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A LIQUID HELIUM COOLED UHV SAMPLE MANIPULATOR FOR SURFACE SCIENCE WORK

by

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Abstract.

A liquid helium cooled ultra-high-vacuum sample manipulator has been designed and built for surface science work at Daresbury. It cools samples from room temperature to 25K in 20 minutes. After 1 to 2 hours it reaches a base temperature of 20K after which it requires a liquid helium supply of 1 to 1.5 litres/hour. The reduction in temperature decreases the thermal contribution to the measured width of the tantalum Fermi level (FWHM) from 91meV to 7meV. The sample mounting plate can be heated to 1300K for sample cleaning and annealing. Proposals for future development include a sample mount with a much reduced thermal load from ambient radiation and thermal conduction, an improved sample mount geometry for better access to electron energy analysers and electronic control of temperature between 20K and 300K.

1. Introduction.

A sample manipulator is needed for performing UHV surface science experiments with very low sample temperatures. Examples of experimental phenomena with such a requirement are:

(i) Investigating temperature dependent phenomena at low temperatures (e.g. high Tc superconducting materials);

(ii) The reduction of thermal broadening of features in photoemission spectra;

(iii) The study of surface interactions at very low temperatures;

(iv) In surface X-ray diffraction, to reduce the background due to thermal diffuse scattering.

Perhaps the most urgent need for an instrument of this type is in the characterisation of the newly discovered high Tc superconductors. Photoemission experiments have played a major role in the studies of the electronic structure of these materials. Typical early studies on the LSCO (Tc~35K) and YBCO (Tc~90K) systems failed to reveal any significant emission just below the Fermi level[1]. This absence of states within a few kTc

of the Fermi level implied that these materials should not even exhibit metallic behaviour much less that of superconductors. This prompted the conclusion that band calculations on these materials were inadequate since they predicted an appreciable density of states at the Fermi level. The experimental situation changed dramatically with the work on low temperature (20K) cleaved $EuBa_2Cu_3O_7$ where a clear Fermi edge was observed[2,3].

Thermal broadening in photoemission work can limit the energy resolution of an experiment[4]. A simple example of this is the width of the Fermi edge as a function of temperature[5]. At room temperature (~300K) this effect will contribute 91meV* to the width of the Fermi edge in a photoemission spectrum; the remaining contribution to the width coming from the energy distribution of the photons (i.e. monochromator resolution) and the resolution of the electron energy analyser. If a liquid nitrogen cooled manipulator is used a base temperature of approximately 120K can be achieved, reducing this effect to 37meV*. At 20K the thermal contribution is only 7meV* thus features with a considerably narrower natural width can be resolved than would otherwise be possible. Eliminating the thermal contribution to a photoemission feature such as a Fermi edge allows monochromators and electron energy analysers to be characterised. New science can also be done, such as the separation of closely overlapping core level spectra in surface core level shifts work[6] if the instrumental resolution is also good enough.

Temperatures only achievable with liquid helium cooling are necessary when studying certain "condensed" gases (such as condensed nitrogen and argon) which can also be used for monochromator characterisation purposes by measurement of absorption edges in the soft x-ray region.

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*Widths given taken from FWHM of differentiated Fermi functions for these temperatures (equivalent to 112, 46 and 11meV respectively using width between 10% and 90% of height of Fermi function).

Surface reconstruction or changes in the properties of surfaces and surface/adsorbate systems may only occur at such low temperatures. These systems could not otherwise be studied.

2. Practical Considerations.

With many surface science experiments it is necessary to heat the sample to high temperatures for cleaning and surface restructuring purposes. With this in mind the requirements are:

(i) the sample should be in good thermal contact with the liquid helium cooled heat exchanger at low temperatures to attain the best base temperature;

(ii) it should be thermally isolated from it at high temperatures to allow rapid heating cycles for sample cleaning without significantly affecting the temperature of the heat exchanger and;

(iii) the sample should be electrically isolated to allow drain current measurements and sample biasing.

Other cryostat-sample manipulators have been designed specifically for a certain type of sample such as that of Palmer et al[7]. In their design the sample is heated by passing a current directly through it. This limits the sample type to materials with a suitable resistivity (in their case pyrolytic graphite, the current passing in a direction parallel to the atomic planes). The sample size is also constrained due to the method of mounting whereas in the design presented here the sample type and size are relatively unconstrained.

An example of a commercially available manipulator is the Vacuum Generators Liquid Helium Cooled Sample Manipulator[8]. The most significant difference between this and the manipulator described here is that the Vacuum Generators sample holder features rotation of the sample about its surface normal. This means however, that the radiation shield is necessarily far larger in order to accommodate this movement, in fact 95mm in diameter. A radiation shield of this size would be impractical in most of the UHV vacuum systems at Daresbury and with the shield removed the base temperature is limited to 45K at best (manufacturers data). The heat transfer from the sample plate to the heat exchanger in this design is by copper braid in the same way as

their standard SM range of sample stages, necessary again because of the azimuthal sample rotation. This limits the base temperature of the sample due to the length of the path between sample and heat exchanger, increased heat loading from external radiation and the extra temperature drops across the junctions of the braid to sample plate and heat exchanger.

3. Mechanical Construction.

The manipulator consists of a standard Vacuum Generators HPLT series XYZ table with a differentially pumped rotary feedthrough (DPRF) and an Oxford Instruments CF1100 special continuous flow cryostat[9] (see figure 1). Unlike a standard HPLT manipulator the manipulator needs a DPRF to allow axial rotation as the helium entry is along the manipulator axis.

The sample stage (see figure 2) is radially compact to allow the sample to be positioned at short focal lengths (10 to 20mm typical) of the electron energy analysers used for photoemission experiments. The design features an OFHC (oxygen free high conductivity) copper heat exchanger (for good thermal conductivity at low temperatures and UHV compatibility) on which are mounted both the sample plate and heater. The sample plate is machined from high purity molybdenum which is a good thermal conductor and outgasses little (after conditioning) when heated to high temperatures during sample cleaning. A recess machined into the back of the sample plate aids local heating of the sample and minimises the effect of the heater on the heat exchanger (see figure 3). The plate is screwed to the heat exchanger by stainless steel M2 screws electrically isolated with ceramics.

Sandwiched between the sample plate and the heat exchanger is a 1mm thick polished synthetic sapphire plate (see figure 4) made by BDH Ltd.[10]. This material is an excellent electrical insulator but its thermal conductivity varies considerably with temperature (see figure 5) in exactly the way required for this application: it thermally insulates the heat exchanger from the sample plate when hot but provides a good thermal contact between the two when both are cold. For instance, if the sample plate is held at 1300K for extended periods (1 hour or more) the heat exchanger reaches a steady state temperature of only 800K.

Behind the sample plate and bolted to the other side of the heat exchanger is the heater. This is electrically isolated from the sample plate and heat exchanger with ceramics. The heater is of the electron beam type; that is the sample plate is biased to a high voltage (500-1000V) and electrons emitted from a hot filament (thoriated tungsten wire) bombard the rear of the sample plate thus heating it. The filament is well shielded to improve efficiency and reduce sputtering onto the ceramics. The ceramics are also protected either by being placed out of "line of sight" of the heater or by the use of a molybdenum shielding cup.

The temperature of the sample is monitored using a type K Chromel-Alumel thermocouple (range 3K to 1640K) clamped to the sample plate. This choice of temperature monitoring was chosen to cover the wide range of temperatures of the cryostat. Other methods can be used to measure low temperatures accurately but they are either not suitable for use in UHV systems or unable to withstand the high temperatures used in sample cleaning.

Around the sample stage is a detachable OFHC copper radiation shield of all-welded construction. This shield is cooled by the cold helium gas returning from the heat exchanger and intercepts the vast majority of ambient radiation from the surroundings at room temperature. A small (15mm diameter) hole over the sample allows photons to hit the sample and electrons to leave it and enter an analyser. Hence the sample plate receives little radiation from outside the radiation shield.

The cryostat is cooled with liquid helium from a storage dewar at (or slightly above) atmospheric pressure. It is delivered by an ultra-low loss transfer tube the delivery end of which is inserted into the cryostat. The helium is delivered to the "flow" tube of the heat exchanger where it boils, cooling the cryostat. The cold exhaust gas flows back through the "return" tube to a secondary heat exchanger where it cools the radiation shield (see figure 6). From here the gas return passes back through the transfer tube leg in a tube surrounding the helium flow capillary, shielding the incoming helium from ambient radiation (see figure 7) and so minimising evaporation and consumption of liquid helium.

Although similar to a normal manipulator such as the Vacuum Generators HPLT series in use, the cryostat, due to its design

constraints and construction, differs in some areas. It has the normal range of movement in the x, y and z directions and unrestrained axial rotation as the liquid helium delivery is along the axis and the rotary feedthrough has no limit of rotation. The sample plate can not however rotate azimuthally, that is about the surface normal of the sample. This is because radial size limitations are imposed by the radiation shield and also that an azimuthal drive would result in a poorer thermal path between the heat exchanger and the sample, which would reduce heat transfer and limit the base temperature of the cryostat. The primary rotation, as previously stated, is via a differentially pumped rotary feedthrough which requires a rotary pump to maintain a vacuum of 10^{-2}mbar or less in the interspace between the vacuum seals.

