

# oxford cryosystems

## Cryostream Cooler Troubleshooting v1.5

PLEASE READ: [Very important guidelines for using this document](#)

[How to make contact with Oxford Cryosystems](#)

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1. [Condensation and/or ice covering the outside of the blue Coldhead or the flexible transfer line](#)
2. [Localised ice spot on the flexible line](#)
3. [Localised icing on the Coldhead SUCT connector and tubing](#)
4. [Localised ice formation around the neck of the dewar and the Cryostream leg](#)
5. [Inability to reach lower temperatures](#)
6. [Inability to reach low temperatures and the base temperature begins to rise](#)
7. [Controller Status reads 'Ice Block warning will trip at 2 l/min'](#)
8. [Ice formation on the sample](#)
9. [Feather of ice forming from inside the cold stream nozzle and extending towards sample](#)
10. [Ice formation on outer edge of the nitrogen gas cold stream nozzle](#)
11. [Concentric formation of ice around the nozzle](#)
12. [Spitting of liquid nitrogen from the nozzle](#)
13. [Unstable gas temperature](#)
14. [Cryostream maintains an error of a few degrees that disappears when the dewar is topped up](#)
15. [Cryostream Shutdown due to low flow after a few seconds of running](#)
16. [Cryostream Shutdown due to low flow - no explicable reason](#)
17. [Cryostream Shutdown due to ice block](#)
18. [Cryostream Shutdown due to large temperature error](#)
19. [Cryostream Shutdown due to large temperature error with unusually high or low GasT](#)
20. [Cryostream Shutdown due to large temperature error when refilling the dewar](#)
21. [Cryostream fails to initialise due to Gas Heater >64 ohms or <30 ohms](#)
22. [Cryostream fails to initialise due to Gas Sensor Error](#)
23. [Cryostream fails to initialise due to Evap Heater or Evap Sensor error](#)
24. [Gas Pump is making a strange noise](#)

[Nitrogen Circuit Gas Leak Test](#)

## [Flashlight Test](#)

## [Stainless Steel Line Drier Unit](#)

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# Very important guidelines for using this document

1. This document 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 600 Series Cryostream Cooler.
  2. This document 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 your Cryostream 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 including GasT, Error, EvapT, Heat%, gas flow rate from the flowmeter 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.
  5. This document may be copied freely and distributed to users of the Oxford Cryosystems Cryostream Cooler.
  6. Be sure to check the [Oxford Cryosystems Web Site](#) for notification of the latest update to this document.
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# Condensation and/or ice covering the outside of the blue Coldhead or the flexible transfer line

## Cause

Condensation and/or ice over the outside of the Cryostream transfer line or blue can indicates 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. If these symptoms arise more frequently than twice a year, there is a chance the Pumpout Port and Relief Valve under the two blue dust caps at the top of the rigid section of the transfer leg may be freezing during refilling of the dewar. These ports contain rubber O-rings which can harden and leak when sprayed with cold gas or liquid. This freezing will not permanently damage the O-rings as they will defrost, but it will cause them to leak.
3. 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 and *be sure to protect the Pumpout Port and Relief Valve when refilling.*

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 servicable.

## Associated Symptoms

[Inability to reach low temperatures and the base temperature begins to rise](#)

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# 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

Cryostream 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.

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# Localised icing on the Coldhead SUCT connector and tubing

## Cause

This is due to an imbalance in the heat exchanger inside the Coldhead of Cryostream.

Cryostream requires 5 litres/minute of gas along both paths of its heat exchanger so that the 5 litres of gas in the SUCT path can absorb the heat from the 5 litres of gas in the FLOW path. If gas has leaked out between the gas exiting the SUCT side of the heat exchanger (SUCT connector on the top of the Coldhead,) and entering the FLOW side of the heat exchanger (FLOW connector on the top of the Coldhead,) excess cold gas spills out into the nylon tubing in the SUCT connector causing condensation and icing.

