QMC Instruments Ltd

Liquid Helium Bath Cryostat Operating Manual

Model TK1840M
Split-case Kevlar-threaded cryostat

QMC Instruments Ltd

Contents

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First - A Word of Warning

Lifting and Handling the Cryostat

Take care when moving / lifting the cryostat because it is heavy. You should ensure that provision is made for manipulation of the cryostat.

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 cryostat that was pressurised because the boiling helium could not escape. 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.

Introduction

- ## The liquid helium cryostat. Type TK1840M

This is a liquid helium bath cryostat supplied by our sister company Thomas Keating Ltd.

This cryostat is of a special design to house particular equipment. The outer vacuum case extension and radiation shields extensions are quite large (see later photos) and are detachable at the position of the cold plate (split-case) to allow easy access. For improved cryogen efficiency the stages are supported by low thermal conductivity and strong Kevlar thread, which also gives more useable room on the cold plate.

- ## Serial numbers

Item	Serial Number
Thomas Keating Ltd Type TK1840M cryostat identification number	xxxxx

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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.

Thomas Keating Ltd. Type TK1840M cryostat

- Cryostat fitted with
  - Transit protection fixtures
  - Safety pressure relief valve
- Cryostat central neck safety baffle which includes:
  - Over-pressure relief valve
- Outer vacuum case extension and base-plate with O-ring
- 77K radiation shield extension and base-plate
- Liquid nitrogen blow-out tube with washer and O-ring
- Non-return valve
- Two off preamplifier mounting screws
- Two horns mounted on an aluminium plate and associated bolts / fixings
- Assorted bolts and screws
- Spares kit which includes:
  - 1 off 10 pin socket and shroud
  - Complete set of O-rings and washers
  - Set of screws
  - 2 off brass disks for the blow-out tube
  - Blanking disks and circlips
  - NW16 clamp, O-ring and 16mm KF adaptor nozzle type pipe fitting
  - M3 and M4 Allen keys
- 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 the cryostat 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.

Please note that some of the photos included are general photos that may not be specific to your particular cryostat.

Initial Inspection

Please inspect the box 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.

Photo 1.1. Transit base-plate

Removing the transit-plate

Refer to **Photo 1.1**

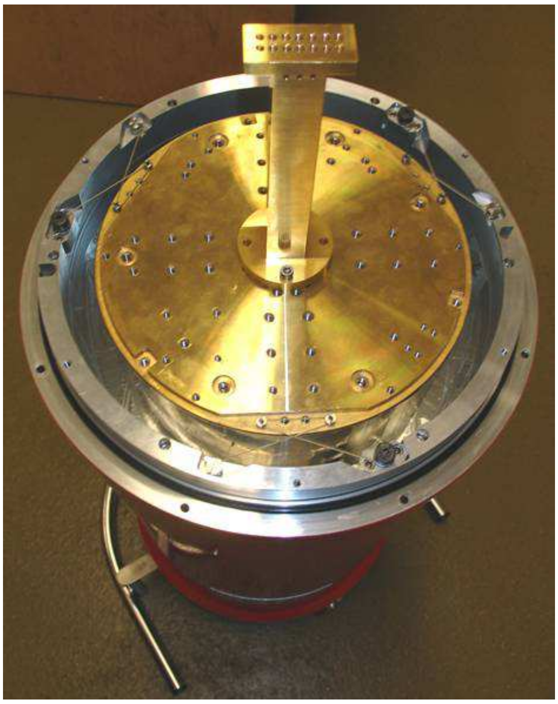
Invert the cryostat to allow access to the bottom-plate. The weight of the cryostat should not be allowed to rest on any of the top-plate fittings. Rather it should be supported by the top lifting handle. To avoid marking the chrome handle stand the cryostat on something to protect it such as soft tissue / cloth or bubble wrap.

