



OXFORD CRYOSYSTEMS

700 Series Cryostream Cooler

Operation & Instruction Guide

700 SERIES CRYOSTREAM COOLER

Operation & Instruction Guide 5.2

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Introduction.....	4
Please Register your Cryostream Cooler!	4
How the Cryostream Cooler Works.....	5
Items Required for Assembling your Cryostream Cooler	7
Items Supplied for the Cryostream Cooler Maintenance.....	7
Getting Started.....	8
Setting Up	9
Using the Varibeam Coldhead Support Stand	9
Assembly and Positioning of the Varibeam Coldhead Support Stand	9
Mounting of the Cryostream Cooler Coldhead on the X-ray System.....	9
Connecting Up the Cryostream Cooler.....	12
The Teflon Tubing and Connecting the System Together	13
Connecting up a Dry Air Shroud Gas	14
Dewar Vessels	15
Stainless Steel Line Drier Units.....	16
Before Fitting and Using your Line Drier Unit.....	16
Fitting and Using your Line Drier Unit.....	16
Regenerating the Line Drier Unit.....	16
Running the 700 Series Cryostream Cooler.....	18
700 Series Cryostream Cooler Temperature Controller	18
Final Checks Before Switch On.....	19
How to Switch the Cryostream Controller On.....	19
Cryostream Cooler Initialisation and Screen Options	19
3 Steps to 100K.....	19
700 Series Cryostream Cooler Front Panel.....	20
Display Modes.....	21
Programming the 700 Series Cryostream Cooler	26
Using the Quick Start Facility and the COOL Function.....	26
Further Programming of the 700 Series Controller	26
Phase Table.....	30
Variable Gas Flow and the TURBO Button	30
HOLD and 'Un-HOLD'	31
Alarm Conditions	31
Safety Features During Power Failures	31
Refilling the Dewar Vessel from a Storage Vessel.....	32
How to Shut Down the Cryostream Cooler.....	32
Cryostream Cooler Shut Downs.....	33
Measuring the True Crystal Temperature.....	34
How the 700 Series Cryostream Measures the Crystal Temperature.....	34
How to Measure the Crystal Temperature at your Crystal Position.....	34
700 Series Serial Line Communications	35
Overview.....	35
Wiring	35

Serial Line Settings	35
Status Packets	35
Notes.....	36
Command Packets.....	37
Pumping Down the Vacuum Space in the Cryostream Cooler	39
Cryostream Cooler Service	41
Liquid and Gaseous Nitrogen Safety Sheet	42
General	42
General Properties.....	42
Fire and Explosion Hazards	42
Health Hazards	42
Precautions	43
Personnel Protection.....	44
Emergencies	44
Asphyxiation.....	44
Treatment of Cold Burns and Frostbite.....	44
First Aid.....	44
Treatment by Medical Practitioner or Hospital	44
Hypothermia.....	45
Liquid Nitrogen Spillage.....	45
Cryostream Cooler Troubleshooting Guide	46
Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line	46
Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes	47
Localised ice spot on the flexible line	47
Localised ice formation around the neck of the Dewar and the Cryostream Cooler leg	47
Inability to reach low temperatures	48
Inability to reach low temperatures and the base temperature begins to rise.....	48
Ice formation on the sample	48
Ice formation on outer edge of the nitrogen gas cold stream nozzle	50
Concentric formation of ice around the nozzle.....	50
Positive Gas Temperature Error	50
Cryostream Cooler Shutdown due to low flow after a few seconds of running.....	51
Gas Pump Unit is making a strange noise	51
Flow rate will not rise to 10 l/min when the TURBO button is pressed	51
EPROM fail on initialisation	52
Flashlight Test.....	53
Technical Support	54

Introduction

Welcome to the Oxford Cryosystems 700 Series Cryostream Cooler operating and instruction guide. The Cryostream Cooler is the world's leading nitrogen gas low temperature attachment for x-ray crystallography and is designed for use in the freezing of macromolecular, small molecule and powder samples during the collection of x-ray data. It can be used for all applications from shock cooling to lengthy data sets lasting many weeks.

Its versatility and flexibility means it can be fitted to practically any x-ray system including an Image Plate System, CCD Detector, Eulerian Cradle Four Circle Diffractometer, Kappa Diffractometer or Powder Diffractometer. Due to its unique design, the Cryostream Cooler operates from 80K to 400K, with a gas stability of 0.1K and a very economic liquid nitrogen consumption also low at 0.6 litres of liquid nitrogen per hour at 5 l/min of gas flow. The outstanding performance of the Cryostream Cooler has been achieved by drawing the liquid nitrogen out of an unpressurised Dewar vessel using a continuous flow gas pump.

Before using the Cryostream Cooler, please read the section, 'Liquid and Gaseous Nitrogen Safety Sheet'.

Please Register your Cryostream Cooler!

Help us to help support you when you need it. Please visit our Cryostream Cooler Registration Page at <http://www.OxfordCryosystems.co.uk/cryo/cryostream/customer/index.html> and register your Cryostream Cooler. This is very important as it allows us to track your enquiry and tie this up with the technical notes we have on your particular system.

Please fill in the fields on the form as comprehensively as possible to allow us to make contact with you.

How the Cryostream Cooler Works



The figure below illustrates the gas flow circuit of the Cryostream Cooler.

Cryostream Cooler Layout

Liquid nitrogen is drawn up by the action of the Diaphragm Pump from an unpressurised Supply Vessel, through a flexible vacuum insulated Transfer Line, into the Cryostream Cooler Coldhead. The Supply Vessel can be any convenient container of liquid nitrogen such as a conventional metal Dewar. Please note that the Dewar vessel is *not* sealed.

The liquid nitrogen, once inside the Cryostream Cooler Coldhead, passes through a heater called the Evaporator Heater, which evaporates most of the liquid into vapour at the boiling point of liquid nitrogen. This vapour then flows outward along one path of the Heat Exchanger, through the Cryostream Cooler Temperature Controller, to arrive at the inlet of the Diaphragm Pump at approximately 10K below room temperature. The nitrogen gas from the pump is then dried with a Line Drier Unit. The flow rate of the gas from the pump is then regulated by a Variable Flow Controller in the Cryostream Cooler Temperature Controller. This gas flows back into the Cryostream Cooler Coldhead where it is recooled along the second path of the Heat Exchanger.

Therefore, the main Heat Exchanger carries the gas streams to and from the constant-flow pump that operates at room temperature. The nitrogen flow rate is set to either 5 or 10 l/min by the Variable Flow Controller. After returning to the cold end of the Heat Exchanger, the gas temperature is regulated by a heater and sensor before entering the nozzle of the Cryostream. The gas then flows along the isothermal nozzle and out over the sample. The temperature indicated on the Controller is a mapped temperature for the crystal position, (see *How the 700 Series Cryostream Measures the Crystal Temperature*.)

The flexible liquid nitrogen Transfer Line, the Heat Exchanger, Evaporator Heater and delivery nozzle share a common high-vacuum insulation jacket which is pumped out and sealed before use. An Adsorption Cryopump is built into the end of the rigid part (leg) of the Transfer Line to increase the strength of the vacuum when plunged into liquid nitrogen.

The 700 Series Cryostream Controller is designed specifically to manage the Cryostream Cooler.

The Cryostream Cooler flow rate is variable and can be set to either 5 or 10 l/min of gas, this equated to approximately 0.6 and 1.2 litres / hour of liquid nitrogen. This means an average 25 litre Dewar will last for more than one day at 5 l/min and about 20 hours at 10 l/min. It is worth noting that different Dewars have different rates of boil off, so it is worthwhile taking this into consideration when running the Cryostream Cooler and leaving it unattended for long periods. Also note that below 90K, the lower flow rate of 5 l/min is slowly increase to achieve the very low temperatures.

Because the Cryostream Cooler 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 by pouring or using a mildly pressured secondary storage vessel or an automatic refilling system.

Items Required for Assembling your Cryostream Cooler

The component parts of the system are:

1. The Cryostream Cooler Coldhead and flexible transfer line (integral)- Supplied
2. 700 Series Cryostream Controller - Supplied
3. A Gas Pump Unit - Supplied
4. Interconnecting Teflon tube set (Read the Tech Note supplied with the tube set on how to fit the tubes to the system.) Including:
 - Two Long Tubes with fittings - Supplied
 - Two medium length tubes with fittings – Supplied
 - One short piece of tube with fittings - Supplied
 - One red dry air tube with right angled connector - Supplied
5. One Coldhead cable - Supplied
6. A Varibeam Coldhead Support Stand - Optional Extra
7. An AD51 Dry Air Unit- Optional Extra (or alternative dry gas supply).
8. Line Drier Units – Optional Extra
9. Dewar Vessel – Either an Oxford Cryosystems ES-60 Dewar or a suitable alternative (See later.)

Items Supplied for the Cryostream Cooler Maintenance

1. A CRH25 Reactivation Heater - Supplied
2. A Pumpout Adaptor - Supplied

Getting Started

Operating Voltage

It is essential that the Cryostream Cooler is configured to operate on the local mains electrical supply. Check the following:

Item	Operating Voltage Requirements
Cryostream Controller	Switch-mode power supply automatically selects the correct voltage and frequency, 200-240 Volts AC, 50Hz, 3 Amps 100-120 Volts AC, 50-60Hz, 6 Amps. Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted. The correct fuse is fitted as standard.
Gas Pump Unit	Three voltage variants are currently available: 220-240 Volts AC, 50 Hz 115 Volts AC, 60Hz 100 Volts AC, 50-60Hz Make sure you have the correct pump.
CRH25 Reactivation Heater	Two voltage variants are available: 230 Volts AC 115 Volts AC

Setting Up

Using the Varibeam Coldhead Support Stand

The Varibeam is an extremely robust and rigid stand that will support the Cryostream Cooler Coldhead on almost all x-ray systems. The Varibeam has a leadscrew positioner and an X-Y Positioner that allows the Cryostream Cooler nozzle to be positioned very accurately at the crystal.

The stand can be assembled in various configurations; the rotation of the horizontal arm; angling the nozzle; and the block gripping the Cryostream Cooler Coldhead can be removed and fitted onto the other side of its support plate. This stand will support and guide the Coldhead in all configurations.

Assembly and Positioning of the Varibeam Coldhead Support Stand

The Varibeam column and cross-arm are anodised aluminium to give an extremely tough finish. When the stand is being put together, try to avoid bolting the Varibeam in a position which causes obstruction where access to the crystal is important, collision with any microscopes or circles of the diffractometer, or obstruction to x-ray tube housing.

If you are unsure of exactly where you want to position the Cryostream Cooler on your system, try setting it up on a desktop first to give you a chance to consider all the options. Alternatively, use the blue Mounting Pin supplied with the support stand as a guide for aligning the real Coldhead nozzle. For determining the exact crystal position, simply place a fibre in the small hole at the end of the blue Mounting Pin, or use the Nozzle Alignment Tool (available separately).

The Varibeam is supplied with bolts to allow the user to securely fix the stand to the cabinet tabletop. Users are often reluctant to drill holes without first experimenting with various positions first. If this is so, use a G-clamp (C-clamp) to temporarily fix the Varibeam to the tabletop. If the positioning point of the Varibeam base is not close enough to the edge of the table top, try bolting the stand to a flat sheet of metal which can be clamped to the edge of a cabinet top.

In many cases bolting the Varibeam to a cabinet top can prove quite a lot of work or is just not practical, so try using tapping screws to fix the support stand in place. Make sure the cabinet top is sufficiently strong enough to prevent the tapped screws from tearing from it.

