QMC Instruments Ltd

Model TK1813 Cryostat

Operating Manual

Date

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First - A Word of Warning

Lifting and Handling the Cryostat

Please take care when moving and lifting the cryostat. It is designed give great cryogenic performance for your convenience and is, as a consequence, rather heavy.

Using Cryogens

Cryogenic liquids are potentially dangerous. If you are not already familiar with the standard procedures appropriate for the use of liquid nitrogen and liquid helium, please seek advice before proceeding.

Operating this equipment involves the use of vacuum and cryogenic liquids. Please read this manual carefully before you operate the cryostat – although this is not a safety instruction manual, the text describes our own procedures and this may help to avoid accidents.

The photo below shows part of a damaged cryostat. We do not want this to happen to you. Please ensure that all personnel involved in the use of the cryostat are fully accustomed with the techniques involved.

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Introduction

This is a QMC Instruments Ltd. type TK1813 liquid helium cryostat built to our specification by our sister company Thomas Keating Ltd.

Item	Serial Number
TK1813 cryostat	xxxxx-x / xxxx

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Packing List

The following items are included in this shipment. Please check the contents against this list and contact QMC Instruments as soon as possible if you suspect that any items are damaged or missing.

Type TK1813 Thomas Keating Ltd cryostat

- Cryostat fitted with:
  - Transit protection fixtures
  - Over-pressure relief valve fitted to the cryostat top plate
  - Non-return valve
- Cryostat central neck safety baffle which includes:
  - Over-pressure relief valve
- Outer vacuum case base plate with O-ring
- 77K radiation shield base plates
- Liquid nitrogen blow-out tube
- Spares kit which includes:
  - O-rings
  - Set of screws
  - M3 and M4 Allen keys
- Cryostat operating manual

1. Unpacking and Preparing the System for Operation

The cryostat is not supplied in a condition that renders it ready for immediate use. A temporary base-plate has been installed to protect it from damage during its journey. The following procedure must be carried out to prepare the cryostat for operation. To prepare the cryostat for transportation the following procedure should be followed in reverse.

Photographs included in this manual are general photos that may not be specific to your particular cryostat.

Initial inspection

Please inspect the flight case in which the goods were shipped, and the contents, for any obvious sign that damage has occurred in transit. If you think that the package has been damaged in some way, please contact us before proceeding further. Your equipment is guaranteed for two years against failure resulting from effects beyond your control, and we will be happy to make any repairs at no cost to you during this time.

The O-rings, bolts, screws etc, which are required to prepare the cryostat, can be found in the spares kit.