## Solution

Check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector on the Cryostream and then perform the [Nitrogen Circuit Gas Leak Test](#) . Run the system again and if the problem persists, contact [Oxford Cryosystems](#) or your local agent of your findings.

## Associated Symptoms

[Unstable gas temperature](#)

[Spitting of liquid nitrogen from the nozzle](#)

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# Localised ice formation around the neck of the dewar and the Cryostream 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 leg, venting and refilling. If the dewar is sealed up, a vent hole is VERY important otherwise Cryostream will NOT work.

## Associated Symptoms

[Controller Status reads 'Ice Block warning will trip at 2 l/min'](#)

[Cryostream Shutdown due to ice block](#)

[Cryostream Shutdown due to low flow after a few seconds of running](#)

[Cryostream Shutdown due to low flow - no explicable reason](#)

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## 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 Cryostream will only reach 105K but is stable and not rising,) there are two possible causes:

1. The Evaporator Heater setting is too high. This is usually only the case when the required temperature is below about 95K.
2. If the minimum gas temperature has been rising over a period of time and it is not possible to reach the required temperature, the vacuum may be failing.

### Solution

Manually adjust your Evaporator setting. Please see your manual for this procedure. If the base gas temperature that Cryostream reaches continues to rise, it is likely 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 blue Coldhead or the flexible transfer line](#)

[Inability to reach low temperatures and the base temperature begins to rise](#)

[Cryostream maintains an error of a few degrees that disappears when the dewar is topped up](#)

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

## Cause

These symptoms indicate a loss of vacuum. 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. If these symptoms arise more frequently than twice a year, there is a chance the Pumpout Port and Relief Valve under the two blue dust caps at the top of the rigid section of the transfer leg may be freezing during refilling of the dewar. These ports contain rubber O-rings which will turn to glass when sprayed with cold gas or liquid. This freezing will not permanently damage the O-rings as they will defrost, but it will cause them to leak.

## Solution

Repump the vacuum and be sure to protect the Pumpout Port and Relief Valve when refilling the dewar with nitrogen.

## Associated Symptoms

[Condensation and/or ice covering the outside of the blue Coldhead or the flexible transfer line](#)

[Inability to reach low temperatures](#)

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# Controller Status reads 'Ice Block warning will trip at 2 l/min'

## Cause

Cryostream has detected the beginnings of an ice block inside the Coldhead. The ice block is detected by a pressure switch in the controller and is designed to give the user an advance warning of an ice block that will eventually cause the system to trip.

This is due to a build up of ice in the FLOW path capillary of the heat exchanger. The water vapour in the nitrogen gas freezes out onto the wall of the capillary of the heat exchanger and eventually blocks it. It is not blocked by large particles of ice by the slow narrowing of the capillary wall as the water vapour freezes out. If the Cryostream has been running well up to this point, it may continue to run for long enough to finish a data collection. The next symptom to be seen is a gradual fall in the nitrogen gas flow rate.

There are two possible sources for the water vapour:

1. Ice in the nitrogen supply - small particles of ice are sucked up the leg and flexible transfer line into the Coldhead. These particles pass through the Evaporator Heater and along the SUCT path of the heat exchanger. The ice then passes out of the Coldhead along the tubing marked SUCT, through the controller, through the pump, back through the controller and back into the Coldhead along the tubing marked FLOW or DEL. During its course along the nylon tubing, the ice melts to water vapour which, on entry into the FLOW path of the heat exchanger, refreezes causing narrowing of the capillary and an impending ice block. **ICE PARTICLES DO NOT CAUSE THE BLOCK, WATER VAPOUR THAT FREEZES CAUSES THE BLOCK.**  
**Please note: There is no easy way of completely removing particles of ice from your nitrogen supply. This means you will get this warning eventually, so do not panic!!**
2. An inward leak - Atmospheric moisture is sucked into the gas stream on the SUCT path of the gas flow circuit at some point. The likely cause of this is a nylon tube NOT pushed its full 15-16mm into the quick-fit connector. If all the tubing has been reseated properly, it is possible the diaphragm in the gas pump has split.