Mounting of the Cryostream Cooler Coldhead on the X-ray System

The mounting of the Cryostream Cooler Coldhead depends on the particular x-ray system being used. Oxford Cryosystems recommend the use of the Varibeam Coldhead Support Stand to mount the Cryostream Cooler Coldhead on all x-ray systems.

Note:

The following rules and instructions are guidelines only and if a user has an alternative technique for mounting the Cryostream Cooler then they are welcome to use it. If the user is in anyway unsure of the mounting of the Cryostream Cooler contact your local Cryostream Cooler supplier or Oxford Cryosystems for advice.

General Rules

There are a few general rules the user should consider when mounting the Coldhead and fixing the support stand in position.

1. Do not point the nitrogen cold stream at the detector.
2. Try to limit the amount the Cryostream nozzle infringes the path of the x-rays.
3. Do not point the cold stream at any optical device or gearing. (These devices need to be more than 15cm away).
4. Do not mount the cold stream coaxial with the goniometer head, this will cause icing problems.
5. Do not fix the stand to your cabinet top so it prevents access to your x-ray tube, prevents the detector being swung in theta or makes access to the crystal difficult.
6. The crystal should be less than 8mm from the end of the nozzle in the centre 3-4mm of the gas stream, this will help to prevent icing. The Oxford Cryosystems Nozzle Alignment Tool is idea for this application (available separately).
7. The Transfer Line is made out of flexible tubing containing a very fine continuous metal capillary. This should not be flexed to a radius of less than 200mm so **DO NOT BEND THE FLEXIBLE TRANSFER LINE TOO SHARPLY**. Obviously, the more the Transfer Line is flexed the more likely it is to fatigue.

Setting Up on Eulerian Cradle Four Circle Geometry (eg Bruker AXS P4, Stoe Stadi-4)

The Cryostream Cooler Coldhead should be mounted so that the nozzle enters the chi circle over or close to the collimator at an angle of 45°. There maybe a need to reduce the movement of the omega circle so that the goniometer does not hit the nozzle of Cryostream Cooler. The Varibeam stand should be mounted behind the x-ray housing and toward the back of the cabinet, with respect the path of the x-rays.

In the Bruker AXS cabinets, there is often a slot in the back for the cooling hose of their CDD Detector. This also makes for a very useful access point for the

Transfer Line. It is likely the Dewar vessel will need to be raised off the floor so the Transfer Line can reach the bottom of the vessel. This slot is also a very good port for the tubing and Coldhead Cable.

Setting Up on Kappa Four Circle Geometry (eg Nonius KappaCCD)

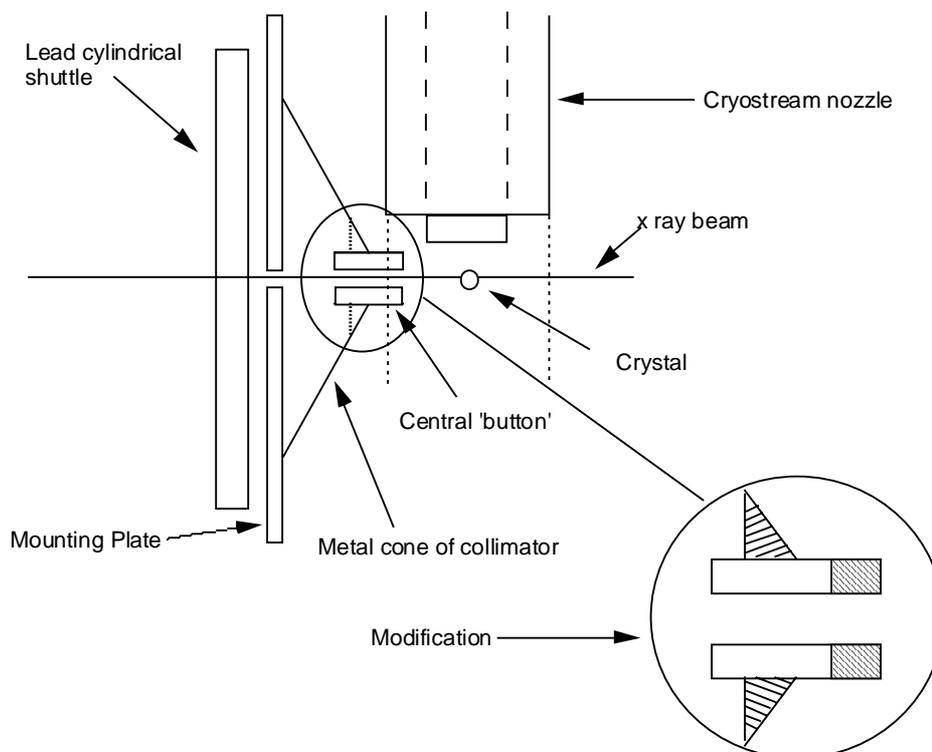
The Varibeam Support Stand should be mounted either to the left or right of the x-ray housing to reduce interference when accessing the crystal and the movement of the detector. The Cryostream Cooler Coldhead should then be mounted at about 65°. This set up will require a hole to be machined in the cabinet and it is likely the Dewar vessel will need to be raised off the floor so the Transfer Line can reach the bottom.

Setting Up on the Marresearch Image Plate Detector

There is plenty of access into the crystal on the Marresearch image plate system and although there is a temptation to mount the Cryostream Cooler vertically over the horizontal goniometer one must be careful to avoid pointing the cold stream at the CCD camera pointing up from underneath. We recommend the user tilts the Coldhead from 90° vertically to about 55° towards the goniometer housing. The position will depend on which side the user wishes to gain access to the crystal. With the 700 Series Cryostream Cooler it is possible to point the stream upward.

On the Marresearch image plate the crystal is mounted very close to the collimator and often results in the collimator infringing on the dry air shroud or cold stream of the Cryostream Cooler. This can cause icing and temperature fluctuations, so it may be necessary to modify the collimator. Do not rush into this modification.

Modification to Marresearch Image Plate Collimator



Setting Up on the Rigaku R-Axis Image Plate Detector

The Cryostream Cooler Coldhead should be mounted opposite the microscope, perpendicular to the x-ray beam path, at a fairly unrestricted angle of between 55° and horizontal (ideally about 45°). If the R-Axis is fitted with both a CCD camera

and a microscope, then one may need to be (re)moved depending on the amount of space available. If the Coldhead is any steeper than 55° the goniometer may interfere with the Laminar Flow of the cold stream and cause icing to occur. The Varibeam Coldhead Support Stand is ideal for this application.

If the R-Axis is fitted with an inverted Phi-Axis, the Cryostream Cooler can then be pointed upward at the sample to maintain the pin mount - cold stream orientation.

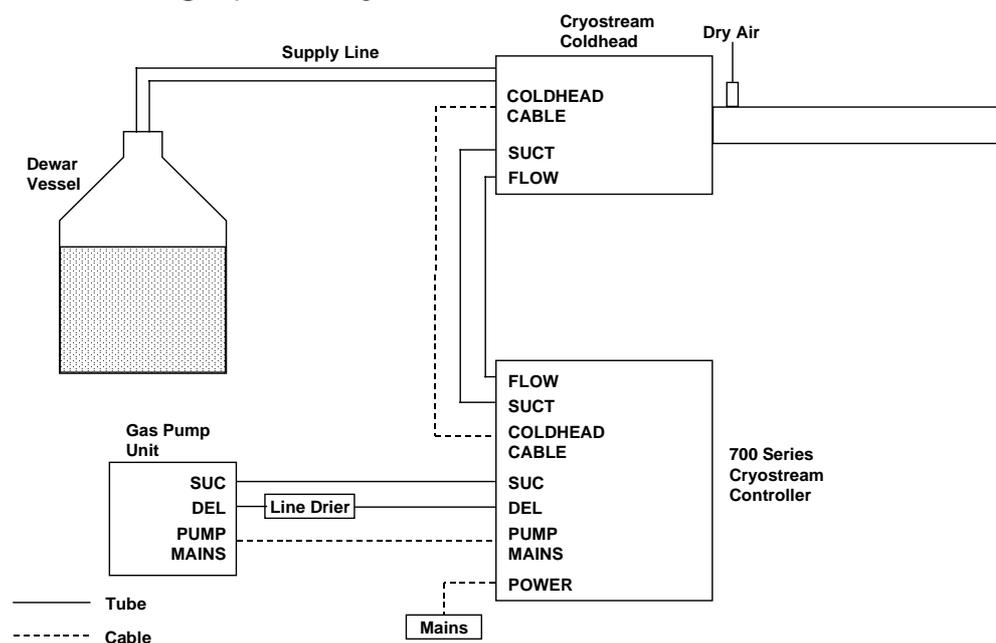
Setting Up on the Bruker SMART / APEX CCD System

The Cryostream Cooler should be mounted so the Coldhead is as close to vertical as possible. If 90° is vertical, then mount the Coldhead at about 80°. This steep angle is necessary as manufacturers continue to increase the diameter of the detector. The Varibeam stand should be mounted behind the x-ray housing and toward the back of the cabinet, with respect the path of the x-rays.

In the newer Bruker AXS cabinets, there is often a slot in the back for the cooling hose of their CDD Detector. This also makes for a very useful access point for the Transfer Line. It is likely the Dewar vessel will need to be raised off the floor so the Transfer Line can reach the bottom of the vessel. This slot is also a very good port for the tubing and Coldhead Cable.

Connecting Up the Cryostream Cooler

Tube and Cable Connection Scheme



The diagram above illustrates all the connections that have to be made, full lines denote 6mm (outside diameter) tubing and the dotted lines denote electrical cables. (The table below outlines which tubes go in which connectors). The tube set provided with the Cryostream Cooler consists of six 6mm (outside diameter) tubes:

1. Two long tubes
 - a) One with a fitting on the end marked with a BLUE collar and a 'fitting-less' end
 - b) One with a fitting on the end marked with a GREEN collar and a 'fitting-less' end
2. Two medium length tubes
 1. One marked with BLUE collars
 - a) One marked with GREEN collars
3. One short tube - Marked with GREEN collars
4. One red tube with right angled connector.

The Teflon Tubing and Connecting the System Together

The 700 Series Cryostream Cooler has been upgraded from previous 700 Series systems with new Quick-Release fittings. These new fittings are designed to be more predictable and easier to use.

In an attempted to make the Cryostream as simple to assemble as before, all the connectors have been marked at each end to match their corresponding fittings on the system.

Important Note

Before connecting the system together exactly as illustrated in the diagram on Page 12 and with the information in the table below, the system should be run without a Line Drier fitted into the circuit. This means that the tube marked Line Drier OUT will connector onto the fitting on the Pump marked DEL. Once it is time to fit the Line Drier, the tube marked Line Drier OUT it plugged onto the OUT connector on the Line Drier and the short tube with fittings is fitted between the Pump DEL connector and the Line Drier IN connector as marked.

