscopy (STM) in ultra-high vacuum (UHV) and magnetic field up to 15 Tesla are presented. Their design details, including differences between top and bottom sample loading features are described. Base temperature, cooling power, internal vacuum and other experimental results are discussed.

1. Introduction
Choosing a cooling system configuration for STM depends on application specific demands, such as needed base temperature, intrinsic heat release and total heat load, vacuum conditions at room and base temperatures, magnetic field strength and preferred sample delivery scheme to the magnet centre. If a solenoid configuration of the magnet is used, the only practical solutions are the so called top- and bottom sample loading refrigerators. Below we describe two corresponding designs, both for UHV STM, and both able to cool STM mounting plates to below 15 mK in a high magnetic field. As a general approach, the systems are made UHV compatible by using Conflat flanges (CF) or copper wire sealed flanges and VCR couplings instead of indium, totally avoiding soft soldering and non-UHV compatible plastics and metals, especially zinc-containing in UHV space. PEEK is used where plastics are unavoidable. All stainless steel parts are made from austenitic 300 series alloy, critical weld joints are annealed, and most parts are electro-polished. All copper and copper alloy parts are 24 carat gold-plated without a nickel under layer. Low- or non-magnetic materials, such as phosphor and silicon bronze, beryllium copper are widely used for fasteners hardware.

2. JDR-500 with sample top-loading port
The first system was built for the Center for Nanoscale Science and Technology, NIST, Gaithersburg, USA. As shown on the Figure 1, this group has opted for sample loading through the top of the cryostat via a 40 mm i.d. clear shot access port to the magnet center. As can be seen, this system requires a long UHV manipulator to deliver/exchange samples, tips or even the whole STM if needed from the UHV preparation chamber at the top (not shown) to the silver STM support, attached to the mixing chamber. It also needs an intricate mechanism of cooled shutters to block any infrared light propagating downwards along loading tube after the sample and STM tip are delivered.

The system also has an easily removable insert as in other conventional refrigerators. The insert is installed inside a vapor shielded dewar with a 290 mm diameter neck and a 250 liter helium belly capacity. The dewar has six super-insulated shields in its vacuum space, resulting in a static boil-off rate of approximately 0.6 l/hr. A 90 mm bore 15 Tesla superconducting solenoid with compensation coil around the mixing chamber is loaded into the dewar through an indium sealed bottom flange.
The insert is sealed through a 10 inch CF at the top of the UHV compatible space in the IVC. It has a pre-cooling loop with inlet and outlet ports for pre-cooling the DR stage to 4 K without heat-exchange gas. The pre-cooling is done by delivering cryogens to copper anchors at the 1 K pot, Still, intermediate cold plate (ICP) and mixing chamber (MC).

While outside, the insert can be baked and pumped in a dedicated “furnace” at temperatures close to 100° C, while keeping the DR stage at lower temperature through the pre-cooling loop. After baking, the DR insert can be inserted into the dewar, even with liquid helium in the main reservoir.

There are two methods of condensing the 3He-4He mixture in this system. Its mixture condensing unit contains a large volume 1 K Pot, as well as a Joule-Thomson counter-flow heat-exchanger. The 1 K pot is quipped with two needle valves for 4He delivery from the main reservoir. One valve has a manual operator only for open-close operation, while the second has a computer controlled step motor for fine tuning its flow impedance.

Two independent mixture return lines have flow impedances of approximately $3 \times 10^{10}$ cm$^{-3}$ and $1 \times 10^{11}$ cm$^{-3}$. The first line is used for the 1 K pot mode operation, while the second parallel mixture return line, is used for JT operation mode. Both lines merge before entering the still heat-exchanger where the common secondary flow impedance is used.

A customized JDR-500 dilution stage was built for this system, with its Still and continuous heat exchanger moved from the central axis, while having a large (40 mm) central opening through ICP, silver all heat-exchangers and mixing chamber. Infra-red light entering the IVC from the top has been
blocked during tests at Janis by blank flanges at IVC, Still and ICP plates (blank flanges were installed).

All wiring was made through a dedicated UHV wiring chamber at the top of the insert. About 100 superconducting shielded wires were brought down and attached to the mixing chamber as single coaxial lines and twisted pairs. They were used for monitoring temperature sensors at the still and mixing chamber, the UHV compatible cartridge heaters, as well as application needs. Five special UHV compatible RuO sensor packages were supplied by NIST, and UHV compatible CMN and superconducting fixed point thermometry were developed by Janis.