The normal bakeout temperature for the DPRF is 150°C and steps should be taken to ensure the temperature of the feedthrough does not exceed this if the rest of the vacuum system requires a higher bakeout temperature. Alternatively it is possible to release the pressure on the top bearing of the DPRF which will allow the feedthrough to be baked at up to 200°C (see appendix 2).

The molybdenum sample plate accommodates samples of up to a size of 20mm x 15mm x 3mm. These are either clamped to the sample plate with tantalum clips or glued in place with UHV compatible silver loaded epoxy resin (Ablebond 36-2, Manufactured by The Dage Group[11]), the latter method providing a better thermal contact. The plate is 2mm thick with a 1mm recess cut into the back behind the sample area to facilitate rapid local heating of the sample without significantly affecting the heat exchanger.

4. Performance.

In use the cryostat reaches its base temperature (approximately 20K) in around 1 to 2 hours (see figure 8) although its temperature can drop slightly further with time as the radiation shield continues to cool. Once cold it requires approximately 1 to 1.5 l/h of liquid helium to maintain its base temperature.

The sample plate can be "flashed" to high temperature from cold using the electron beam heater. This is useful for removing contaminants that have been adsorbed onto the

sample when it has been cold for a long time or during the cooling-down period. Figure 9 shows the recovery of the sample plate to almost the base temperature in around 10 minutes after rapidly heating (about 1 minute) from 20K to 800K.

5. Data Taken With the Manipulator.

The manipulator has been used extensively for characterising the surface science experimental stations of the SRS (Synchrotron Radiation Source) at Daresbury Laboratory.

The difference this instrument can make to energy resolution in photoemission work is demonstrated in figures 10 and 11 which are from work done on a VUV (vacuum ultra-violet) station (TGM 6.2) with a toroidal grating monochromator (energy range 15 to 90 eV). Figure 10 shows photoemission spectra of the Fermi edge of a polycrystalline tantalum sample at room temperature and at 20K. Figure 11 shows the derivatives of these two spectra. The FWHM of these are used to assign a width to the Fermi edges; these are 125meV and 68meV respectively. The contribution to the width of the Fermi edge at 20K from thermal broadening is negligible (7meV); the measured width is effectively a combination of monochromator and electron energy analyser resolution and thus can be used to asses their performance.

The data in figure 12 were taken on a soft X-ray station (NEX 1.1: energy range 220 to 1200eV) and shows an absorption feature of condensed argon (at approximately 50K) taken with two different beam currents in the SRS storage ring. The feature shown is an X-ray absorption peak corresponding to a transition from the ^2P level ($^2P_{3/2}$) to the ^4S level ($^1S_0$) of argon. The increase in width of the peak with stored beam current is thought to be due to a change in source size with stored beam current although a change in the properties of the beamline optics with heat loading from the X-ray beam could be an important factor.

The spectrum in figure 13 is again of condensed argon at 50K and was taken on the undulator beamline (beamline 5U) of the SRS. As well as the peak in figure 12 the spectrum shows other absorption features. The X-ray absorption and photoemission spectra of condensed gases such as argon and nitrogen have been under investigation in an effort to explain the changes in lineshapes of electronic levels

between the gas phase and condensed phase[12].

The manipulator has been used in high Tc superconductor work on TGM 6.2 investigating the surface reactivity of a $Bi_2Sr_2CaCu_2O_8$ single crystal surface in ultra-high vacuum[13] although in this case only 90K was required for the experiment.

This manipulator is now in routine use for surface science experiments and station characterisation.

6. Future Development.

Some auxiliary equipment would be desirable to facilitate the use of the cryostat, in particular more accurate temperature measurement and control of temperature between the base temperature and ambient. Purpose built type K thermocouple linearisation electronics are planned to overcome the problem of the non-linearity of the thermocouple at low temperatures (see figure 14). A temperature controller with three term control (proportional, differential and integral) is needed to stabilise the temperature between the base temperature and ambient. This would probably use the electron beam heater filament as a radiant heat source in conjunction with constant liquid helium flow to produce a steady temperature anywhere between 20 and 300K.

7. Design Considerations for a Second Liquid Helium Cooled Manipulator.

A second similar manipulator has been proposed and is being designed with several improvements:

The copper radiation shield would be replaced by a liquid nitrogen cooled shroud thus reducing further the radiative heat reaching the sample plate. At present the radiation shield, which intercepts almost all of the incident thermal radiation from the surroundings, is only cooled at the top by cold helium gas returning from the primary heat exchanger. The shield reaches a temperature of around -50°C (~220K) which is enough to reduce the radiation reaching the heat exchanger and sample plate by around a factor of 4 due to the T^4 dependence of absorbed radiation (Eqs 1 and 2).

$$Q_{Rad} = \sigma_B \varepsilon_{SP} A_{SP} (T_{RS} - T_{SP})^4 \qquad (1)$$

$$Q_{Rad} = \sigma_B \varepsilon_{HE} A_{HE} (T_{RS} - T_{HE})^4 \qquad (2)$$

where σ_B is the Stefan-Boltzmann constant, ε_{SP} and ε_{HE} are the total emissivities of the sample plate and heat exchanger, A_{SP} and A_{HE} are the surface areas of the sample plate and heat exchanger, T_{RS} is the temperature of the radiation shield and T_{SP} and T_{HE} are the temperatures of the sample plate and heat exchanger. The absorption of radiation can be reduced further if consideration is given to the emissivity of total radiation of materials used. The heat exchanger is made of copper which has an emissivity of 0.6 and stainless steel which has an emissivity of 0.35. If it were gold plated (by electroplating or vapour deposition) this would be reduced to around 0.02. The other source of heat load to the sample plate and heat exchanger is by conduction via the thermocouple and bias/drain-current wires and from the heater (Eq. 3).

$$Q_{Cond} = \frac{A_x K \Delta T}{L} \qquad (3)$$

where A_x is the cross-sectional area, K is the thermal conductivity of the wires, L is the path length of conduction along the wire and ΔT is the temperature difference across L. The new design will minimise the number of wires attached to the sample plate and heat exchanger and limit their cross sectional area to reduce heat conduction along them. A compromise will obviously have to be made between reducing the thermal load and the mechanical strength of the wires. Conduction can can also be reduced by minimising $\frac{\Delta T}{L}$. This is best achieved by clamping all wires to the top of the liquid nitrogen cold shield. Sapphire plates can be used for this to provide a good thermal contact and maintain electrical isolation.

The heater on the existing sample stage is designed to be in electrical isolation from the heat exchanger using alumina ceramics. These have a low thermal conductivity at low temperatures (see figure 5). This is now considered to be a design problem as the mass of

the heater plate may provide a slow "leak" of heat to the reservoir which will prolong the cool-down time. Also, if the heater plate remains significantly warmer than the sample plate it will provide thermal radiation which will limit its base temperature.

The heat exchanger will be redesigned to allow positioning of the sapphire plate away from the heater as contamination of its lower edge currently limits the time the heater can be used before shorting occurs between the sample plate and heat exchanger.

The first cryostat was designed with the axis of polar rotation coincident with the axis of the heat exchanger. This meant that the a sample stage had to be built with the front of the sample plate lying along the centre-line of the cryostat in order that, when the polar angle was changed, the centre of any sample mounted on the plate would change position as little as possible. As a result of this it is difficult to get the sample close to large electron energy analysers and the limit of off-normal-emission angles is very small (see figure 15).

The geometry of the second manipulator will be such that the axis of rotation is in front of the centre of the heat exchanger (see figure 16). This will enable the sample to be positioned closer to electron energy analysers and allow larger electron emission angles as the sample plate will be able to be rotated further before the radiation shield fouls the analyser.

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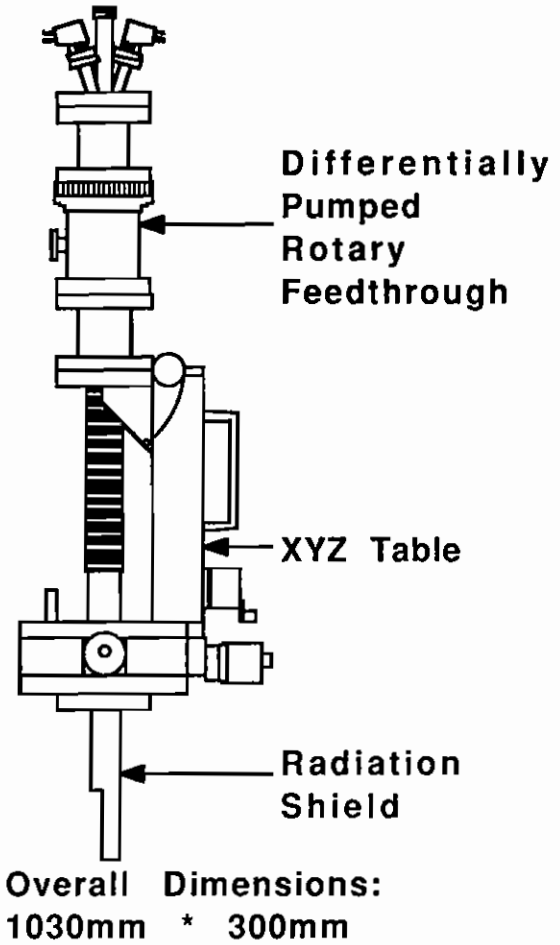


Figure 1. The Liquid Helium Manipulator.