To assist with the marking on the tubes :

Tube	Tube Marking	Fits onto:
1 (a)	Long tube with BLUE collar	SUCT Quick Release fitting on the Coldhead
1 (a)	Long tube with BLUE collar	SUCT Quick Release fitting on the Controller
1 (b)	Long tube with GREEN collar	FLOW Quick Release fitting on the Coldhead
1 (b)	Long tube with GREEN collar	FLOW Quick Release fitting on the Controller
2 (a)	Medium tube with BLUE collar	SUC Quick Release fitting on the Controller
2 (a)	Medium tube with BLUE collar	SUC Quick Release fitting on the Gas Pump Unit
2 (b)	Medium with GREEN collar	DEL Quick Release fitting on the Gas Pump Unit
2 (b)	Medium with GREEN collar	DEL Quick Release fitting on the Controller

When fitting a Line Drier Unit, firstly unplug DEL Quick Release fitting on the Controller:

2 (b)	Medium tube GREEN with Quick-Release fitting on end	DEL Quick Release fitting on the Gas Pump Unit
2 (b)	Medium tube GREEN with Quick-Release fitting on end	IN Quick Release fitting on the Line Drier
3	Short tube GREEN with Quick-Release fitting end	OUT Quick Release fitting on the Line Drier
3	Short tube GREEN with Quick-Release fitting end	DEL Quick Release fitting on the Controller

Connecting up a Dry Air Shroud Gas

The system is now set up to deliver nitrogen gas but in order to operate in an ice free environment at low temperatures, the nitrogen gas stream requires a shroud gas as it exits the nozzle. This gas can be either dry air with a dew point of less than -60°C or an inert gas such as nitrogen that is often fed in and piped around

laboratories. Oxford Cryosystems manufacture the AD51 Dry Air Unit that is a stand-alone unit that provides a constant stream of clean dry air and is ideal for this application.

Attach the red nylon tube from the tube set to the side connector on the delivery nozzle of the Cryostream Cooler Coldhead, a right angle connector is provided.

Insert the rigid leg at the end of the flexible Transfer Line into an open Dewar vessel (see next section on Dewar Vessels) containing liquid nitrogen. Always make sure that there is enough nitrogen in the Dewar vessel to cover the bottom five or six inches of the rigid Transfer Line. This is vital, as the Cryopump in the leg needs to be plunged in liquid nitrogen to insure a good vacuum.

Press some insulating material into the neck of the Dewar, (a white Teflon bung is provided with the Oxford Cryosystems ES-60 Dewar Vessel), and around the Transfer Line in order to minimise boil off and to prevent the development of ice. However, the Dewar must not be sealed! More elaborate neck fittings can be constructed, but always remember to vent the Dewar.

Dewar Vessels

The purpose of the Dewar vessel is to hold the liquid nitrogen supply used by the Cryostream Cooler. There are a number of variables to consider when deciding on which Dewar vessel to purchase for use with the Cryostream Cooler.

1. Capacity - this is the most important consideration when deciding on a Dewar.

Note

It is important to remember that the rigid Transfer Line of the Cryostream Cooler is plunged into the Dewar and is 900mm long, so it can only utilise the top 850mm of a Dewar vessel. It is possible to use a 100 litre Dewar, but if the rigid Transfer Line does not reach the bottom, the Cryostream Cooler will not utilise its full capacity.

The recommended capacity is between 30 and 60 litres. The liquid nitrogen consumption of the Cryostream does vary with flow rate so make sure the Dewar is topped up.

2. Construction – either stainless steel or aluminium (aluminum).
3. 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.

Stainless Steel Line Drier Units

Line Drier Unit



Before Fitting and Using your Line Drier Unit

When first installing your Cryostream Cooler, run the system without fitting the Line Drier. If there are any inward leaks or a high level of ice in the liquid nitrogen, the Cryostream Cooler will block in a few days but will not contaminate the Line Drier with water. If this occurs, recheck all the Quick-Release Connectors (pressing them in 16-17mm,) check the liquid nitrogen in the Dewar vessel for ice contamination then rerun the system. The Cryostream Cooler should run for about 5-7 days before a block occurs, although poor quality nitrogen can reduce this to 2-3 days. (The ALARM Lamp will flash and on pressing the ALARM button the STATUS Screen will indicate a Pressure Warning on the 700 series Cryostream Cooler.) If none of these signs appear then the quality of the liquid nitrogen is very good.

Once the system has run for about a week, fit your Line Drier.

Fitting and Using your Line Drier Unit

The Line Drier is designed to remove traces of water vapour from the nitrogen stream. It will not cope with gross contamination of the nitrogen supply or a large inward leak.

Also warm up and clean out the liquid nitrogen vessel at regular intervals, keep the vessel neck covered to prevent atmospheric moisture and 'rubbish' getting into the Dewar. Also ensure the liquid nitrogen is of good quality.

Remove the Blanking Plugs, (these plugs must be fitted to the Line Drier during transit and storage.) Fit the Line Drier Unit in the DEL gas circuit pipework. This is between the DEL connector on the Cryostream Gas Pump Unit and the DEL connector on the Cryostream Controller. Fit the Line Drier as close to the Gas Pump Unit DEL connector as possible. Do not leave the Line Drier open to the atmosphere; this will avoid contamination.

Regenerating the Line Drier Unit

Eventually the Line Drier will become saturated by water and require regenerating, this is indicated by a Pressure Warning in the Controller (see *Alarm Conditions*.) To regenerate, disconnect the Line Drier and unscrew the Metal End Fittings at each end to avoid heat damage to the rubber 'O' ring seals.

There are two important things to achieve when reactivating Line Driers:

To heat the Line Drier evenly all around its body to at least 250°C. Do not exceed 300°C. This is achieved most successfully using:

1. Heating Tape / Strip (1.5 metres, 240 watts).
2. Oven with access for the purging gas stream.

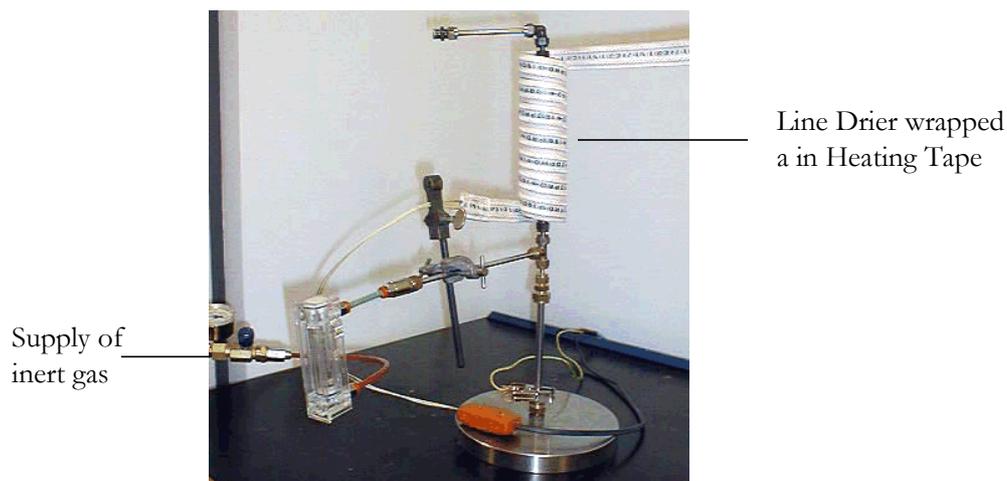
DO NOT USE:

1. Sealed oven with no gas flow through the Line Drier.
2. Heating Mantle.
3. Naked flame!!

Purge the Line Drier with a steady stream of inert cylinder gas (eg. Nitrogen, helium etc). Inject the purge gas through a short (300mm) length of stainless steel tube to act as a heat break to the OUT connector of the Line Drier, small leaks at the couplings do not matter. The OUT connector size is 1/4" BSP. The gas flow should be set to 1-2 litres/minute. DO NOT USE DRY AIR FROM THE DRY AIR UNIT. THIS GAS IS NOT DRY ENOUGH.

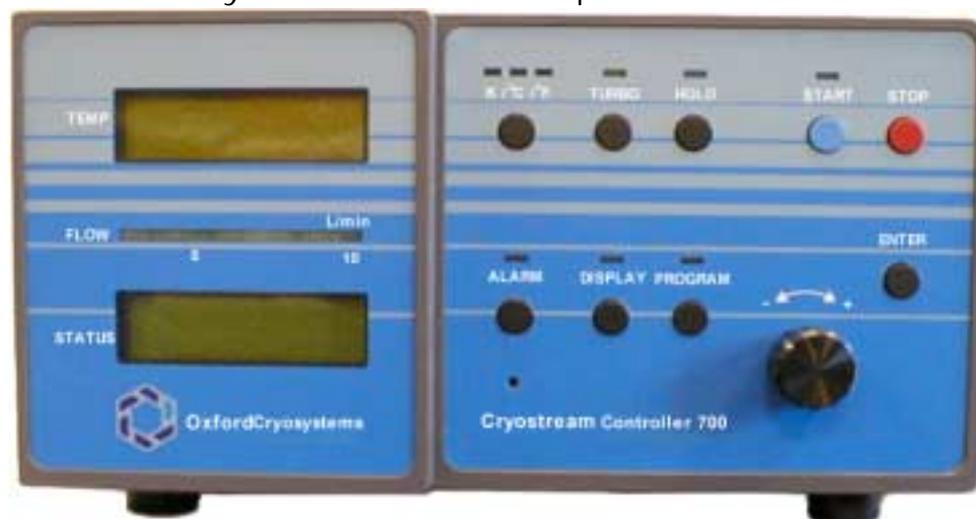
After a minimum of 4 hours switch off the supply of heat, but allow the gas flow to continue until the Line Drier is cool (approx 2 hours). Replace the Metal End Fittings and Blanking Plugs immediately.

Typical Reactivation
Set Up

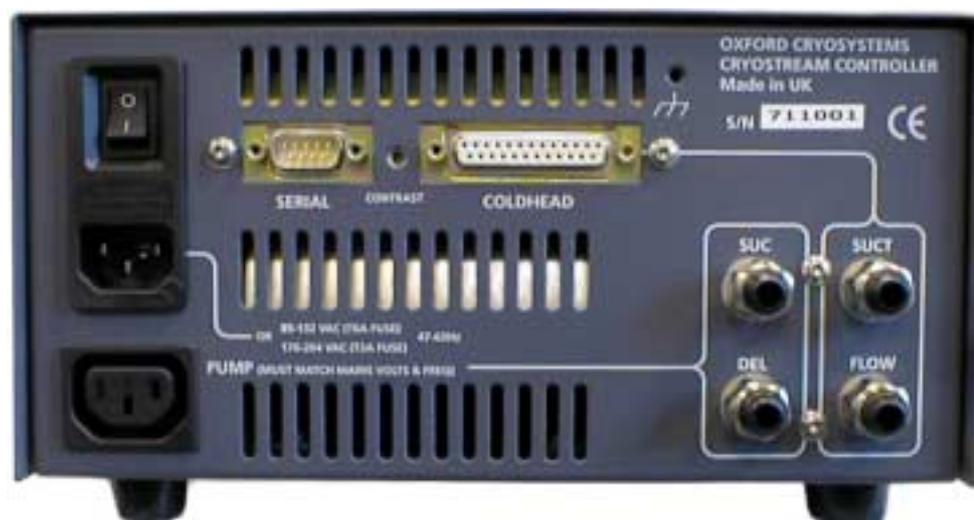


The picture above illustrates an example of the reactivation procedure used by Oxford Cryosystems.

Running the 700 Series Cryostream Cooler 700 Series Cryostream Cooler Temperature Controller



700 Series Cryostream Controller- Front View



700 Series Cryostream Controller- Back View

The 700 Series Cryostream Cooler Temperature Controller is designed to provide a completely flexible means of controlling the Cryostream Cooler. This is achieved by allowing the user to enter up to 8 phases.

Final Checks Before Switch On

Make sure:

1. All the Teflon tubes are pushed firmly into their Quick-Release Connectors. Each one should be seated 16-17mm into its connector.
2. There is liquid nitrogen in the Dewar and the Cryostream Cooler rigid Transfer Line is in the Dewar vessel.
3. That if a dry air shroud is required, it is connected to the Quick-Release connector at the top of the Cryostream nozzle.

