User Manual - Original Instructions

LLT Siphon

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LLT Transfer Siphon Manual

Oxford Instruments Nanoscience

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1 LLT Siphon - Principles of Operation

Low-Loss transfer siphon for liquid helium.

1.1 Revision history

Always use the latest issue of the manual. Check for updates online at https://support.myoxinst.com.

1.2 Copyright

1.3 Contents

1.4 Introduction

This manual contains user and technical information for the LLT liquid Helium transfer siphon.

The information provided in this manual supplements the information given in the other manuals supplied with your system. The cryostat manual will explain how to set up and operate your system, and the Mercury iTC manual will give details of the temperature controller. If you have purchased a complete system then you should not need to use this manual unless you need to make any adjustments.

This manual contains important information for the safe operation of your system. We recommend that you read this manual carefully before operating the system for the first time.

Please keep all the manuals supplied with your system and make sure that you check for updated information and incorporate any amendments. If you sell or give away the product to someone else, please give them the manuals too.

If you have bought a complete system from Oxford Instruments, separate manuals will have been supplied describing the other components. Please ensure you have reviewed the information supplied in all of the manuals before you attempt to operate your system.

1.5 Copyright Notice

1.6 Statement of Intended Use

The equipment has been designed to operate within the process parameter limits that are outlined in the user manual. The equipment is intended to be installed, used and operated only for the purpose for which the equipment was designed, and only in accordance with the instructions given in the manual and other accompanying documents. Nothing stated in the manual reduces the responsibility of users to exercise sound judgement and best practice. It is the user's responsibility to ensure the system is operated in a safe manner. Consideration must be made for all aspects of the system's life-cycle including, handling, installation, normal operation, maintenance, dismantling, decontamination and disposal. It is the user's responsibility to complete suitable risk assessments to determine the magnitude of hazards.

The installation, use and operation of the equipment are subject to laws in the jurisdictions in which the equipment is installed and in use. Users must install, use and operate the equipment only in such ways that do not conflict with said applicable laws and regulations. If the equipment is not installed, used, maintained, refurbished, modified and upgraded as specified by the manufacturer, then the protection it provides could be impaired. Any resultant non-compliance damage, or personal injury would be the fault of the owner or user.

Use of the equipment for purposes other than those intended and expressly stated by Oxford Instruments, as well as incorrect use or operation of the equipment, may relieve Oxford Instruments or its agent of the responsibility for any resultant non-compliance damage or injury. The system must only be used with all external covers fitted.

1.7 Restrictions on Use

The equipment is not suitable for use in explosive, flammable or hazardous environments. The equipment does not provide protection against the ingress of water. The equipment must be positioned so that it will not be exposed to water contact.

1.8 Maintenance and Adjustment

Only qualified and authorised persons should service or repair this equipment. Under no circumstances should the user attempt to repair this equipment while the electrical power supply is connected.

1.9 Warranty

The Oxford Instruments customer support warranty is available to all our customers during the first 12 months of ownership from date of delivery. This warranty provides repair to faults that are a result of manufacturing defects at Oxford Instruments NanoScience.

1.10 Acknowledgements

All trade names and trademarks that appear in this manual are hereby acknowledged.

1.11 Support

If you have any questions, please contact us with the following details :

- **System type** :
- **Serial number** : The Sales Order (SO) number and/or other identifiers of your system.
- **Installation/Shipment Address** :
- **Contact information** : How we can contact you, email/telephone details.
- **Details of your query** : The nature of your problem, part numbers of spares required, etc.

Please contact Oxford Instruments first before attempting to service, repair or return components.

1.12 Contact information

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1.13 Acronyms

A number of acronyms may be used throughout this document. Please refer to the document Practical Cryogenics for a glossary of terms.

1.14 Safety information

Before you attempt to install or operate your system, please make sure that you are aware of all safety precautions listed in this manual together with the warnings and cautions set out in other documents supplied with the system.

All cryogenic systems are potentially hazardous and you must take precautions to ensure your own safety.

The general safety precautions required when working with cryogenic systems are given in the OINS' *Safety Matters* document. We recommend that all users should read this document, become thoroughly familiar with the safety information provided and be aware of the potential hazards.