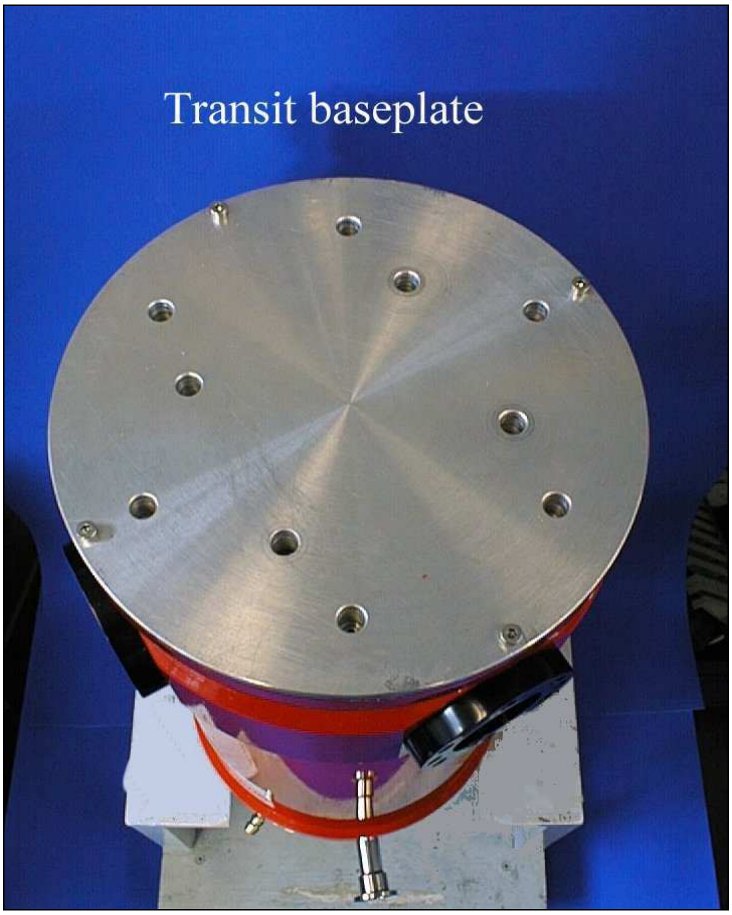

Photo 1.1. Transit base-plate

Removing the Transit Plate

Refer to Photos 1.1 and 1.2.

To allow access to the bottom-plate invert the cryostat so that it rests on the stainless lifting ring. To avoid marking the ring, stand the cryostat on something to protect it such as soft tissue, cloth or bubble wrap.

The aluminium transit base-plate should be removed by unscrewing all the socket head screws holding it in place and carefully lifting it from the cryostat.

Then, remove the four support pillars.