This DR used a manual gas handling system, with Alcatel 601 Roots, 2063H rotary vane pump and bellows compressor for the mixture circulation system. Its computer control included resistive and CMN thermometry, based on LCSI 370S resistance bridge and Agilent 4263B LCR meter, as well as pressure and flow meters from MKS. One 1 K Pot needle valve had a step motor with GPIB controlled driver.

3. **JDR-500 with sample bottom-loading port**

This system was been made for Max Plank Institute in Stuttgart, Germany. The bottom delivery method dictated that the insert had to be attached to the magnet bobbin at the bottom, combining the OVC and IVC, and thus demanding that all the vacuum space inside should be UHV compatible (see Figure 2). The dewar in this system is a liquid nitrogen shielded helium dewar with no super-insulation, with a static boil-off rate of approximately 0.9 litre/hour.

![Figure 2 JDR-500 with bottom loading port](image-url)
A system of individually manipulated shutters was built into the bottom of the cryostat. The still shield employed a linear manipulator, while the 4 K and 77 K shields had rotary UHV manipulators. This system requires much shorter bottom manipulator for sample exchange. A sample loading port at the bottom was used as the evacuation port. As shown in Figure 2, the 14 Tesla magnet with saddle and compensation coils was welded into a bobbin and attached to the dewar and insert via a copper wire sealed flange and a 10” CF flange. This system used a conventional dilution refrigerator scheme, where a model JDR-500 stage with all-stainless supports was attached to somewhat larger than usual 1 K pot. A single mixture return line was used with $3 \times 10^{10}$ cm$^{-3}$ flow impedance allowed a maximum circulation of more than half a mmole/sec, produced by an Alcatel 601 Roots backed 2063H rotary pump. A removable hydrogen tubular trap was included in this system.

Again, a pre-cooling system was used, but this time using a heat-pipe. After some gas (nitrogen or helium) is introduced inside a single tube with heat-anchors, liquid forms in the tubular heat-exchanger in the belly full of corresponding cryogen and drops down, where it cools the heat-anchors below. A similar wiring chamber with UHV compatible SMA and multi-pin circular connectors was used to bring all the needed wiring, including thermometry, heaters and customer wiring. Again, the thermometry included UHV compatible RuO thermometers based on bare chips from Lake Shore, as well as Janis CMN and FPD.

4. First test results

During the successful tests at Janis, both systems reached base temperatures near 9 mK as measured in zero magnetic field by CMN thermometers outside their mixing chambers, at optimal circulation rates of about 300 micromole/sec. There was no difference in base temperature when first system was operated in the 1 K pot or JT mode. In the JT mode the system could be operated with level of helium near the bottom of the belly. It had about 0.8 litre/hour total boil-off rate in each mode of operation in zero field, while second system showed about 1.1 litre/hour at maximum circulation rate in zero field.

The silver STM support plate of the second system cooled to below 14 mK in zero field, as indicated by another CMN thermometer mounted there. RuO thermometers in both systems were calibrated against CMN thermometers on their mixing chambers, and also indicated that both STM supports were below 20 mK. The main heat leak to the supports is clearly associated with infrared light leaking through shutters imperfections.

When the magnetic field was swept, both systems showed some overheating by eddy-currents, although insignificant at speeds of few tens of mT/sec. At constant field of 10 – 15 Tesla, the mixing chamber temperature was within 0.5 mK from its zero field value after an hour or so of equilibration.

Cooling power measurements revealed that the first system had a maximum power of about 300 microwatts at 100 mK, no matter what mode it is in. It took the compressor to develop 1.3 – 1.5 bar absolute pressure to ensure the needed circulation rate of 300 – 500 micromole/sec in JT mode. Even without the compressor, the first system delivered 200 microwatts at 100 mK at 0.86 bar absolute pressure in JT mode. Further increase of the still temperature, and thus, the circulation rate did not cause any increase in cooling power. The second system had a cooling power of 500 microwatts at 100 mK and a at a circulation rate of about 400 micromole/sec. At this rate the return pressure was approximately 0.6 bar absolute.

It should be noted here that both systems were equipped with only passive super fluid film suppressors (sharp edge diaphragms). Maximum cooling power is typically limited by increased 4He contribution to circulation when still temperature is raised up. Although the same pumps were used for both systems, any restriction in the pumping path was limiting the power even further. The spiralled capillary in JT heat exchanger of the first system is thought to significantly reduce the rate at the same still temperature, thus limiting the overall maximum cooling power of the first system.

The best vacuum achieved at Janis was approximately 1E-9 mbar after a rather quick insert bake-out. More data are being taken now at the NIST site, including vibration measurements with accelerometers. The system for MPI is yet to be characterised in terms of final vacuum, including performance of its built-in baking heaters.