How to Switch the Cryostream Controller On

Once the system has been connected up correctly and the Transfer Line has been placed into the liquid nitrogen Dewar vessel, press the 'ON' switch for the Cryostream Controller. This is on the top, left-hand side of the rear panel of the Controller, (right-hand side when viewing from the front of the Controller).

Cryostream Cooler Initialisation and Screen Options

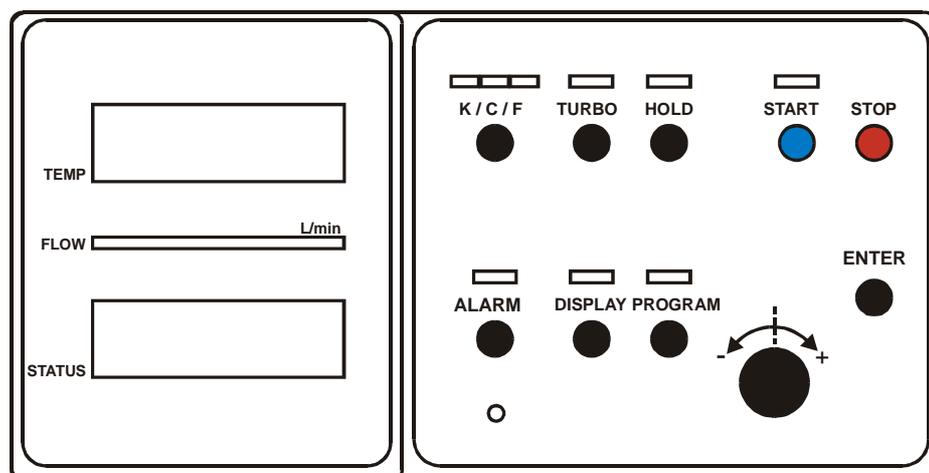
As the Controller is switched on, it undergoes an Initialisation process and a Self-Check procedure. During the Self-Check, the Cryostream Controller checks to make sure all parts of the Cryostream Cooler system are working properly. If a part of the system passes the check, a tick will appear next to it. If the part fails, a cross will appear next to it and a diagnostic message will appear.

3 Steps to 100K

1. Perform 'Final Checks Before Switch On'.
2. Read about 'How to Switch the Cryostream Cooler On'.
3. Once the system has finished initialising adjust the 'Cool to:' temperature to 100K using the IntelliKnob if necessary and press the START button.

700 Series Cryostream Cooler Front Panel

The 700 Series Cryostream Cooler Front Panel includes the following items



- TEMP screen. Whilst the Cryostream Cooler is running this screen displays the temperature of the nitrogen gas stream. During start-up and shutdown the screen is used for status messages.
- FLOW meter. The flow meter indicates the nitrogen gas flow in l/min. If no lamps are illuminated, the gas flow is zero, or less than 3 l/min. Three lamps indicates 5 l/min, all eight lamps indicates 10 l/m.
- STATUS screen. This screen displays information described in detail under the heading *Display Modes*.
- K/°C/°F button and lamps. The button allows the temperature units used by Cryostream to be switched between Kelvin, Centigrade and Fahrenheit at any time. The current choice of unit is indicated by the illuminated K/°C/°F lamp. For the purposes of this manual, temperatures are indicated in Kelvin (K).
- TURBO button and lamp. The button allows the nitrogen gas flow rate to be adjusted between a normal value (5 l/min except at temperatures below 90 K) and a 'Turbo' value (10 l/min except above 310 K). If the TURBO lamp is OFF this indicates normal flow, whereas ON indicates Turbo flow. In addition, during a COOL phase the Turbo lamp will flash. This indicates a 'Machine Turbo', in which the Cryostream Cooler fixes the flow rate at the Turbo value in order to minimise the cooling time.
- HOLD button and lamp. Pressing the HOLD button will execute a Hold (see *Programming the 700 Series Cryostream Cooler*) and illuminate the HOLD lamp. If Cryostream is already in a Hold, pressing HOLD again will release it.
- START button and lamp. The START button switches the Cryostream on, executing the start-up phase or the current Phase Table (See *Programming the 700 Series Cryostream Cooler*). This button is also used to re-start the control program after it has been halted.

- STOP button. The STOP button will immediately halt the Cryostream Cooler, turning off the pump and all the heaters. The Controller may then be safely switched off, or else re-started by pressing START.

Note

The approved method of shut down is via an END phase

- ALARM button and lamp. If an alarm condition develops (see *Alarm Conditions*), the ALARM will be illuminated and a buzzer may sound. Pressing the ALARM button will display the cause of the alarm in the STATUS screen, and will also cancel the buzzer.
- DISPLAY button and lamp. The DISPLAY button is used to toggle the Display Mode (see *Display Modes*), indicated by the corresponding lamp.
- PROGRAM button and lamp. The PROGRAM button is used to toggle Program Mode (see *Programming the 700 Series Cryostream Cooler*), indicated by the corresponding lamp.
- IntelliKnob. This knob is used to scroll the contents of the STATUS screen. In Program Mode it is also used together with the ENTER button to input information. The IntelliKnob is speed sensitive. This means the faster the Knob is turned, the greater the increment in the number and the slower the Knob is turned, the smaller the increment in the number.
- ENTER button. This button is used during Program Mode (see *Programming the 700 Series Cryostream Cooler*) to input information.

Display Modes

The STATUS screen displays a variety of information depending on the Display Mode and on whether Cryostream is Running or Idle. In each case the contents of the STATUS screen may be scrolled using the IntelliKnob.

The various situations are summarised below.

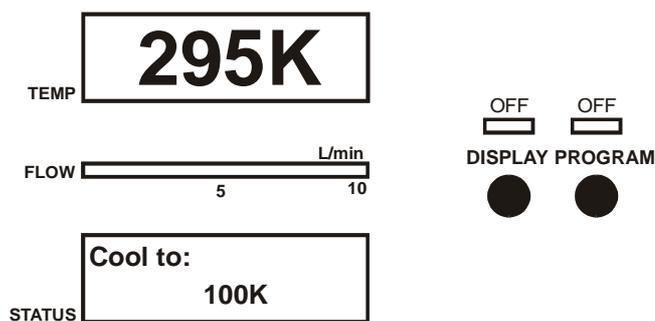
Display Mode 1

Cryostream State: **IDLE (Power on, not running)**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **OFF**

Description:



Idle Phase Table Mode

If a program has not been entered, use the IntelliKnob to adjust the temperature and press **START** to begin. The Cryostream Cooler will then enter a **COOL** function and achieve the required temperature as quickly as possible. This is done by using Machine Turbo where the system automatically increases the flow to 10 l/min (l/min) and cools the system down.

If a program has been entered, use the IntelliKnob to scroll the **STATUS** screen and press **START** to begin.

Press **DISPLAY** or **PROGRAM** to enter the modes below.

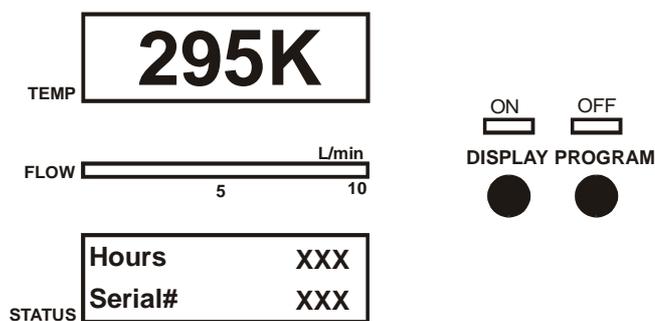
Display Mode 2

Cryostream State: **IDLE**

DISPLAY Lamp: **ON**

PROGRAM Lamp: **OFF**

Description:



Idle Phase Table

Use the IntelliKnob to scroll through the following information:

Hours – the cumulative time the system has run since manufacture.

Serial# - Controller Serial Number

Software – the version of the Controller software

Shutdown – This indicates the last reason for shutdown. Options are:

STOP – The STOP button has been pressed

END – The system has been shut down due to a programmed END

PURGE – The system has been shut down due to a programmed PURGE
(see *Further Programming on the 700 Series Controller*)

POWER – The power has been switched off at the mains

FLOW – Shutdown due to low flow
 TEMP – There has been a large temperature error
 SENSOR – This indicates ADC latches or a sensor failure
 SINK – Controller Overheating
 PSU – Power Supply Overheating

LAST ERR – this stores the last reason for the shutdown but does not include STOP or POWER.

If there has been an unexpected shutdown, the following list of items are recorded on this list after the LAST ERR:

Set T, Gas T, Gas Heat, Gas Flow, Pressure, EvapT, Heat%, SuctT, Suct Heat, Run Time.

These are recorded to allow the user to diagnose the reason for the shutdown and are stored until there is another erroneous shutdown.

Press DISPLAY or PROGRAM to alter the Display Mode.

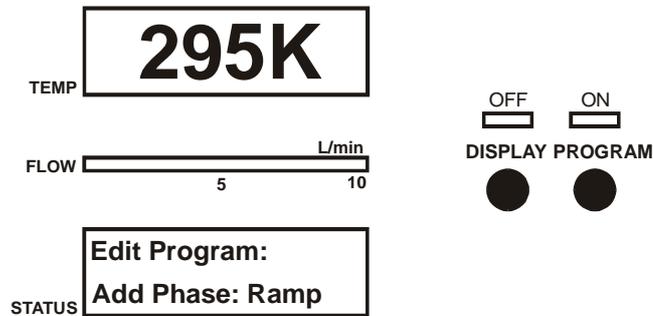
Display Mode 3

Cryostream State: **IDLE**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **ON**

Description:



Program Mode

This mode allows the user to program the Cryostream as described in *Programming the 700 Series Cryostream Cooler*. The list of phases also give you the option to save or load a program. ‘Save Program’ will save the current program and ‘Load Program’ will load the last saved program.

Press DISPLAY or PROGRAM to alter the Display Mode.

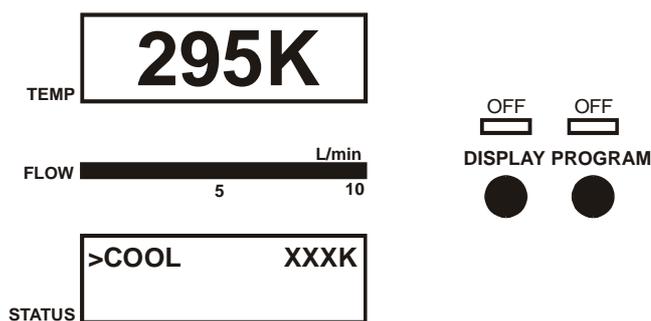
Display Mode 4

Cryostream State: **RUNNING**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **OFF**

Description:



Phase Table Mode

The Cryostream Cooler lists the phases in the current program, with the current phase at the top of the list. The current phase is indicated with a '>'. Use the IntelliKnob to scroll through the list.

Press DISPLAY or PROGRAM to enter the Display Modes 5 & 6.