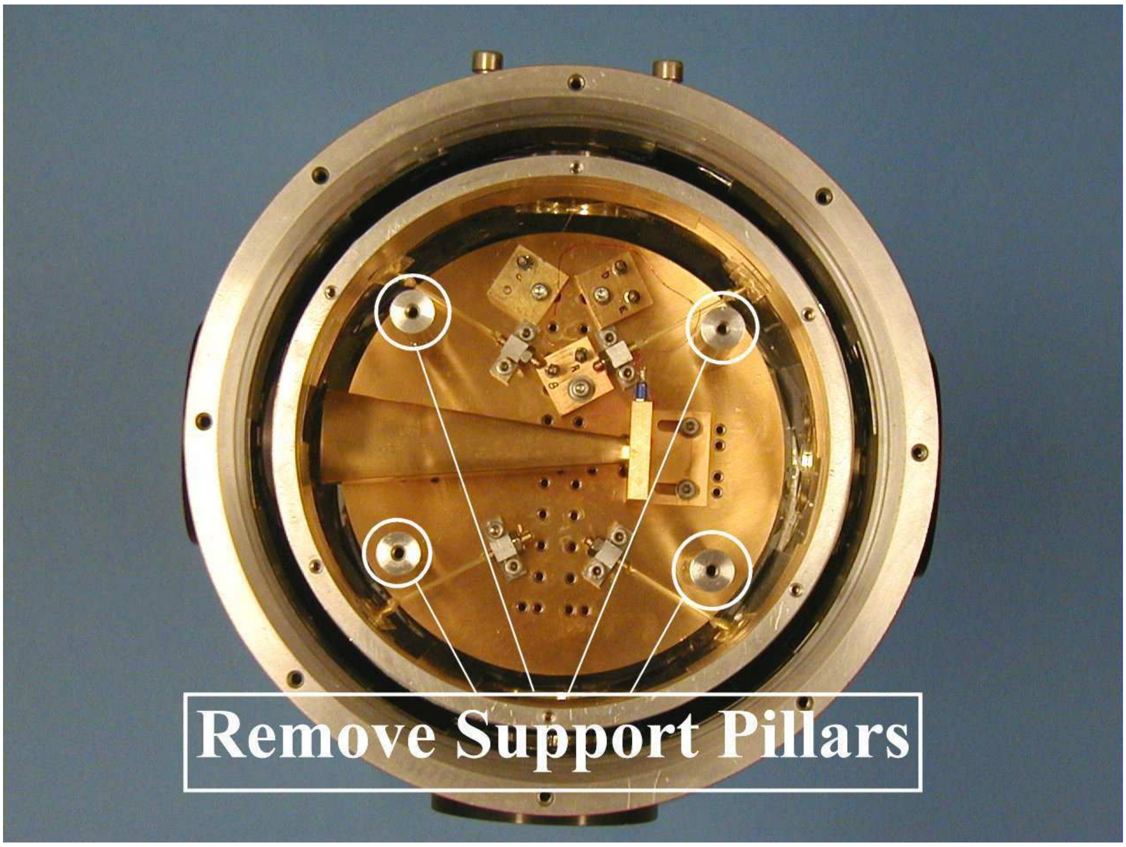

Photo 1.2. The four support pillars

Fitting the cryostat base-plates

Refer to Photo 1.3 and 1.4

The TK1813 cryostat has a liquid nitrogen cooled 77K radiation shield base-plate, and a room temperature outer vacuum casing (OVC) base-plate. The 77K shield is located using the set of M3 screws provided. The black OVC base-plate is located using the M4 socket headed screws provided. It is important to check that the O-ring is in place, that it is clean, well greased and that its seating is free of marks and scratches. The screws locating the OVC base-plate should not be over-tightened because this can distort the O-ring and may cause vacuum leaks. If the screws are equally tightened, it is normal for a small gap to show between the lip of the OVC base-plate and the bottom of the cryostat casing.