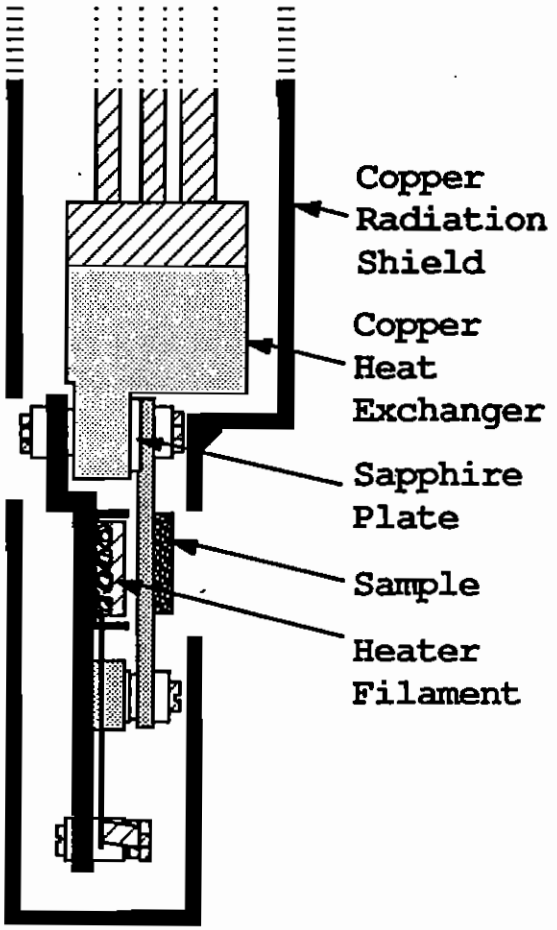


Figure 2. Sample Stage (cross-section).

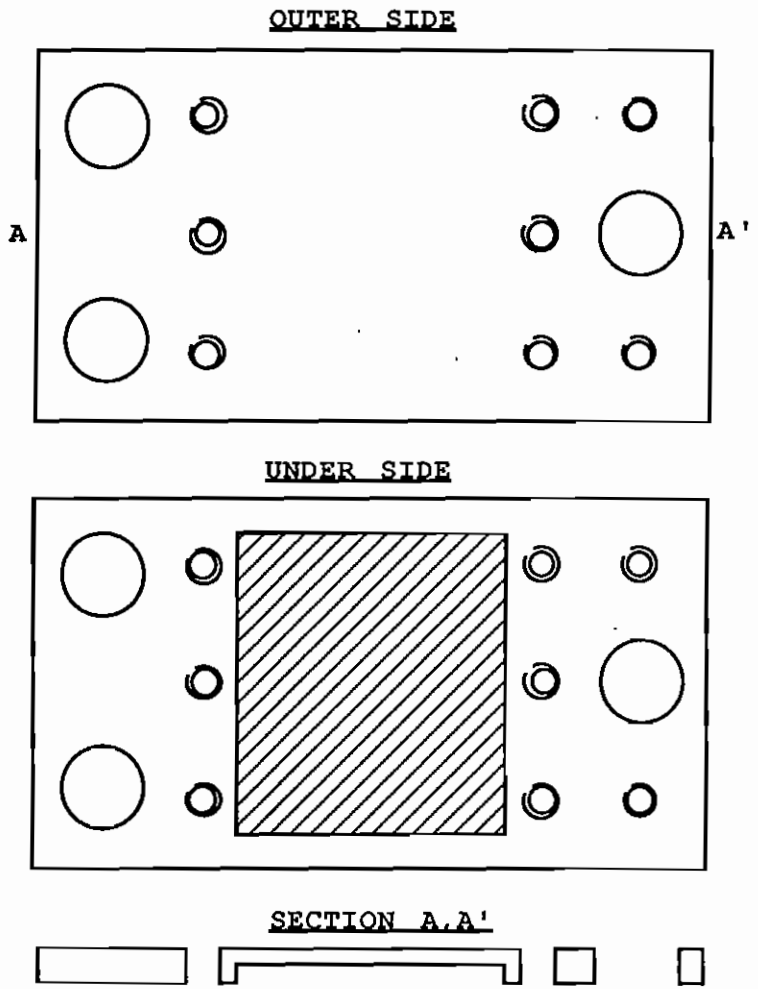
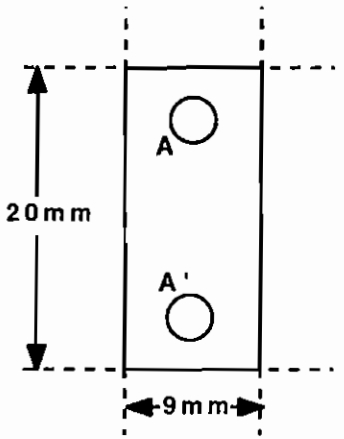


Figure 3. Molybdenum Sample Plate.



Thickness 1.0mm
Holes A & A'
3.0mm dia.

Figure 4. Sapphire Plate.

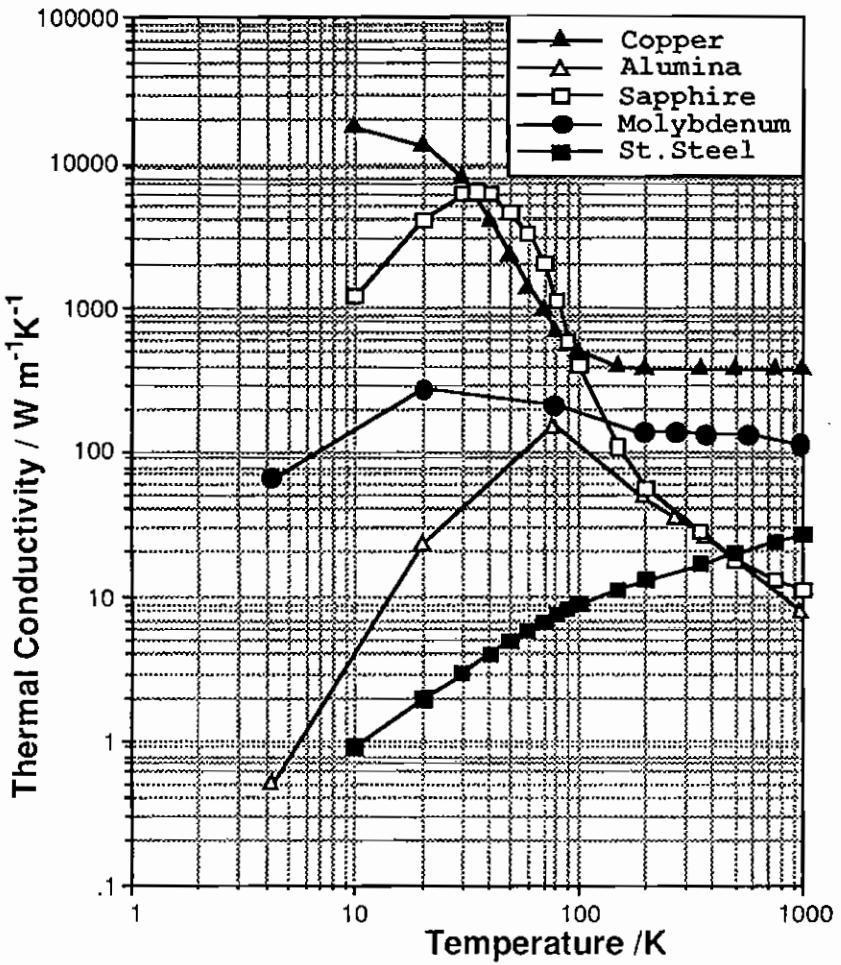


Figure 5. Thermal Conductivity of Various Materials.

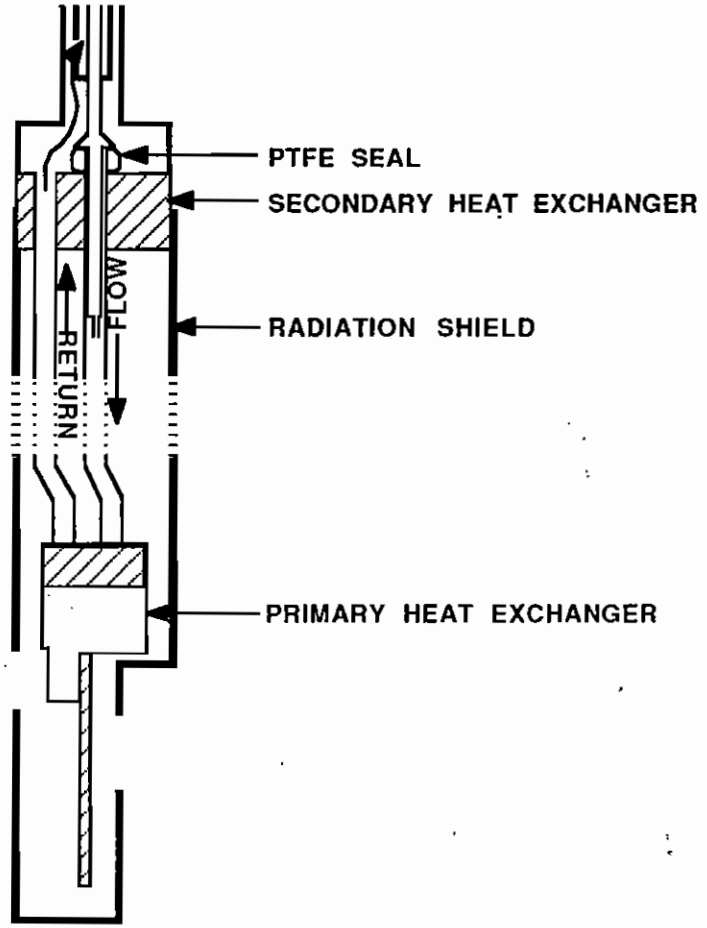


Figure 6. Liquid Helium Flow Diagram.

