OxfordCryosystems

800 Series Cryostream Cooler

Operating & Instruction Guide

Operating and instruction guide for:
800 Series Cryostream Cooler
800 Series Cryostream Plus
800 Series Cryostream Compact
Varibeam Support Stand
Cryostream Accessories

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Contents

1 Safety Conventions

1.1 Safety Symbols

DANGER

Information displayed under this heading must be conformed to in order to avoid death or serious injury.

WARNING

Information displayed in these sections relates to the prevention of equipment and/or environmental damage.

CAUTION

Information displayed in these sections relates to correct handling or use. Disregarding these instructions or cautions can lead to malfunctions or minor equipment damage.

NOTE

Information displayed under this heading provides useful or helpful tips.

1.2 Personnel Qualifications

SKILLED PERSONNEL

All work described in this document may only be carried out by persons who have suitable technical training and the necessary experience or who have been instructed by the custodian of the product.

2 Liability and Warranty

Oxford Cryosystems assumes no liability and the warranty becomes null and void if the custodian or third parties:

- disregard the information in this document.
- use the product in a non-conforming or improper manner.
- make any kind of changes (modifications, alterations etc.) to the product.
- use the product with accessories not listed in the product documentation.

The custodian assumes the responsibility in conjunction with the documentation and media supplied.

Oxford Cryosystems - Warranty Certificate

This warranty is subject to the Oxford Cryosystems Ltd's (OCL) Terms and Conditions of Sale.

OCL warrants to the Buyer that the goods sold for use hereunder will be free from defects in material and workmanship under normal use and operation for 12 months from the date of shipment from OCL's premises.

In order to obtain the benefits of the warranty the Buyer must first notify OCL of the defects. An OCL representative will verify the nature of the defect and if it is covered by this warranty, OCL will issue the Buyer with a RMA number and provide the Buyer with instructions on how to return the goods to OCL. The Buyer must return the goods according to instructions from OCL, complete with a written description of the claimed defect and RMA number. The goods should be packed safely, preferably in its original packaging prior to return.

The Buyer shall meet the cost of shipping the defective goods to OCL and OCL will pay any return costs to the Buyer.

OCL's obligation under this warranty is limited to its option to repair or replace goods that are proven to be defective when used under normal operating conditions and within specification. This warranty does not cover any changes made by the customer, depreciation of the goods or claims for compensation.

No warranty is given for damage resulting from misuse or fair wear and tear. In addition, this warranty does not cover any costs incurred in damage arising from the dismantling or reassembly of any of the goods, or for consequential losses of time or materials caused by Cryostream failure.

Registration

In order for us to be able to provide fast and effective service, you should register your system with us. Please send the serial number of the system (found engraved on the Coldhead) to support@oxcryo.com, together with your full contact details.

To make contact with Oxford Cryosystems you can telephone, fax, or email us at:

Oxford Cryosystems Ltd, 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN, UK
Tel: +44 1993 883488 Fax: +44 1993 883988
Email: support@oxcryo.com

3 Safety Information

3.1 General Safety and Environmental Use

The 800 Series Cryostream Cooler including the Controller and Pump Unit is designed to be operated within the following environmental conditions:

- Environmental Temperature Range: 15°C to 35°C
- Relative Humidity: 10 - 70%

In addition to the above, please ensure the following safety conditions are followed:

- The equipment should not be placed close to any source of potential water leak or spillage. The Controller and Pump Unit must also be operated well away from the possibility of any splashes from liquid nitrogen.
- The 800 Series Controller and the Pump Unit (if, for any reason, it is powered independently) must be connected to an earthed power supply.
- Ensure that all vents around the Controller and Pump Unit are kept clear during usage to prevent any overheating and damage to the system.
- It is important that the mains power cable to the back of the controller and access to the isolation switch are kept clear and unobstructed to the user at all times.
- The mains power cable used shall be appropriately rated and approved according to the regulations of the country of use.

3.2 Use of System around X-rays

DANGER

The Cryostream Cooler is designed for use with an X-ray system for the purposes of cooling crystalline samples for study by X-rays. In order to safely use the Cryostream within this environment, it is assumed that all users are trained in and certified to local X-ray safety standards and that all necessary precautions have been taken with respect to X-ray safety when both installing and using the Cryostream system.

3.3 Liquid Nitrogen Handling

DANGER

When using the Cryostream system it will be necessary to refill the Dewar vessel with liquid nitrogen. It is assumed that all users have had local safety training in the handling and transport of liquid nitrogen. Liquid nitrogen for use with the Cryostream Cooler should not be handled by anyone who has not received the correct training or is not authorised to do so by the local Health & Safety Officer.

NOTE

Please refer to Appendix 5 Liquid and Gaseous Nitrogen Safety Sheet.

3.4 Use of Dewar

WARNING

The Cryostream Cooler is designed for use with an unpressurised Dewar vessel. Do not, under any circumstances, seal or pressurise the Dewar vessel. The Cryostream is designed for use with Dewar vessels supplied by Oxford Cryosystems. Although the Cryostream can be used with a variety of unpressurised Dewar vessels, Oxford Cryosystems does not take responsibility for vessels that users source themselves or have been supplied to them other than by Oxford Cryosystems. Oxford Cryosystems also does not take responsibility for the performance of the Cryostream system if the alternatively sourced Dewar has an adverse effect on the system performance.

3.5 Refilling

DANGER

Refilling of the Cryostream Dewar should only be carried out by someone trained in the handling of liquid nitrogen.

3.5.1 Points of Consideration when Refilling the Dewar Vessel

- Ensure the operator has had appropriate training and is wearing the relevant Personal Protective Equipment as specified during the training.
- If the Dewar is being refilled manually, make sure the nozzle of the liquid nitrogen supply transfer line is at least 30cm inside the neck of the Dewar.
- Do not push the nozzle of the transfer line down into the liquid as this can cause excessive boil off and liquid turbulence.
- Slowly open the supply valve from the self-pressurised vessel to prevent sudden surging of liquid nitrogen. Note that the liquid will not flow straight away as the transfer hose from the self-pressurised vessel to the Cryostream Dewar will need to be cooled first.
- Do not leave the refilling of the Cryostream Dewar unattended at any time.
- Do not let the Cryostream Dewar overflow.
- Once the Cryostream Dewar has been filled, cover the vessel as best as possible with the cap provided to prevent atmospheric moisture migrating inside.

WARNING

The Cryostream Dewar should be refilled from a secondary self-pressurised storage Dewar. The pressure in the self-pressurised storage Dewar should not exceed 2 bar. This is because highly pressurised vessels can cause excessive turbulence of the liquid nitrogen within the Cryostream Dewar resulting in the system shutting down.

3.5.2 Refilling of Dewar within the X-ray Enclosure

If the Cryostream Dewar has been installed inside the X-ray enclosure, boil-off from the refilling process could fill the enclosure with vapour. To keep this vapour to a minimum, ensure the pressure in the self-pressurised storage vessel is kept as low as possible; preferably no more than 1 bar, and consider venting the Dewar outside the enclosure.

3.6 Contact with the Stream of Nitrogen Gas

Although the Cryostream produces a stream of cryogenic gas, the amount of cooling is relatively small so short exposures from the stream on the surface of the skin are very unlikely to cause any injuries. However, prolonged skin exposure to the stream at low temperatures (or high temperatures in the case of the Cryostream Plus) could result in skin burns.

3.7 Automatic Refill

The Cryostream can be used in conjunction with an automatic liquid nitrogen refill system. These systems are designed to measure the level of the liquid in the Dewar. Refill of the Cryostream Dewar is initiated once the level of the liquid inside drops below a predefined level and then stops once an upper level is reached. This is achieved by controlling a solenoid valve on the liquid nitrogen supply transfer line between the self-pressurised storage vessel and the Cryostream Dewar. (See separate Setting Up and Using the Automatic Refill System for further details)

WARNING

Oxford Cryosystems does not take responsibility for automatic refill systems that are left unattended.

3.7.1 Automatic Refill Liability

Oxford Cryosystems is not responsible for damage to equipment or injury to any persons caused by either the misuse of, or the failure of, any Automatic refill system or any of its component parts. Oxford Cryosystems has supplied the system in good faith but the user installs and uses the system at their own risk. It is recommended that the user only operate the system in 'Automatic mode' while someone is in attendance. If the unit is left unattended in Automatic mode, e.g. overnight, the user does this at their other risk.

4 Outline of Intended Use

The Cryostream Cooler is designed:

- To be used with liquid nitrogen only.
- For use with an unpressurised Dewar vessel. The Cryostream must never be inserted into a pressurised Dewar vessel.
- To cool a sample of less than $1mm^3$ in an open stream of gas. The nozzle is not designed to be enclosed or to cool samples that are enclosed inside any kind of sample holder, chamber or vessel.
- To be used with the components and accessories supplied with the system or separately by Oxford Cryosystems. Use of accessories not outlined in this manual can only be used with the written agreement of Oxford Cryosystems.

4.1 Improper Use

Improper use of the system will forfeit claims for liability and any warranties on the system. Improper use would include:

- Not using the product as described within this technical guide
- Using the product with other liquids or liquid gases other than liquid nitrogen
- Inserting the Cryostream into a pressurised Dewar vessel
- Using the system when not authorised to do so or without the correct training
- Using support stands other than the Varibeam Support Stand, unless with prior authorisation from Oxford Cryosystems.
- Using a different dry air system from the AD51 without authorisation from Oxford Cryosystems
- Any attempt to modify or exchange parts or components within the system with non-standard parts that have not been supplied by Oxford Cryosystems

5 Technical Data

5.1 Standard Cryostream Technical Data

800 Series Cryostream Coldhead	
Temperature Range	80-400 Kelvin (or 80-500 Kelvin for Cryostream Plus)
Nitrogen Gas Flow Rate	5 or 10 litres/minute
Liquid Nitrogen Consumption	0.6 litres/hour at 5 litres/minute gas flow 1.2 litres/hour at 10 litres/minute gas flow
Temperature Stability	0.1 Kelvin
Cool Down Time to 100 Kelvin	20 minutes
Standard Length of Transfer Line	1500 mm (see section 5.2)
800 Series Controller	
Dimensions & weight	263 mm W x 141 mm H x 299 mm D, 6.25 kg
Mains power supply	100-120V 50/60 Hz, 200-240 V 50/60 Hz
Fuse	100-120V - 3.15A HBC T Fuse 200-240V - 6.3A HBC T Fuse
Power consumption	400VA
800 Series Gas Pump	
Dimensions & weight	263 mm W x 184 mm H x 272 mm D, 6.75 kg
Fuse	1.6A HBC T Fuse
AD51 Dry Air Unit	
Dimensions & weight	660 mm x 300 mm x 415 mm, 42 kg
Mains power supply	220-240 V or 100-115 V, 50/60Hz
Power Consumption	750VA
Varibeam Support Stand	
Max. table to sample height (Cryostream vertical)	600 mm
Max. horizontal distance to sample from Varibeam column (Reach)	430 mm
Weight	6 kg
ES60 Dewar vessel	
Volume	60 litres
Construction	Welded stainless steel
Overall dimensions	725mm H x 457 D (650 mm Internal Depth)

Weight	36 kg empty, approx. 96 kg full
Neck size	NW50 KF fitting 50mm diameter bore

5.2 Alternative Cryostream transfer line options

Cryostream Version	Flexible Length	Rigid Leg Length	Total Length
Standard	1500mm	905mm	2405mm
Long Transfer Line	3000mm	905mm	3905mm
Short Transfer Line	1160mm	770mm	1930mm

6 Setting Up the Cryostream

6.1 Before you Begin

6.1.1 Items Supplied

Qty	Description
1	800 Series Cryostream Coldhead (with integral transfer line)
1	800 Series Cryostream Controller
1	800 Series CSPU200 Cryostream Pump Unit
2	Power Cables
1	Coldhead Cable (3.5m)
1	Computer Cable set comprises:
1	Serial Cable for CryoConnector (5m)
1	USB Cable for CryoConnector (5m)
1	Ethernet Cable for CryoConnector (5m)
1	Tube Set comprises:
2	PTFE Tubes with Fittings (3m)
2	PTFE Tubes with Fittings (2m)
1	PTFE Line Drier Connection Clear Tube (0.7m)
1	Red PTFE Dry Air Tube with Connector (3m / 6mmOD)

Note: Tube and cable lengths may differ for specialised systems, depending upon the specific system configurations.

6.1.2 Components supplied for maintenance

Qty	Description
1	CRH25 Reactivation Heater
1	Pump-out Adaptor

NOTE

The pump-out adaptor is unique to the 800 Series Cryostream. Therefore this adaptor is not compatible with the 700 series Cryostream and vice versa.

6.1.3 Optional extras

Components
Varibeam Coldhead Support Stand
AD51 Dry Air Unit (or dry air regulator kit for your own dry air supply)
Nozzle Alignment Tool
Automatic Refill System
Line Drier Units
Dewar Vessel – Oxford Cryosystems 60 L Dewar*

** Note: A smaller 13 L vessel may be supplied depending upon specific system configurations.*

6.2 Cryostream Set Up – Overview

Task	Section
Stage One - Varibeam Set up	6.3
Stage Two - Handling of Cryostream Cooler Coldhead and Transfer Line	6.4
Stage Three - Cryostream Dewar Vessel	6.5
Stage Four – Positioning 800 Series Cryostream Controller and Gas Pump	6.6
Stage Five - Cable and Tube Connections	6.7
Stage Six - Setting up the Dry Air supply	6.8
Stage Seven - Line Drier Installation	6.9

Figure 1: Overview of Cryostream layout

PTFE Tubing
Cryostream Coldhead – to – Controller Cable
Serial Cable OR Ethernet Cable OR USB Cable
Power Cables
Red PTFE Tubing

WARNING

Please note this is an overview only, full instructions MUST be read before the system is assembled to avoid causing damage to the system.

6.3 Stage One - Varibeam Set up

6.3.1 Assemble the Varibeam

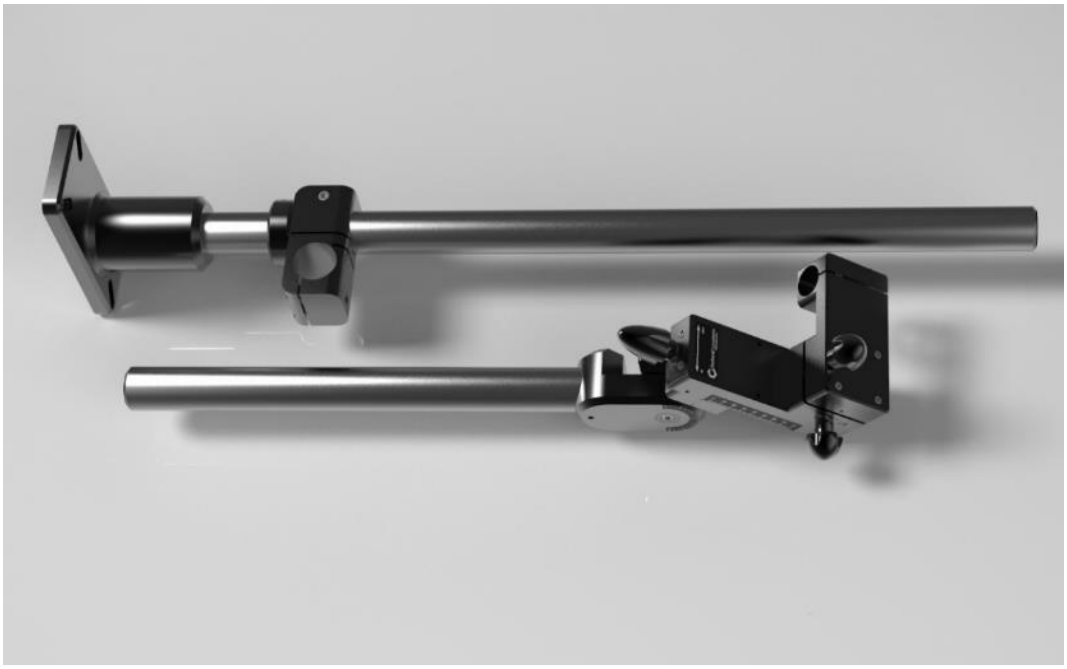


Figure 2: Varibeam stand as packed

For packing purposes, the Varibeam support stand is disassembled and will be found as 2 separate pieces. Using the Hex key provided, move the locking collar (found below the 90 degree block on the vertical pole) and 90 degree block to an appropriate height away from the foot, and insert the horizontal arm. Use the Hex key to tighten the 90 degree block and locking collar to the vertical pole.