A schematic of the cryostat is shown in **Figure 1.1.**

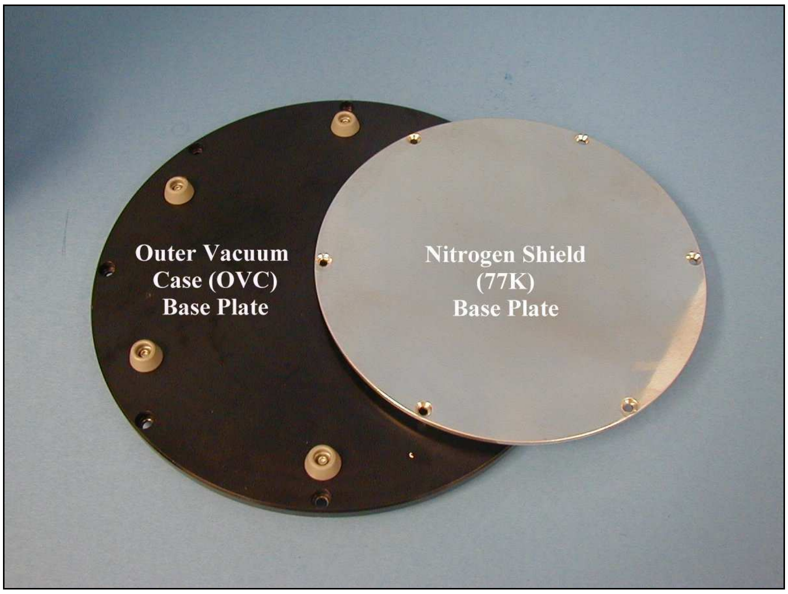

Photo 1.3. Cryostat base-plates

Photo 1.4. Liquid nitrogen base-plate in position

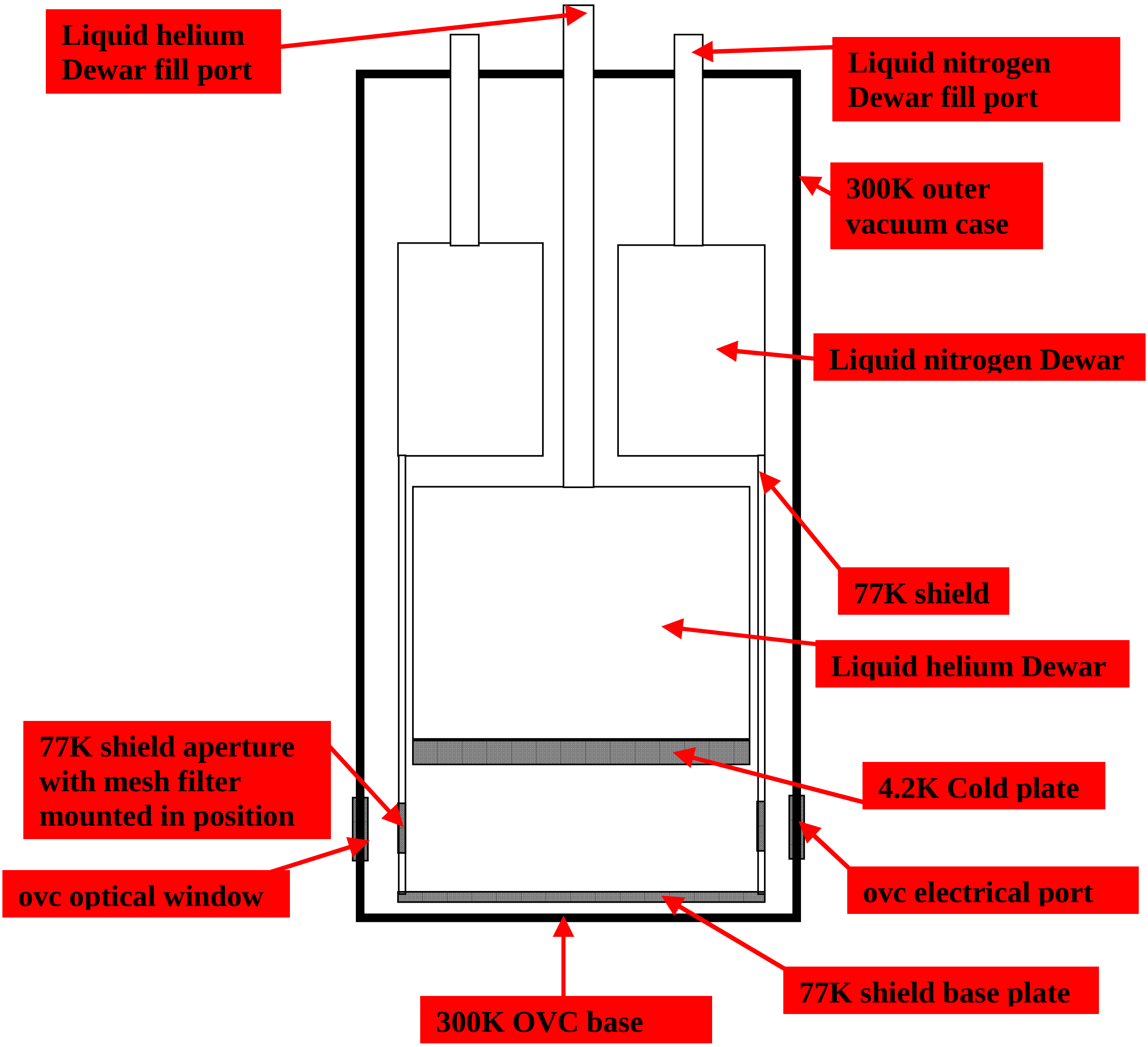

Figure 1.1. Cryostat schematic showing the main features of the cryostat. Not to scale

2. Evacuating the Cryostat

Please refer to **photo 2.1.** Before cooling the cryostat, the vacuum chamber must be evacuated by connecting a suitable pump to the evacuation port located on the top-plate. The pump should be capable of reducing the pressure in the cryostat to below 10^{-1}mbar. This can with time be achieved by using a rotary pump only, but for optimum cryogenic performance of the cryostat it is better to use a diffusion or turbo-molecular pump to reduce the pressure still further.

The pumping system should ideally have a pressure gauge measuring the pressure as close to the cryostat as possible. The spare NW16/KF16 port located on the top-plate of the cryostat can be used to attach a pressure gauge to monitor the pressure in the cryostat directly.

Always check the quality of the pump system and pumping line prior to opening the cryostat valve.

The vacuum valve should be opened very slowly when the pressure in the cryostat is at or close to atmospheric pressure. This prevents rapid pressure changes that risk damage to the delicate components inside the cryostat.

Typically, the cryostat could be ready for pre-cooling (refer to Section 3) after pumping for thirty minutes using a two stage pumping station.