It is the responsibility of customers to ensure that the system is installed and operated in a safe manner. It is the responsibility of customers to conduct suitable risk assessments to determine the nature and magnitude of hazards.

1.15 Disclaimer

Oxford Instruments assumes no liability for use of any document supplied with the system if any unauthorised changes to the content or format have been made.

Oxford Instruments policy is one of continued improvement. The Company reserves the right to alter without notice the specification, design or conditions of supply of any of its products or services. Although every effort has been made to ensure that the information in this document and all accompanying documents is accurate and up to date, errors may occur. Oxford Instruments shall have no liability arising from the use of or reliance by any party on the contents of this these documents (including this document) and, to the fullest extent permitted by law, excludes all liability for loss or damages howsoever caused.

Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in this manual and the other manuals supplied with the system. The warranty may be affected if the system is misused, or the recommendations in the manuals are not followed.

1.16 General hazards

The following general hazards must be considered when planning the site for installation. Please take notice of the following relevant warnings.

1.16.1 Warning notices

Warning notices draw attention to hazards to health. Failures to obey a warning notice may result in exposure to the hazard and may cause serious injury or death. A typical warning notice is shown below:

The warning triangle highlights dangers which may cause injury or, in extreme circumstances, death.

1.16.2 Caution notices

Caution notices draw attention to events or procedures that could cause damage to the equipment. Failure to obey a caution notice may result in damage to the equipment. A typical caution notice is shown below:

The general caution symbol highlights actions that you must take to prevent damage to the equipment. The action is explained in the text.

1.17 Specific hazards

Each cryogenic system will have a number of specific hazards associated with the product. For full details please refer to the system manual. You will receive this at the time of installation and it should be read by all users before operating the system.

1.18 Description

The LLT liquid helium transfer siphon is a gas-flow shielded, vacuum-insulated siphon designed for use with continuous flow cryostats. The exhaust gas from the cryostat is used to cool a radiation shield around the liquid delivery tube, reducing the heat load on the liquid helium.

Two versions of the LLT are available, with schematic diagrams for each shown below,

- The LLT600, designed for use with cryostats which have horizontal or nearly horizontal entry arms.
- The LLT700, designed for use with cryostats which have vertical entry arms.

Both versions have a flexible section so that the cryostat can be moved relative to the storage dewar. A needle valve is fitted in the leg that fits into the storage dewar. Both versions of the siphon may be upgraded to incorporate an automatic needle valve, for which the part numbers are LLT650 and LLT750 respectively. This option allows the siphon to be used in conjunction with a Mercury iTC to provide automatic control of the liquid helium flow rate. Refer to the auto needle valve manual for further details.

1.19 Installation

1.19.1 Unpacking the transfer siphon

Carefully remove the transfer siphon from the packing and inspect it to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford Instruments immediately.

1.19.2 Evacuating the vacuum space

The vacuum space has to be pumped to high vacuum to make sure that it gives the required thermal insulation. When the transfer siphon is new all the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This does not mean that the transfer siphon is leaking, just that the new materials are being cleaned by the vacuum. The transfer siphon has been pumped to high vacuum and baked before it left the factory. However, the vacuum space should be pumped occasionally, especially when the transfer siphon is new.

If water condenses on the outside of the transfer siphon while it is being used, or if the cryostat does not reach base temperature or needs a high flow rate to reach base temperature, this may indicate that the transfer siphon vacuum space needs to be pumped again.

The LLT transfer siphon is fitted with a vacuum valve with an integral NW16 flange.

Connect the pumping system to the NW16 flange on the valve. We recommend that you use a diffusion pump or turbo-molecular pump. If a rotary pump is used to back the high vacuum pump, the gas ballast valve should be left closed unless there is reason to believe that the transfer siphon is badly contaminated with water vapour.

Evacuate the pumping line to a medium vacuum with the rotary pump before you open the valve. Open the valve by turning the knob anti-clockwise a few turns, and pump the vacuum space to a high vacuum. Leave it to pump for at least one hour, and preferably overnight. Close the valve firmly before you vent the pumping line and remove the pumping system.