For packing purposes, the Black Mounting Pin may be mounted backward in the Varibeam Nozzle Clamp. Remove it using the Hex key supplied and fit it into the Nozzle Clamp the right way around. Ensure that the Mounting Pin is pushed all the way into the Varibeam Nozzle Clamp.

WARNING

Do not use the Varibeam to mount any other type of device.

6.3.2 General guidelines for fitting the Varibeam and mounting the Cryostream coldhead

WARNING

The Varibeam weighs 6kg and is unwieldy when assembled, so use the Mounting Pin supplied with the Varibeam rather than the coldhead as a guide for aligning the nozzle. Once the Mounting Pin has been aligned to the sample position, it can be removed from the Varibeam and the Cryostream coldhead put in its place.

There are some general rules which should be considered when mounting the Varibeam in position and then fitting the Cryostream coldhead in place:

- Do not fix the stand to the cabinet top so it prevents access to the X-ray tube, restricts the detectors movements or makes access to the crystal difficult.
- It is not necessary to perfectly align the nozzle of the Cryostream in place when locating and fitting the Varibeam. The fine X, Y and Z translations of the stand allow for accurate alignment once the stand is in place.
- Do not point the nitrogen cold stream at the X-ray detector.
- Try to limit the amount that the end of the Cryostream gas delivery nozzle infringes the path of the X-rays.
- Do not point the cold stream so that it is cooling any optical devices or gearing (these devices need to be more than 15 cm away).
- The crystal should be less than 8 mm from the end of the Cryostream nozzle (optimum distance is 5 mm) in the centre 2 mm of the gas stream; this will help to prevent icing. The Oxford Cryosystems Nozzle Alignment Tool is ideal for determining the correct position.
- Make sure that when the Cryostream coldhead is fitted into the Varibeam, the Transfer Line can reach the bottom of the Cryostream Dewar vessel

The Varibeam is supplied with a kit of nuts and bolts to fit in the slotted base allowing the user to securely fix the stand to the enclosure table top or hang securely from the roof of the enclosure, if appropriate.

WARNING

Always use the Locking Collar to support the Horizontal Arm in the 90 degree block to ensure it does not slip when the Arm is loosened.

6.4 Stage Two - Handling of Cryostream Cooler Coldhead and Transfer Line

Please ensure that;

a) The rigid section of the Cryostream Transfer Line can be fed through a port in the cabinet or labyrinth (if applicable).

DANGER

Please ensure that any ports made in your cabinet adhere to your local Health and Safety laws and policies and have been approved by your local Health and Safety Officer and the original enclosure manufacturer.

b) The rigid section of the Cryostream Transfer Line can be fed into and reach the bottom of the Dewar

* Some configurations of the Cryostream Compact include a small Dewar that is mounted inside the X-ray enclosure. For these configurations, it is not necessary for the Cryostream transfer line to exit the enclosure, it should instead be fed directly into the Dewar.

The Cryostream coldhead is designed to be fitted into the Nozzle Clamp of the Varibeam. Make sure the Cryostream coldhead is pushed all the way into the Nozzle Clamp before tightening the bolts on the Nozzle Clamp. It might be necessary to retract the nozzle using the Z-motion of the Varibeam stand so that it doesn't interfere with the pre-aligned crystal position.

NOTE

Use the Z-translation on the Varibeam to retract the nozzle from the crystal position. Do not move the Cryostream Nozzle by sliding out of the Nozzle Clamp.

CAUTION

It is important to handle the Cryostream Coldhead and transfer line assembly as carefully as possible.

- The minimum bend radius of the transfer line is 200mm. Do not bend the flexible line more tightly as this will result in possible damage of the internal capillary.
- Try to avoid turning the rigid part of the transfer line upside down.
- Do not try to disconnect the flexible transfer line from the coldhead. The will cause a loss of vacuum and will damage the system.
- During use, do not remove the rigid leg section from the Dewar until the Cryostream coldhead is warm. If the rigid leg needs to be removed more quickly from the Dewar, program a PURGE.

6.5 Stage Three - Cryostream Dewar Vessel

Fill the Dewar with liquid nitrogen.

The purpose of the Dewar vessel is to hold the liquid nitrogen to be used by the Cryostream. The ES Dewar vessels supplied by Oxford Cryosystems have been designed for use with the Cryostream cooler. Other Dewars can be used with the Cryostream, however, Oxford Cryosystems does not take responsibility for other Dewar vessels or their suitability.

DANGER

Section 3 of this document refers to the safety aspects of the handling of liquid nitrogen. It is assumed that these instructions and guidelines have been followed. Do not, under any circumstances, handle liquid nitrogen unless authorised and suitably trained to do so by the local Health & Safety Officer. Further safety information on the handling of liquid nitrogen can be found in *Appendix 5 Liquid and Gaseous Nitrogen Safety Sheet*.

If a Dewar other than the ES60 is being used, be aware of the following points:

- Capacity –The recommended capacity is between 30 and 60 litres. Smaller Dewar vessels will require refilling more often than larger Dewar vessels. This will result in greater boil off during refilling and, therefore, a greater overall consumption of liquid nitrogen.

NOTE

When considering a large Dewar, it is important to remember that the rigid transfer line of the Cryostream is plunged into the Dewar and is ~900 mm long, so it can only utilise the top 850 mm of a Dewar vessel. It is possible to use a 100 L Dewar, but if the rigid transfer line does not reach the bottom, the Cryostream will not utilise the full capacity of the vessel.

- Construction – either stainless steel or aluminium.

- Neck opening size – Dewar necks vary in size. If the opening is too small, there may be problems refilling the Dewar. If the Dewar opening is too large, then the rate of boil off will be very high and contaminants will get into the liquid nitrogen.

6.5.1 Fitting the Cryostream Rigid Transfer Line into the Dewar

It is recommended that the end of the Transfer Line does not sit on the bottom of the Dewar vessel. Generally, particulate matter will concentrate in the bottom of the vessel and, although rare, this can block the flow of liquid nitrogen and stop the system.

Because the Cryostream applies the same gas pressure at the crystal as it does in the Dewar, it is safe to replenish the supply of liquid nitrogen to your Dewar at any time without any fluctuations in gas temperature. This can be simply done manually or by using an automatic refilling system (available from Oxford Cryosystems).

NOTE
Please ensure that the Dewar is at least ~20% full at all times.

6.6 Stage Four – Positioning 800 Series Cryostream Controller and Gas Pump

6.6.1 Placement of Controller

The 800 Series Cryostream Controller is designed to manage the Cryostream system. It is important to place the controller so the user has easy access to the front panel and can clearly see the display on the front. As the controller is connected to other components in the system, it should be placed:

- Less than 3 metres from the coldhead
- Less than 2 metres from the 800 Series Gas Pump

6.7 Stage Five - Cable and Tube Connections

6.7.1 Controller to Cryostream Coldhead Connections

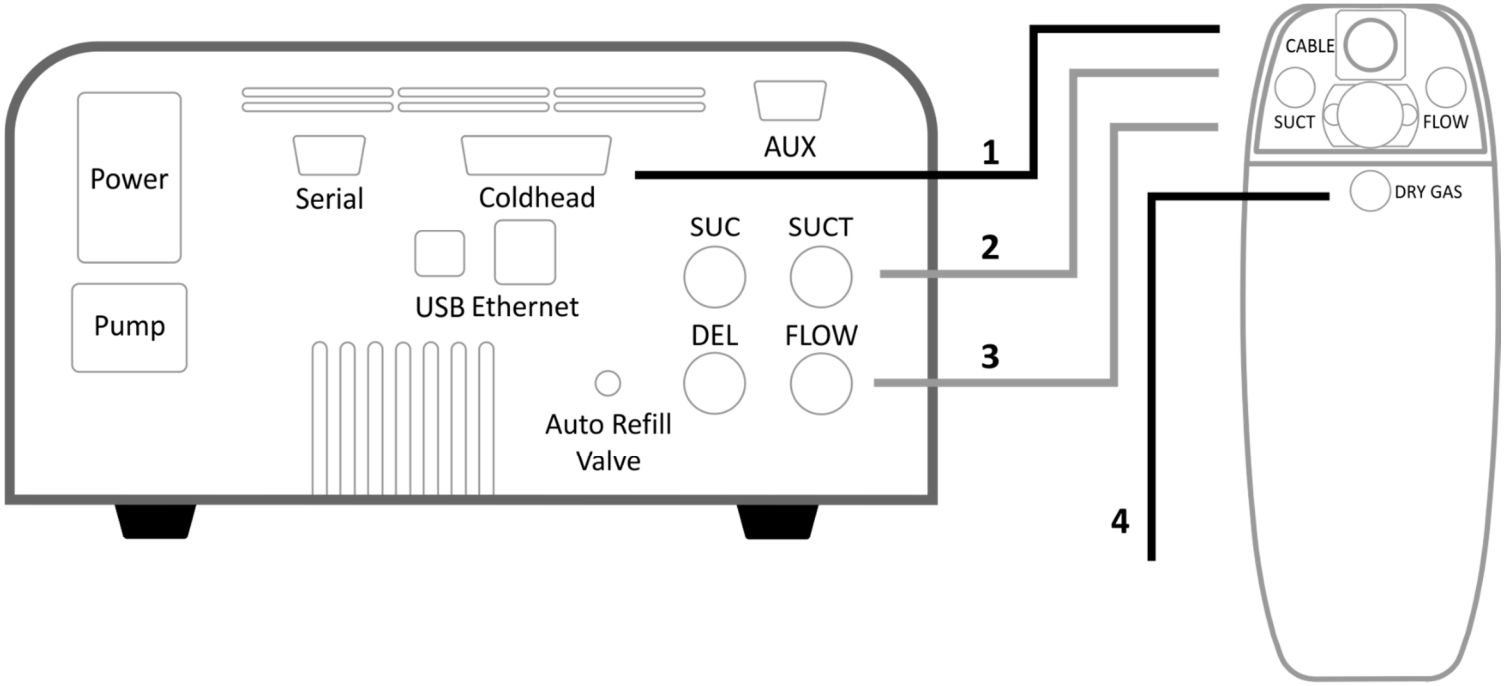


Figure 3: Controller to Cryostream connections

Number	Connection	From	To Coldhead
1	Coldhead Cable	'Coldhead' socket on controller	Electrical socket
2	3m white tubing with connector	'SUCT' connector on controller	SUCT connector
3	3m white tubing with connectors	'FLOW' connector on controller	FLOW connector
4	Red dry air tubing	Dry gas supplier e.g. AD51 Dry Air Unit	Dry gas connector

6.7.2 Gas Pump to Controller Connections

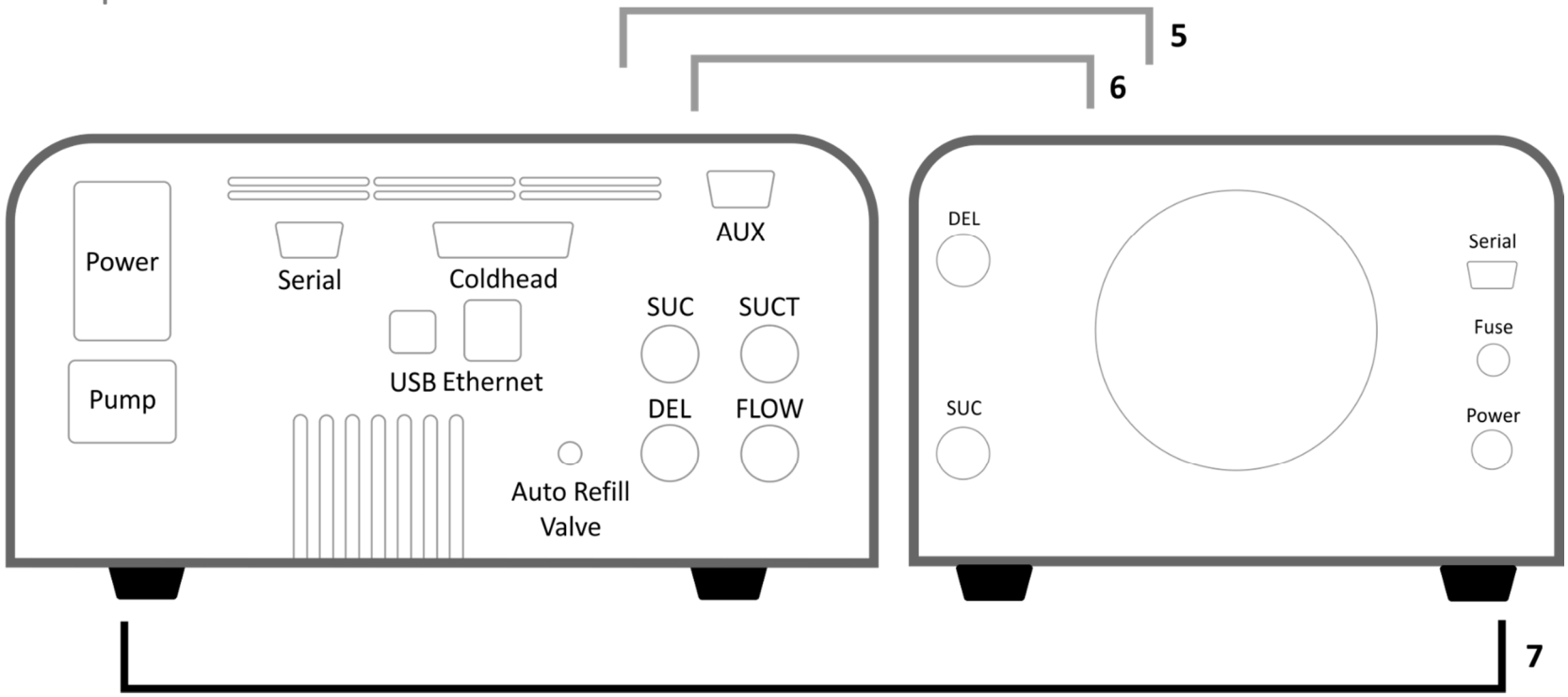


Figure 4: Gas pump to controller connections

Number	Connection	From Pump	To Controller
5	2m white tubing with connector	'SUC' connector	'SUC' connector
6	2m white tubing with connectors	'DEL' connector	'DEL' connector
7	Mains power cable	Integral power cable outlet	Pump mains power inlet