## Solution

First, to remove the ice block, let your system warm up over night. The EvapT value on the controller must reach room temperature as this indicates that the ice block has melted. When the system is restarted, the water vapour will blow, unnoticed, out of the end of the nozzle. If the Cryostream is required for use and it can not be left over night, disconnect the dry air supply for Cryostream at the top of the nozzle and reconnect it to the FLOW connector on the top of the Coldhead. **IMPORTANT:** Reduce the flow on the dry air supply to 5 litres/minute to avoid any damage to the Gas Sensor in the nozzle. The dry air should be left running for a few hours until the EvapT value on the controller reaches room temperature, this will blow out all the water vapour. Once this process has finished, reconnect all the tubing.

Ice in your nitrogen supply - empty the dewar vessel and let it warm up to room temperature. Mop up any moisture in the bottom. Check on the purity of your liquid nitrogen by shining a flashlight into the dewar. The liquid should look clean like water but do not worry if you see a few particles in the bottom, this is

fairly normal. If your liquid nitrogen is milky, you have a chronic contamination of ice, Cryostream can not handle this so talk to your liquid nitrogen supplier. Try to avoid pushing the rigid leg of Cryostream into the bottom of the dewar, this prevents particles in the bottom of the dewar being sucked up the leg. Dewars that are continually topped up should be emptied and cleaned out at least every three months, more often if possible.

An inward leak - Check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector. It is worth marking each tube with a marker pen to be sure that each tube is seated correctly. Perform the [Nitrogen Circuit Gas Leak Test](#) and inform [Oxford Cryosystems](#) or your local agent of your findings. Do not rush into changing a diaphragm as opening up the gas pump can often cause a leak. The diaphragm in the grey gas pump will last for approximately one year of continuous running. Speak to Oxford Cryosystems first.

Once the ice contamination problem has been rectified, fit an Oxford Cryosystems [Line Drier Unit](#). If there is already one in circuit, it will now be contaminated and will require reactivation ([see instructions](#)). DO NOT fit a new one or a reactivated one until you have rectified the ice contamination. You will create unnecessary work and confusion for yourself.

## Associated Symptoms

[Cryostream Shutdown due to ice block](#)

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# 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.

**Please note: Ice on the sample does not come from the nitrogen gas travelling down the nozzle. Nitrogen gas from Cryostream 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.
  - Too much mother liquor results in dilution of the cryoprotectant to the point where it is no longer adequate.
  - A thick film around the crystal may result in a larger thermal mass which 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 which 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 velocity 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](#)

[Feather of ice forming from inside the cold stream nozzle and extending towards sample](#)

# Feather of ice forming from inside the cold stream nozzle and extending towards sample

## Cause

An ice build up of this type is caused by a disturbance of the laminar flow system where the cold stream mixes with the outer dry air stream and atmospheric moisture. This is due to drafts in the laboratory or a foreign body in the Cryostream nozzle.

## Solution

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 outer dry gas stream. In a draft-free environment 7-8 litres/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 ice build up continues, look *carefully* up the nozzle to make sure there are no obvious foreign bodies disturbing the laminar flow system (eg. blob of solder or modelling clay). If there is, contact [Oxford Cryosystems](#) or your local agent, immediately; DO NOT TRY TO REMOVE THE OBJECT WITHOUT SPEAKING TO US FIRST. Damage to the Gas Sensor is expensive and time consuming to fix.

## Associated Symptoms

[Ice formation on the sample](#)

[Concentric formation of ice around the nozzle](#)

[Ice formation on outer edge of the nitrogen gas cold stream nozzle](#)

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# 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. This is not the same as a [feather of ice growing from inside the cold nitrogen gas nozzle](#).

## Solution

Look up the nozzle of the Cryostream 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.