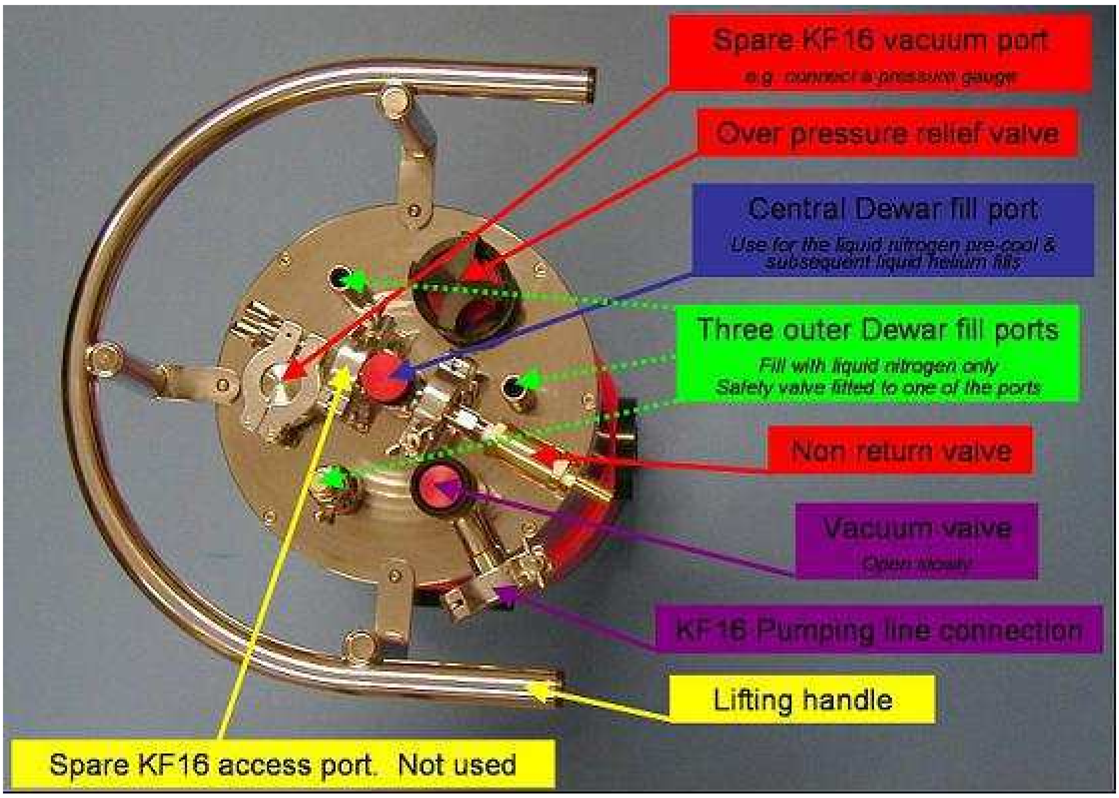

Photo 2.1. Cryostat top-plate fittings

3. Liquid Nitrogen Pre-cool

IMPORTANT: Refer to the warning at the front of the manual before proceeding with cryogenic cooling of this cryostat.

A word about your vacuum pump

The pressure in the cryostat should drop rapidly when filling with liquid nitrogen because some of the gas, mainly oxygen, begins to cryopump (condense onto the cold surfaces). The cryostat can remain attached to the pump during the pre-cool period if the pump you are using is an oil diffusion or turbomolecular type pump with a base pressure lower than 10^{-6}mbar. If you are only using a rotary pump, then the pressure in the cryostat will be lower during the pre-cool period than the pump is capable of generating, and the pump must therefore be detached immediately prior to cooling.

The need to pre-cool the central reservoir with liquid nitrogen

When a satisfactory pressure has been reached in the cryostat vacuum chamber, it is necessary to pre-cool the cryostat with liquid nitrogen before cooling with liquid helium. This will reduce the amount of liquid helium used.

Fill both liquid nitrogen and liquid helium reservoirs with liquid nitrogen using the appropriate ports, **photo 2.1**. Liquid nitrogen need only be poured in through one of the three liquid nitrogen ports. The neck baffle assembly should be unscrewed and removed from the central liquid helium port to enable the liquid nitrogen cryogen to be poured into the liquid helium reservoir.

For preference, transfer the liquid nitrogen directly from a pressurized liquid nitrogen storage Dewar which should take around 15 minutes to complete. Alternatively, pour the liquid nitrogen using a bucket and a funnel, as shown in **photo 3.1**, which may take in excess of an hour to complete. In this case, the funnel must be attached to a pipe which extends down into the neck and well into the reservoir itself. For a TK1813 cryostat a length of at least 200mm is needed. The pipe diameter should be about 6mm (1/4 inch) to allow both reasonable throughput and space outside of the pipe for boiling nitrogen gas to escape.

Safety valves

The top-plate fittings are shown in **photo 2.1**. The helium reservoir access port should always be fitted with the non-return valve to stop the condensation of moisture within the neck. This moisture could freeze and block the neck of the cryostat which in turn could lead to failure and damage.

The cryostat neck baffle is shown in **photo 3.2**. The baffle incorporates an overpressure release valve. Should an ice blockage form in the central neck of the cryostat, gas will be unable to escape through the non-return valve. Such an event will cause the overpressure relief valve, located at the top of the baffle, to open thereby releasing pressure from the inner reservoir.