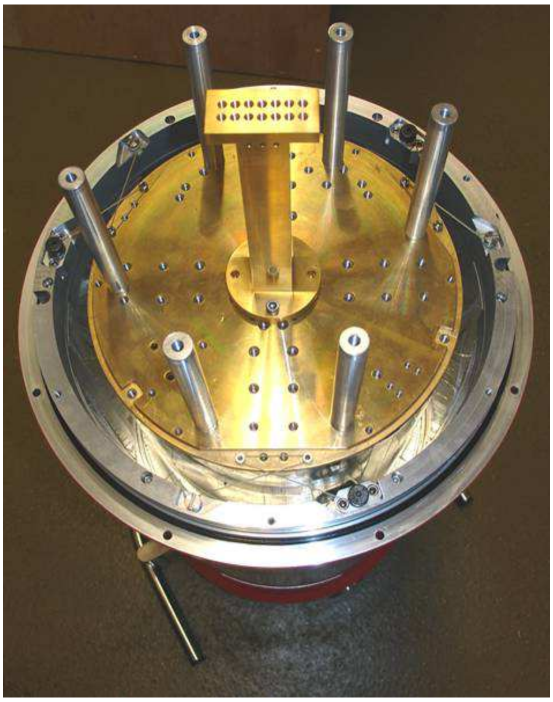
The aluminium transit base-plate should be removed by removing the socket head screws holding it in place and carefully lifting it off of the cryostat.

Fitting the included components, OVC and 77K radiation shield, and base-plates

Refer to **Photos 1.2, 1.3** and **1.4** which show the stages of assembly of the components accompanying this cryostat.

The TK1840 split case cryostat has a liquid nitrogen cooled 77K radiation shield extension and base-plate, and a room temperature outer vacuum casing (OVC) extension and base-plate. These can be fixed in place using the screws provided. It is important to check that the OVC O-rings are in place, are clean, well greased and that the seatings are free of marks and scratches. The screws locating the OVC base-plate should not be over-tightened, as this can distort the O-ring and perhaps 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.





Photos 1.2. Component assembly

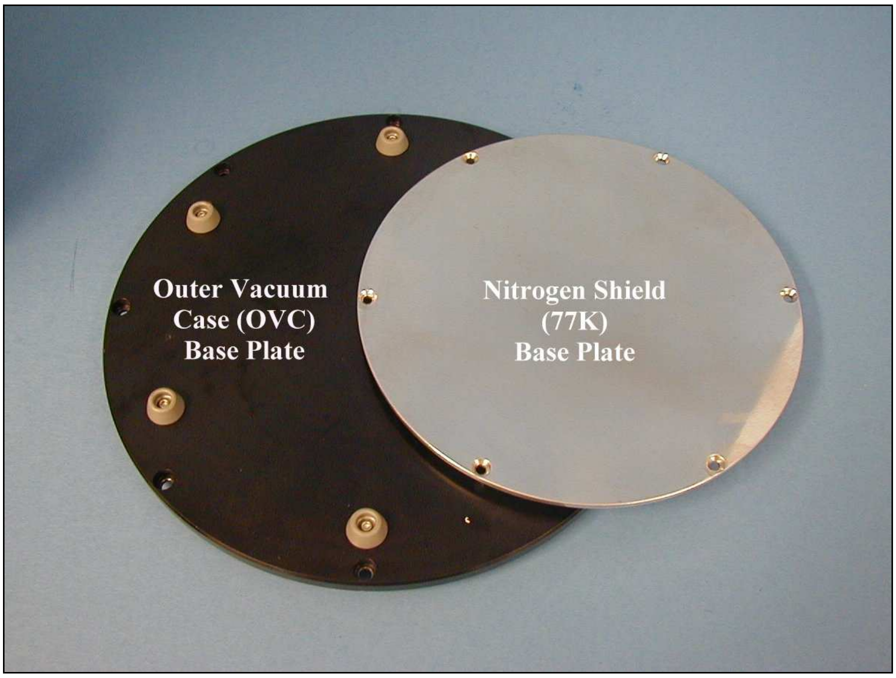


Photo 1.3. Cryostat radiation shield base-plates

Photo 1.4. Liquid nitrogen base-plate in position

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 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. This prevents rapid pressure changes that risk damage to the delicate components inside the cryostat.