## Associated Symptoms

[Feather of ice forming from inside the cold stream nozzle and extending towards sample](#)

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# 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  $-60^{\circ}\text{C}$ . 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 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](#)

[Feather of ice forming from inside the cold stream nozzle and extending towards sample](#)

[Ice formation on outer edge of the nitrogen gas cold stream nozzle](#)

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## Spitting of liquid nitrogen from the nozzle

### Cause

Spitting of liquid nitrogen is caused by recondensation of nitrogen gas in the FLOW path of the heat exchanger. This can be caused by a number of factors:

1. The gas flowing through the heat exchanger in the Coldhead is unbalanced. The amount of gas flowing through the SUCTION path of the heat exchanger is greater than the amount of gas flowing along the FLOW path. This causes the gas in the FLOW path to get too cold and recondense. This is commonly due to an outward gas leak.
2. The Evaporator heater is set too low. If the Evaporator has been adjusted to a lower setting, there is more cooling in the heat exchanger than is required. This causes recondensation of nitrogen in the FLOW path and liquid nitrogen is spat out of the nozzle.
3. The Coldhead is angled too close to horizontal. Cryostream relies a little on gravity so that any condensed liquid nitrogen can run unnoticed from the nozzle. If the Coldhead is running horizontally or upward, there is often a small build up of pressure behind droplets of liquid nitrogen that are then spat from the heat exchanger out of the nozzle.

### Solution

It is worth considering these solutions in order:

1. Adjust the angle of your Coldhead to greater than 10° from the horizontal.
2. Increase your Evaporator setting until the spitting has stopped. (600 Series Users: If you are running at around 90K your Heat% value should be no more than a 2-5%.)
3. Check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector then perform the [Nitrogen Circuit Gas Leak Test](#) and contact [Oxford Cryosystems](#) or your local agent with your findings.

## Associated Symptoms

[Localised icing on the Coldhead SUCT connector and tubing](#)

[Unstable gas temperature](#)

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## Unstable gas temperature

### Cause

There are a couple of possible causes of an unstable gas temperature:

1. When Cryostream experiences recondensation of nitrogen in its heat exchanger the liquid spits down the nozzle and hits the Gas Heater and Gas Sensor causing the temperature to jump around as it reacts to keep the temperature stable. This will also be associated with a fluctuation in the Heater power (Heat%). This imbalance in the heat exchangers can be caused by an outward gas leak or an Evaporator Heater set too low.
2. The Gas Sensor in the Cryostream nozzle is misaligned.
3. If after months of use, Cryostream starts to lose its vacuum insulation, then it will struggle to maintain its base temperature and the gas temperature error will increase, positively.

### Solution

It is worth considering these solutions in order:

1. Increase your Evaporator setting by a few tenths of a degree at a time until the spitting has stopped. (600 Series Users: If you are running at around 90K your Heat% value should be typically 5-7%).
2. Look up the end of the nozzle. The Gas Sensor is a small chip that lies in the gas stream. It is possible to just make out the edge of it if you look carefully. **DO NOT, UNDER ANY CIRCUMSTANCES, TOUCH THIS SENSOR. IT CAN BE EASILY DAMAGED AND IS EXPENSIVE TO FIX.** If the sensor is not in the centre of the stream, the temperature will jump around as it is not seeing the true gas temperature. Contact [Oxford Cryosystems](#) who will be able to fix the problem.
3. If you suspect an outward gas leak, check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector then perform the [Nitrogen Circuit Gas Leak Test](#) and contact [Oxford Cryosystems](#) or your local agent with your findings.
4. As a very last resort, repump your vacuum insulation in accordance with the instructions in the

manual.

## Associated Symptoms

[Condensation and/or ice covering the outside of the blue Coldhead or the flexible transfer line](#)

[Spitting of liquid nitrogen from the nozzle](#)

[Localised icing on the Coldhead SUCT connector and tubing](#)

[Inability to reach low temperatures and the base temperature begins to rise](#)

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## Cryostream maintains an error of a few degrees that disappears when the dewar is topped up

### Cause

In the bottom five inches of the Cryostream transfer leg there is a Cryopump. This maintains the vacuum in Cryostream when the leg is plunged into liquid nitrogen. If the nitrogen level gets sufficiently low to uncover the Cryopump, the vacuum insulation can be weakened to cause the gas temperature to rise.