1.19.3 Protective cover

All LLT transfer siphons are supplied with a protective cover to prevent damage to the delicate part that fits into the cryostat arm. Unscrew and remove this cover before you use the transfer siphon, but keep it in a safe place for future use.

1.20 Operation

If the transfer siphon has been supplied as part of a system, please refer to your cryostat manual which will explain in more detail how to use the transfer siphon with your system.

If the siphon has been supplied separately, the following instructions explain how to cool it down safely.

1.20.1 Adjusting the cryostat entry arm lock nuts

Normally you only need to follow this procedure if the transfer siphon has to be adjusted to fit a different cryostat. The components are labelled below.

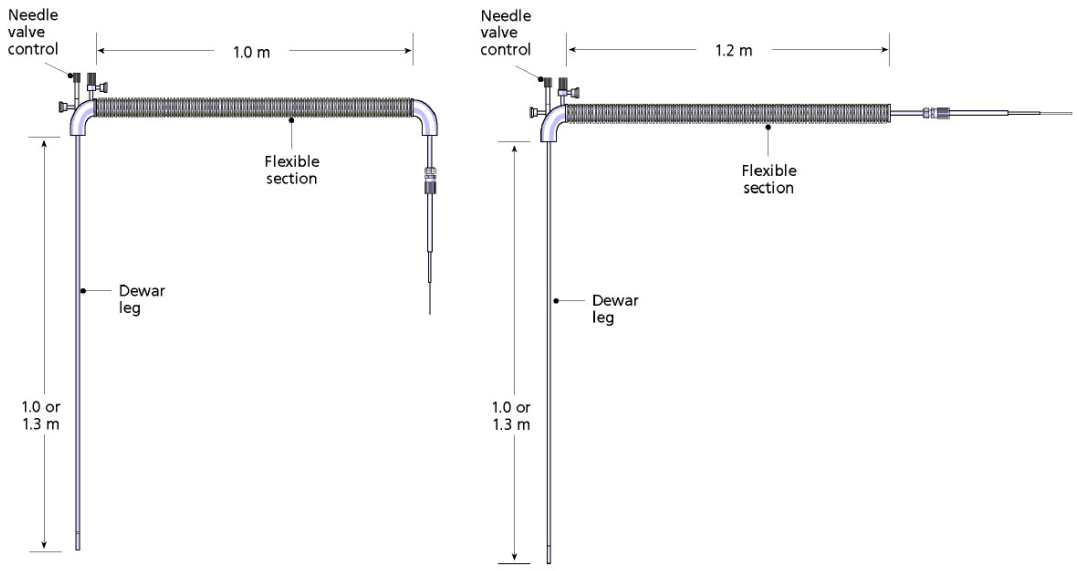

Figure 1.1: LLT transfer siphon options

The nut and lock nuts are used to compress a collet, so that the assembly is firmly fixed to the transfer siphon arm.

1. Hold the nut and loosen the lock nut using two spanners so that the assembly can slide freely along the transfer siphon delivery arm.
2. Push the transfer siphon delivery arm fully into the cryostat arm, so that the PTFE seal meets the mating surface inside the cryostat. Some cryostats do not have entry arms and no PTFE seal is required. Push the transfer siphon in as far as it will go.
3. Screw the knurled nut fully onto the thread on the entry arm, and then unscrew it by two or three turns.
4. Gently push the transfer siphon delivery arm into the cryostat to make sure that the PTFE seal is seated (or the siphon is pushed as far as possible into the cryostat) while you tighten the lock nut onto the transfer siphon again.

1.20.2 Cooling down the transfer siphon

Close the needle valve on the transfer siphon fully by rotating the knurled nut clockwise (viewed from above) or setting a gas flow of 0% on the Mercury iTC. Then open the valve by a by six turns, or set the gas flow to 100%.

Check that the PTFE seal (if required) on the end of the transfer siphon is clean, de-greased and undamaged.

Open the exhaust valve of the liquid Helium dewar to release any pressure, keeping your hands and face away. Slowly lower the dewar leg of the siphon into the dewar, engaging the nut on the syphon with the dewar fitting. Switch on the GF4 pump. Some liquid will be used to cool the leg, and the dewar exhaust must be open to allow the boil-off to escape. If you try to cool the leg too quickly a large amount of liquid will be wasted, and there is a risk of being burnt by the cold gas.