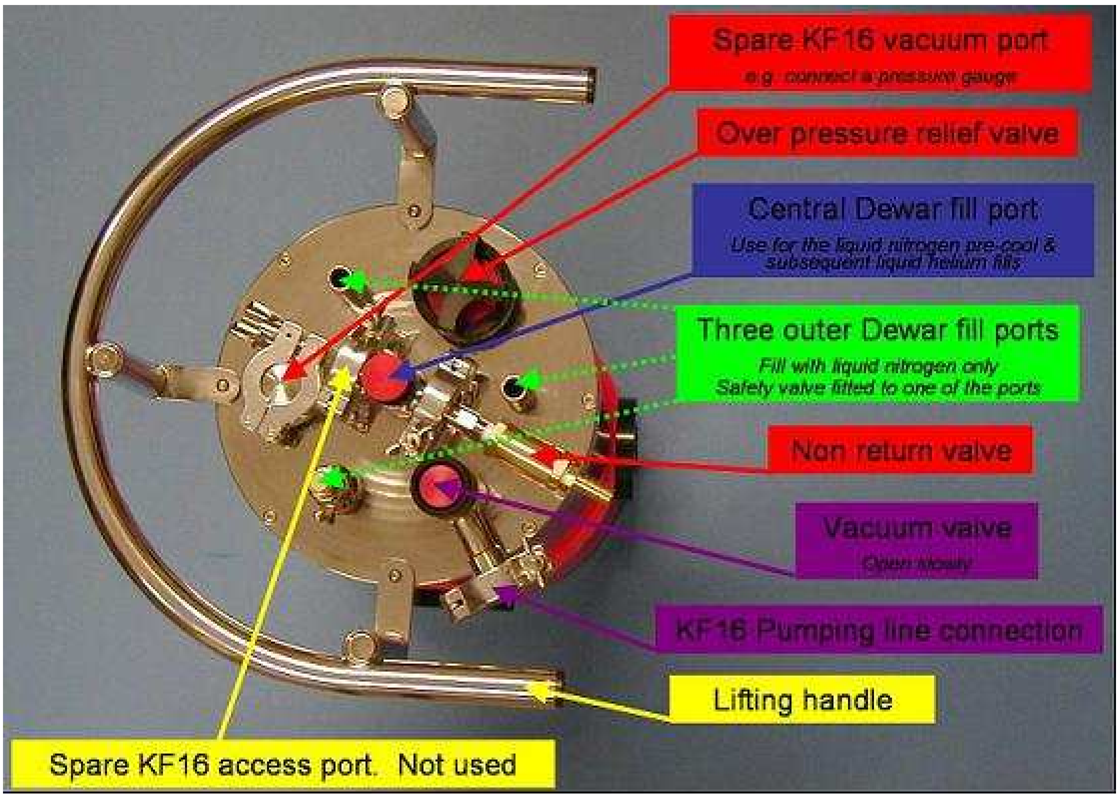


Photo 2.1. Cryostat top-plate fittings

3. Liquid Nitrogen Pre-cool

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

Vacuum pumping prior to cooling

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.

Liquid nitrogen pre-cool

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 pressurised liquid nitrogen storage Dewar which should take around 15 minutes to complete. You may have to 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 deep into the reservoir itself. For a TK1840 a length of at least 300mm 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.

When both reservoirs are full of liquid nitrogen, ensure that the safety valves are all in place

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.

Time to cool the cryostat to liquid nitrogen temperature (77K)

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 and TK1865) 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. The largest, Model TK1875, cryostat should ideally be pre-cooled for 24hrs.

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 around the central reservoir access port 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 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.

Ensure that all of the liquid nitrogen is removed

All liquid nitrogen must be 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 helium, and the ice forms an effective insulating layer which will prevent the cold components reaching their intended operating temperature. A large amount of expensive liquid helium will also be wasted in creating a small amount of very cold nitrogen ice!

At this stage therefore, 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 reaches the bottom of the helium reservoir.