### Solution

Top up the dewar. If the dewar is fitted with a level detector, make sure it is set up to refill before the Cryopump is exposed.

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

### Cause

If, after a few seconds of running the pump 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 SUCT connector on top of the Coldhead. Set the gas flow to about 5 litres/minute. This will blow air down the Cryostream 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.

## Associated Symptoms

[Cryostream Shutdown due to low flow - no explicable reason](#)

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# Cryostream Shutdown due to low flow - no explicable reason

## Cause

There are two possible causes Cryostream can not diagnose itself:

1. A restriction in transfer line from the dewar caused by a solid piece of matter (usually ice) restricting the flow of liquid nitrogen.
2. The optical Gas Flow Sensor in the flowmeter has been subjected to a source of infra red light which will fool it and cause the Cryostream to trip.

## Solution

1. Wait for the Cryostream to warm up over a few hours (EvapT must reach room temperature) then take the leg out of the dewar vessel. Disconnect the dry air shroud gas from its connector at the top of the nozzle and reconnect it to the SUCT connector on top of the Coldhead. Set the gas flow to about 5 litres/minute. This will blow air down the Cryostream transfer line and will both remove any solid matter or ice from the line and dry it out. **DO NOT CONNECT THIS DRY AIR SUPPLY TO THE FLOW PATH WITH A FLOW RATE OF >5 LITRES/MINUTE. THIS MAY DAMAGE THE GAS SENSOR IN THE NOZZLE.**
2. Make sure the front panel of Cryostream is not facing a window or a bright light. Turn it away

from the light source.

## Associated Symptoms

[Cryostream Shutdown due to low flow after a few seconds of running](#)

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# Cryostream Shutdown due to ice block

## Cause

This is due to a build up of ice in the FLOW side of the heat exchanger. This ice has come from water vapour frozen out on the wall of the capillary of the heat exchanger. It is not a block caused by large particles of ice but by the slow narrowing of the FLOW path capillary wall as the water vapour freezes out.

There are two possible sources for the water vapour:

Ice in the nitrogen supply - small particles of ice are sucked up the leg and flexible transfer line into the Coldhead. These particles pass through the Evaporator Heater and along the SUCT path of the heat exchanger. The ice then passes out of the Coldhead along the tubing marked SUCT, through the controller, through the pump, back through the controller and back into the Coldhead along the tubing marked FLOW or DEL. During its course along the nylon tubing, the ice melts to water vapour which, on entry into the FLOW path of the heat exchanger, refreezes causing narrowing of the capillary and an impending ice block. **ICE PARTICLES DO NOT CAUSE THE BLOCK, WATER VAPOUR THAT FREEZES CAUSES THE BLOCK.**

**Please note: There is no easy way of completely removing particles of ice from your nitrogen supply. This means you will get this warning eventually, so do not panic!!**

An inward leak - Atmospheric moisture is sucked into the gas stream on the SUCT path of the gas flow circuit at some point. The likely cause of this is a nylon tube NOT pushed its full 15-16mm into the quick-fit connector. If all the tubing has been reseated properly, it is possible the diaphragm in the gas pump has split.

## Solution

First, to remove the ice block, let your system warm up over night. The EvapT value on the controller must reach room temperature as this indicates that the ice block has melted. When the system is restarted,

the water vapour will blow, unnoticed, out of the end of the nozzle. If the Cryostream is required for use and it can not be left over night, disconnect the dry air supply for Cryostream at the top of the nozzle and reconnect it to the FLOW connector on the top of the Coldhead. **IMPORTANT:** Reduce the flow on the dry air supply to 5 litres/minute to avoid any damage to the Gas Sensor in the nozzle. The dry air should be left running for a few hours until the EvapT value on the controller reaches room temperature, this will blow out all the water vapour. Once this process has finished, reconnect all the tubing.