6.7.3 Other Controller Connections

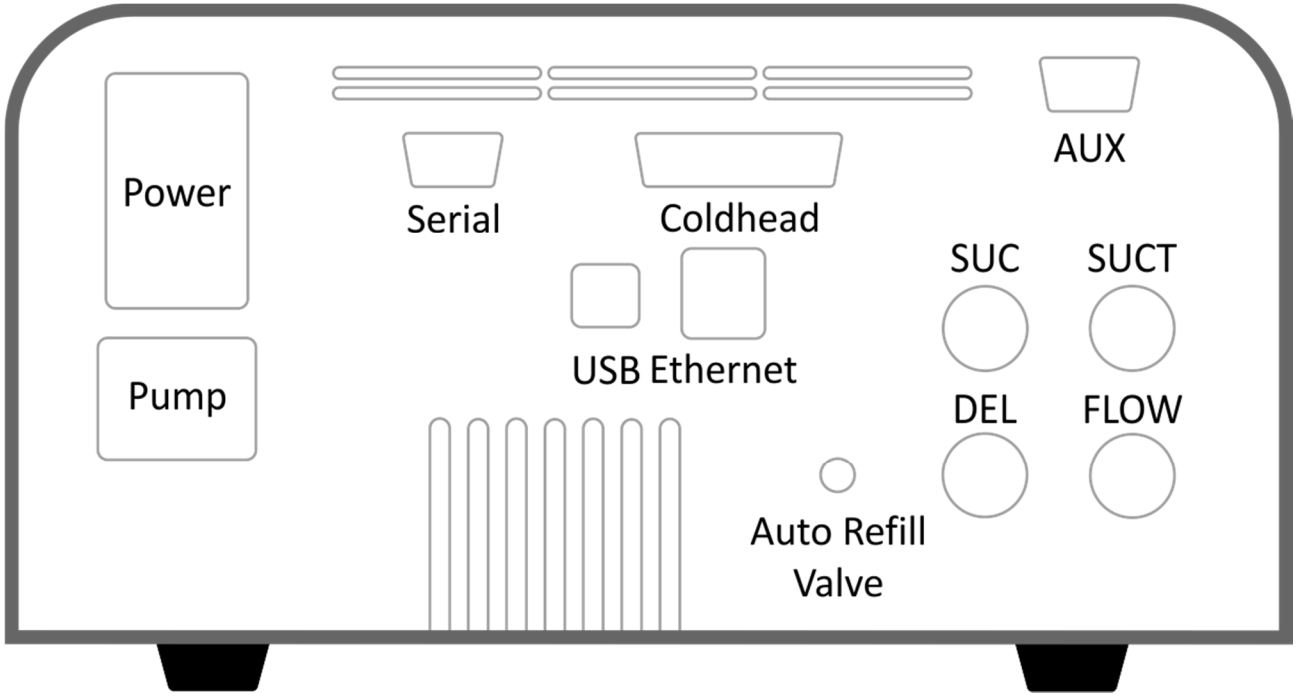


Figure 5: Other controller connections

Optional Connections	Connector	To
Serial	9 Way Serial Cable socket	PC
USB	USB cable socket	PC
Ethernet	Ethernet cable socket	PC, network hub or router
Auto Refill Valve	Auto Refill Valve cable	Auto Refill Solenoid Valve
Aux	9 Way Serial Cable	Auto Refill Capacitance Probe

6.8 Stage Six - Setting up the Dry Air supply

6.8.1 Setting up the AD51 Dry Air Unit

1. Ensure that the AD51 is situated in the laboratory or service room such that there is a free flow of air around the unit and the vents are not blocked.

2. The AD51 is supplied with 5 m of 8 mm OD red tubing to connect into the back of the AD51.

3. Couple the 5 m of 8 mm OD red tube to the 3 m of 6 mm OD red tube supplied with the Cryostream Tube Set using the 6 mm-8 mm adapter provided with the AD51 Dry Air Unit.

4. Fit the other end of the 6 mm OD red tube into the quick release connector on the Cryostream Coldhead, see 6.7.1 Controller to Cryostream Coldhead Connections

5. Connect the AD51 to a power outlet using the power cable provided.

NOTE

Best practice is to allow for the AD51 Dry Air Unit to be run for 12 hours prior to connecting to the Cryostream

6.8.2 Setting the Flow of Dry Air Unit

Switch on the AD51 Dry Air Unit and set the flow to approximately 12-13 litres per minute using the flow meter on the front of the unit. This is the ideal flow rate for use with the Cryostream system although adjustments may be required.

CAUTION

Do not exceed 25 litres per minute (LPM) mark on the AD51 Dry Air Unit flow meter, above this mark the unit will be incapable of producing sufficiently dry air.

NOTE

If the AD51 Dry Air Unit has been switched off for a long period of time, best practice is to allow the unit to run for 12 hours before using in conjunction with the Cryostream.

6.8.3 In-house Dry Air Supply

The cold nitrogen gas stream requires a shroud of dry gas as it exits the nozzle to ensure an ice-free environment on and around the sample while at low temperatures. If an AD51 dry air unit has not been purchased, in-house dry air or nitrogen gas may be used instead. The gas should have:

- A dew point of less than -60˚C
- Regulated delivery pressure of 1 bar /14.5psi (Max. 1.4 bar /20.3psi)
- A minimum of 12 L/min gas flow

Use the 3m length of 6mm OD tubing provided to connect your dry air source to the Cryostream. A dry air regulator kit is available from Oxford Cryosystems if required.

6.9 Stage Seven - Line Drier Installation

Line Driers are designed to extend the continuous running time of the Cryostream. The Line Drier removes traces of water vapour from the nitrogen stream; it will not cope with gross contamination of the nitrogen supply or a large inward leak. A system should be capable of running continuously for a week or more without a line drier. By using a line drier in the nitrogen flow path of the Cryostream, you can extend the continuous run period to around 2 weeks.

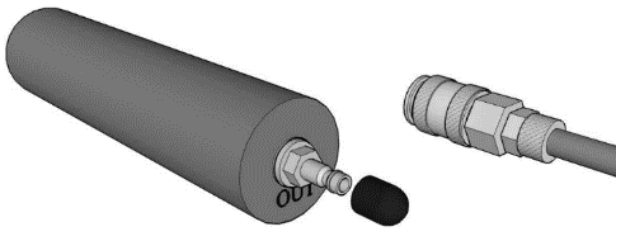


Figure 6: Line Drier with blanking cap and connection hose

6.9.1 Before Fitting a Line Drier

Upon installing your Cryostream, we strongly recommend running the system WITHOUT the Line Drier at first, as otherwise, any initial issues with the system will be masked. For example if there are any inward leaks or a high level of ice in the liquid nitrogen, the Cryostream will block within a few days. (Refer to Appendix 2 Cryostream Troubleshooting Guide for further details). The Cryostream should run for at least 7 days before signs of a block, although poor quality nitrogen can reduce this.

Once the system has run for about a week and there are no leaks in the system, fit the Line Drier.

6.9.2 Fitting the Line Drier

1. Remove the blanking caps (these caps must be fitted to the Line Drier during transit and storage to prevent contamination).

2. Disconnect the FLOW connector from the back of the controller

3. Plug this connector into the OUT fitting on the Line Drier.

4. Connect the short Line Drier tube (supplied) between the FLOW connector on the controller and the IN fitting on the Line Drier.

NOTE

Do not leave the Line Drier open to atmosphere as this will cause contamination and shorten its service life.

NOTE

It is good practice to turn the Cryostream system off overnight or program a PURGE around once every two weeks to avoid blockages interrupting the use of your Cryostream.

6.10 Cryostream Nozzle Alignment

The Nozzle Alignment Tool has been designed to allow the exact centring of a crystal sample in the nitrogen gas stream. Accurate alignment of the stream is vital to avoid ice formation, as it ensures that the crystal stays in the coldest part of the stream as it rotates.

1. Correctly align and centre an example crystal or empty loop as normal by using the adjustments on the goniometer head and telescope or camera crosshairs to focus sharply on the crystal's position.

2. Remove the sample and fit the Nozzle Alignment Tool to the end of the nozzle and tighten the thumb screw to hold it in place.

3. Use the Varibeam Coldhead Support Stand to adjust the position of the Cryostream so the tip of the point of the Nozzle Alignment Tool is in focus at the centre of the crosshairs.

4. Carefully remove the Nozzle Alignment Tool as soon as the nozzle is aligned. The point of the Nozzle Alignment Tool corresponds to the ideal distance the crystal should be from the end of the Cryostream Cooler nozzle.

WARNING

- Care should be taken when handling pointed objects.
- Please ensure the Nozzle Alignment Tool is stored appropriately.

6.11 Temperature at the Crystal Position

In contrast to other cooling systems and indeed some previous versions of the Cryostream, the Cryostream displays the gas temperature at the position of the crystal, rather than at a point within the Coldhead. Oxford Cryosystems has established a method of mapping the true temperature at 5 mm from the end of the nozzle. Because of the superior laminar flow of the Cryostream gas stream, one can place the sample up to 8 mm from the end of the nozzle, although 5mm is recommended as the optimum position.

The correction which needs to be applied is a function of gas flow, which means that switching between Normal and Turbo flows will cause a temporary change in the crystal temperature. The Cryostream will compensate for this change. Therefore the reading of "Gas Temperature" on the Cryostream Controller, CryoConnector software or Oxford Connect is the true temperature of the nitrogen gas at the crystal position.

7 Programming the Cryostream 800 Series Controller

7.1 Final Checks Before Switching on the Controller

- Ensure that all the tube and power connections are correctly made.
- Ensure that there is liquid nitrogen in the Dewar and the Cryostream rigid transfer line is in the Dewar vessel.
- Make sure that the dry air supply is switched on, connected to the Cryostream and the flow rate is set correctly.

7.2 Starting up the 800 Series Controller

The 800 Series controller is switched on using the power switch on the rear of the controller.

The 800 Series controller is designed to be intuitive and simple to use. The front screen incorporates a single illuminated Start/Stop button and a touch screen.

Figure 7: Touch screen (shown above in Command screen) and illuminated Start/Stop Button

The button is used for starting, stopping and restarting the controller. Depending on the state of the controller it will be illuminated according to the table below.

Illumination	Cryostream State	Result of button press
Solid green	Ready	Run controller
Pulsing green	Running	Stop controller
Solid red	Stopped normally	Restart controller
Flashing red	Stopped with error	Restart controller

7.3 Cryostream Controller Home Screen

After the controller has completed its self-check the Home screen is displayed, as shown in Figure 8.

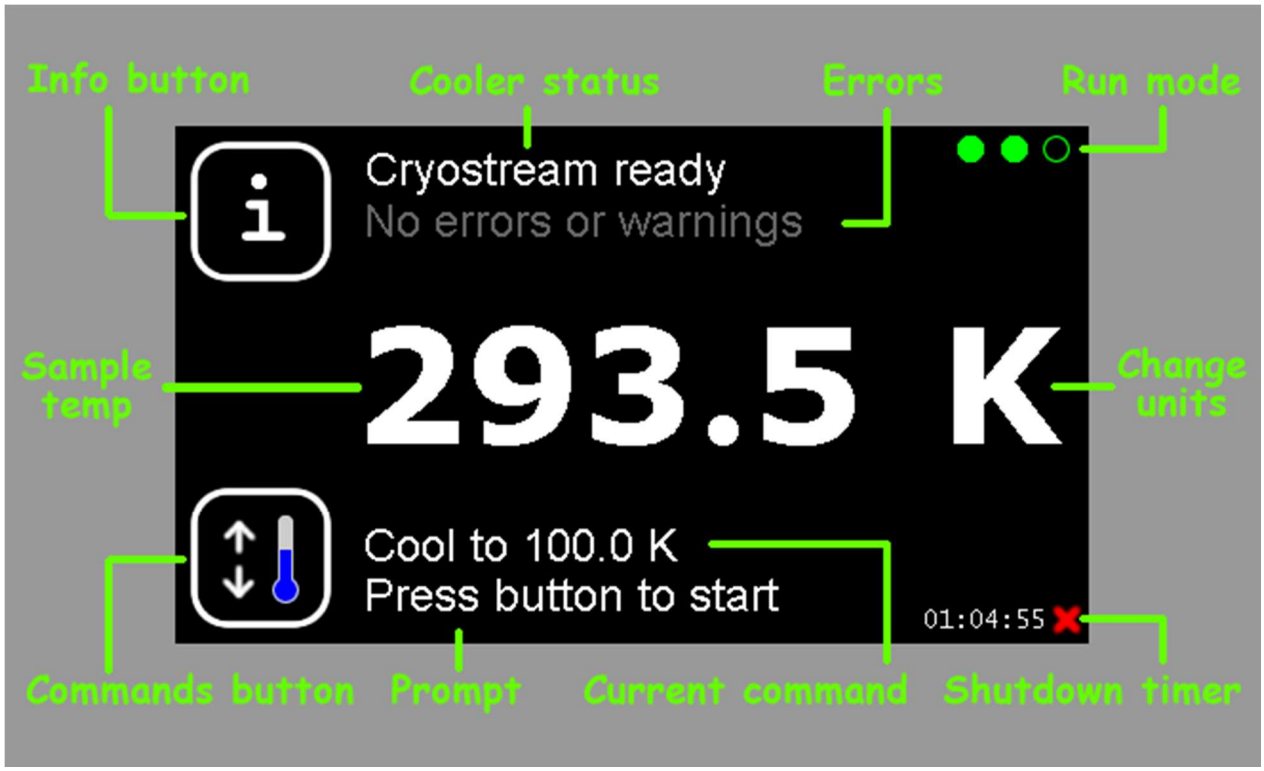


Figure 8: Key to 800 controller home screen

NOTE
Pressing the screen lightly with the fingernail tip gives the best response.

Once the home screen is shown, the cooler may be started by pressing the Start/Stop button, and will execute the command indicated by the Current command text (in this case "Cool to 100.0 K"). To specify a different command (e.g. a different temperature or a controlled ramp rate), press the Commands button to display the “Set temperature” screen. Setting the temperature is described in 7.4.1 Changing the Set temperature and Ramp rates.

NOTE
The Cryostream controller will default to a Command of “Cool to 100.0 K “on first start-up. To execute this command, simply touch the illuminated Start/Stop button.

Once the cooler is running the Home screen will look as shown in Figure 9.

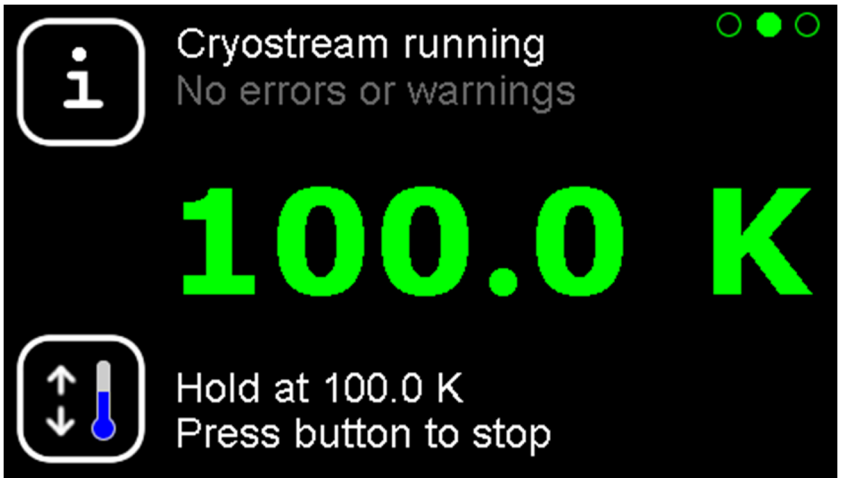


Figure 9: Home screen when running

7.3.1 Changing Temperature Units

The Cryostream 800 Series is set to Kelvin by default. To switch between Kelvin, Celsius and Fahrenheit, simply press the current temperature unit ("K", "C" or "F").