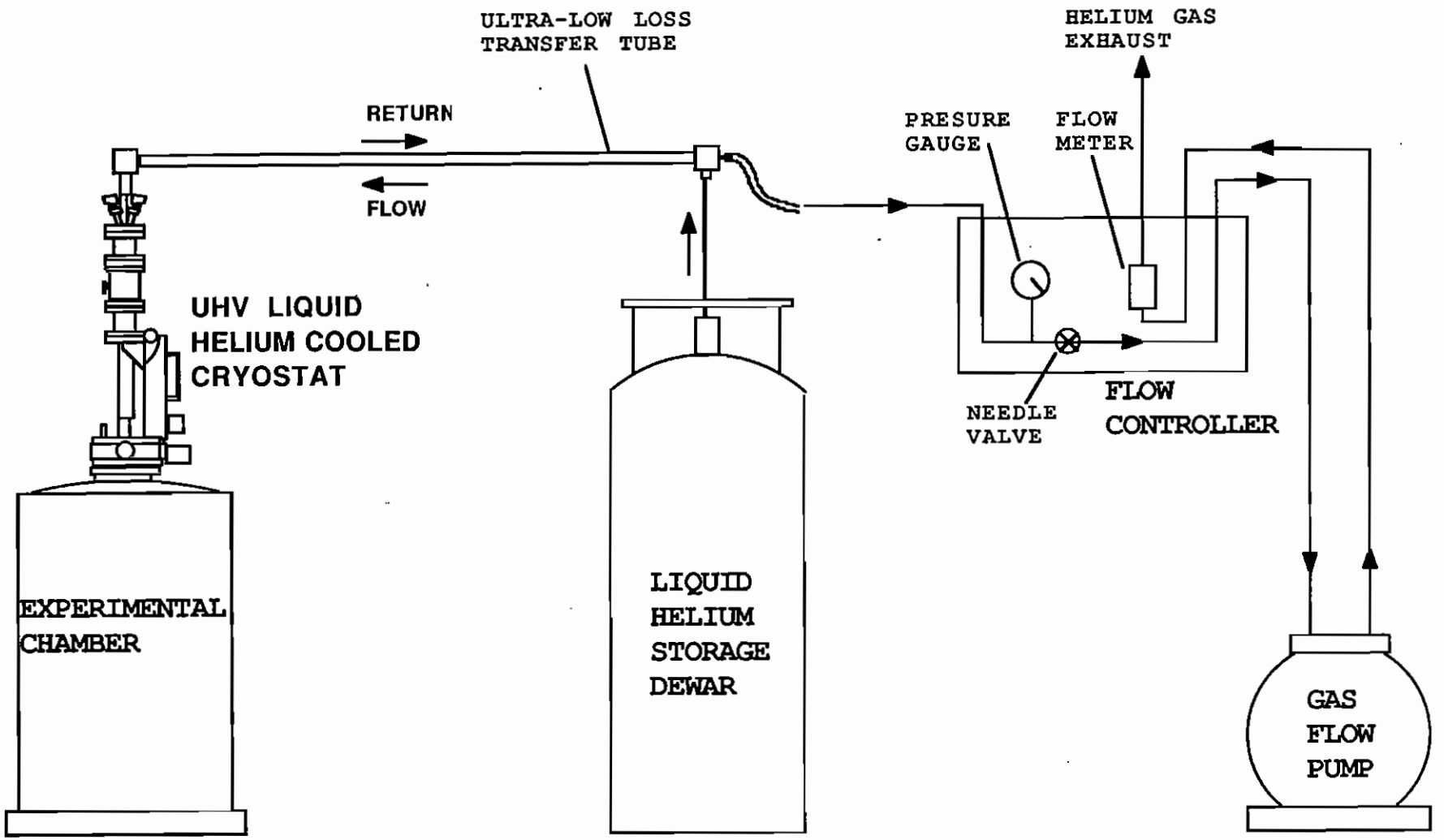


Figure 7. Liquid Helium Transfer Control.

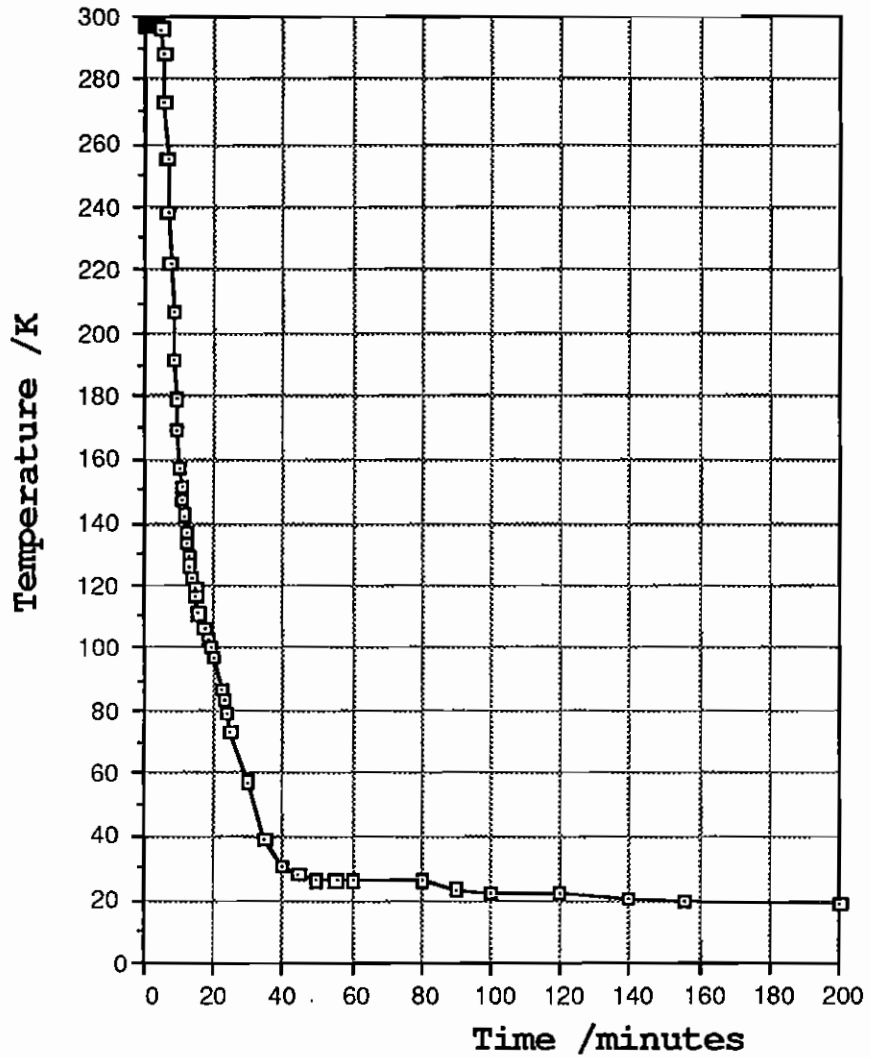
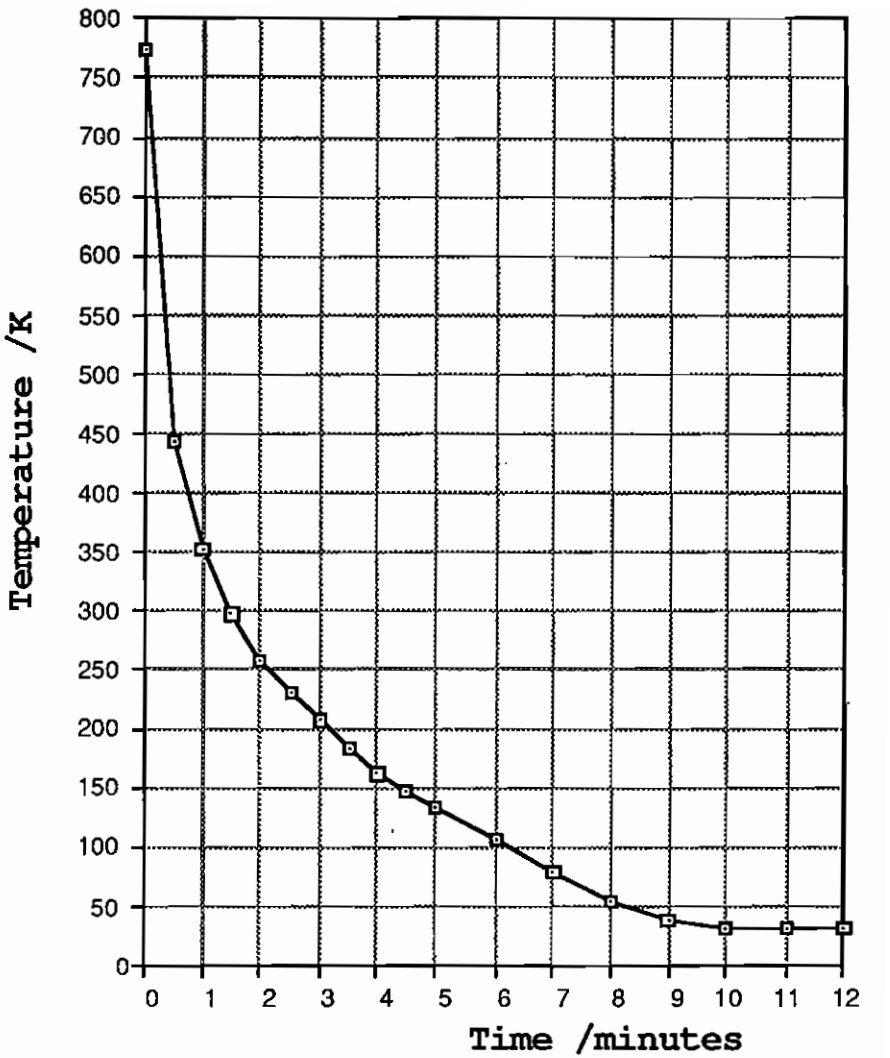


Figure 8. Cryostat Cooldown From Room Temperature.