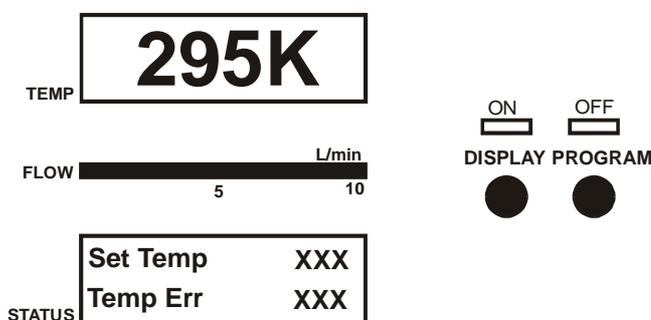
Display Mode 5

Cryostream State: **RUNNING**

DISPLAY Lamp: **ON**

PROGRAM Lamp: **OFF**

Description:



Running Display Mode

Use the IntelliKnob to scroll through the following information:

Set Temp - Temperature

Temp Error - Temperature

Run Time – How long system has been running since START was last pressed.

Flow Rate – gas flow in l/min

Pressure - in bar.

Gas Heat – %. The instantaneous value is given and the average is in brackets.

EvapT - Temperature

Evap Heat – %. The instantaneous value is given and the average is in brackets.

Evap Shift – Is the amount, in %, the Evap heater has shifted from its ideal operating rate and is used to compensate and then indicate a failing vacuum.

Suct Heat – On or Off

Suct Set Point – Temperature

Suct Temp - Temperature

Press DISPLAY or PROGRAM to alter the Display Mode.

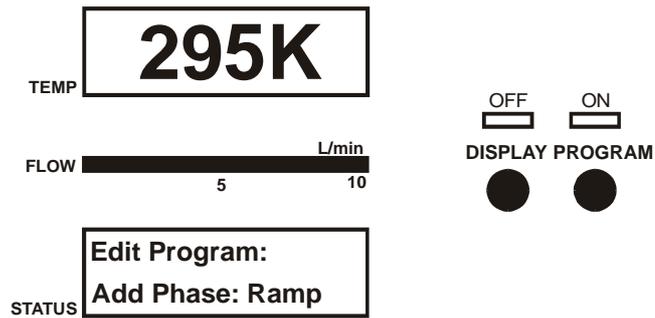
Display Mode 6

Cryostream State: **RUNNING**

DISPLAY Lamp: **OFF**

PROGRAM Lamp: **ON**

Description:



Program Mode

This mode allows the user to program the Cryostream Cooler as described in *Programming the 700 Series Cryostream Cooler*. New phases are added at the end of the list of phases. If the system is in a HOLD, press HOLD to begin the next phase.

Press DISPLAY or PROGRAM to alter the Display Mode.

Programming the 700 Series Cryostream Cooler

Switch the Cryostream Controller on according to the instructions above and wait for the system to initialise.

Using the Quick Start Facility and the COOL Function

To cool as quickly as possible simply rotate the IntelliKnob to the appropriate temperature and press START. The Cryostream will remember the last value requested here and store it for the next time the Quick Start facility is used.

Note

The system uses the COOL function to get cold as quickly as possible. The COOL function uses a flow rate of 10 l/min to draw the maximum amount of liquid out of the Dewar. This is called Machine Turbo and is indicated by the TURBO lamp flashing on and off. Once the system is cold the flow rate will drop to 5 l/min above 90K. To maintain 10 l/min, press the TURBO button so that lamp remains on permanently.

Once the Cryostream reaches the desired temperature, the Controller will automatically enter a HOLD in the Phase Table.

With the system now running, press DISPLAY to view *Display Mode 5* discussed above. This displays all the parameters of the system. Alternatively, press PROGRAM to enter *Display Mode 6* and add additional phases to your program (see *List of Phases* and *Further Programming of the 700 Series Controller*).

Further Programming of the 700 Series Controller

While the system is idle or running, it is possible to program more detailed phases.

Press PROGRAM (this illuminates the PROGRAM lamp). The STATUS screen then displays the following:

Edit Program: Add Phase: Ramp
--

Spin the IntelliKnob to see all the phases. Press ENTER at any time to accept a particular phase.

Here is a list of the possible phases and other parameters that each one requires. For an explanation of each phase, see *List of Phases*.

Phase and Description

STATUS Screen Modes

Phase: RAMP

Description

Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the Cryostream Cooler to follow, the Controller will automatically enter the RAMP / WAIT mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5K of the gas temperature.

The Ramp Rate may be anything between 1 and 360K / hr

**Edit Program:
Add Phase: Ramp**

**Ramp Rate:
120K/hr**

**Final Temp:
100K**

Phase: COOL

Description

COOL is designed to get the system as cold as quickly as possible. It uses a flow rate of 10 l/min to draw the maximum amount of liquid out of the Dewar. This is called Machine Turbo and is indicated by the TURBO lamp flashing on and off. Once the system is cold the flow rate will drop to 5 l/min above 90K. To maintain 10 l/min, press the TURBO button so that the lamp remains on permanently.

It is not possible to spin the IntelliKnob above the end temperature of the previous phase or the current gas temperature.

**Edit Program:
Add Phase: COOL**

**Cool to:
100K**

Phase: PLAT

Description

Maintain temperature fixed for a certain time. The user is prompted to enter a temperature at which to plateau and to specify a time to plateau.

Below 10hr 00min the PLAT function will start to count down in seconds and this will be displayed in the STATUS screen during running.

Edit Program:

Add Phase: PLAT

Plat Length:

1:00 (hh:mm)

Phase: HOLD

Description

Maintain temperature fixed indefinitely until the START button is pressed (a programmed HOLD should not be confused with the HOLD button).

Edit Program:

Add Phase: HOLD

Phase: PURGE

Description

This function is designed to warm up the Coldhead as quickly as possible. It applies maximum power to the three heaters in the Coldhead to get to 300K as quickly as possible. The PURGE is replaced by a SOAKING as the heaters run for a further 10 minutes.

Edit Program:
Add Phase: PURGE

Phase: END

Description

System shutdown. You are asked to enter a Ramp Rate back to a final temperature of 300K and then the system is shut down. This is the controlled way to finish an experiment and should be used whenever possible.

Edit Program:
Add Phase: End

Ramp Rate:
120K/hr

Once an END function has been programmed, it is not possible to enter any more phases. The only options available are to load or save a program or delete the last phase.

Delete Phase

Description

To delete a phase at any time, in Program Mode, spin the IntelliKnob to 'Delete Phase' and press ENTER. This will delete the last phase entered. If the system happens to be executing this last phase, the phase will be replaced by a HOLD.

Edit Program:
Delete Phase

Load Program / Save Program

Description

While the system is idle, it is possible to load or save a program. Loading a Program simply loads the last saved program.

Edit Program:
Load Program

Edit Program:
Save Program

Note

Turning the IntelliKnob also offers the option to load a program or save the current program. This is only possible when the system is idle.

Press ENTER on completing each screen. To cancel programming at any time, press PROGRAM or Display, (the PROGRAM lamp will go out).

Once the phases have been entered, press START to begin the first phase in the Phase Table. The Controller will automatically enter a HOLD phase at the end of the program if one has not already been programmed. If the system is running and already in a HOLD phase, pressing the HOLD button will pass to the next instruction in the Phase Table.

Phase Table

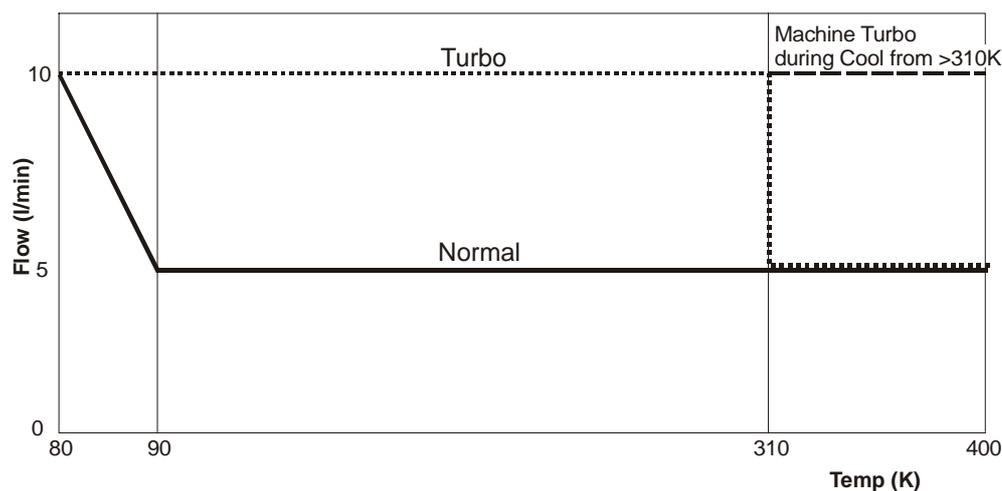
Press PROGRAM at any time during running to look at the Phase Table and enter more phases. This will enter *Display Mode 6* but will not give the option of loading or saving a program while the system is running.

If there is a list of phases longer than the screen in the Phase Table, this will be indicated by small characters on the left of the STATUS screen pointing up or down.

↑ ↓	This indicates it is possible to scroll up and down through the phases using the IntelliKnob.
↓	This indicates it is possible to only scroll down through the phases using the IntelliKnob.
↑ └	This indicates that the end of the Phase Table has been reached and it is only possible to scroll up through the phases using the IntelliKnob.
T	This indicates that the top of the Phase Table has been reached.

Variable Gas Flow and the TURBO Button

Cryostream offers two gas flow settings: 'Normal' and 'Turbo', selected using the TURBO button and indicated by the TURBO lamp. For temperatures between 90 and 310K these correspond to flow rates of 5 and 10 l/min respectively. Below 90K the Normal flow rate is steadily increased from 5 l/min at 90 K to 10 l/min at 80K. Above 310K the Turbo flow rate is 5 l/min – identical to the Normal flow. This is illustrated in the diagram below.



In addition to the Normal and Turbo settings, Cryostream enforces a 'Machine Turbo' during COOL phases. This corresponds to a flow rate of 10 l/min and is indicated by a flashing TURBO lamp.

HOLD and 'Un-HOLD'

A program can be paused at any time using the HOLD button, this will illuminate the HOLD lamp. To continue the program simply press the HOLD button at any time and the HOLD lamp will go out.

It is also possible to release the HOLD phase by pressing the START button.

Alarm Conditions

An Alarm Condition is indicated by an illuminated ALARM lamp. There are two reasons why the ALARM lamp will light up:

1. The ALARM lamp will flash slowly to indicate potential problems with the system. It is possible to see the cause of the potential problem by pressing the ALARM button. This allows you to take the appropriate action. Possible causes are:
 - a) Pressure Warning
 - b) Temp Error Warning – this indicates there is a large temperature error and when the system reaches an error of 25K the system will shut down.
 - c) Poor Vacuum – this indicates that the system has detected a failing vacuum and works in conjunction with the 'Evap Shift' parameter.
2. If the system shuts down, the ALARM lamp will flash quickly and the buzzer will sound. See the diagnostics by pressing DISPLAY and report these to Oxford Cryosystems.

Safety Features During Power Failures

The 700 Series Cryostream Controller is designed to protect itself and the sample during power interruptions.

It is possible for the Controller to maintain gas flow and not reset the Controller during electrical interruption indicated by the term 'Brown-Out' of between 0-2 secs. If a 'Brown-Out' is detected, this is indicated on the screen.

Refilling the Dewar Vessel from a Storage Vessel

Although it is possible to simply refill the Cryostream Dewar by pouring liquid nitrogen directly from a secondary Dewar, it is recommended that a self-pressurised storage vessel be used where possible.

The Cryostream Cooler Dewar Vessel can be refilled at any time. Although the Cryostream Cooler operates at atmospheric pressure, it is important that the liquid nitrogen is delivered into the Cryostream Cooler Dewar Vessel correctly.