The pre-cool period

The length of pre-cool period will determine the initial efficiency of use of liquid helium. For a TK1813 we recommend a minimum pre-cool of four hours, but it is often convenient to leave a cryostat overnight if, for example, it has been attached to a pump throughout the day. Larger cryostats (TK1840, TK1865 and TK1875) require a longer minimum pre-cool period because the additional gas cooled radiation shield is only weakly linked to the other temperature stages and therefore cools slowly. For these larger cryostats, a twelve hour minimum pre-cool period is recommended.

Photo 3.1. Using a funnel to fill the cryostat with liquid nitrogen

Photo 3.2. Cryostat neck baffle

Removing the liquid nitrogen from the central reservoir

When the pre-cool period is complete the liquid nitrogen in the helium reservoir should be removed. This is best done using a supply of compressed dry nitrogen gas and the blow out tube supplied. The O-ring and tightening ring from the central reservoir access port, and brass washer from the spares kit, should be arranged on the blow out tube as shown in **photo 3.3**. The non-return valve should be replaced with the adaptor nozzle. The liquid nitrogen can now be removed from the central reservoir by applying (through the adaptor nozzle) a small overpressure within the reservoir as shown in **photo 3.4.** The liquid nitrogen is directed into a safe container, and can be used to replenish the outer reservoir.

It is important that all of the liquid nitrogen is removed from the central reservoir before the liquid helium transfer is started. Any liquid nitrogen remaining in the central reservoir will be frozen by the liquid helium. Nitrogen ice forms an effective insulating layer which will prevent the cold plate reaching its intended operating temperature. A large amount of expensive liquid helium will also be wasted in creating a small amount of very cold nitrogen ice!

The supply of dry nitrogen gas can be continued until the stream of ejected liquid nitrogen ceases. Ensure that the blow out tube does not block, that it is properly located and it reaches the bottom of the helium reservoir.