Once the siphon leg has been loaded into the Helium dewar, push the other end of the siphon into the cryostat entry arm, but do not engage the nut straight away.

Connect the exhaust gas line and, if the flow is being monitored (using an Oxford instruments VC-U gas flow controller, for example), watch the flow gradually increase as the siphon coold. After a few minutes, once the flow rate is around 1.5 litres/hour, engage the nut on the siphon with the thread on the cryostat arm. Take care not to over-tighten the nut.

The flow will initially drop, then rise again as the cryostat cools. If the flow does not reach about 1.5 litres/hour after 20 minutes, this indicates that the transfer siphon may be blocked, or the needle valve may not be opening correctly.

It is good practice to fit a soccer-ball bladder to the exhaust of the storage dewar and squeeze and release it occasionally to keep the pressure slightly above atmospheric pressure. This encourages the transfer of helium through

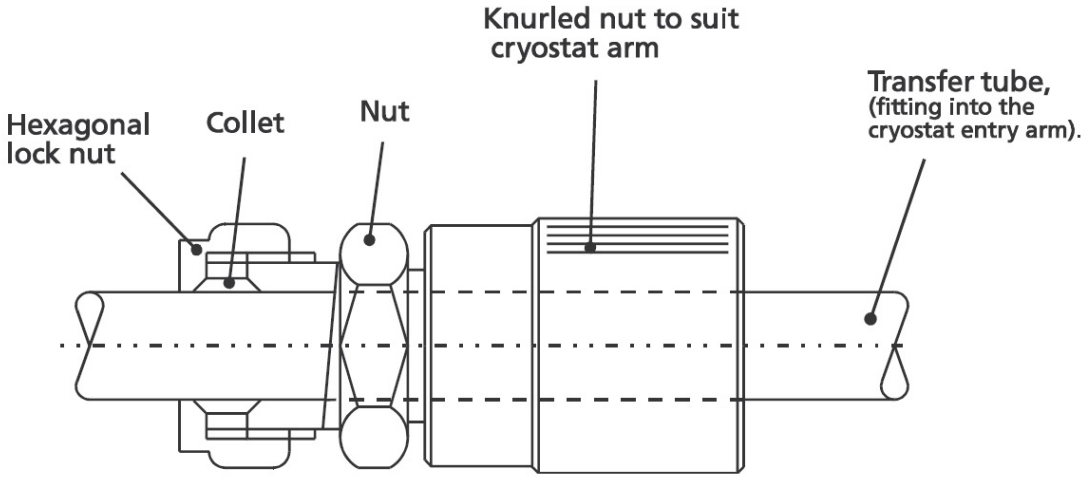

Figure 1.2: Adjusting the cryostat entry arm nut

the siphon and helps to prevent air from getting inside the storage dewar.

If you have persistent problems with blockages in the transfer siphon you may be able to reduce the risk by using a pump (such as the GF4 pump) and VC-U to draw Helium gas through the siphon as it is being lowered into the Helium dewar. As the connections to and from the siphon need to be adjusted in this case, your cryostat system manual will explain this process in further more detail.

Once your cryostat heat exchanger and sample is cooling steadily, the transfer siphon and cryostat arm may contract by different amounts. The knurled nut on the cryostat arm should be tightened occasionally to ensure the seal in the cryostat is maintained, preventing liquid helium from by-passing the cryostat.

1.21 Maintenance

1.21.1 Clearing blockages in the transfer siphon

If the transfer siphon becomes blocked during use, remove it from the cryostat and storage dewar with the needle valve open. Connect a supply of clean helium gas to the delivery arm end of the siphon with rubber tube and pressurise it to around 500mbar. Allow the transfer siphon to warm up, warming gently with an air gun or fan heater if necessary. The heating is unlikely to affect the inner tube due to the insulating vacuum, but warming the ends of the tube and the needle valve is likely to clear most blockages. Make sure that you can feel gas escaping through the inlet hole in the dewar leg.

In an extreme case there may be a sudden pressure rise which blows the rubber tube from the transfer siphon. Do not hold the transfer siphon unless you are wearing the correct protective equipment.