7.3.2 Stopping the Cooler

The cooler may be stopped at any time by pressing the Start/Stop button, at which point the Home screen will look as shown in Figure 10. Pressing the Start/Stop button once again will initiate self-check and return the controller to the Ready state.

Figure 10: Home screen when cooler stopped

NOTE
Other ways of shutting down your Cryostream cooler are described in Section 7.4.3 Set shutdown options

7.4 Command Screens

The controller has a number of screens that can be cycled through for both control and monitoring of the Cryostream. To access the Command Screens, press the Commands button, and use the left and right arrows to scroll through the screens. These are as follows:

- Command screen 1: Changing the Set temperature and Ramp rates
- Command screen 2: Setting the gas flow
- Command screen 3: Set shut-down options

7.4.1 Changing the Set temperature and Ramp rates

This screen is accessed by pressing the Command button from the Home screen

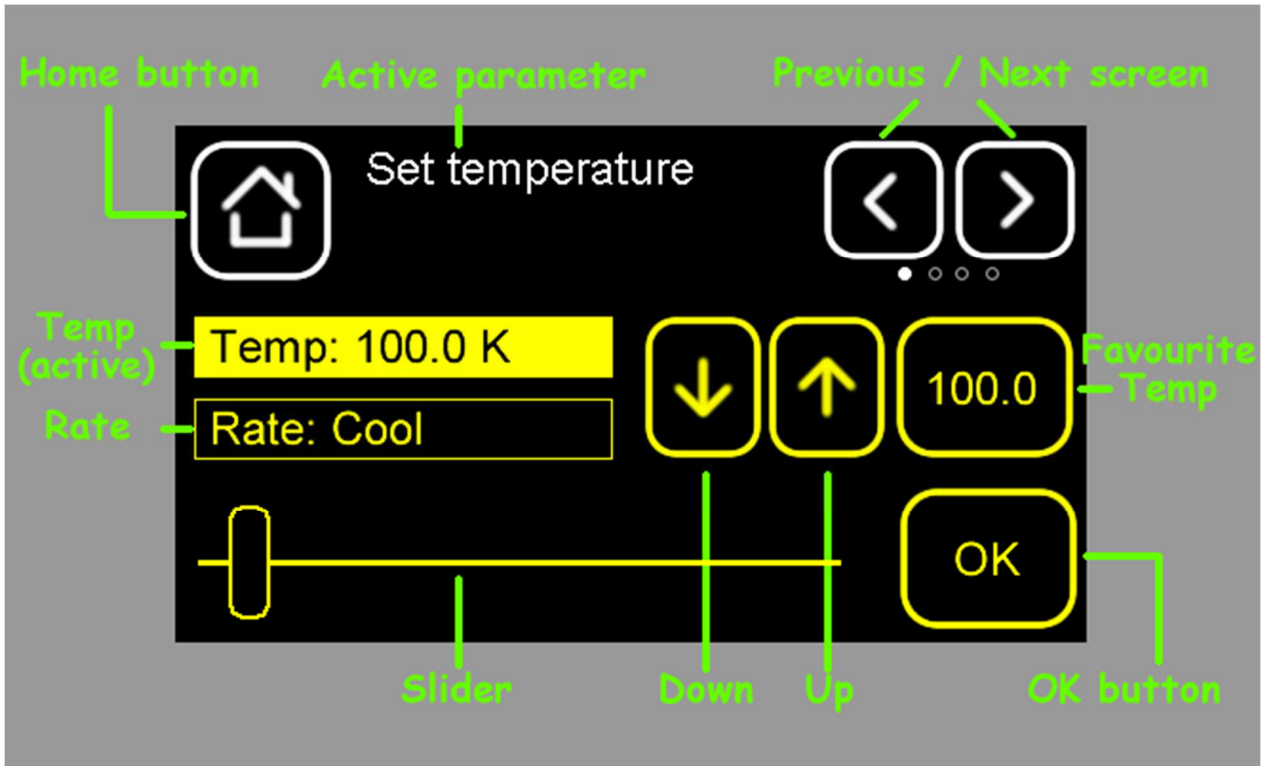


Figure 11: Key to command screen 1

Use this screen to adjust the set temperature and the cooling rate. The Slider together with the Down and Up buttons are used to adjust the active parameter, which is the one in black text against a yellow background ("Temp" in the illustration above). Pressing OK sets the parameter and activates the next parameter to be edited.

Temp: This parameter sets the gas temperature at the crystal position.

Rate: This parameter sets how quickly the Cryostream will change from the current gas temperature to the set gas temperature. Using the “Cool” rate will command the Cryostream to change temperature as quickly as possible.

NOTE
Using the Cool rate to decrease the gas temperature will cause the Cryostream to increase the nitrogen gas flow to 10 l/min.

Using the Slider

Use the Slider to set the parameter quickly to an approximate value (e.g. in the case of temperature the Slider allows setting to the nearest 10 K). Fine-tuning is achieved using the Down and Up buttons.

> **NOTE**
>
> Pressing the slider lightly with the fingernail tip gives the best response.

Using the Down and Up buttons

A single press will change the active parameter by the smallest allowable amount (0.1 K in the case of temperature). A longer press will result in changes of 1 K, and if the button is held down then the step size will increase.

The Favourite button

Pressing the Favourite button will set the active parameter to the value displayed (in Figure 11, and as default upon start-up, the favourite temperature is set to 100.0 K). The controller will learn your favourite values for the various parameters.

The OK button

Press this when you are happy with the value of the active parameter. If you are adjusting the temperature, then next the Ramp rate is activated. Pressing OK again will complete the process of setting the temperature and return you to the Home screen.

7.4.2 Setting the gas flow

It is possible to set the nitrogen flow rate of the Cryostream using Command screen 2; this screen is accessed by pressing the Command button from the Home screen and then using the Next screen button. It is used to switch between Auto and Turbo gas flows. Note that Auto is the default setting.

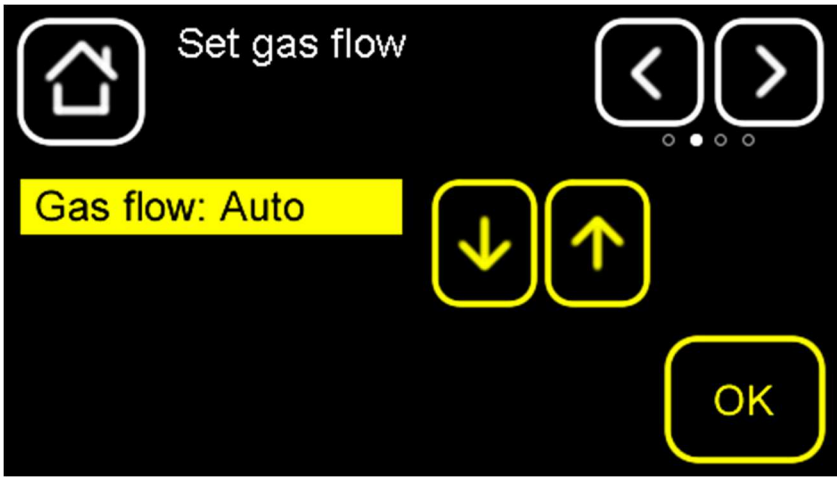


Figure 12: Command screen 2 showing gas flow settings

The two options are toggled using the Down or Up buttons. Pressing OK will implement the current selection and return to the Home screen. The meanings of the two options are given in the table below.

Auto	This is the default flow setting. The system will use 5 l/min gas flow except in a Cool or below 90 K. In a Cool 10 l/min is used to maximise the cooling speed. Flow rates greater than 5.0 l/min are also used to achieve temperatures < 90 K
Turbo	The system will use 10 l/min gas flow except above 310 K. Above 310 K available heater power limits the maximum achievable flow to 5 l/min

7.4.3 Set shutdown options

The Cryostream can be programmed to shut down in a number of ways using Command screen 3; this screen is accessed by pressing the Command button from the Home screen and then using the Next screen button. It is used to program a shutdown.

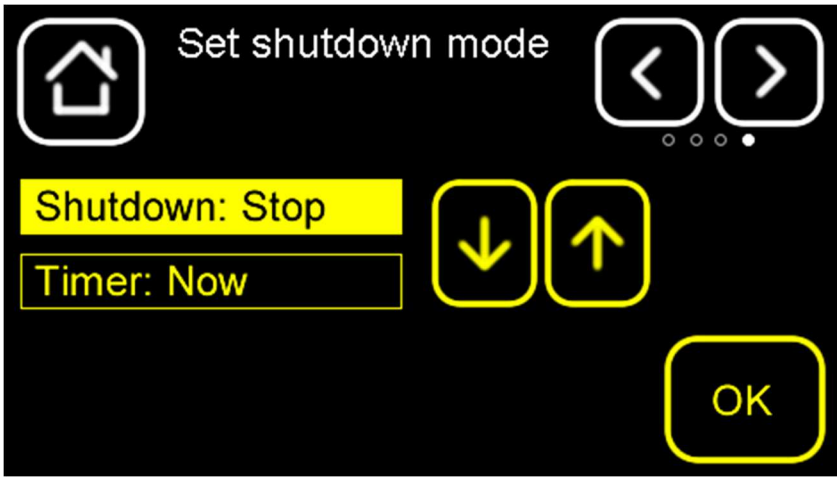


Figure 13: Command screen 3 showing shut-down options

When the Shutdown mode is the active parameter there is no Slider or Favourites button, the three options (explained below) are toggled using the Down or Up buttons. Pressing OK will implement the current selection and activate the Timer parameter. This will cause the Favourites button and the Slider to be shown, enabling the time period before the chosen shutdown method is activated to be edited. Pressing OK again will activate the chosen shutdown command after the selected period.

If a timed shutdown is selected by choosing a Timer period other than Now, the Home page will display the Shutdown timer. Pressing the red cross on the home screen will cancel the timer.

Stop Gas flow is halted and the system is stopped at the current temperature.

End The sample is warmed to 300 K, at which point the gas flow is halted and the system stops.

Purge Gas flow is halted and the whole system brought up to 300 K using its internal heaters, at which point the system stops. We recommend programming a Purge once every 2 weeks on average, in order to avoid ice blockages in the Cryostream.

NOTE

Pressing the Start/Stop button whilst running will immediately stop the system.

7.5 Info Screens

The controller has a number of screens that can be cycled through for monitoring of the Cryostream. These are not necessary during normal operation, but you may be asked to view these by our technical support team to collect various information about the system. To access the Info Screens, press Info and use the left and right arrows to scroll through the screens. There are 6 info screens in total, we show only two examples below. However if the technical support team ask for various parameters to be read from the controller, they will clearly specify which screen(s) should be accessed.

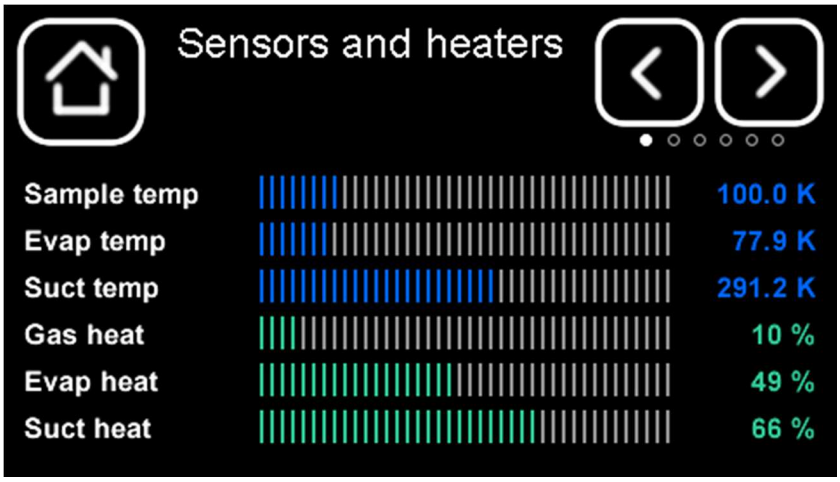


Figure 14: Info screen 1: Sensors and heaters

Displayed are the live readings from the three temperature sensors together with the current heater outputs

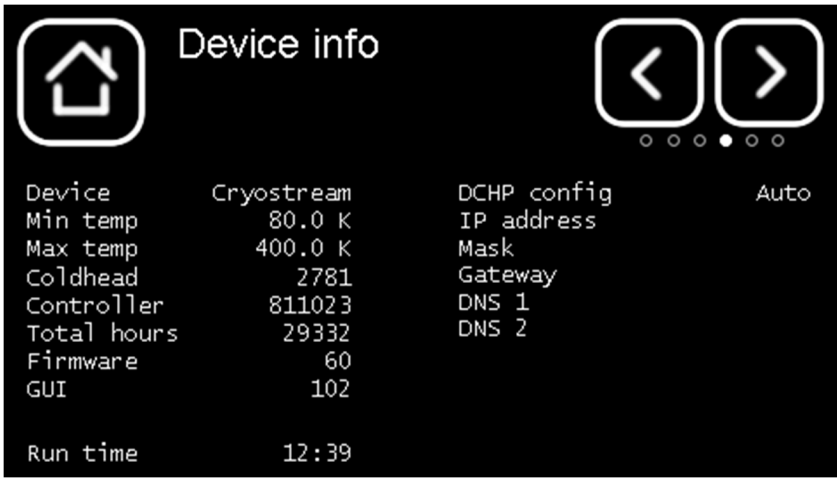


Figure 15: Info screen 4: Device Info

Displayed here are various properties of your cooler including serial numbers and hour count. Also shown are the current settings used for the Ethernet connection.

7.6 Errors and Warnings on the Cryostream Controller

The Cryostream controller will change its appearance when it displays an error or warning.

Should an error condition occur which causes the system to shut down, the Start/Stop button will flash red and the controller will display the Home screen, which will appear similar to Figure 16.

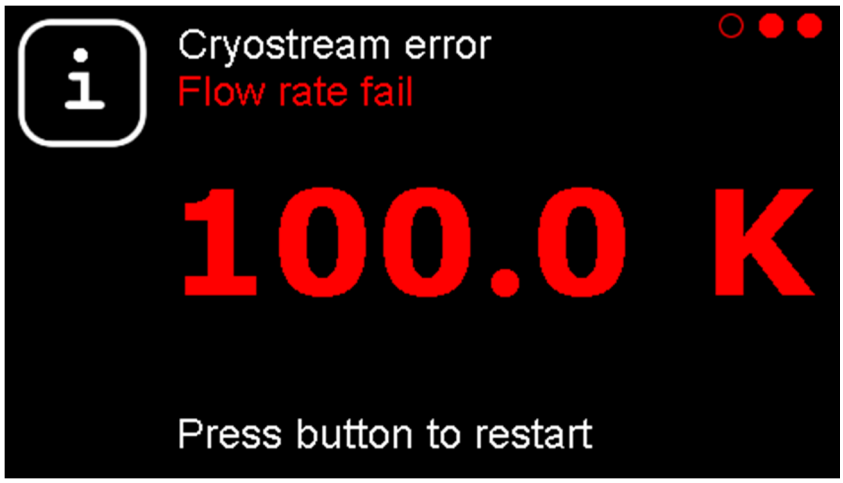


Figure 16: Home screen after system has shut-down with error

If your cooler displays an error or warning, please refer to Appendix 2 Cryostream Troubleshooting Guide or contact Oxford Cryosystems' Support Team by e-mailing support@oxcryo.com or calling +44 (0)1993 883 488.