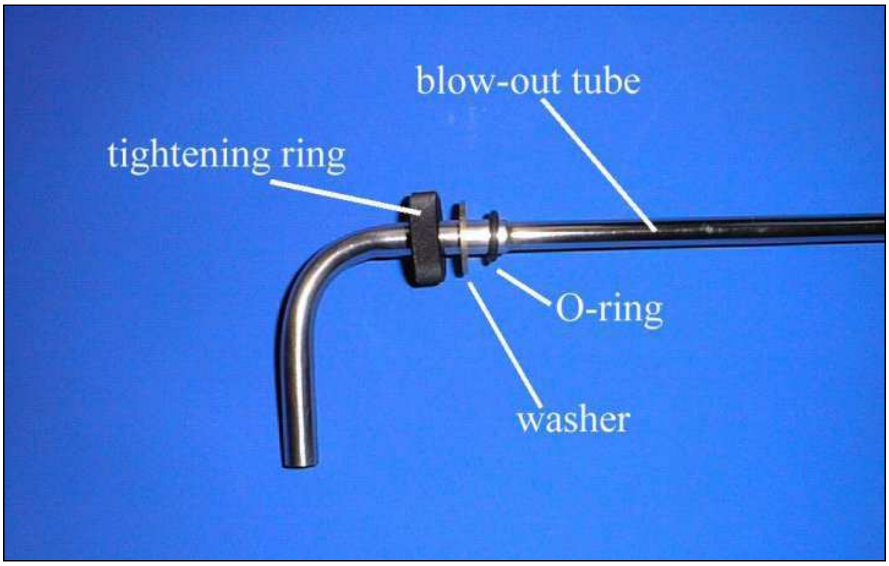

Photo 3.3. The blow out tube

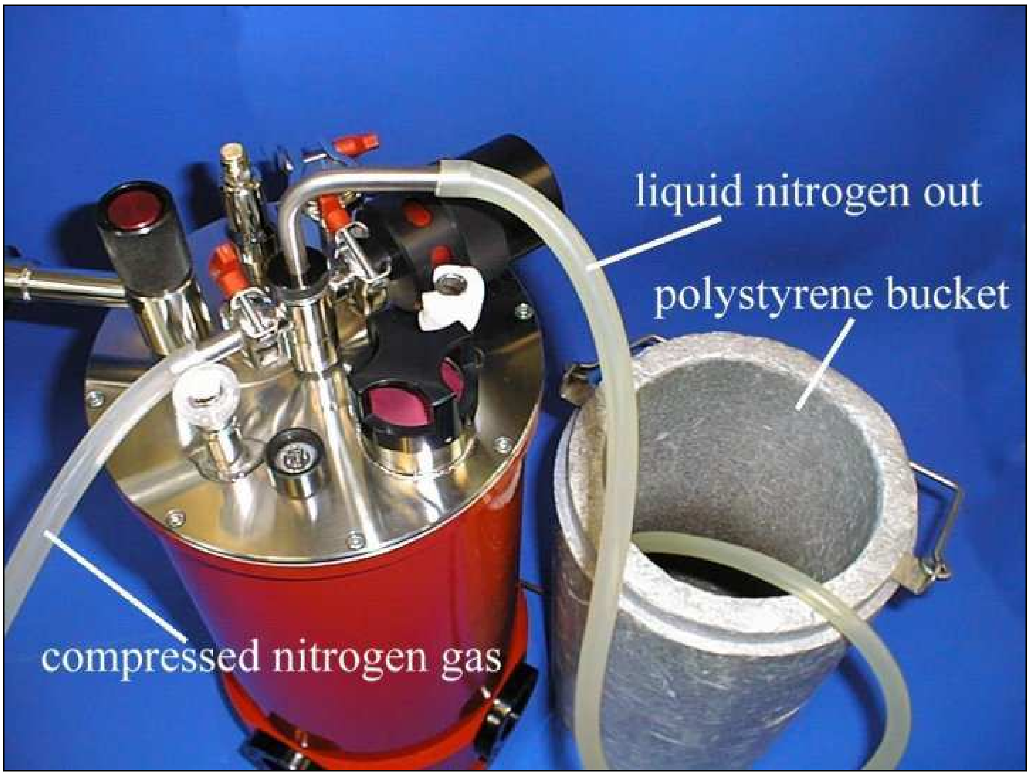

Photo 3.4. Arrangement to blow the liquid nitrogen out of the liquid helium reservoir

4. Liquid Helium Transfer

When you are certain that all liquid nitrogen has been removed from the central reservoir the cryostat can be filled with liquid helium. The blow-out tube should be removed from the central neck and the cryostat should be arranged such that the transfer tube reaches the bottom of the cryostat and the storage Dewar simultaneously.

It is wasteful to transfer liquid helium too quickly. A rubber bladder can be used to control the pressure driving the transfer, and the rate of filling can be judged from the size of the plume of exhaust helium gas rising from the cryostat.

The liquid helium transfer tube

It is important that the liquid helium transfer tube used is designed to suit both the cryostat and the liquid helium storage Dewar. The delivery end of the transfer tube should have a fully evacuated section with diameter approximately 6mm (¼ inch) and length at least 200mm. It should therefore permit liquid helium to be delivered efficiently into the central reservoir while at the same time leave space for spent helium gas to escape without a build-up of pressure within the cryostat.

QMC Instruments Ltd. can arrange to supply a suitable liquid helium transfer tube for your cryostat. We offer a rigid transfer tube, product code QTT/R, and a flexible transfer tube, product code QTT/F, with a reach in excess of 1000mm. Please contact us, or your supplier, if you have any questions regarding the suitability of your equipment.

Photo 4.1 depicts a liquid helium transfer in progress. **Photo 4.2** shows a typical boil-off plume in the phase when the cryostat is cooling between 77 K and 4.2 K. **Photo 4.3** shows the larger, cloudier and more erratic plume, which results when the liquid helium reservoir is full. At this stage the transfer should be terminated. It should take about thirty to forty minutes for a TK1813 cryostat to cool down from 77K to 4.2K and to fill with liquid helium; and the whole process should consume about six litres of liquid helium.