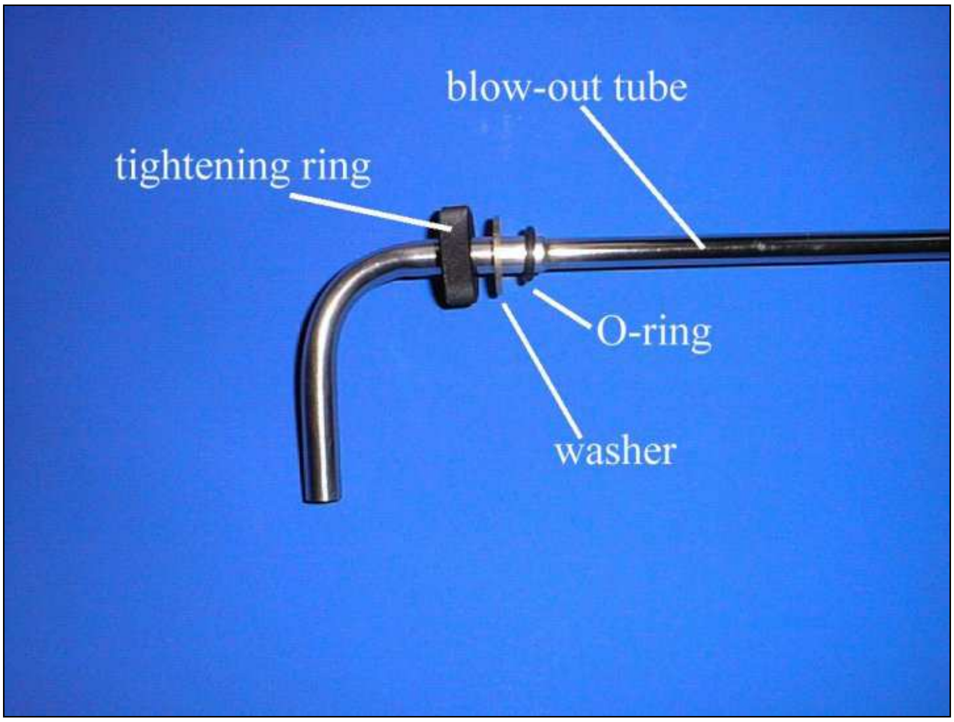


Photo 3.3. The blow out tube

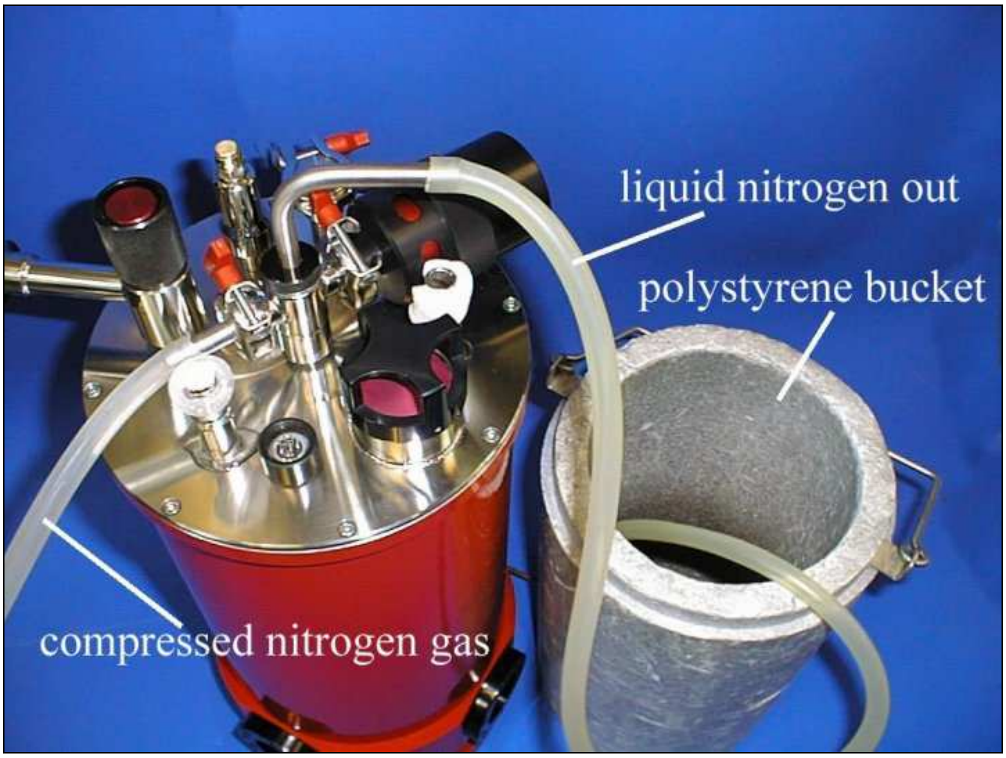


Photo 3.4. Arrangement to blow the liquid nitrogen out of the liquid helium reservoir

4. Liquid Helium Transfer

When the pre-cool liquid nitrogen has been removed 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 with a reach of 800mm, product code QTT/R, and a flexible transfer tube with a reach of 1000mm, product code QTT/F. 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 77K and 4.2K. **Photo 4.3** indicates 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 30 to 40 minutes for a TK1840 cryostat to cool down from 77K to 4.2K and to fill with liquid helium; and 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 flowmeter 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

Notes on 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. Hold times for both the liquid helium in the central reservoir and liquid nitrogen in the outer reservoir are 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.

The cryostat is ready for operation as soon as the liquid helium fill is complete

The performance may improve very slightly during the first hour or so after the first fill helium transfer while the internal components cool to their final operating temperature.