Once the issue has been resolved, the controller can be re-started by pressing the Start/Stop button.

8 Maintenance

CAUTION

It is recommended that a PURGE is scheduled every 1-2 weeks to avoid ice blockages in the system and help prevent any interruption to your work.

8.1 Service Intervals

Procedure	Typical Service Interval	Comments
Pump down of Vacuum	6-18 months	It will be necessary to re-pump the vacuum space as this will deteriorate over time. Please note that the vacuum space in 3m transfer lines will deteriorate faster than the standard 1.5m lines so these Cryostreams may need more regular pumping. Details about the vacuum pump down procedure are found in Appendix 3 . For signs of vacuum deterioration, see Cryostream Troubleshooting Guide.
Replacement of Line Drier	6-12 months	Replacement of the line drier units are recommended as they will eventually become saturated with water vapour from the nitrogen supply.
Service of Dry Air Unit	12,000 Hours	The Counter on the front of the AD51 indicates how many hours the system has run. An AD51 Service Kit can be obtained from Oxford Cryosystems or your local agent.
Dewar Cleaning	2-3 months	

For further advice or information on maintenance procedures, please contact **support@oxcryo.com**

While all of our devices are designed to be as efficient and economical to maintain as possible, as with any mechanical system, some level of maintenance will be required. As always, Oxford Cryosystems continues to offer free unlimited technical support to allow you to carry out minor service work; however, in response to demand from our customers, we are pleased to now offer a choice of pre-paid maintenance contracts.
Please contact support@oxcryo.com for more information on the service contracts available.

Appendix 1 Remote control and monitoring of the Cryostream

The 800 Series Cryostream may be controlled and monitored from a PC using our CryoConnector software. CryoConnector replaces the previous Cryopad software, it is available free of charge from connect.oxcryo.com and is also found on the CD provided with your Cryostream.

Oxford Connect is a free online platform that can be used with the CryoConnector software to control and monitor a Cryostream from any web-enabled device such as a tablet, smartphone or remote PC. By registering your new device at Oxford Connect, you will be able to:

- Connect to your Cryostream via the web.
- Remotely monitor and control your Cryostream from a PC, smartphone or tablet.
- Automatically log your device data.
- Enable Oxford Cryosystems to remotely view your device performance for faster tech support.
- Receive status notifications when your device status changes.

NOTE

The Cryostream can only be monitored and controlled by one piece of PC software at a time through each port; e.g. if you are using the USB port to connect to the controller through CryoConnector, you will need to use the serial or Ethernet ports to connect to the Cryostream using software made by your diffractometer manufacturer.

Installing CryoConnector

NOTE
Oxford Cryosystems' CryoConnector software requires Internet Explorer 8 or later and Windows XP or later.

CryoConnector can be downloaded from the Oxford Connect website (connect.oxcryo.com) or installed from the CD provided with your Cryostream.

Oxford Connect Website:

1. Create an account with the Oxford Connect website using the Device ID for your Cryostream (found on a card on top of the Cryostream controller).
2. Once registered, press the "Download CryoConnector" button on the homepage.
3. Open the downloaded file from your downloads folder (Ctrl+J open your download folder this in most browsers).
4. An installation wizard will walk you through installing CryoConnector.

Software CD:

Your Cryostream system will come with a CD containing an electronic copy of our manuals and software.

1. Insert this into your PC and run the CD to access these files.
2. Select the CryoConnector file to initiate an installation wizard that will install CryoConnector.

Logging in to Oxford Connect using CryoConnector

NOTE

The PC connected to your Cryostream (which is running CryoConnector) will need a stable internet connection to successfully use Oxford Connect.

Press F6 when CryoConnector is running to open the Oxford Connect window (Figure 17). From this window, you can log in to Oxford Connect to link your Cryostream to your Oxford Connect account:

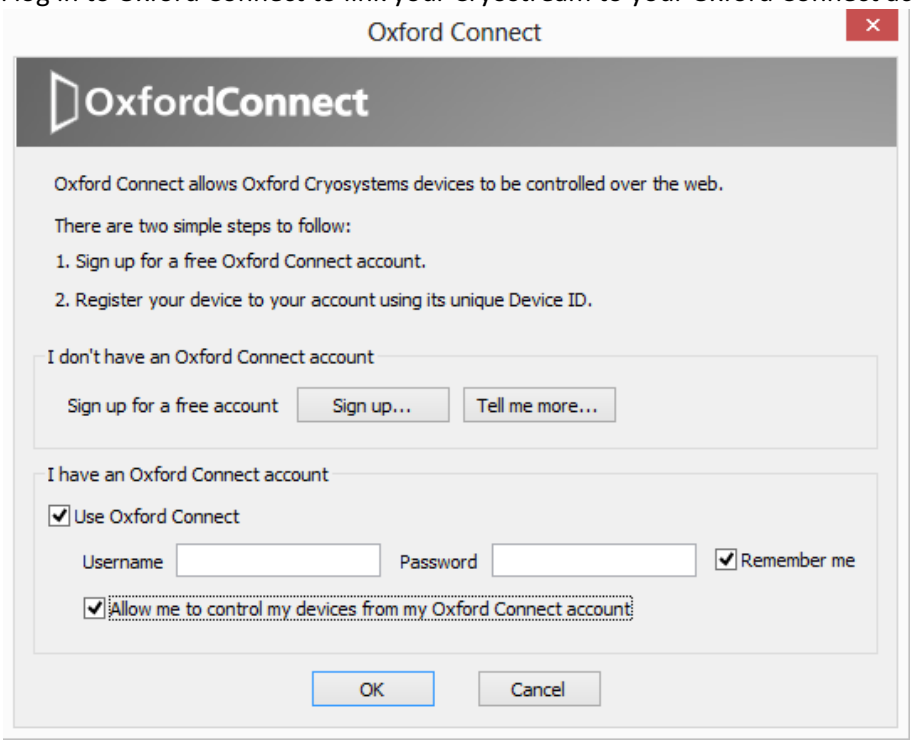


Figure 17: CryoConnector Oxford Connect Window

1. If you don't have an account, press the "Sign up…" button to go to the Oxford Connect website and create an account.
2. If you have an account, select "Use Oxford Connect" tick box to allow connection to Oxford Connect.
3. Fill in your Username and Password that you chose when setting up your account.
4. Selecting the "Allow me to control my devices from my Oxford Connect account" tick box will allow you to send commands from online devices to your Cryostream
5. Selecting the "Remember Me" will allow CryoConnector to remember your log-in details.

Monitoring and Controlling the Cryostream through CryoConnector

CryoConnector consists of three sections accessible in a single window: Overview, Commands and Display. The Overview section is always visible, while the Commands and Display sections can be hidden or revealed by pressing the "Commands" and "Display" buttons.

If multiple Oxford Cryosystems devices are connected to the same PC, they will all appear in the same CryoConnector window.

NOTE

CryoConnector will be continuously updated by our software engineers and so the appearance may change and new features may be added as new versions are released. For information on any new features or to suggest new features that would be useful, contact Oxford Cryosystems (support@oxcryo.com).

CryoConnector Overview Section

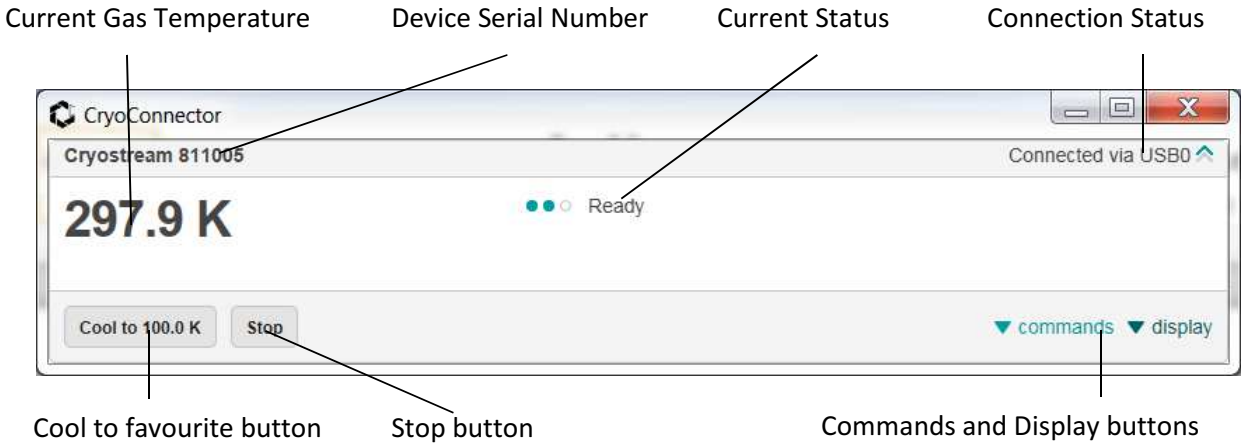


Figure 18: CryoConnector showing Overview Section only

- **Current Gas Temperature:** Shows the current nitrogen gas temperature at the crystal position..
- **Device Serial Number**: Displays the model and serial number of the device.
- **Current Status:** When running, shows total run time of current Cryostream run, what the current target temperature and rate of change is and any errors or warnings; otherwise shows the current status of the Cryostream.
- **Connection Status:** Shows which port of your PC the Cryostream is connected to CryoConnector through. Moving blue chevrons confirm an active connection to Oxford Connect; a red cross shows that there is no connection with Oxford Connect.
- **Cool to favourite button:** Commands the Cryostream to change temperature to the temperature displayed as quickly as possible, overwriting the existing command.
- **Stop button:** Commands the Cryostream to stop running immediately.
- **Commands and Display buttons:** Show (and hide) more CryoConnector functions.

CryoConnector Commands Section

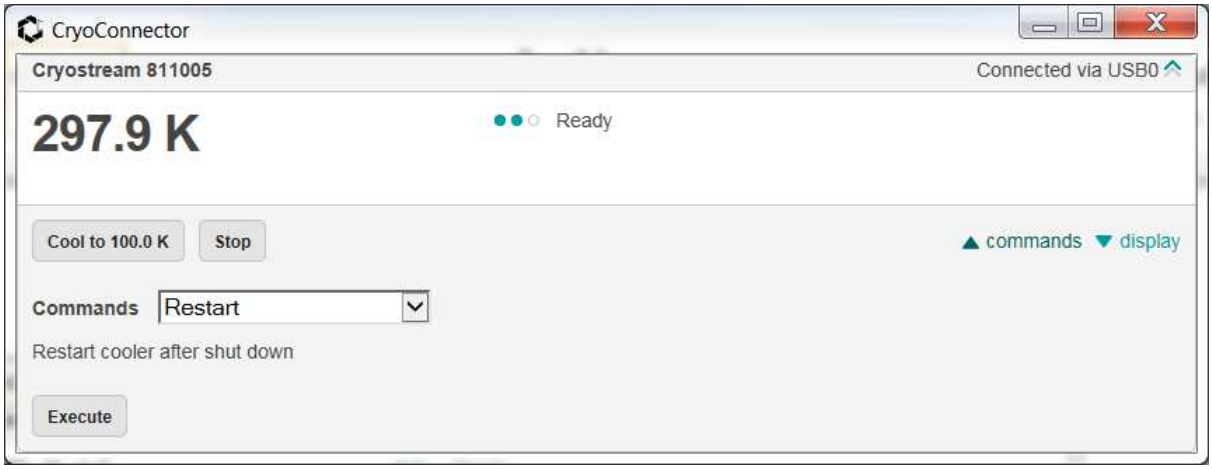


Figure 19: CryoConnector showing Commands Section

The Commands section can be used to give commands to the Cryostream.

The options in the Commands drop-down list are:

Command Name	Meaning
RESTART	Stop Cryostream and re-initialise system back to “Ready”.
RAMP	Change gas temperature to a set value at a controlled rate.
COOL	Make gas temperature decrease to a set value as quickly as possible.
PLAT	Maintain the current temperature for a set amount of time.
SUSPEND	Interrupt the current commands and maintain the current gas temperature until instructed otherwise by a RESUME command.
RESUME	Resume the previous command before the SUSPEND command was given.
HOLD	Stay at the current temperature indefinitely with no ability to resume the previous command (unlike the SUSPEND and RESUME functions).
END	Bring the gas temperature to 300 K, then shut down.
PURGE	Bring the gas temperature and the internal temperature to 300 K then shut down.

Pressing the “Execute” button will cause the 800 Series Cryostream to carry out the phase currently shown.

CryoConnector Display Section

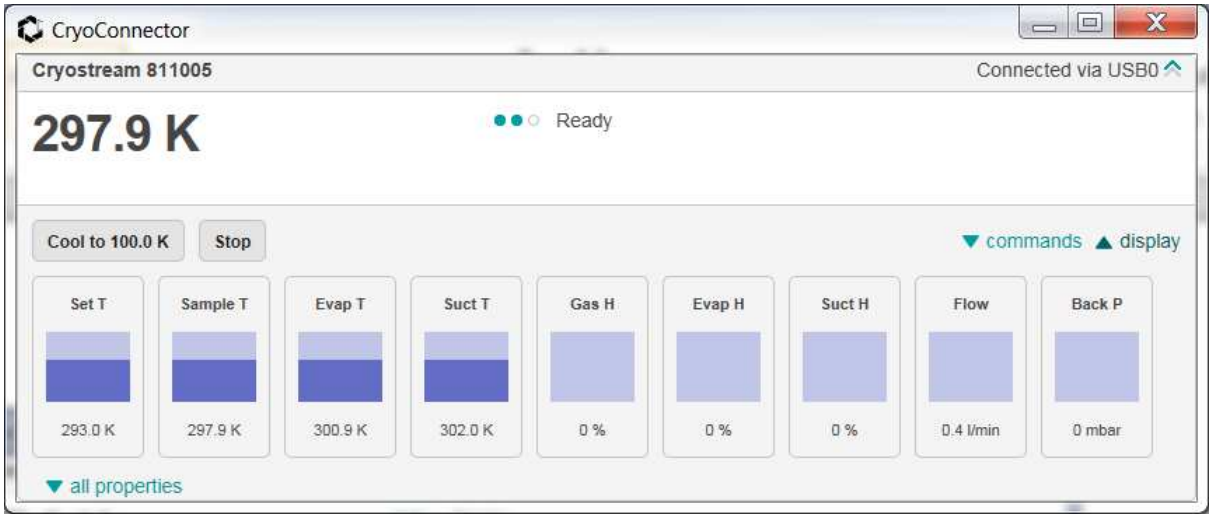


Figure 20: CryoConnector showing Display Section

Pressing the "Display" option will display a range of 800 Series Cryostream parameters (as per 7.5 – Info Screens). Further parameters can be viewed by pressing the "all properties" button; these may be useful to report when liaising with Oxford Cryosystems support.