Ice in your nitrogen supply - empty the dewar vessel and let it warm up to room temperature. Mop up any moisture in the bottom. Check on the purity of your liquid nitrogen by shining a flashlight into the dewar. The liquid should look clean like water but do not worry if you see a few particles in the bottom, this is fairly normal. If your liquid nitrogen is milky, you have a chronic contamination of ice, Cryostream can not handle this so talk to your liquid nitrogen supplier. Try to avoid pushing the rigid leg of Cryostream into the bottom of the dewar, this prevents particles in the bottom of the dewar being sucked up the leg. Dewars that are continually topped up should be emptied and cleaned out at least every three months, more often if possible.

An inward leak - Check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector. It is worth marking each tube with a marker pen to be sure that each tube is seated correctly. Perform the [Nitrogen Circuit Gas Leak Test](#) and inform [Oxford Cryosystems](#) or your local agent of your findings. Do not rush into changing a diaphragm as opening up the gas pump can often cause a leak. The diaphragm in the grey gas pump will last for approximately one year of continuous running. Speak to Oxford Cryosystems first.

Once the ice contamination problem has been rectified, fit an Oxford Cryosystems [Line Drier Unit](#). If there is already one in circuit, it will now be contaminated and will require reactivation ([see instructions](#)). DO NOT fit a new one or a reactivated one until you have rectified the ice contamination. You will create unnecessary work and confusion for yourself.

## Associated Symptoms

[Controller Status reads 'Ice Block warning will trip at 2 l/min'](#)

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# Cryostream Shutdown due to large temperature error

## Cause

If the Cryostream has shut down after a period of time during which the gas temperature has risen steadily

and the Heat% has read zero, there is a good chance the vacuum is deteriorating.

When Cryostream experiences recondensation of nitrogen in its heat exchanger, the liquid will spit down the nozzle and hit the Gas Heater and Gas Sensor causing the temperature to jump around as it reacts to keep the temperature stable, (this instability may cause the shutdown after a period of time.) This will also be associated with a fluctuation in the Heater power (Heat%). This unbalance in the heat exchangers can be caused by an outward gas leak or an Evaporator Heater set too low.

## Solution

It is worth considering these solutions in order:

1. If you suspect an outward gas leak, check to make sure all the nylon tubing is pushed 15-16mm into each quick-fit connector then perform the [Nitrogen Circuit Gas Leak Test](#) and contact [Oxford Cryosystems](#) or your local agent with your findings.
2. Increase your Evaporator setting by a few tenths of a degree at a time until the spitting has stopped. (600 Series Users: If you are running at around 90K your Heat% value should be typically 5-7%).
3. Repump your vacuum insulation in accordance with the instructions in the manual.

## Associated Symptoms

[Condensation and/or ice covering the outside of the blue Coldhead or the flexible transfer line](#)

[Spitting of liquid nitrogen from the nozzle](#)

[Cryostream Shutdown due to large temperature error with unusually high or low GasT](#)

[Cryostream Shutdown due to large temperature error when refilling the dewar](#)

[Cryostream maintains an error of a few degrees that disappears when the dewar is topped up](#)

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# Cryostream Shutdown due to large temperature error with unusually high or low GasT

## Cause

If the Cryostream has shutdown due to a large gas temperature error and either the error or gas temperatures are nonsense values, (eg 30K or 600K,) the system is suffering from electrical noise. Possible sources of electrical noise are a Rotating Anode generator, noisy mains supply or static electricity.

## Solution

Often moving a large (100-200 litre) storage dewar across a lab on rubber wheels can cause enough static to build up when the transfer hose is stuck in the opening of the dewar. Be sure to earth the nitrogen storage dewar before placing the transfer hose into the dewar used by the Cryostream.

If the problem persists, contact [Oxford Cryosystems](http://www.oxfordcryosystems.co.uk) or your local agent.