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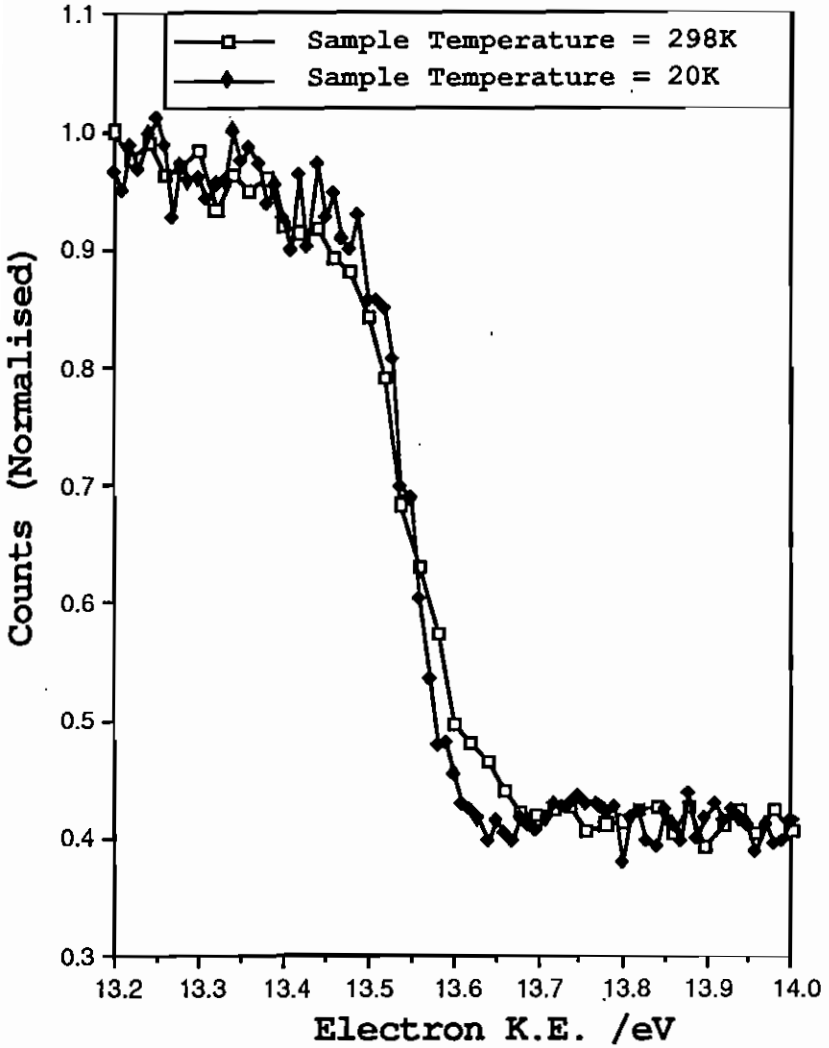


Figure 10. Tantalum Fermi Levels at 298K and 20K.

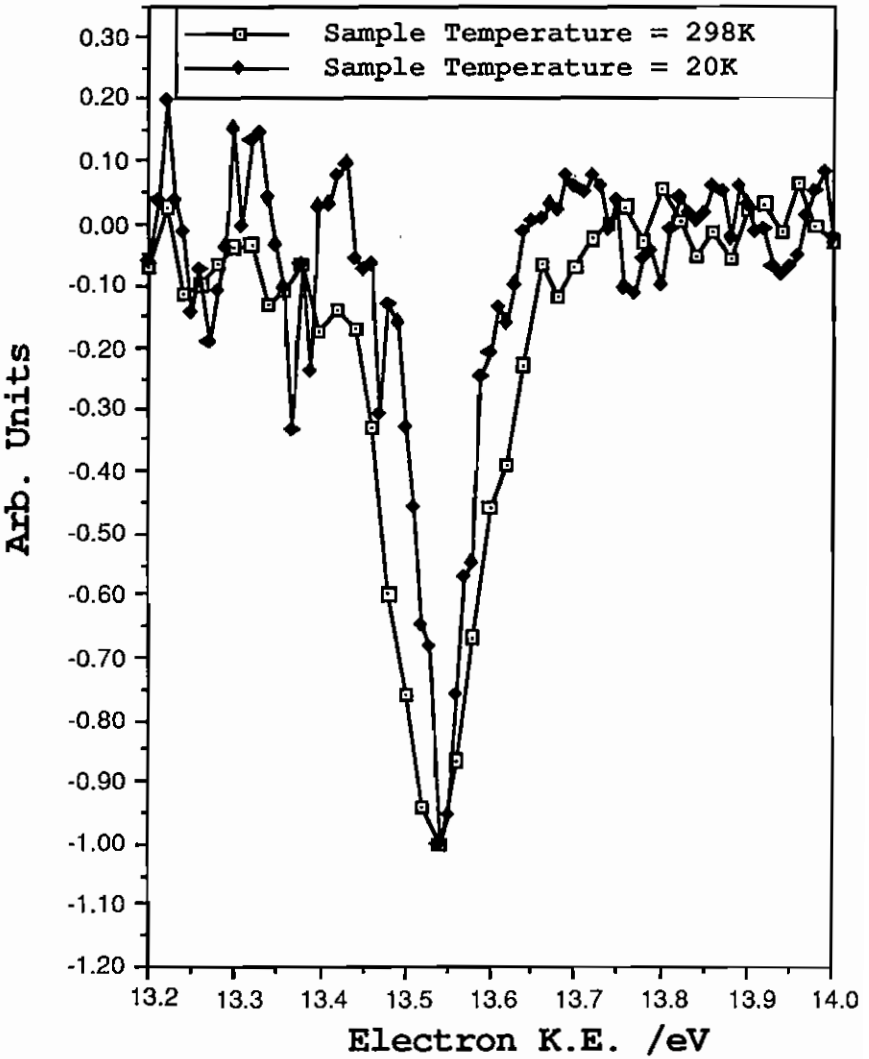
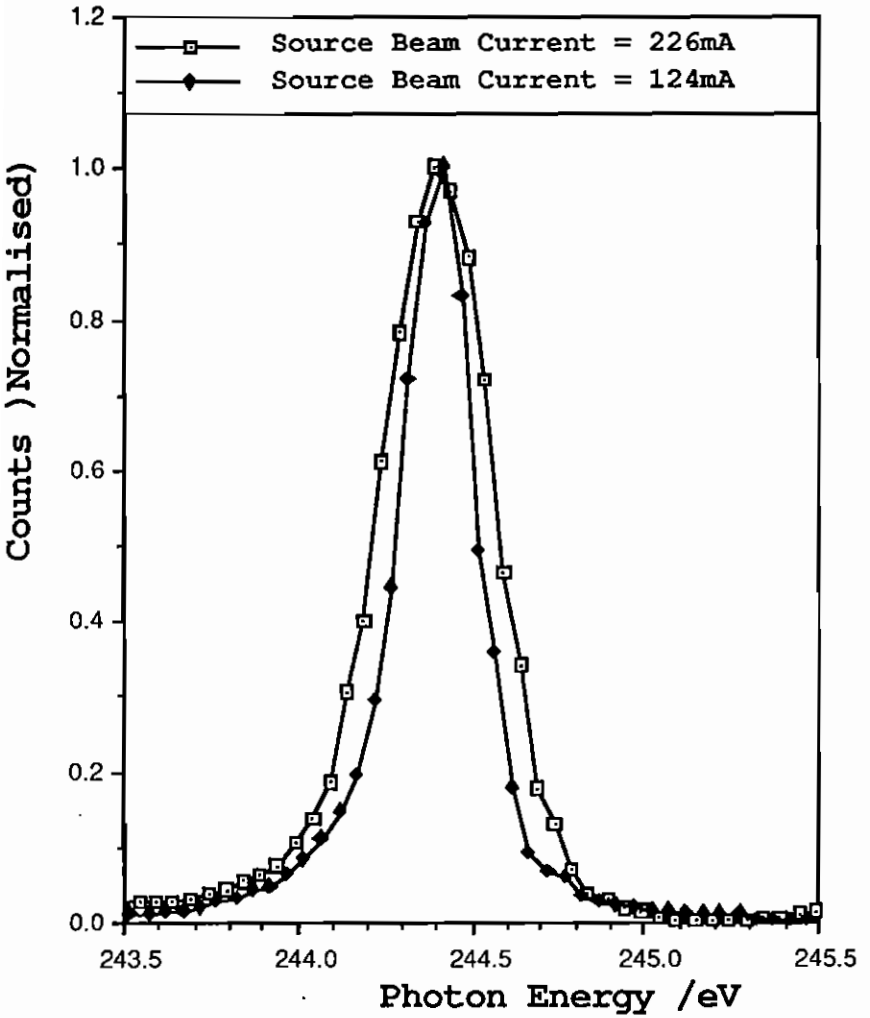


Figure 11. Derivatives of Tantalum Fermi Level Spectra at 298K and 20K.