Using Oxford Connect

Your Oxford Connect account can be accessed by visiting connect.oxcryo.com using your web browser. Oxford Connect is compatible with a wide range of internet browsers.

Once you have logged in, click on "My Devices" to view the Oxford Cryosystems devices registered to your account as well as information on those that are currently connect to Oxford Connect through CryoConnector.

Any devices connected to Oxford connect through CryoConnector can be controlled through Oxford Connect. The interface on Oxford Connect is the same as the CryoConnector interface described above, but is viewed in your browser window.

If you have any issues with CryoConnector or Oxford Connect, please contact Oxford Cryosystems (support@oxcryo.com) for further information.

Other CryoConnector Functions

CryoConnector has a range of other options that can be accessed by pressing a function button or by accessing the Menu.

> **NOTE**
>
> Access the Menu by single-clicking on the hexagonal Oxford Cryosystems logo in the top left of the window.

Option Name	Shortcut	Use
Help	F1	Access online help about using CryoConnector and Oxford Connect.
Connections	F3	View devices currently connected to CryoConnector.
Settings…	F4	Set the data log storage location for each device.
Scan for Devices	F5	Check all available computer ports for Oxford Cryosystems devices.
Oxford Connect Settings	F6	Log in and change settings for connection with Oxford Connect.
Visit Oxford Connect	F7	Open the Oxford Connect webpage in your browser.
Set Device Alias		Name or rename your Cryostream so it is easier to identify on CryoConnector and Oxford Connect.
Check for Updates		Check the Oxford Cryosystems server for updates to the CryoConnector software.
About…		Show current version of CryoConnector software.

> **NOTE**
>
> CryoConnector is frequently updated by the Oxford Cryosystems' Software development team to include new features. CryoConnector can be set to automatically check for updates on start-up; the option for this is found in the "About CryoConnector…" option on the menu.

Appendix 2 Cryostream Troubleshooting Guide

NOTE

Oxford Cryosystems offers free-of-charge technical support for all of its products; please do not hesitate to contact us with any problems.

support@oxcryo.com +44 (0) 1993 88 34 88

The following information is intended for use by operators of the Cryostream. It is intended to provide the correct solution to a range of common technical problems, but it does not cover every technical possibility for the Cryostream.

Do not rush into changing components in an attempt to fix the Cryostream as changes may mask existing symptoms and increase the length of time taken to resolve a problem.

Some major issues will be detected by the Cryostream controller and will produce an error and will cause the Cryostream to stop; the controller screen text will turn red, the controller button LED will also turn red and the error will be displayed on the home screen. Please see the table on the next page if you have an error.

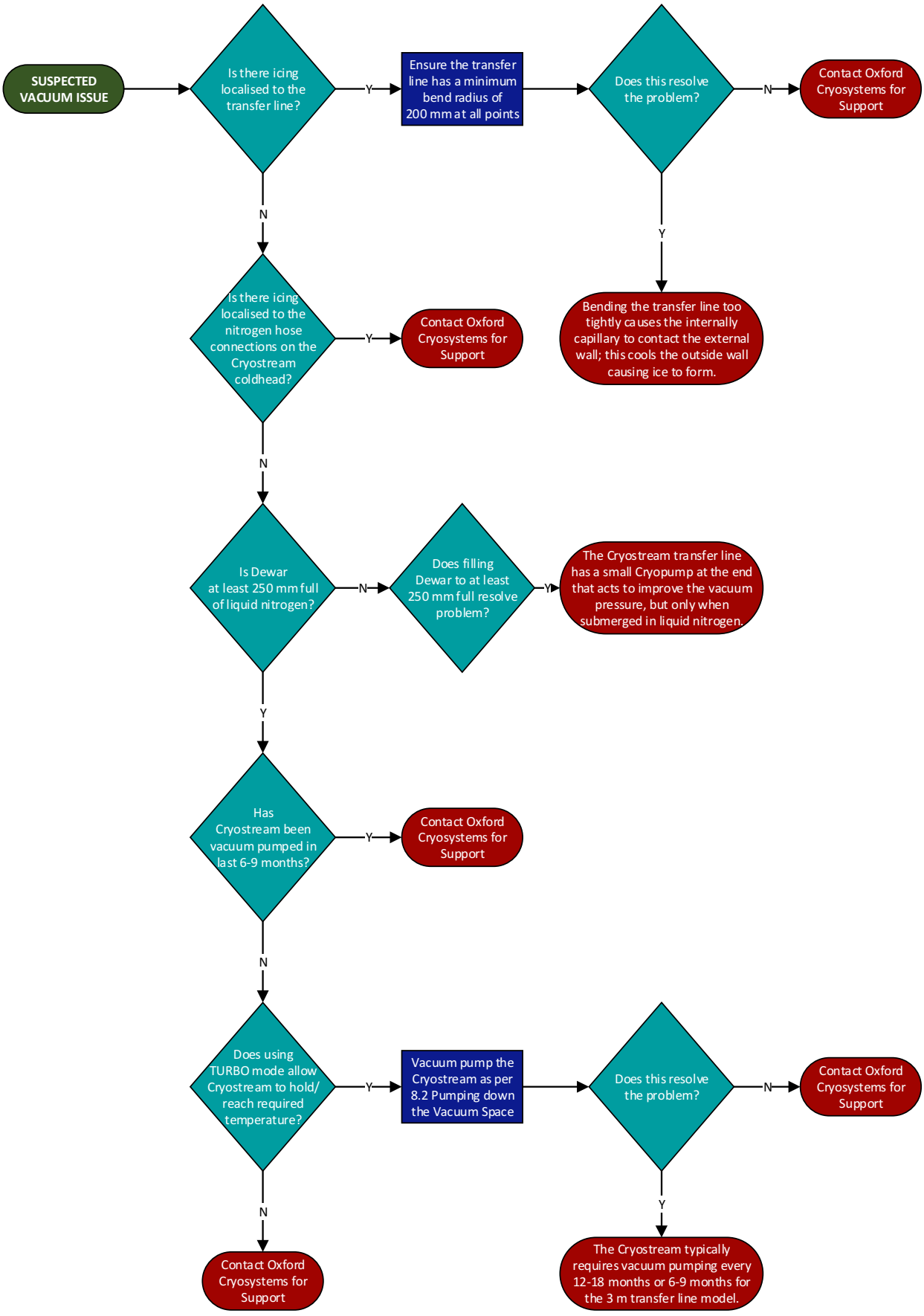
Some minor issues will be detected by the Cryostream controller and will produce a warning; these will be displayed on the home screen, but will not cause the Cryostream to stop. Please see the table on the next page if you have a warning.

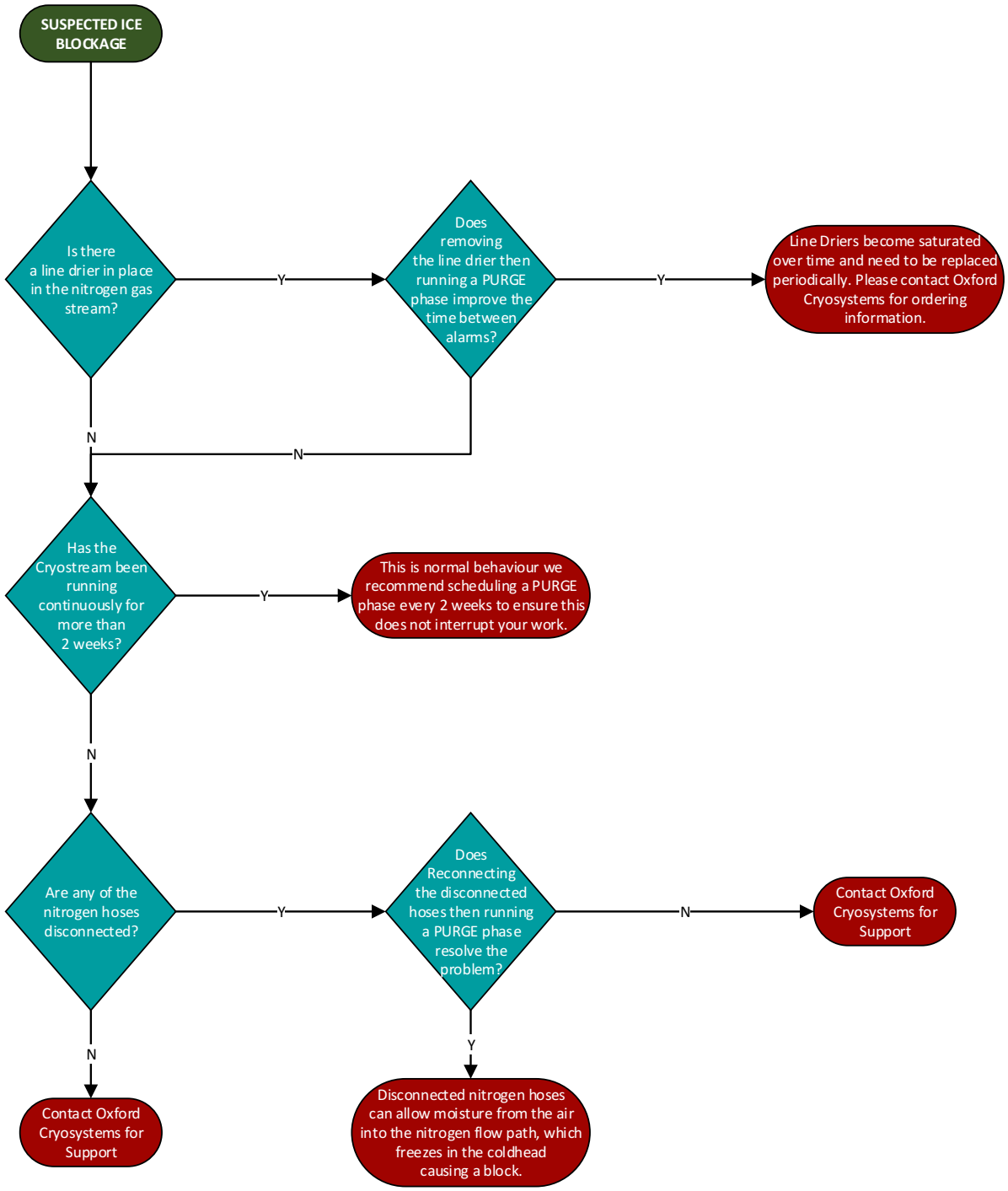
Some issues are not detected by the Cryostream controller and so do not give an error or warning status; please see the “Symptoms” table on the next page if you have an issue but no error or warning.

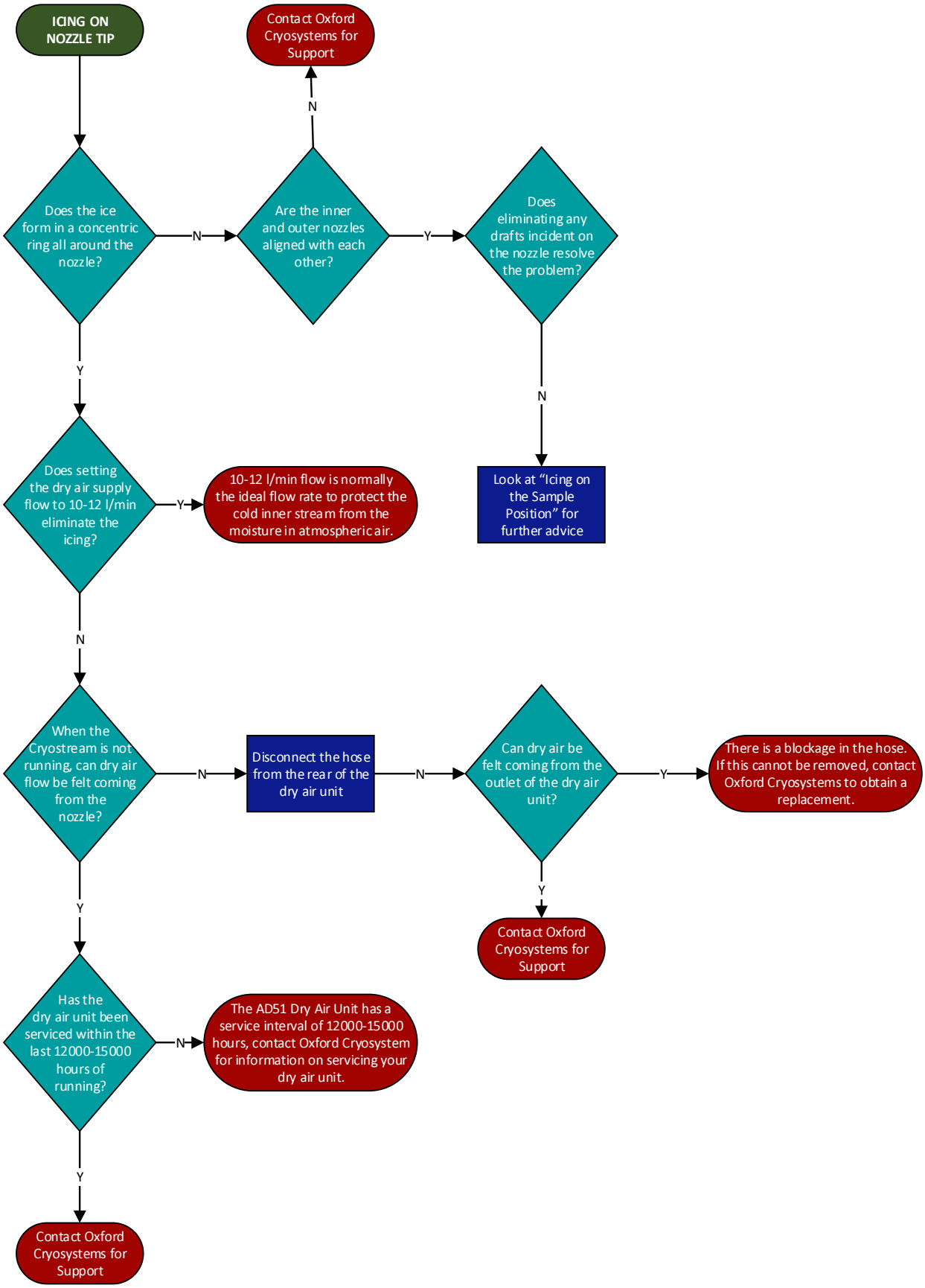
Further information on some of these issues is given on the subsequent pages.