1. Make sure the pressure in the Storage Tank being used to refill the Cryostream Cooler Dewar vessel is no greater than 2 bar.
2. The Transfer Line from the Storage Tank to the Cryostream Cooler Dewar vessel is less than 2 metres long.
3. Vent the Transfer Line to begin with to avoid spraying warm air from the Transfer Line in the Cryostream Cooler Dewar vessel. This will evaporate the liquid from the Dewar.
4. The end of the Transfer Line should not be submerged into the liquid nitrogen in the Cryostream Cooler Dewar vessel.
5. Any large storage vessel should be grounded (earthed) before using it to top up the Cryostream Dewar Vessel. This avoids any large static discharges from the vessel in to the Cryostream Controller.

How to Shut Down the Cryostream Cooler

To shut down the Cryostream Cooler correctly, the user should program in an END phase into the Phase Table. The Cryostream will ramp to 300K and will then shut down. This will allow the nozzle to become warm, and thus help prevent moisture from the air migrating up the nozzle.

In the case of an emergency, press the red STOP button. This can also, if required, be used if the gas temperature is close to room temperature, although a programmed END maybe more convenient.

Once the Cryostream Cooler has shut it can simply be reprogrammed to start the system up again. **IT IS NOT NECESSARY TO SWITCH THE SYSTEM OFF.** Simply press START to re-initialise the system.

After the Cryostream Cooler has been switched off it is advisable to leave the liquid nitrogen leg in the LN2 Dewar (e.g. until the next day) so that the Cryopump can maintain a good vacuum while the Heat Exchanger warms up gradually. This will prevent condensation or ice forming on the grey Coldhead which may cause nuisance to surrounding equipment.

Cryostream Cooler Shut Downs

The 700 Series Cryostream Controller has been designed to fully protect the Cryostream Cooler under its normal mode of operation as outlined in this manual. The Controller will shut down the Cryostream Cooler if:

1. The Gas Temp or registers an error greater than ± 25 K.
2. Gas flow drops below 2 litres/minute.
3. The Controller or power supply overheat.
4. A temperature sensor (Gas temperature, Evaporator, or SUCT temperature) fault occurs.
5. External electrical noise (from a rotating anode for example) is too great to allow accurate temperature readings to be taken.

In each case the run time variables are saved (see *Display Mode 3*) and a suitable error message is displayed.

In the case of an unexpected shut down, record the reason for the shutdown as indicated on the STATUS screen and press START to have the option to reprogram it. Pressing START will again, reinitialise the Controller. To see a full list of diagnostics at the time of shut down, press DISPLAY which will take you into *Display Mode 2*. Use the IntelliKnob to scroll through the 'Last Shutdown Diagnostics' listed after the 'Last Err' entry.

If the shutdown is due to (2) then the Controller will automatically enter and execute a PURGE phase.

Measuring the True Crystal Temperature

How the 700 Series Cryostream Measures the Crystal Temperature

In contrast to other cooling systems and indeed previous versions of the Cryostream Cooler, the 700 Series Cryostream displays the gas temperature at the position of the crystal, rather than a point within the Coldhead. Oxford Cryosystems has established a method of mapping the true temperature at 5mm from the end of the nozzle. Because of the superior laminar flow of the 700 Series gas stream, one can place the sample up to 10mm from the end of the nozzle.

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 genuine change in the crystal temperature. The Cryostream Cooler will compensate for this change, but nevertheless unnecessary changes in the flow rate should be avoided if small changes in gas temperature at the crystal are not desirable.

How to Measure the Crystal Temperature at your Crystal Position

If it is important to know the actual temperature at the position of your crystal for an experiment, you can always check it. However, we do not recommend measuring the temperature with a thermocouple placed in the stream. In the heterogeneous environment of a narrow cold gas stream, there are several factors that lead to spurious voltages on the thermocouple, creating errors in apparent temperature of possibly tens of degrees! For instance, conduction of heat down the wires creates a heat leak. At the point of entry of the thermocouple wires into the stream a cold junction is formed whose temperature is much lower than the room temperature assumed by the Controller, thus making the measured temperature *appear* to be much higher than indicated. Also, the sharp temperature change at the interface between the cold stream and the surrounding warm air can induce stresses into the thermocouple wires and then generate spurious EMF.'s. We believe that the only satisfactory way to find the error in absolute temperature at the crystal position is to calibrate with a sample that undergoes a known phase transition or change of state. For instance, we have found that the low temperature phase transition in the langbeinite $(\text{NH}_4)_2\text{Cd}_2(\text{SO}_4)_3$ was observed from intensity measurements to be in the range 88-89K (established elsewhere to be at 88K). Similarly, lattice parameter measurements of sodium ammonium tartrate tetrahydrate (Rochelle Salt) gave a transition temperature in agreement to within 0.5K of the published value of 109K. A most useful compilation of transitions in hundreds of crystals has been published by P. Tomaszewski (*Phase Transitions*, **38**, 127).

700 Series Serial Line Communications

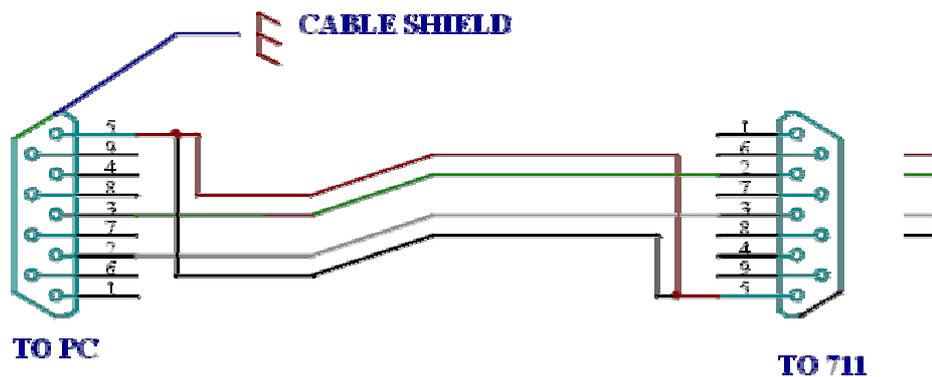
Overview

The 700 series controller incorporates an RS232 interface allowing communication with a remote computer. The nature of these communications is a good deal simpler than that in earlier Cryostreams. The communications fall into two categories.

- Cryostream issues 'status packets' at regular (1 second) intervals. This does not require any 'status request' command as in previous versions of the controller. The status packets are of a fixed length and format detailed below.
- Cryostream listens for 'command packets' sent by the remote computer. The available commands for version 1.0 are listed in detail below. There is no 'acknowledge' or 'complete' packet, instead the remote computer must monitor the status packets.

Wiring

700 series controllers are shipped with a suitable connecting wire, the details of which are illustrated here.



Serial Line Settings

700 series controllers use the following R232 settings: 9600 baud, no parity, 1 stop bit.

Status Packets

As indicated above, the controller issues status packets of a fixed length at 1 second intervals. The format of the status packet is given below.

```
typedef struct {
    unsigned char Length; /* Length of this packet = 32 (bytes) */
    unsigned char Type; /* Status Packet ID = 1 */
    unsigned short GasSetPoint; /* Set Temp 100*K */
    unsigned short GasTemp; /* Gas Temp 100*K */
    signed short GasError; /* Error 100*K */
    unsigned char RunMode; /* The current 'run mode' */
    unsigned char PhaseId; /* theState.PhaseTable[0].Id */
    unsigned short RampRate; /* theState.PhaseTable[0].Temp */
    unsigned short TargetTemp; /* theState.PhaseTable[0].Temp */
    unsigned short EvapTemp; /* Evap temp, 100*K */
    unsigned short SuctTemp; /* Suct temp, 100*K */
    unsigned short Remaining; /* Time remaining in phase */
    unsigned char GasFlow; /* Gas flow, 10*l/min */
    unsigned char GasHeat; /* Gas heater, % */
}
```

```

    unsigned char EvapHeat; /* Evap heater, % */
    unsigned char SuctHeat; /* Suct heater, % */
    unsigned char LinePressure; /* Back pressure, 100*bar */
    unsigned char AlarmCode; /* Indicates most serious alarm condition */
    unsigned short RunTime; /* Time in minutes pump has been up */
    unsigned short ControllerNumber; /* Controller number, from ROM */
    unsigned char SoftwareVersion; /* Software version */
    unsigned char EvapAdjust; /* EvapAdjust vacuum compensation */
} CryostreamStatus ;

```

Notes

- chars have a size of 1 byte, shorts have a size of 2 bytes.
- All temperatures are in centi-Kelvin, i.e. 80 K is reported as 8000.
- The RunMode member make take the following values:

```

enum {
    StartUp, /* = 0: Initial transient value - run through system checks */
    StartUpFail, /* = 1: Some failure in system checks - leave results on
screen */
    StartUpOK, /* = 2: System checks OK - awaiting Start button */
    Run, /* = 3: Gas is flowing */
    SetUp, /* = 4: Special commissioning mode */
    ShutdownOK, /* = 5: System has shut down cleanly */
    ShutdownFail /* = 6: System has shut down due to hardware error */
};

```

The PhaseId member may take the following values, whose meaning should be obvious from the manual. This parameter is meaningless unless iRunMode = Run. Parameters of the current phase are stored in the RampRate, TargetTemp and Remaining members.

```

enum {
    Ramp, /* = 0: Current phase is a Ramp */
    Cool, /* = 1: Current phase is a Cool */
    Plat, /* = 2: Current phase is a Plat */
    Hold, /* = 3: Current phase is a Hold */
    End, /* = 4: Current phase is an End */
    Purge, /* = 5: Current phase is a Purge */
    DeletePhase, /* = 6: Internal use only */
    LoadProgram, /* = 7: Internal use only */
    SaveProgram, /* = 8: Internal use only */
    Soak, /* = 9: Part of the Purge phase */
    Wait /* = 10: Part of Ramp/Wait */
};

```

The AlarmCode member make take the following values.

```

enum {
    AlarmConditionNone, /* = 0: No alarms exist */
    AlarmConditionStopPressed, /* = 1: Stop button has been pressed */
    AlarmConditionStopCommand, /* = 2: Stop command received */
    AlarmConditionEnd, /* = 3: End phase complete */
    AlarmConditionPurge, /* = 4: Purge phase complete */
    AlarmConditionTempWarning, /* = 5: Temp error > 5 K */
    AlarmConditionHighPressure, /* = 6: Back pressure > 0.5 bar */
    AlarmConditionVacuum, /* = 7: Evaporator reduction at max */
    AlarmConditionStartUpFail, /* = 8: Self-check fail */
    AlarmConditionLowFlow, /* = 9: Gas flow < 2 l/min */
    AlarmConditionTempFail, /* = 10: Temp error > 25 K */
    AlarmConditionTempReadingError, /* = 11: Unphysical temp. reported */
    AlarmConditionSensorFail, /* = 12: Invalid ADC reading */
    AlarmConditionBrownOut, /* = 13: Degradation of power supply */
    AlarmConditionHeatsinkOverheat, /* = 14: Heat sink overheating */
    AlarmConditionPsuOverheat, /* = 15: Power supply overheating */
    AlarmConditionPowerLoss /* = 16: Power failure */
};

```

Command Packets

Command packets are small, variable length packets sent by the remote computer to Cryostream. If the packet contains a valid command, then Cryostream will immediately act upon that command, potentially over-writing any existing command and starting the gas flow if necessary. There is no need for Cryostream to be started manually or put into SLAVE mode as was the case in earlier versions. In order to determine whether a command has been received and acted upon the remote computer must monitor the information contained in the status packets - there is no 'command acknowledge' response. Likewise, the status packets should be monitored to determine when the command is complete, because Cryostream no longer issues a 'command complete' packet.