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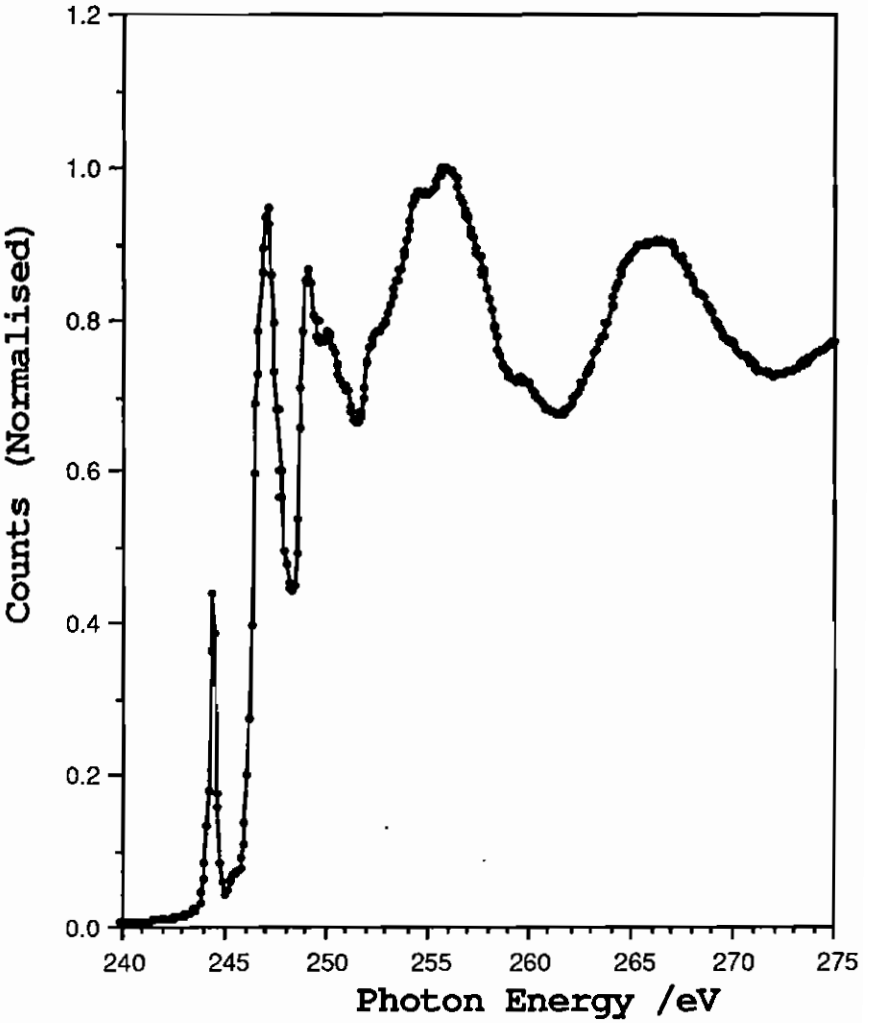


Figure 13. Total Electron Yield Spectrum of Condensed Argon.

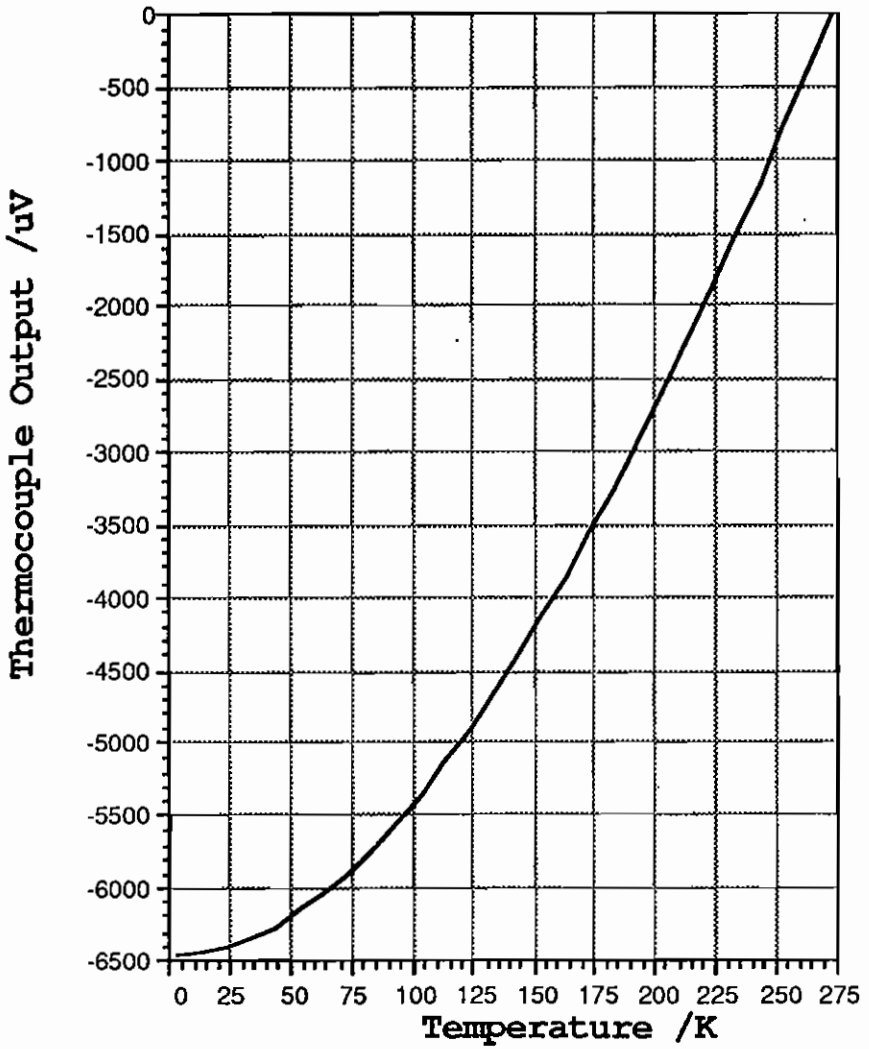
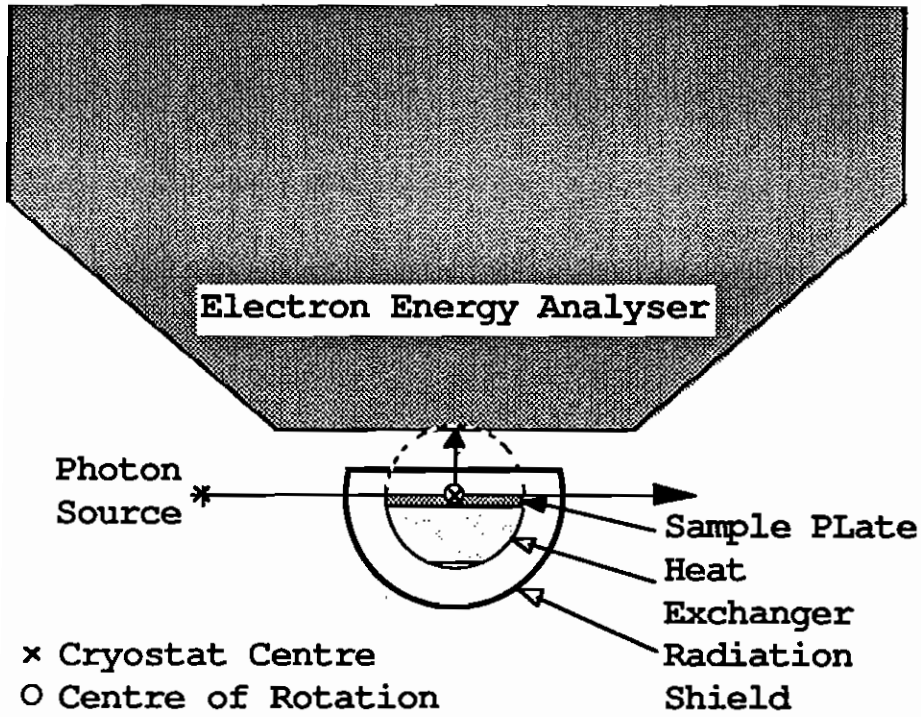
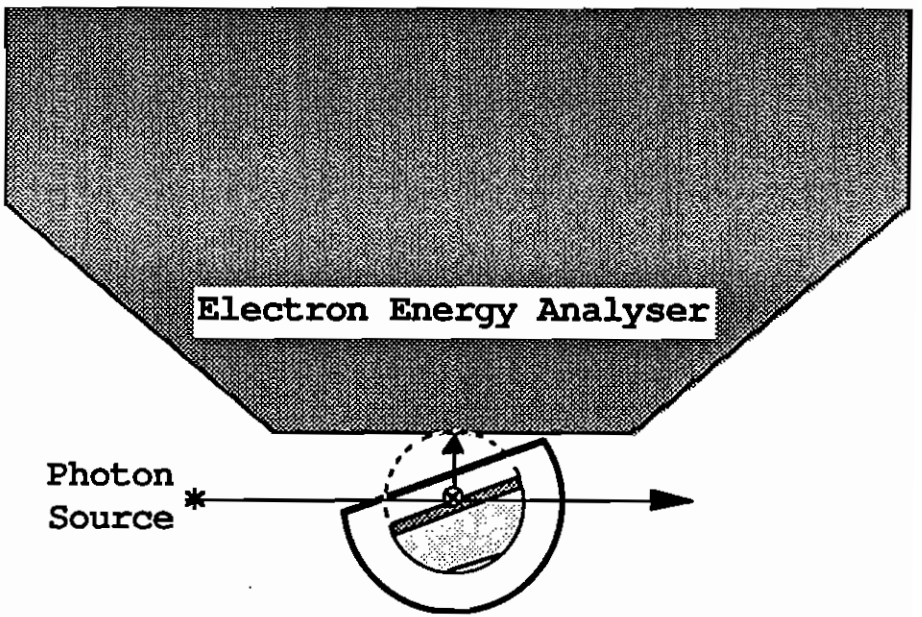