Helium gas recovery

Here in Cardiff we have no facilities for recovering spent helium gas, hence all the liquid helium transfers undertaken in our laboratories are “open” in the manner shown in the photos. However some installations offer recovery facilities whereby a helium return line is attached to the exhaust port of the cryostat. Use the black anodized aluminium tightening ring and O-ring from the central neck fitting to make a seal around the liquid helium transfer tube. Under such circumstances, a coarse flow meter could be inserted in the return line to indicate flow rate from the transfer. Usually a steady flow-rate is indicated during the cool and fill phases of the transfer. When the reservoir is full however, the flow rate becomes erratic, and the transfer should be terminated. When the transfer is complete the transfer tube should be removed carefully but swiftly and the safety valves fitted without delay.

Photo 4.1. Liquid helium transfer

Photo 4.2. Helium gas exhaust during fill

Photo 4.3. Helium plume when complete

Keeping the cryostat cold

It is important to keep all the neck fittings and safety valves in place whenever the cryostat is cold. If these are removed for liquid helium transfer, they should be removed only at the last moment when all other preparations have been made. They should be replaced as soon as the transfer tube is removed.

The cryostat can be kept continuously cold by repeatedly replenishing the cryogens. The subsequent fill hold time for the liquid helium is shown in **Table 5.1** in **Section 5.** Note that the liquid nitrogen in the outer reservoir will require topping up more often than the liquid helium, and that the first fill liquid helium hold time may be shorter. This is because the initial liquid helium boil-off rate may be high if significant further cooling takes place when the transfer is complete.

When transferring liquid helium into a cryostat that already contains liquid helium, the transfer tube should be fully cooled before it is inserted into the cryostat neck. This prevents the warm transfer tube and warm helium gas from boiling away excessive amounts of the liquid helium already in the cryostat. In this case the transfer tube is inserted into the storage Dewar and the pressure control bladder inflated slightly to pass gas through the tube to cool it. When the transfer tube has cooled, thick milky helium gas emerges from the delivery end, **photo 4.4**, and the transfer tube can then be manoeuvred carefully to the cryostat and lowered into the central neck. The refill can then proceed in the way described above.

Photo 4.4. Liquid helium emerging from a cold tube

5. Cryostat Cryogenic Performance

The liquid helium hold-time of the cryostat is measured in QMC Instruments Ltd. tests and tabulated in **Table 5.1**. The liquid helium boil-off is measured as the cryostat cools and reaches thermal equilibrium. The base boil-off at equilibrium is recorded and used to determine the liquid helium hold-time of the cryostat. Note that a first fill will not last as long as a subsequent fill hold-time due to the high initial boil-off when the cryostat is cooling from liquid nitrogen temperature.

In order to achieve these figures it is important that the operating instructions laid out in this manual are followed, and that care is taken to ensure that the cryostat is completely full before the liquid helium transfer is terminated.

Liquid helium reservoir capacity / litres	1.79
Liquid nitrogen reservoir capacity / litres	1.75
Base helium boil-off / litres of gas per min at STP	0.23
Liquid helium subsequent fill hold-time / hours	98 ± 10
Liquid nitrogen hold-time / hours	22 ± 4

Table 5.1. System cryogenic performance of the TK1813 cryostat

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Contract details and guarantee

This equipment is guaranteed for a period of two years from the date of delivery against failure caused by defective materials or workmanship. Defective parts will be repaired or replaced on return to the final supplier at no cost, provided that failure is not due to misuse or mishandling after delivery. QMC Instruments Limited will assume no liability for loss of life or damage to property arising from the use or misuse of its products.

Purchase Order Number
Purchase Order Date
QMCIL Reference
System Serial Number

On receipt of your shipment

Please check that your equipment has arrived safely. Please advise QMC Instruments if you suspect any damage has been incurred during transport and delivery or if any of the items are missing.

This operating manual contains instructions for operation of the cryostat, against which our guarantee is given as stated above. The user is advised to read this document carefully prior to operation of the cryostat and is reminded that our guarantee will be invalidated if the equipment is damaged through misuse.

Signed.. Date...
Ken Wood - Director, QMC Instruments Ltd.

QMC Instruments technical staff will be happy to advise you if you have any questions or difficulties. The contact details are:

Ken Wood (Sales and Marketing Director)
QMC Instruments Ltd
Cardiff University
School of Physics and Astronomy
Queens Buildings
The Parade
Cardiff
CF24 3AA
UK

T. +44 (0) 29 2045 1071
F. +44 (0) 29 2045 1271
E. k.wood@terahertz.co.uk
www.terahertz.co.uk