The structure of a valid command packet is as follows:

```
char Size; /* The variable size of the command packet */
char Id; /* The command packet identifier */
char *Params; /* Possible variable number of parameters */
```

The Size parameter must reflect the size of the command packet, as listed below. As of version 1.0 the Id parameter must be one of the following values:

```
CSCOMMAND_RESTART=10, /* Restart a Cryostream which has shutdown */
CSCOMMAND_RAMP=11, /* Ramp command identifier - parameters follow */
CSCOMMAND_PLAT=12, /* Plat command identifier - parameter follows */
CSCOMMAND_HOLD=13, /* Hold command identifier - enter programmed Hold
*/
CSCOMMAND_COOL=14, /* Cool command identifier - parameter follows */
CSCOMMAND_END=15, /* End command identifier - parameter follows */
CSCOMMAND_PURGE=16, /* Purge command identifier */
CSCOMMAND_PAUSE=17, /* Pause command identifier - enter temporary
Hold */
CSCOMMAND_RESUME=18, /* Resume command identifier - exit temporary
Hold */
CSCOMMAND_STOP=19, /* Stop command identifier */
CSCOMMAND_TURBO=20, /* Turbo command identifier - parameter follows */
```

The Params[] array contains the parameters associated with the particular command. In most of the above cases no parameters are required, and thus the command packet is a simple two-byte one in which Size=2. For example, a command packet to stop the Cryostream would be created as follows:

```
char buf[2] = { 2, CSCOMMAND_STOP }; /* Create a Stop command packet
*/
```

For those commands requiring parameters, the Params[] array takes various forms, illustrated by the following examples.

```
/*
 * The CSCOMMAND_TURBO command packet, size = 3
 * The Params[] array consists of a single char taking the value
either 0
 * (switch Turbo off) or 1 (switch Turbo on)
 */
char buf[3] = { 3, CSCOMMAND_TURBO, 1 }; /* Switches Turbo on */
```

The Ramp, Plat, Cool and End commands are a little more complicated, because the parameters which are passed are shorts - 16 bit integers. These need to be assembled as illustrated in the following examples, which use the standard HIBYTE and LOBYTE macros reproduced below.

```
/*
 * The CSCOMMAND_PLAT command packet, size = 4
```

```

    * The Params[] array consists of a short containing the duration of
    the
    * Plat in minutues
    */
char buf[4] = { 4, CSCOMMAND_PLAT, HIBYTE(720), LOBYTE(720) }; /* 720
minute plateau */
/*
    * The CSCOMMAND_COOL command packet, size = 4
    * The Params[] array consists of a short containing the end
    temperature in centi-Kelvin
    */
char buf[4] = { 4, CSCOMMAND_COOL, HIBYTE(9000), LOBYTE(9000) }; /*
Cool to 90 K */
/*
    * The CSCOMMAND_END command packet, size = 4
    * The Params[] array consists of a short containing desired ramp
    rate in K/hour
    */
char buf[4] = { 4, CSCOMMAND_END, HIBYTE(360), LOBYTE(360) }; /* End
rate = 360 K/hour */
/*
    * The CSCOMMAND_RAMP command packet, size = 6
    * The Params[] array consists of a short containing desired ramp
    rate in K/hour,
    * followed by a short containing the end temperature in centi-Kelvin
    */
char buf[6] = { 6, CSCOMMAND_RAMP, HIBYTE(120), LOBYTE(120), /* Rate
= 120 K/hour */ HIBYTE(25050), LOBYTE(25050), /* Final temp = 250.5 K
*/ };
HIBYTE

#define HIBYTE(w) (((unsigned char)(((unsigned short)(w) >> 8) &
0xFF))

LOBYTE

#define LOBYTE(w) ((unsigned char)(w))

```

Pumping Down the Vacuum Space in the Cryostream Cooler

From time to time you may need to repump the vacuum insulation space of the Cryostream Cooler Coldhead. This will be apparent when the outside of the Coldhead and Transfer Line becomes excessively cold or wet all over during operation or the Cryostream Cooler is unable to reach the required gas temperature and the Heat% value is zero.

Do not be misled by similar symptoms that are not due to a vacuum problem. For instance, localised condensation or icing of the SUCT line is not likely to be caused by poor vacuum. Similarly, if the liquid nitrogen supply runs low or when the nitrogen leg is removed from the storage vessel the operation of the built-in cryopump is defeated and the Coldhead may become cold or damp externally.

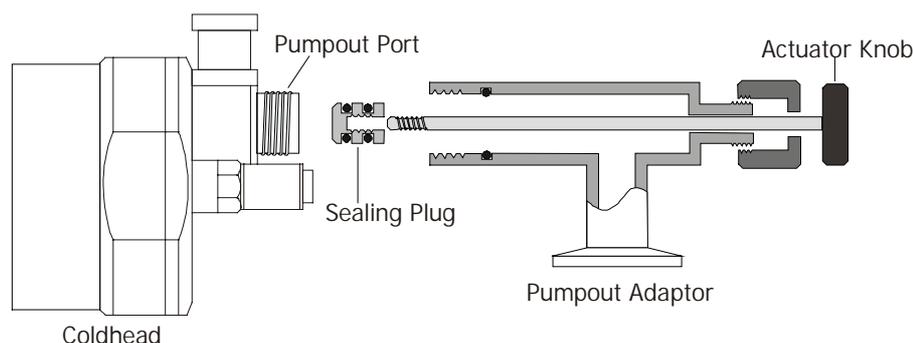
It has been established that the Cryostream Cooler vacuum can be repumped using a good rotary vacuum pump (with air ballast valve), a Pirani gauge and the CRH25 Reactivation Heater. It is not necessary to use a diffusion pump or turbomolecular pump – in fact, backstreaming of diffusion pump fluid could well contaminate the coldhead.

The correct procedure is as follows:

1. Determine that the unit really does require repumping as described above (see Troubleshooting Guide or contact Oxford Cryosystems or your local agent if you are unsure.)
2. Switch the Cryostream Cooler off and leave it standing for 24 hours. This will allow it to warm up internally.
3. Take the rigid section of the transfer line out of the storage vessel. Leave the rigid leg to warm up for an hour and then dry it carefully.
4. Place the end of the nitrogen leg into the hole in the hot block of the CRH25 heater. Ensure that nothing else can come into contact with the heater to cause damage. Do not switch the heater on at this stage.
5. Connect a good rotary pump (preferably 2-stage) to the Cryostream Cooler Pump-out Port using the Pumping Adaptor (see picture below) supplied. Screw the adaptor onto the Pumpout Port positioned at the top of the rigid leg of the transfer line.

Do not withdraw the sealing plug at this stage.

Attaching the Pumping Adaptor



6. Start the rotary pump and ensure that a pressure of 0.1 mbar can be obtained up to the pumping adaptor. It may be necessary to run the pump with its air ballast valve open for about 30 minutes.
7. When the rotary pump pressure is 0.1mbar, use the Pumping Adaptor Actuator Knob to locate and withdraw the Sealing Plug very slowly to avoid a rush of gas. An extra valve in the pumping line would help in this operation.
8. Wait until the pressure falls towards 0.1mbar - this may take 60 minutes. If necessary, use the rotary pump air ballast again. If you cannot obtain a sufficiently low pressure at this stage you may have a leak (or bad pump!) which should be investigated. Contact your Cryostream Cooler supplier if all else fails.
9. When a pressure near to 0.1mbar is obtained, the CRH25 reactivation heater can be switched on. The heater raises the temperature of the cryopump situated in the bottom of the nitrogen leg to 150-200°C in order to drive off water and other condensable vapours. This is likely to cause the vacuum space pressure to rise and it may well be necessary to use the rotary pump air ballast yet again to purge the pump of these condensables.
10. The vacuum should be pumped and baked for approximately 6 hours. The final pressure should be 0.1mbar (or better) with a 2-stage rotary pump. You may well achieve 0.01 mbar but do not worry too much as Pirani gauges are not always very accurate.
11. To finish pumping and baking, the Sealing Plug should be pushed back in using the Actuator Knob of the Pumping Adaptor.

This must be done whilst the CRH25 heater is still hot.

12. Switch the CRH25 heater off immediately and allow to cool. Unscrew the Actuator Knob from the Sealing Plug and remove the Pumping Port Adaptor. Replace the Pumping Port Cover.

Cryostream Cooler Service

The Cryostream Cooler has been designed to be as easy to use as possible and should run without the need for constant attention, once the user is accustomed to the system. However, there are some important points to look out for:

1. If the outer case of the Cryostream Coldhead becomes wet or frosty, this probably indicates a loss of vacuum. The remedy is to pump out the system as described in Pumping 'Down the Vacuum Space in the Cryostream Cooler'.
2. Always make sure all the Teflon tubes are pushed firmly into each of their Quick-Release connectors. Each tube should fit in 16-17mm. Make sure that none of the tubes are strained to one side, this can sometimes cause a leak in an otherwise reliable vacuum connector. Regularly trimming these tubes will help keep them seated correctly.
3. Regular maintenance of the Line Drier Units is recommended, as they will become saturated with water from the nitrogen supply. Use of, and instructions for maintaining the Line Driers can be found in the section 'Stainless Steel Line Drier Units'.
4. The Cryostream Cooler Gas Pump Unit contains a rubber diaphragm. This diaphragm is designed to last 10000 hours of running. Possible symptoms of a diaphragm failure can be found in the Troubleshooting Guide. Do not rush to change this diaphragm as failure to replace it properly can result in the introduction of gas leaks.
5. The AD51 Dry Air Unit (optionally supplied for use as a dry air supply) has a service interval of 10000 hours. The Counter on the front of the AD51 indicates when this system needs servicing. If demand on the AD51 is high, plan to service the AD51 before it fails at a time that is convenient. Instructions for servicing the AD51 are in its manual. An AD51 Service Kit can be obtained from Oxford Cryosystems or a local Cryostream Cooler agent.

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 Cooler. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

General Properties

1. 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.
2. 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.
3. Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
4. Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
5. Gaseous and liquid nitrogen is non-corrosive.
6. Nitrogen does not support life and acts as an asphyxiant.
7. 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 nor oxygen depletion are 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. 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 uninsulated 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 10°C 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 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 HEAT 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 is 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.

Cryostream Cooler Troubleshooting Guide

Very important guidelines for using this document

1. This guide is designed for operators responsible for looking after Cryostream Coolers and have, at least, some experience of them. It is aimed at users of the 700 Series Cryostream Cooler.
2. This guide is not designed to cover every technical eventuality but to provide the correct interpretation of, and solution to, a variety of common symptoms. As a user, symptoms may arise that are not covered here. If, at any time, you are unsure of the cause of the Cryostream Cooler problem, contact your local agent or Oxford Cryosystems directly.
3. If you experience a shutdown or unusual behaviour from your system, please record as much information as possible and any physical symptoms you feel are a concern. Then contact Oxford Cryosystems or your local agent.
4. DO NOT rush into changing components or fixing something until you have spoken to Oxford Cryosystems (remember technical support and advice are free of charge) or your local agent. Changing components can VERY OFTEN create more problems and mask the original fault.

Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line

Cause

Condensation and/or ice over the outside of the Cryostream Cooler Transfer Line or can indicate a loss of vacuum. This should not be confused with localised spots of ice or condensation. Remember, it's impossible to lose a vacuum from one small area of vacuum space! This loss of vacuum can be for two reasons:

1. Natural out-gassing over a period time depending on the physical treatment of the Coldhead.
2. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

Solution

Consult the instruction manual on repumping the vacuum.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated Symptoms

Inability to reach low temperatures and the base temperature begins to rise

Inability to reach low temperatures

Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

Cause

This is caused by a failing of the vacuum insulation in the Coldhead and is determined by the Controller diagnostics. This is done by analysing the power to the various heaters in the Coldhead :

3. Natural out-gassing over a period time depending on the physical treatment of the Coldhead.
4. Vacuum leak at an internal or external joint or cracked flexible line, although this is rare.

Solution

Consult the instruction manual on repumping the vacuum.

If it is believed there is a leaky joint or cracked flexible hose, contact Oxford Cryosystems or your local agent as this is not user serviceable.

Associated Symptoms

Inability to reach low temperatures and the base temperature begins to rise

Inability to reach low temperatures

Localised ice spot on the flexible line

Cause

This is due to the flexible line being bent too sharply beyond its 200mm minimum radius so that the transfer capillary inside the flexible vacuum jacket touches the wall of the vacuum jacket.

Solution

The Cryostream Cooler will continue to run happily like this, but it may be worth contacting Oxford Cryosystems or your local agent. Try to increase the bend radius at the point where the ice spot occurs.

Localised ice formation around the neck of the Dewar and the Cryostream Cooler leg

Cause

If the Dewar is open to the atmosphere, it is common for ice to build up at the interface between the warm air and the cold gas emanating from the Dewar. This ice can fall into the Dewar and contaminate the nitrogen as its concentration builds up.

Solution

Find a way to cover the Dewar opening. This can be done quite comprehensively but **BE SURE NOT TO SEAL THE DEWAR**. Simply plugging the Dewar with a cloth will prevent most atmospheric moisture getting into the Dewar, but more elaborate setups involve clamping a bung on to the top of the Dewar and then drilling holes for the Cryostream Cooler leg, venting and refilling. If the Dewar is sealed up, a vent hole is **VERY** important otherwise the Cryostream Cooler will **NOT** work.

Associated Symptoms

Cryostream Cooler Shutdown due to low flow after a few seconds of running

Cryostream Cooler Shutdown due to low flow - no explicable reason

Inability to reach low temperatures

Cause

If the gas temperature will not drop below a certain temperature, (for example, the programmed gas temperature is 100K and the Cryostream Cooler will only reach 105K but is stable and not rising,) or has been rising over a period of time, the vacuum may be failing. The 700 Series is designed to monitor vacuum failure as it has control over the heat in the system. If there are vacuum problems when trying to run at 100K, the controller Alarm will ring and the vacuum warning will appear on the screen.

Solution

If the base gas temperature that the Cryostream Cooler reaches continues to rise, it is likely that the vacuum is degrading and will require repumping. See the manual for this procedure.

Associated Symptoms

Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line

Inability to reach low temperatures and the base temperature begins to rise

Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

Inability to reach low temperatures and the base temperature begins to rise

Cause

These symptoms indicate a loss of vacuum due to the natural out gassing over a period time depending on the physical treatment of the Coldhead. At lower temperatures, the Vacuum Warning Alarm should sound. At higher temperatures, a temperature error is more likely.

Solution

Repump the vacuum.

Associated Symptoms

Condensation and/or ice covering the outside of the Coldhead or the flexible transfer line

Inability to reach low temperatures

Cryostream Controller indicates 'Poor Vacuum' and ALARM lamp flashes

Ice formation on the sample

Cause

Ice formation on the sample can begin at the point of flash cooling the sample or it can build up over time to eventually cover the sample and thus ruin the diffraction image.

Note:

Ice on the sample does not come from the nitrogen gas travelling down the nozzle. Nitrogen gas from the Cryostream Cooler is very dry (circa 0.1ppm of water vapour).

Ice on the thin film supporting the crystal in the loop can arise from a number of sources.

1. Insufficient cryoprotection of the buffer solution.
 - a. Too much mother liquor results in dilution of the cryoprotectant to the point where it is no longer adequate.
 - b. A thick film around the crystal may result in a larger thermal mass that must then be cooled.
2. Rate of flash cooling is too slow.
3. 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.
4. The loop is unclean. Any particles on the loop will propagate ice formation.
5. A wet dry air supply or a disturbance of the laminar flow system due to drafts in your laboratory or an oversized sample mount (ie capillary or pin is too thick).
6. It is important that the velocities of the two gases are the same. If they are grossly unmatched, atmospheric moisture will encroach the streams and cause ice to build up. A true laminar flow will prevent attack from atmospheric moisture.

Solution

To increase the effectiveness of the cryoprotectant, increase its concentration.

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 6mm from the end of the nozzle and the centre 2mm cross section. Be sure to clean the loop before use as ice build up will only compound the problem.

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. This will greatly reduce the turbulence. If you are unsure of the source of the draft, try the Flashlight Test.

Try adjusting the flow of the outer dry gas stream. In a draft-free environment 7-8 litres per minute is fine, but when the air is more turbulent, try turning the outer stream flow rate up to 12 or 13 litres per minute; this can often cure the problem. (See Flashlight Test)

If the icing persists and there is also a concentric build up of ice on the nozzle, the most likely cause is a wet dry air supply. If you have an Oxford Cryosystems Dry Air Unit, change the Compressor Filter Delivery Element. If the icing persists, contact Oxford Cryosystems or your local agent about a Dry Air Unit service.

Associated Symptoms

Concentric formation of ice around the nozzle

Ice formation on outer edge of the nitrogen gas cold stream nozzle

Cause

The likely cause of ice on one side only of the nitrogen nozzle is a misaligned dry air shroud tube.

Solution

Look up the nozzle of the Cryostream Cooler and check to make sure the outer dry air shroud is concentric with the inner nitrogen nozzle. A small misalignment may be corrected by pushing the inner nozzle to one side. The shroud tube is locked into its 26mm diameter mounting bush using a low strength retainer compound (eg Loctite 222e). To release the shroud tube, grasp it gently and push to one side to release the retainer bond. Movement of the shroud tube will be limited as it touches the outside of the inner nitrogen nozzle - this prevents the shroud tube from kinking. Once the outer dry air shroud has been removed, refit the shroud tube using a little retainer compound, check that the tube is concentric and allow the retainer to set.

Concentric formation of ice around the nozzle

Cause

This is likely to be wet air from the dry air supply or a high flow rate from the dry air supply. The cold stream requires a dry air shroud of dewpoint -60°C . If the stream is wet, the moisture in the air will freeze onto the nozzle and sample.

Solution

Make sure an unruly student has not turned the flow rate up to 25 litres/minute!! If the Cryostream Cooler utilises an Oxford Cryosystems AD31 Dry Air Unit, change the Compressor Delivery Filter Element; a spare is provided. If the ice persists, contact Oxford Cryosystems or your local agent who will supply a service kit.

Associated Symptoms

Ice formation on the sample

Ice formation on outer edge of the nitrogen gas cold stream nozzle

Positive Gas Temperature Error

Cause

If after months of use, the Cryostream Cooler starts to lose its vacuum insulation, then it will struggle to maintain its base temperature and the gas temperature error will increase, positively.

Solution

Repump your vacuum insulation in accordance with the instructions in the manual.

Associated Symptoms

Condensation and/or ice covering the outside of the Coldhead or the flexible Transfer Line

Inability to reach low temperatures and the base temperature begins to rise

Cryostream Cooler Shutdown due to low flow after a few seconds of running

Cause

If, after a few seconds of running the Gas Pump Unit can not generate any gas flow the system will shut down. The most likely cause of this is a restriction in the transfer line from the Dewar caused by a solid piece of matter (usually ice) restricting the flow of liquid nitrogen.

Solution

Take the leg out of the Dewar vessel. Wait 15 minutes for the end of the leg to warm up to room temperature. Disconnect the dry air shroud gas from its connector at the top of the nozzle and reconnect it to the SUCTION connector on top of the Coldhead. Set the gas flow to about 5 litres/minute. This will blow air down the Cryostream Cooler transfer line and will both remove any solid matter or ice from the line and dry it out. This should be done for about half an hour until the leg has warmed up. Once the block is cleared reconnect the tubing to its correct connectors.

Gas Pump Unit is making a strange noise

Cause

Although sounds are often difficult to interpret it is worth noting a few things. The noises from the Gas Pump Unit vary from pump to pump and voltage to voltage. If you are unhappy with the noise your pump is making, possible causes are:

1. a rattling or knocking can often indicate a broken con rod.
2. a grinding usually indicates a failed bearing.
3. a slapping noise can be produced by the diaphragm. IT IS NOT USUALLY A FAULT.

Solution

Contact your local agent or Oxford Cryosystems to get the replacement part or the pump serviced.

Flow rate will not rise to 10 l/min when the TURBO button is pressed

Symptoms

The system is running and when the TURBO button is pressed, the flow does not rise from 5 l/min to 10 l/min. The flow may stop at 7 or 8 l/min.

Cause

One of the likely causes of this problem is that the unit in the controller regulating and monitoring the flow is not functioning correctly. To test for this, do the following:

1. Disconnect the SUCT and FLOW tubes from their connectors on the top of the Coldhead. (You will need to remove the tube from its compression fitting too.)
2. Switch the controller on, (if its not on already!)
3. Program in a RAMP to whatever the current room temperature default reading is (about 290-295K) and press START.
4. Once the system has been running for a minute or so, press the TURBO button several times to cycle the flow rate between 5 and 10 l/min.
5. Press STOP when you have finished.

If the flow does not rise to 10 l/min then there is likely to be a problem with the Flow Controller.

Solution

Contact Oxford Cryosystems for a replacement Flow Controller.

EPROM fail on initialisation

Symptoms

During the initialisation of the controller, there is an EPROM failure.

Cause

The likely cause of this problem is corruption of the EPROM software. If this is the case, this problem can be rectified with the assistance of Oxford Cryosystems.

Take the lid off the controller, be careful of the earth wire that is attached inside when doing so. Move the jumper (JP9) to the SET pins and then turn the controller on.

Once the controller has finished initialising, you will see a list of parameters like those listed below. Write down all the parameters and their corresponding values and send them to CryostreamSupport@OxfordCryosystems.co.uk.

Firmware	Date
Board	Coldhead
Fan	Control
Suct H	SuctSet
Evap H	TestR
Gas Heat	SC.SUCT
Gas Flow	RC.SUCT
Shutdown	SC.EVAP
T Units	RC.EVAP
Cool T	SC.GAS
Hours	RC.GAS

Solution

Oxford Cryosystems will then contact you letting you know if any of these numbers are wrong the how to edit them.

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 than by trying to guess what the flow should be by looking at the numbers.

Turn all the lights off in the x-ray room 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.

Technical Support

To allow Oxford Cryosystems to offer fast and accurate technical support please quote your Cryostream Cooler Serial Number with all technical issues. This is a three digit number and can be found etched on the top of the Coldhead section of the Cryostream Cooler. This number is also on the back panel of the Controller. Please do not confuse it with the six-digit Controller number. It is worth keeping a record of this number in a convenient place:

Cryostream Cooler Serial Number

This Cryostream Cooler Serial Number is

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

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3 Blenheim Office Park
Lower Road
Long Hanborough
Oxford OX29 8LN
UK
Tel: +44 1993 883488 Fax: +44 1993 883988
Email CryostreamSupport@OxfordCryosystems.co.uk