Figure 14. Type K (Chromel-Alumel) Thermocouple Output vs. Temperature.

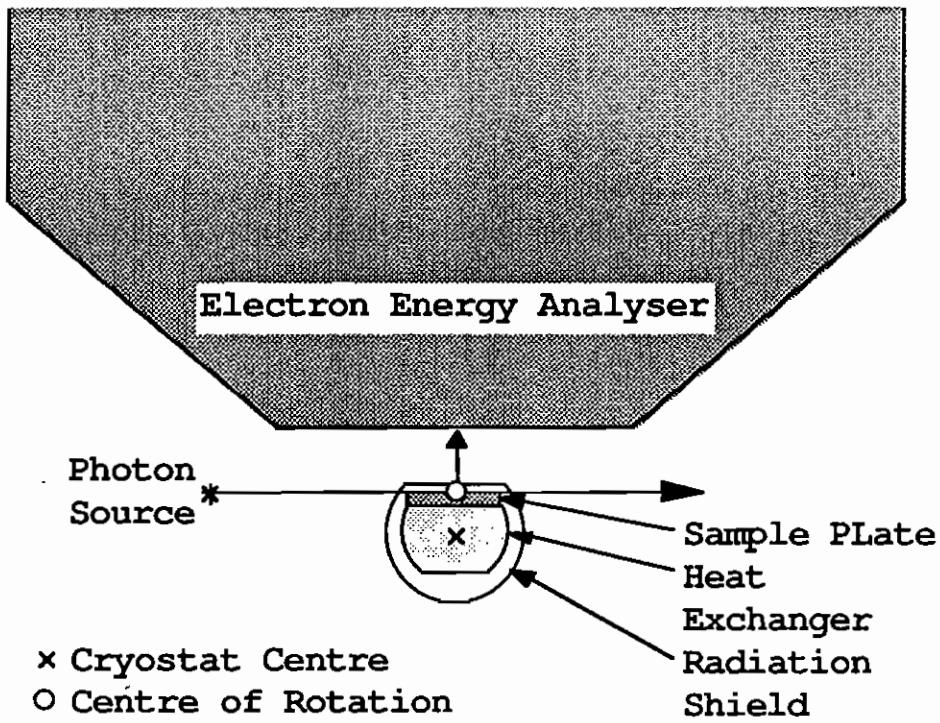


Normal Emission.

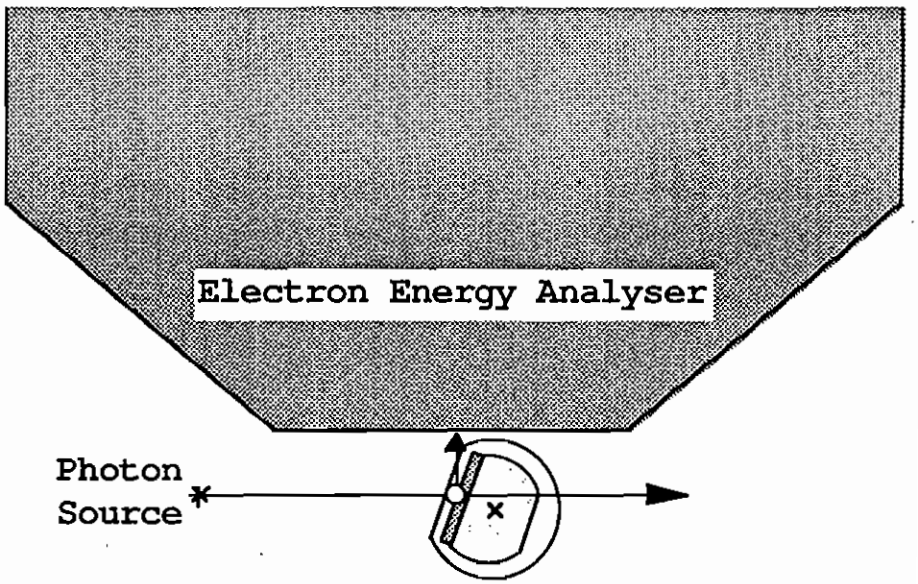


20° Emission Angle.

Figure 15. The Geometry of the First Liquid Helium Manipulator.



Normal Emission.



70° Emission Angle.

Figure 16. Proposed Geometry of 2nd Liquid Helium Manipulator.

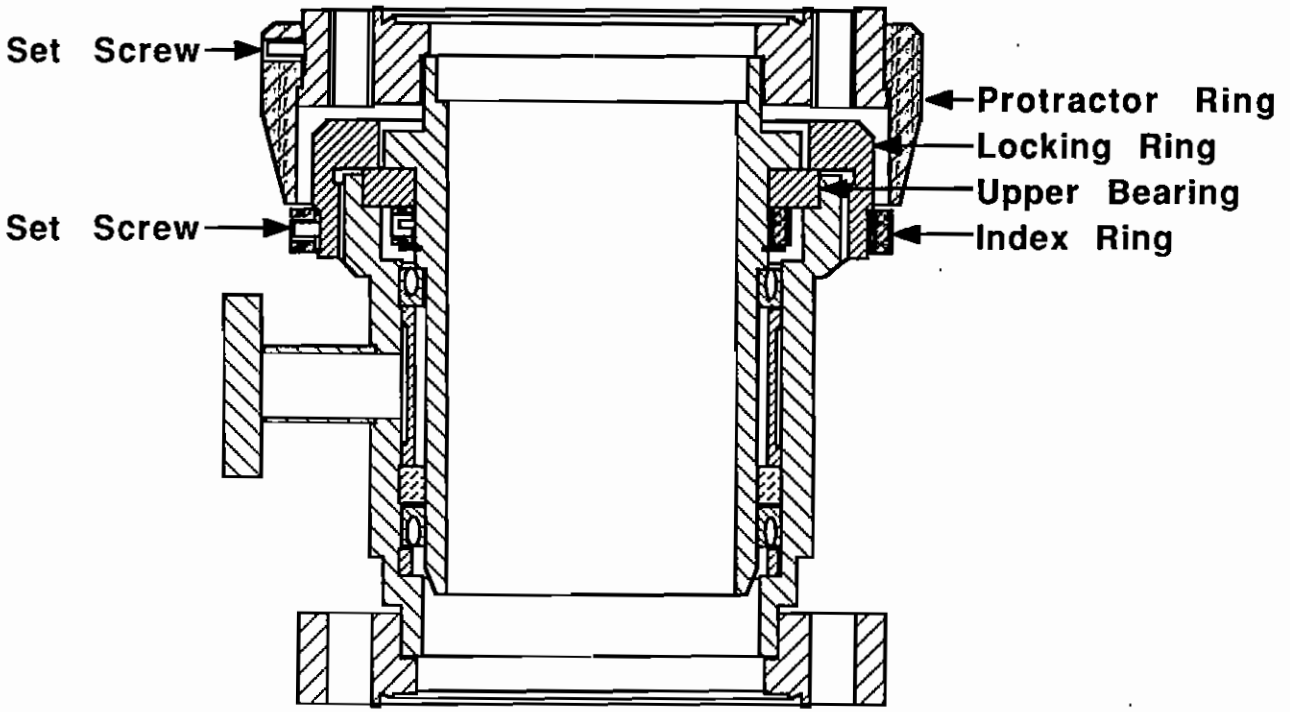


Figure 17. Differentially Pumped Rotary Feedthrough.

APPENDIX 1.

Instructions For The Operation Of The Liquid Helium Manipulator.

If you have not used this manipulator before, please ask for advice beforehand.

If you are unfamiliar with the safe use of liquid helium, advice can be obtained from the Daresbury Laboratory Health and Safety Section. See also the important safety information in appendix 3.

1/ Preparation.