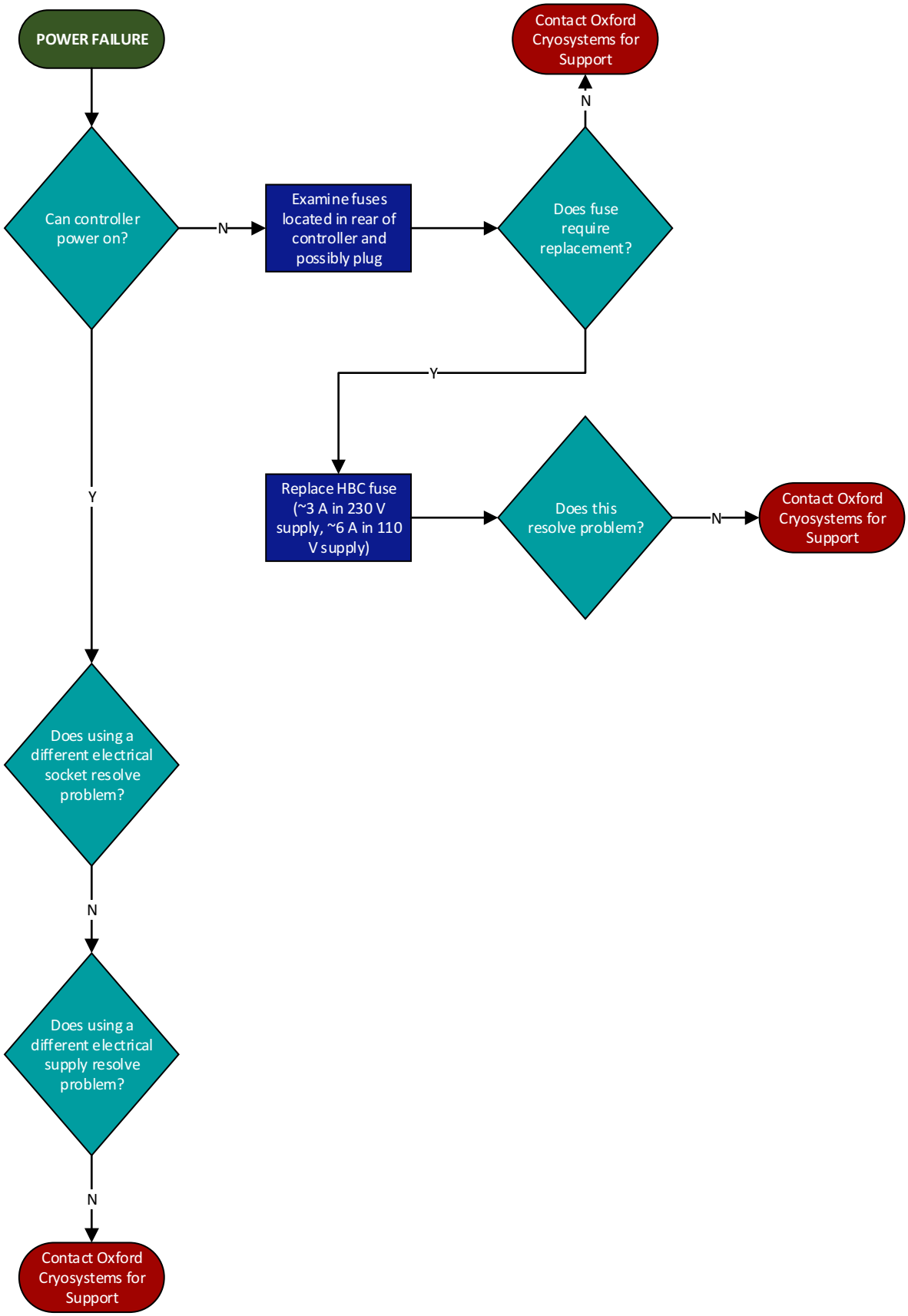
Error	Description	Action
Temp Control Error	Difference between requested temperature and actual temperature is greater than 25 K.	See Flow Chart 1 – Suspected Vacuum Issue.
Flow Rate Fail	Nitrogen gas flow has dropped too low.	See Flow Chart 2 – Suspected Ice Blockage.
Self-Check Fail	Cryostream controller has detected a problem.	Check Cryostream is set up correctly. Contact Oxford Cryosystems for support.
SUCT Temp Error	SUCT connector temperature is too high or too low.	Contact Oxford Cryosystems for support.
Temp Reading Error	Cryostream controller has detected nonsense sensor reading.	Contact Oxford Cryosystems for support.
Sensor Fail	Cryostream controller has detected a faulty sensor.	Contact Oxford Cryosystems for support.
PSU Overheat	Cryostream controller power supply is overheating.	Contact Oxford Cryosystems for support.
Warning	**Description**	**Action**
Temp Warning	Difference between requested temperature and actual temperature is greater than 10 K.	See Flow Chart 1 – Suspected Vacuum Issue.
High back pressure	Cryostream controller has detected a blockage in the nitrogen flow path.	See Flow Chart 2 – Suspected Ice Blockage.

Symptom	Action
Inability to reach low temperatures	See Flow Chart 1 – Suspected Vacuum Issue.
Reaches low temperature, then temperature starts to increase	See Flow Chart 1 – Suspected Vacuum Issue.
Poor temperature stability	See Flow Chart 1 – Suspected Vacuum Issue.
Condensation on the Cryostream coldhead and/or transfer line	See Flow Chart 1 – Suspected Vacuum Issue.
Cryostream carries out a PURGE phase then shuts down	See Flow Chart 2 – Suspected Ice Blockage.
Icing on the Cryostream coldhead nozzle tip	See Flow Chart 3 – Icing on Nozzle Tip
Icing on the sample position	See “Icing on the Sample Position” below.
Liquid nitrogen coming out of the Cryostream coldhead nozzle	Contact Oxford Cryosystems for support.
Controller turns off unexpectedly	See Flow Chart 4 – Power Failure.
Controller will not turn on	See Flow Chart 4 – Power Failure.









Icing on the Sample Position

Icing on the sample position is usually caused by environmental factors or the sample mount itself. Please find below a list of possible causes of icing on the sample or sample mount and suggestions on how this can be avoided.

Cause of Icing	Possible Solutions
The sample is too far away from the nozzle or not aligned in the centre of the cold stream. The cold stream and the dry air stream mix and draw in atmospheric moisture that is frozen out on the sample.	Position the nozzle as close to the sample as possible without affecting the path of the x-rays or casting an image on to the detector. The ideal position is inside the first 6 mm from the end of the nozzle and the centre 2 mm of the flow. A nozzle alignment tool is available from Oxford Cryosystems to aid this.
A disturbance of the laminar flow system due to drafts in your laboratory.	Check the laboratory for drafts; the most likely cause of turbulence is an air conditioning unit, a cooling fan from an x-ray generator or the rotating anode generator. Create a screen between the source of the draft and your cold stream.
An interruption of the laminar flow caused by an oversized sample mount (i.e. capillary or pin is too thick).	If possible, try using a smaller or thinner sample mount.
The inner (cold) and outer (room temperature) gas streams are not creating a laminar flow.	Try adjusting the flow of the outer dry gas stream. In a draft-free environment 10 L/min is fine, but when the air is more turbulent, try turning the outer stream flow rate up to 12 to 15 L/min; this can often resolve the problem. Also, see “Flashlight Test” below.
The loop is unclean. Any particles on the loop will propagate ice formation.	Ensure that the loop is clean and free of particles before use.
The angle between the Cryostream nozzle and the pin is less than 90°. This can cause the pin to interfere with the gas stream prior to it reaching the crystal, disrupting the laminar flow and so cause icing.	Increase the angle between the Cryostream and the pin. Cryostreams can be mounted from below in some situations; please contact Oxford Cryosystems for details.
Insufficient cryoprotection of the buffer solution. Too much mother liquor results in dilution of the cryoprotectant to the point where it is no longer adequate. A thick film around the crystal may result in a larger thermal mass that must then be cooled.	To increase the effectiveness of the cryoprotectant, increase its concentration.

Flashlight Test

To be sure the flow rate of the outer dry air stream is correct, it is often better to set the flow by eye rather that by trying to guess what the flow should be by looking at the numbers. Turn all the lights off in the laboratory and shine a flashlight up towards the nozzle of the Cryostream Cooler in an attempt to highlight the plume created by the cold gas stream. As the gas stream leaves the nozzle it is really made up of two parts; the first 'invisible' 10 or 12 mm and the remaining plume of ice. The object of the exercise is to maximise the length of the 'invisible' section. This should only be done over the first 15 L/min of air from the dry air source. One should not be fooled into thinking that at 25 L/min there is no plume, and therefore, no ice because the ice will build rapidly around the end of the nozzle and blow the sample from its support.

NOTE

Oxford Cryosystems offers free-of-charge technical support for all of its products; please do not hesitate to contact us with any problems.

support@oxcryo.com +44 (0) 1993 88 34 88

Appendix 3 Pumping down the vacuum space in the Cryostream

The vacuum space in the Cryostream will need to be evacuated (or pumped) occasionally to ensure it acts as an effective thermal insulator. Deterioration of vacuum may be noticed as excessively cold, wet or icy Cryostream coldhead and/or transfer line. You may also notice that the Cryostream is unable to hold a low set temperature and the gas temperature drifts slowly upwards. If you have any doubts as to whether a poor vacuum is the cause of these issues, please contact Oxford Cryosystems' support team: support@oxcryo.com, +44 (0) 1993 88 34 88.

NOTE

If you have any questions or concerns, please contact Oxford Cryosystems' support team: support@oxcryo.com, +44 (0) 1993 88 34 88

Tools Required

Pump-out Adaptor	Supplied with Cryostream
5 mm Hex (Allen) key	Supplied with Cryostream
CRH25 Re-activation Heater	Supplied with Cryostream
Vacuum pump (capable of 1x10-2 mBar ultimate pressure, 2.5 m3/hr pumping speed, e.g. Pfeiffer DUO 2.5)	Not supplied with Cryostream. Available from Oxford Cryosystems. Part Number: 22CS-VacRegKitPF
Vacuum hose	
Vacuum hose clamps, centring ring and O-rings (capable of connecting to NW16/KF16 flange on pump-out adaptor)	
Vacuum gauge	

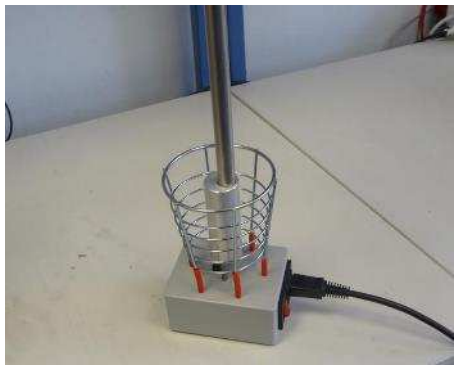
Pump-out Adaptor

CRH25 Re-activation Heater

Instructions

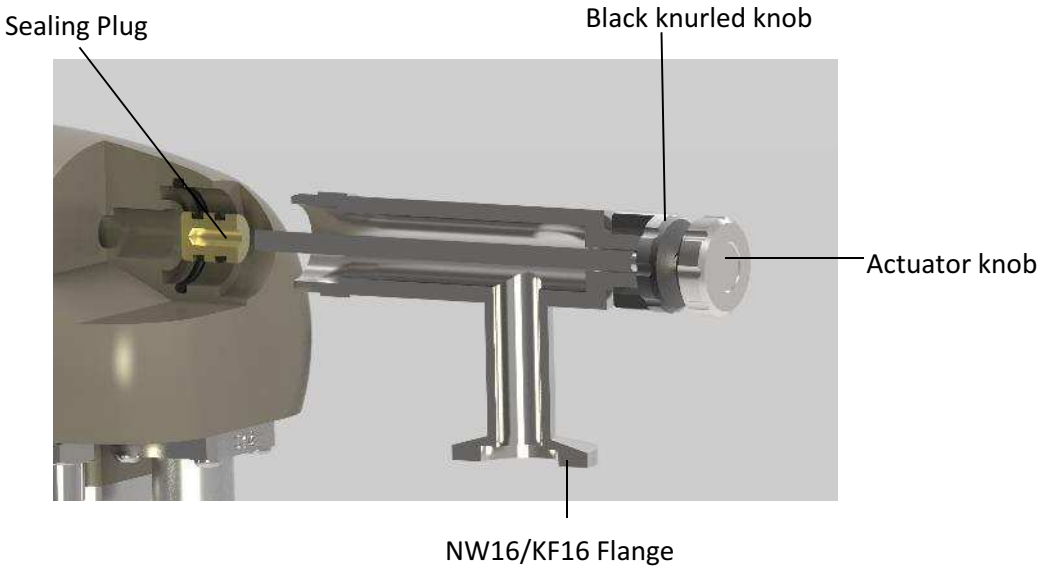
1. Switch off the Cryostream and leave it standing for 24 hours to warm internally or run a PURGE.
2. Remove the rigid section of the transfer line from the liquid nitrogen Dewar.
3. Leave the transfer line out of the liquid nitrogen Dewar for one hour to allow it to warm up, then dry it carefully.
4. Place the end of the rigid section of the transfer line into the hole of the CRH25 re-activation heater.

NOTE
Do not turn the heater on at this stage.



Transfer line in CRH25 Re-activation heater

5. Use the 5mm Hex key to remove the cap covering the pump-out port from the Cryostream coldhead.
6. Screw the pump-out adaptor clockwise into the Cryostream coldhead.



7. When the pump-out adaptor is fully screwed into the fitting, push the actuator knob into the sealing plug and screw in fully clockwise.

CAUTION

Do not withdraw the sealing plug at this stage.

NOTE

The sealing plug is a component of the Cryostream coldhead, not the pump-out adaptor; the sealing plug will already be in the Cryostream coldhead.

8. Connect your vacuum pump and gauge to the flange (NW16/KF16) on the pump-out adaptor.
9. Start the vacuum pump and ensure it is possible to obtain a vacuum pressure of at least 0.1 mBar with the sealing plug still in place to ensure there are no leaks in the vacuum equipment.

NOTE

It may be necessary to use the ballast valve on the vacuum pump to achieve a good vacuum pressure. Refer to the pump manufacturer's instructions for further details.

10. Use the actuator knob on the pump-out adaptor to slowly pull the sealing plug out of the Cryostream coldhead, exposing the Cryostream vacuum space to the vacuum pump.
11. Ensure the black knurled knob on the pump-out adaptor is tight.
12. Wait until the pressure read by the vacuum pump/gauge assembly is 0.1 mBar or lower.
13. Turn on the CRH25 re-activation heater.

WARNING

The CRH25 re-activation heater heats itself and part of the transfer line to 150-200°C. Ensure nothing will come into contact with the heater and do not touch the heater during or for some time after operation.

14. Pump the vacuum for at least 16 hours; ideally, the Cryostream should be vacuum pumped for up to 48 hours.

CAUTION

If the Cryostream is not pumped for at least 16 hours, you may find that the Cryostream does not perform well when re-installed and will require vacuum pumping again sooner than expected. At least 16 hours is required to drive off all contaminants that have been adsorbed by the cryopump in the transfer line.

15. Ensure the vacuum gauge reads 0.01 mBar (1x10-2 mBar) or better.
16. Push the sealing plug back into the Cryostream coldhead using the actuator knob.
17. Switch the CRH25 re-activation heater off immediately.
18. Unscrew the actuator knob from the sealing plug by turning it anti-clockwise (counter-clockwise).

NOTE

By pushing the knob gently into the pump-out adaptor while unscrewing it, you will hear a small click and feel the actuator knob thread leave and re-catch the sealing plug thread once it is fully out of the sealing plug.

19. Pull the actuator knob out, away from the sealing plug.
20. Turn off the vacuum pump.
21. When it is safe to do so (according to the instructions in your vacuum pump), release the vacuum from the vacuum hose.
22. Disconnect the vacuum hose from the pump-out adaptor.
23. Unscrew the pump-out adaptor from the pump-out port.
24. Re-attach the pump-out port cover.
25. Once the end of the Cryostream transfer line has cooled, remove it from the CRH25 re-activation heater; it is now safe to re-install into the liquid nitrogen Dewar.

Appendix 4 Repairs and Returns Procedure

To allow Oxford Cryosystems to offer fast and accurate technical support, please quote the Cryostream Serial Number with all technical issues. It is worth keeping a record of this number in a convenient place:

CRYOSTREAM SERIAL NUMBER
This Cryostream serial number is _____ The four digit serial number can be found etched onto the top flange of the Cryostream Coldhead.