Photo 4.4. Liquid helium emerging from a cold tube

5. System Cryogenic Performance

The liquid nitrogen and liquid helium hold-times of the cryostat are measured in QMC Instruments Ltd. tests and tabulated below.

The liquid helium boil-off is measured over a few days to allow the internal components and radiation shields within the cryostat to reach thermal equilibrium. When equilibrium is reached the base boil-off is measured and used to determine the liquid helium hold-time of the cryostat. The hold-time indicated below is the subsequent fill hold-time. Note that a first fill will not last for as long due to the high initial boil-off when the cryostat is cooling from liquid nitrogen temperatures.

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.

The cryostat test log sheet is given in **Appendix B.** This shows exactly what steps were taken to run the cryostat and the elapsed time between each action.

Liquid helium capacity / litres	4.46
Liquid nitrogen capacity / litres	4.34
Base helium boil-off / litres of gas per min at STP	0.35
Liquid helium hold time / hours	160 ± 20
Liquid nitrogen hold time / hours	38 ± 4

Table 5.1. Cryogenic performance of the TK1840M split-case Kevlar-threaded cryostat

Appendix A. Filter and window transmission

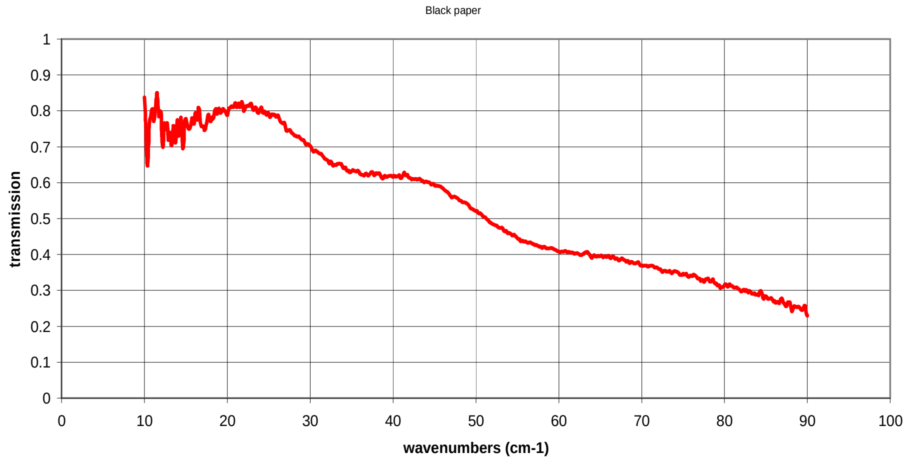


Fig A.1. Measured transmission spectrum of Carbon Loaded Black Paper

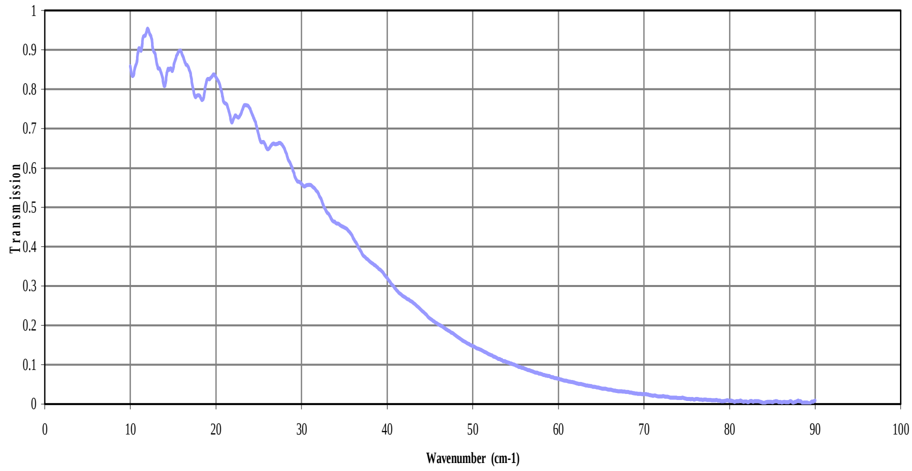


Fig. A.2. Measured transmission spectrum of 0.5mm thick Fluorogold

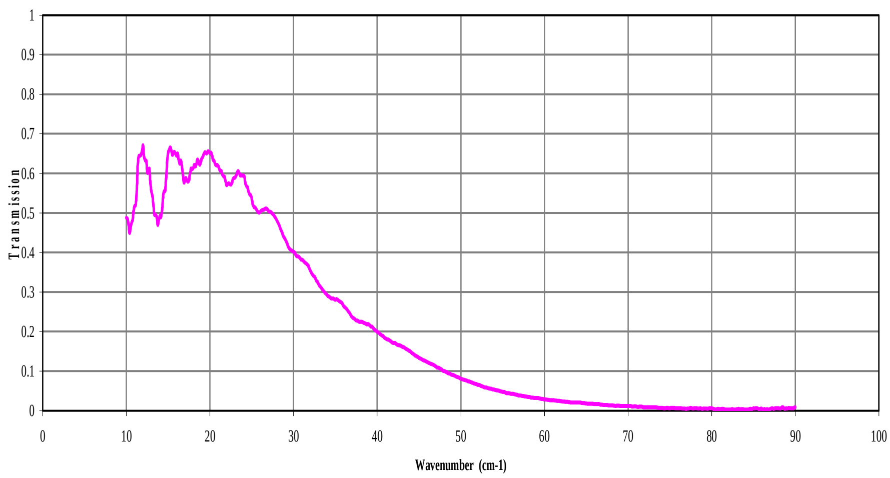


Fig. A.3. Measured transmission spectrum of Black Paper & 0.5mm Fluorogold

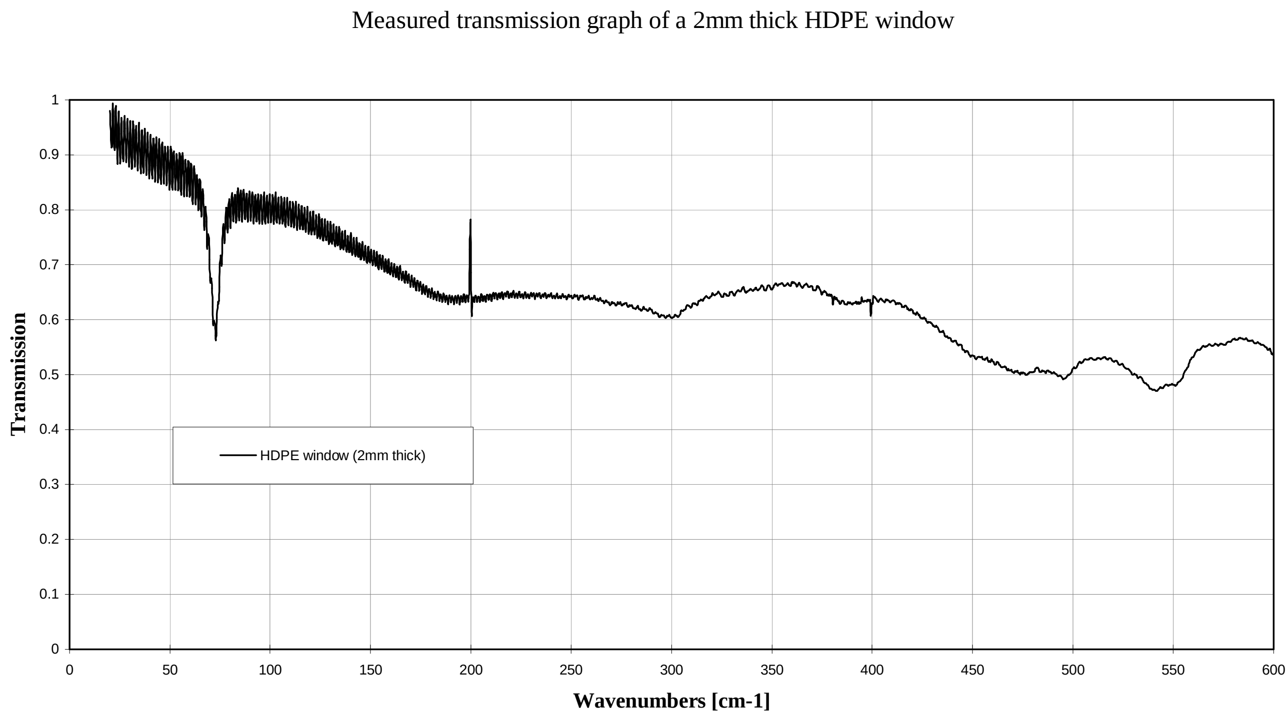


Fig. A.4. Measured transmission of a 2mm thick HDPE window from 20cm^{-1} to 600cm^{-1}. The polyethylene characteristic absorption increase is clearly seen around 73cm^{-1}