Once the cryostat/manipulator is in the chamber ensure that the liquid helium dewar is approximately 1.3 metres away from it (it may occasionally have to be brought closer than this when raising or lowering the transfer tube legs), with the dewar top at around the same height as the top of the manipulator. In order to insert the transfer tube approximately 1 metre of room is required above the manipulator and 1.2 metres above the dewar (a means of raising and lowering the dewar will reduce this and will also make inserting and removing the transfer tube much easier as the bellows section will not have to bend as much).

If a 50 litre dewar is used the dewar leg of the transfer tube should have its standard copper end tube (with baffle) screwed onto it. If a 100 litre dewar is used the copper end should be unscrewed and a stainless steel extension tube of approximately 400mm length should be screwed on in its place. Take care not to overstress the weld on the top of the transfer tube leg when attaching or removing the extension tube. The outer tube of the dewar leg (with needle valve) should be used with 50 litre dewars with 1/2" apertures and may be used with 100 litre dewars with 1/2" apertures for around the first 60-70 litres. For dewars with 3/8" apertures change the fitting to the 1/2" type if possible, otherwise remove the outer tube. The dewar leg outer tube cannot be used in conjunction with an extension tube so for the last 30-40 litres of 100 litre dewars the outer tube must be removed. If the aperture is of the

1/2" type this will make fully sealing the tube to the dewar impossible so change the fitting to the 3/8" type to prevent air being drawn inside and freezing. Check that the plastic tubes to the pump and transfer tube are correctly connected: the helium should be sucked from the helium gas return nozzle on top of the cryostat leg of the transfer tube, through the flow controller (via the needle valve) and to the pump inlet. The helium pump exhaust is then fed back into the flow controller (through the flow meter) and out of the bunsen valve at the back (see figure 7).

2/ Inserting the transfer tube.

Blow off Helium overpressure in the dewar (if any) by opening the manual pressure release valve. High overpressures can build up (up to about 0.5 bar) and may take several minutes to vent. Slacken the sealing nut at the top of the dewar and remove the plug: this may be frozen in place after releasing the overpressure and care must be taken not to break the rubber sealing ring inside: it may be heated gently or allowed to warm up itself).

Remove the brass cover at the end of the cryostat leg of the transfer tube and insert the tube slowly into the cryostat ensuring that:

1) a clean, dry PTFE seal is in place over the end of the tube and;

2) there is no PTFE seal already inside the cryostat as they have a tendency to fall off.

Be particularly careful with the last ten centimetres or so as this is when the narrow section of the transfer tube locates in the cryostat. Care must be taken not to push down too hard or the nozzle of the capillary will be damaged. A gentle rocking of the transfer tube with respect to the manipulator should be all that is necessary to locate the end; otherwise withdraw it to check that it has not become bent.

Allow the cryostat leg to rest in the manipulator but do not tighten it up yet. This allows helium to bypass the reservoir, reducing start-up time. This assumes that there is no moisture in the reservoir (which should be the case after a bakeout). If water vapour is suspected the cryostat leg should be tightened down and the whole system flushed with dry helium.

Insert the dewar leg into the helium

gas space of the dewar i.e. above the liquid, not actually into it, about 400mm down from the top. This may require the cryostat leg to be raised to allow the the dewar leg to enter vertically. Tighten the sealing nut on the dewar to secure the transfer tube and close the manual pressure release valve.

3/ Cool down.

Start the helium pump and fully open the needle valve on the flow controller. Allow helium gas to flush the system in this way for 5 minutes. After this, slowly lower the dewar leg fully into the dewar and seal by tightening the sealing nut and closing the pressure release valve.

It has been known for ice/frozen air to form at the bottom of the neck of the dewar, preventing the transfer tube from reaching the liquid helium. This is potentially very dangerous: see appendix 3 for information on dealing with this situation.

After about 5 to 10 minutes, flow of liquid helium should have started (as seen on the flow meter on the flow controller). As soon as the flow reaches about 1 l/h, fully tighten the large hand "nut" on the top of the cryostat leg to compress the PTFE seal at the bottom and force helium through the heat exchanger. Note: when the the cryostat is fully cooled down, this may require a further, slight tightening due to contraction of the cryostat leg. The sample plate should cool down to approximately 20K (approximately -7.3mV w.r.t. room temperature or -0.63mV w.r.t. a liquid nitrogen cold junction) in about an hour although it may drop slightly further as the radiation shield and heater cool down.

While the sample plate is cooling down and the flow is increasing the needle valve will need to be closed somewhat from the fully open position to avoid using excess liquid helium and to stabilize the flow. A flow rate of 1 to 1.5 l/h should be sufficient.

4/ Warmup.

Fully close the needle valve on the flow controller and turn off the pump.

The system can be left in this state unless it is not to be cooled down again for some time (e.g. overnight), in which case:

If the system has been running for many

hours there may be an overpressure in the dewar: release this as before. Slacken the sealing nut on the dewar and withdraw the dewar leg of the transfer tube. This may require raising the cryostat leg as before, in which case completely slacken the "nut" on the cryostat leg from the top of the manipulator and carefully raise it. N.B. The liquid helium boiloff from the reservoir will act to push the transfer tube upwards. Take care that it is not forced completely out as the liquid helium inside will boil off violently and air will freeze inside the heat exchanger. Cover the end of the dewar leg with, for example, a plastic bag sealed around the tube with tape to prevent air and moisture from condensing inside it. Lower the cryostat leg back into position and tighten the nut. Replace the plug in the dewar top, tighten the sealing nut and close the manual pressure release valve. The system should be left in this state until it has completely warmed back up to room temperature.

When removing the cryostat leg ensure that the PTFE seal is removed at the same time.

APPENDIX 2.

Liquid Helium Manipulator Bakeout Procedure.

The bakeout temperature of the cryostat is limited by the differentially pumped rotary feedthrough (DPRF). The maximum feedthrough bakeout temperature is normally 150°C to protect the PTFE seals inside. Care should be taken therefore to ensure that its temperature does not rise above this, particularly if the rest of the vacuum system is to be baked out at a higher temperature. This can be achieved by leaving the top of the manipulator outside the bakeout shield only allowing heat to reach the DPRF by conduction and also permitting heat to be radiated from it, lowering its temperature with respect to the rest of the system. Alternatively a separate heater tape with aluminium foil insulation can be used to maintain the required temperature which should be monitored with a thermocouple on the DPRF.

If it is required to bakeout the DPRF at temperatures in excess of 150°C but not exceeding 200°C the locking ring (see figure 17) should be released by about half a turn to allow the upper bearing to expand axially. This is achieved by turning the index ring using both hands in an anticlockwise direction when viewed from above. If the index ring is found to be too tight to move, the protractor ring should be freed to allow access to the locking ring. This is best achieved with nothing attached to the DPRF top flange, although it is just possible while the unit is still bolted to the manipulator. To release the protractor ring slacken the two set screws with a 2.5mm Allen key and slide it upwards. Place a suitable piece of rectangular section material in the slot in the periphery of the locking ring and tap it gently with a small mallet until the locking ring is free (again in an anticlockwise direction). Replace the protractor ring and tighten the two set screws. The locking ring should now be able to move by means of the index ring. After bakeout re-tighten the locking ring by turning the index ring until hand tight in a clockwise direction when viewed from above.

APPENDIX 3.

Liquid Helium Dewar Safety Information.

The following information relates to a specific hazard and is in addition to the normal procedures for the safe use of liquid helium and dewars. If in doubt ask for more information.

If air is allowed to enter a helium dewar it can freeze at the bottom of the neck of the dewar and form a plug. When this happens the helium boil-off can not escape through the pressure release safety valve as normal and dangerously high pressures can build up inside the dewar.

It is the responsibility of the user to ensure that the dewar is used in a safe manner (see appendix 1 for instructions on using dewars in conjunction with the liquid helium transfer tube). In particular:

1/ the dewar safety head must not be removed;

2/ the valve to the pressure release safety valve must remain open;

3/ the manual pressure release valve should remain closed except when releasing overpressure;

4/ the transfer tube entry aperture should always be sealed, either onto the transfer tube or the sealing plug (only the plug supplied should be used for this purpose NOT a rubber bung) and

5/ gasses other than helium should never be used to pressurise a liquid helium dewar (pressurising the dewar is not necessary in any case with the type of transfer tube used on UHV manipulators).

As soon as a plug is discovered in a dewar the SRS Crew and Health and Safety Section must be informed. THE USER MUST ON NO ACCOUNT ATTEMPT TO FREE THE ICE PLUG OR MOVE THE DEWAR.

The SRS Crew will contact the Service Engineer at BOC Special Gases (24 hour service: see number below) for advice on dealing with the situation.

IT IS IMPORTANT THAT THE USER ASCERTAINS THE LAST KNOWN TIME WHEN THE DEWAR WAS FREE OF THE ICE PLUG as the Service Engineer needs this information do decide on a course of

action.

The dewar should be made as safe as possible whilst it is awaiting attention using the following procedure:

1/ ensure that the valve to the pressure release safety valve is open;

2/ ensure the manual pressure release valve is closed;

3/ fully seal the transfer tube entry aperture by tightening the sealing nut onto the sealing plug;

N.B. DO NOT MOVE THE DEWAR.

Inform the SRS Crew immediately.

It is the responsibility of last user of the dewar to see that this procedure is observed.

For Service Engineer (24 hour cover) tel. BOC Special Gases (Leeds):

#6 129 (abbreviated dialing)

0532 636381 (STD).