Returns procedure

Do not return any equipment without contacting Oxford Cryosystems in advance. Use the following procedure to return ANY items for repair.

1. Contact Oxford Cryosystems (support@oxcryo.com) and obtain a Return Material Authorisation (RMA) number for the equipment which must then be written on each box being returned. Without this number Oxford Cryosystems may reject the consignment. Oxford Cryosystems will email a form which must be filled out and emailed or faxed back prior to sending the consignment.

2. Remove all traces of dangerous substances and any accessories that will be returned to Oxford Cryosystems. Drain all fluids and lubricants from the equipment and its accessories.

3. Disconnect all accessories from the equipment. Safely dispose of the filter elements removed from inside any oil mist filters.

4. Seal up all of the equipment's inlets and outlets (including those where accessories were attached). It is recommended that they are sealed with blanking flanges or heavy gauge PVC tape.

5. Seal contaminated equipment in a thick polythene bag or polythene sheet.

6. If possible, pack your equipment into its original packaging for return to Oxford Cryosystems.

7. If the equipment is large, strap the equipment and its accessories to a wooden pallet. Preferably, the pallet should be no larger than 510 mm x 915 mm (20"x 35”).

8. If the equipment is too small to be strapped to a pallet, pack it in a suitable strong carton.

9. If the equipment is contaminated, label the pallet (or carton) in accordance with laws covering the transport of dangerous substances.

Appendix 5 Liquid and Gaseous Nitrogen Safety Sheet

General

These safety points are a guideline to outline the potential hazards and procedures involved in the handling of liquid or gaseous nitrogen. Anyone handling liquid or gaseous nitrogen should first inform their departmental or laboratory safety advisor and receive advice about local safety procedures.

All users are requested to read this safety sheet before handling the Cryostream. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

General properties

- Gaseous nitrogen is colourless, odourless and tasteless and is slightly lighter than air at equal temperatures; cold nitrogen vapour is, however, denser than atmospheric air.
- Liquid nitrogen is odourless, colourless and boils at -195.8°C. One volume of liquid nitrogen gives approximately 700 volumes of gas at ambient conditions.
- Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
- Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
- Gaseous and liquid nitrogen is non-corrosive.
- Nitrogen does not support life and acts as an asphyxiant.
- Nitrogen is intrinsically non-toxic.

Fire and explosion hazards

Gaseous and liquid nitrogen are non-flammable and do not, themselves, constitute a fire or explosion risk. However, both gaseous and liquid nitrogen are normally stored under pressure and the storage vessels whether gas cylinders or liquid tanks, should not be located in areas where there is a high risk of fire or where they may normally be exposed to excessive heat.

Health hazards

Asphyxia

Nitrogen, although non-toxic, can constitute an asphyxiation hazard through the displacement of the oxygen in the atmosphere. Nitrogen gas or oxygen depletion is not detectable by the normal human senses.

Oxygen is necessary to support life and its volume concentration in the atmosphere is 21%. At normal atmospheric pressure persons may be exposed to oxygen concentrations of 18% or even less, without adverse effects. However, the response of individuals to oxygen deprivation varies appreciably. The minimum oxygen content of breathing atmospheres should be 18% by volume but to ensure a wider margin of operational safety it is recommended that persons are not exposed to atmospheres in which the oxygen concentration is, or may become, less than 20% by volume.

Symptoms of oxygen deprivation, such as increased pulse and rate of breathing, fatigue, and abnormal perceptions or responses, may be apparent at an oxygen concentration of 16%.

Permanent brain damage or death may arise from breathing atmospheres containing less than 10% oxygen. Initial symptoms will include nausea, vomiting and gasping respiration. Persons exposed to such atmospheres may be unable to help themselves or warn others of their predicament. The symptoms are an inadequate warning of the hazard.

DANGER

Breathing a pure nitrogen atmosphere will produce immediate loss of consciousness and almost immediate death.

Cold burns

Liquid and cold nitrogen vapours or gases can produce effects on the skin similar to a burn. Naked parts of the body coming into contact with un-insulated parts of equipment may also stick fast (as all available moisture is frozen) and the flesh may be torn on removal.

Frostbite

Severe or prolonged exposure to cold nitrogen vapour or gases can cause frostbite. Local pain usually gives warning of freezing but sometimes no pain is experienced. Frozen tissues are painless and appear waxy with a pallid yellowish colour. Thawing of the frozen tissues can cause intensive pain. Shock may also occur if the burns are at all extensive.

Effect of cold on lungs

Prolonged breathing of extremely cold atmospheres may damage the lungs.

Hypothermia

Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing. Hypothermia is possible in any environmental temperature below 10ºC but susceptibility depends on time, temperature and the individual. Older persons are more likely to be affected. Individuals suffering from hypothermia may find that their physical and mental reactions are adversely affected.

Precautions

Operations and maintenance

It is essential that operations involving the use of gaseous or liquid nitrogen particularly where large quantities are used are conducted in well-ventilated areas to prevent the formation of oxygen deficient atmospheres.

Ideally, nitrogen should be vented into the open air well away from areas frequented by personnel. It should never be released or vented into enclosed areas or buildings where the ventilation is inadequate. Cold nitrogen vapours are denser than air and can accumulate in low lying areas such as pits and trenches.

Where large spills of liquid nitrogen occur, a fog forms in the vicinity of the spill caused by the condensation of water vapour in the surrounding air. The fog, in addition to severely reducing visibility may contain oxygen concentrations appreciably lower than that of the air presenting a local asphyxiation hazard.

Personnel protection

Persons handling equipment in service with liquid nitrogen should wear protective face shields, loose fitting gauntlets and safety footwear.

Emergencies

In the event of an accident or emergency the instructions below should be implemented without delay.

Asphyxiation

Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing must receive immediate first aid. Medical assistance should be summoned without delay. First aid measures included inspection of the victim's airway for obstruction, artificial respiration and simultaneous administration of oxygen. These procedures should only be carried out by a trained first aid staff. The victim should be kept warm and resting.

It is important that the personnel carrying out rescue operations should minimise the risk to themselves.

Treatment of cold burns and frostbite

Cold burns should receive medical attention as quickly as possible. However, such injuries are not an everyday occurrence and doctors, hospital staff or works first aid personnel may not be aware of the basic methods of treatment. The following notes describe the first aid treatment and recommended advice for further treatment to be given by a medical practitioner or a hospital.

First Aid

In severe cases summon medical attention immediately. Flush affected areas of skin with copious quantities of tepid water to reduce freezing of tissue. Loosen any clothing that may restrict blood circulation. Move the victim to a warm place but not to a hot environment and do not apply direct heat to the affected parts. Every effort should be made to protect frozen parts from infection and further injury.

Dry, sterilised bulky dressings may be used but should not be applied so tightly that blood circulation is restricted.

Treatment by Medical Practitioner or Hospital

1. Remove any clothing that may constrict the circulation to the frozen area. Remove patient to sick bay or hospital.

2. Immediately place the part of the body exposed to the cryogenic material in a water bath which has a temperature of not less than 40°C but no more than 45°C. Never use dry hear or hot water. Temperatures in excess of 45°C will superimpose a burn upon the frozen tissue.

3. If there has been a massive exposure to the super cooled material so that the general body temperature is depressed, the patient must be re-warmed gradually. Shock may occur during re-warming, especially if this is rapid.

4. Frozen tissues are painless and appear waxy with a pallid yellowish colour. They become painful, swollen and very prone to infection when thawed. Therefore, do not re-warm rapidly if the accident occurs in the field and the patient cannot be transported to hospital immediately. Thawing may take from 15-60 minutes and should be continued until the blue, pale colour of the skin turns to pink or red. Morphine, or some potent analgesic, is required to control the pain during thawing and should be administered under professional medical supervision.

5. If the frozen part of the body has thawed by the time medical attention has been obtained, do not re-warm. Under these circumstances cover the area with dry sterile dressings with a large bulky protective covering.

6. Administer a tetanus booster after hospitalisation.

Hypothermia

Persons suspected to be suffering from hypothermia should be wrapped in blankets and moved to a warm place. Slow restoration of temperature in necessary and forms of locally applied heat should not be used. Summon medical attention.

Liquid nitrogen spillage

If large spills of liquid nitrogen spillage occur, large quantities of water should be used to increase the rate of liquid vaporisation.

Certificate of Test

CERTIFICATE No: TRA-016730-36-01A

ISSUE No: 1

COPY No: PDF

DATE: 23rd June 2014

PURPOSE OF TEST:	EMC EMISSIONS AND IMMUNITY
TRaC PROJECT ID:	TRA-016730
TEST SPECIFICATION(S):	EN61326-2-1:2013 USING THE COMMON TECHNICAL REQUIREMENTS OF EN61326-1:2013 CLASS A EMISSIONS AND INDUSTRIAL IMMUNITY
DEVIATIONS FROM SPECIFICATION(S):	NONE
EQUIPMENT UNDER TEST (EUT):	CRYOSTREAM COOLER WITH CRYODRIVE
DESCRIPTION OF EUT:	THE 800 SERIES CONTROLLER, PUMP UNIT AND CRYODRIVE ARE USED AS PART OF THE CRYOSTREAM COOLER OR COBRA SYSTEM TO OPERATED CRYOGENIC SYSTEMS. THESE CRYOGENIC SYSTEMS ARE THEN USED TO COOL CRYSTALS FOR STUDY BY X-RAYS.
MODEL / TYPE / VERSION No:	800 SERIES
BUILD LEVEL:	PROTOTYPE
EQUIPMENT SERIAL No:	CONTROLLER: 8119002 PUMP: 804002 CRYODRIVE: R76-0641
TEST RESULT:	MEASURED AS COMPLIANT when modified as described in section 21 of TRaC Global Ltd test report TRA-016730-36-00A (Measurement uncertainty as per RF109 current issue)
MANUFACTURER / AGENT:	OXFORD CRYOSYSTEMS LTD 3 BLENHEIM OFFICE PARK LOWER ROAD LONG HANBOROUGH OXFORDSHIRE OX29 8LN
ORDER No / CLIENT REFERENCE:	239804 AND 239815
DATE OF TEST:	28TH, 29TH, 30TH APRIL, 1ST, 7TH, 8TH MAY AND 3RD JUNE 2014

TESTED BY: Digitally signed by Darren Shephard
Reason: I am the author of this document — D SHEPHARD

APPROVED BY: Digitally signed by Richard Ellis
Reason: I am approving this document
Date: 2014.06.23 14:20:52 +01'00' — R ELLIS

COMMERCIAL EMC PRODUCT MANAGER, TRaC Global Ltd

The results herein relate only to the sample tested. Full results are contained in the relevant works order file.

MALVERN
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www.tracglobal.com

UKAS
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Ref. Certificate No.
GB-TRAC 0531

IEC SYSTEM FOR MUTUAL RECOGNITION OF TEST CERTIFICATES FOR ELECTRICAL EQUIPMENT (IECEE) CB SCHEME

SYSTEM CEI D'ACCEPTATION MUTUELLE DE CERTIFICATS D'ESSAIS DES EQUIPEMENTS ELECTRIQUES (IECEE) METHODE OC

CB TEST CERTIFICATE

CERTIFICAT D'ESSAI OC

Product Produit	Cryostream Gas Pump
Name and address of the Applicant Nom et adresse du demandeur	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Name and address of the manufacturer Nom et adresse du fabricant	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Name and address of the factory Nom et adresse de l'usine	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Rating and principal characteristics Valeurs nominales et caractéristiques principales	100-240Vac, 50-60Hz, 240VA
Trademark (if any) Marque de fabrique (si elle existe)	Oxford Cryosystems
Type of manufacturer's Testing Laboratories used Type de programme de laboratoire d'essais constructeur	
Model / Type Ref. Réf. de type	CSPU200 Cryostream Pump
Additional information (if necessary may also be reported on page 2) Les informations complémentaires (si nécessaire, peuvent être indiquées sur la 2ème page)	No mains cord-sets were assessed under this submission. The equipment was accepted on the basis that the installation instructions continue to state the use of only an appropriately rated and approved supply cord-set in accordance with the regulations of the country it is used in.
A sample of product was tested and found to be in conformity with IEC Un échantillon de ce produit a été essayé et été considéré conforme à la CEI	61010-1(ed.3)
National differences / Comments Les différences nationales / Commentaires	EU Group Differences, EU Special National Conditions, EU A-Deviations, CA, JP, US
As shown in the test report Ref. No. which forms part of this certificate Comme indiqué dans le rapport d'essais numéro de référence qui constitue partie de ce certificat	TRA-016723-34-00A

This CB Test Certificate is issued by the National Certification Body:

Ce Certificat d'essai OC est établi par l'Organisme National de Certification

TRaC Global Ltd.
Unit 1 Pendle Place
Skelmersdale
West Lancashire
WN8 9PN
United Kingdom

Date: 2014-10-29

Signature: Stephen Winsor S.P. Winsor

page 1 of 1

Ref. Certificate No.
GB-TRAC 0532

IEC SYSTEM FOR MUTUAL RECOGNITION OF TEST CERTIFICATES FOR ELECTRICAL EQUIPMENT (IECEE) CB SCHEME

SYSTEM CEI D'ACCEPTATION MUTUELLE DE CERTIFICATS D'ESSAIS DES EQUIPEMENTS ELECTRIQUES (IECEE) METHODE OC

CB TEST CERTIFICATE

CERTIFICAT D'ESSAI OC

Product Produit	Cryostream Controller
Name and address of the Applicant Nom et adresse du demandeur	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Name and address of the manufacturer Nom et adresse du fabricant	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Name and address of the factory Nom et adresse de l'usine	Oxford Cryosystems Ltd 3 Blenheim Office Park, Lower Road, Long Hanborough, Oxford, OX29 8LN United Kingdom
Rating and principal characteristics Valeurs nominales et caractéristiques principales	100-240Vac, 50-60Hz, 400VA (Controller)
Trademark (if any) Marque de fabrique (si elle existe)	Oxford Cryosystems
Type of manufacturer's Testing Laboratories used Type de programme de laboratoire d'essais constructeur	
Model / Type Ref. Réf. de type	800 Series Cryostream Controller
Additional information (if necessary may also be reported on page 2) Les informations complémentaires (si nécessaire, peuvent être indiquées sur la 2ème page)	No mains cord-sets were assessed under this submission. The equipment was accepted on the basis that the installation instructions continue to state the use of only an appropriately rated and approved supply cord-set in accordance with the regulations of the country it is used in.
A sample of product was tested and found to be in conformity with IEC Un échantillon de ce produit a été essayé et été considéré conforme à la CEI	61010-1(ed.3)
National differences / Comments Les différences nationales / Commentaires	EU Group Differences, EU Special National Conditions, EU A-Deviations, CA, JP, US
As shown in the test report Ref. No. which forms part of this certificate Comme indiqué dans le rapport d'essais numéro de référence qui constitue partie de ce certificat	TRA-016723-34-00A

This CB Test Certificate is issued by the National Certification Body:

TRaC Global Ltd.
Unit 1 Pendle Place
Skelmersdale
West Lancashire
WN8 9PN
United Kingdom

Ce Certificat d'essai OC est établi par l'Organisme National de Certification

Date: 2014-10-29

Signature: Stephen Winsor S.P. Winsor

page 1 of 1