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# Cryostream Shutdown due to large temperature error when refilling the dewar

## Cause

1. It is important to remember that the inside of the dewar used by the Cryostream is at about 80K. The air in the transfer hose from the storage dewar is at about 300K. Spraying the warm air / nitrogen mixture into the dewar without venting it first, **CAN** evaporate large amounts of liquid in the dewar. This will cause a shutdown due to the presence of no nitrogen.
2. The pressure in the storage dewar should be no greater than 1-2 bar. Refilling a Cryostream dewar with a storage dewar or cylinder under very high pressure will produce warmer liquid nitrogen. This warm nitrogen will cause a large temperature error.
3. Refilling the Cryostream dewar with the transfer hose in the liquid nitrogen can blow enough gas into the liquid to turn it into Cryogenic Soda Water. This effervescence will cause Cryostream to shut down.

## Solution

1. Vent the transfer hose until the exit gas is cold.
2. In the short term, vent the storage dewar to reduce the pressure. In the long term, modify the relief valve so the liquid nitrogen is stored between 1-2 bar.
3. Make use the liquid nitrogen transfer hose from the supplier dewar is out of the liquid nitrogen in the Cryostream dewar during the refilling.

## Associated Symptoms

[Cryostream Shutdown due to large temperature error](#)

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# Cryostream fails to initialise due to Gas Heater >64 ohms or <30 ohms

## Cause

The Cryostream Gas Heater is either open circuit (>64 ohms) or short circuit (<30 ohms). This can be caused by corrosion or contamination from particles or liquids in the lab or letting the dewar frequently run dry.

## Solution

Make sure the Cryostream nozzle is not angled at less than 20° from the horizontal. This will prevent condensable from attacking the Gas Heater when the system is switched off.

Make sure there is always, at least, 5 inches of liquid nitrogen in the bottom of the dewar.

Contact your local agent or [Oxford Cryosystems](#) to arrange of the component to be repaired.

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# Cryostream fails to initialise due to Gas Sensor Error

## Cause

The Cryostream Gas Sensor is a very delicate silicon diode positioned just inside the end of the nozzle. If an object such as a pen, mounting pin or goniometer arc is inserted into the nozzle, this can cause damage to the sensor. Very much like the Gas Heater, damage to the Gas Sensor can be caused by corrosion from particles or liquids in the lab.

## Solution

Warn all users not to push anything up the nozzle. Take extra care when placing and removing Tophats / Caps from the goniometer. No maintenance should take place via the nozzle exit.

Make sure the Cryostream nozzle is not angled at less than 20° from the horizontal. This will prevent condensable material from attacking the Gas Heater when the system is switched off.

Contact your local agent or [Oxford Cryosystems](#) to arrange of the component to be repaired.

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## Cryostream fails to initialise due to Evap Heater or Evap Sensor error

### Cause

As these components are secured tightly inside the blue body of the Coldhead, it is very rare for them to get damaged. The most likely cause of damage to these components is from an electrical surge.

### Solution

Contact [Oxford Cryosystems](#) or your local agent immediately. Do not try to service this yourself as there are no user serviceable parts inside the Coldhead.

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## Gas Pump is making a strange noise

### Cause

Although sounds are often difficult to interpret it is worth noting a few things. The noise from the gas pumps 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.

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## Nitrogen Circuit Gas Leak Test

Wait for the Cryostream Coldhead to reach room temperature. This will usually require leaving the system overnight. Once the inside of the Coldhead has warmed up, remove the leg from the liquid nitrogen and allow that to also warm up (15-20 minutes.) These precautions prevent atmospheric moisture from contaminating the inside of Cryostream.

Disconnect the nylon tubes from the FLOW and SUCT Quick Fit Connectors on the top of the Coldhead flange.

If you are using a Line Drier Unit, remove it from the gas flow circuit. (If you do not remove it, you will contaminate it with atmospheric moisture. Leaving the Line Drier in the circuit will smooth the flowmeter reading.)

Disconnect the two mains cables from the back of the controller. (If you are using a 500 series Cryostream, disconnect the mains cable from the back of the controller and the cable from the back of the Gas Box.) Run the gas pump directly from the mains by connecting the two cables together.

Your gas flowrate should read 5 litres / minute on your flowmeter. The top edge of the float should touch the bottom of the number '6' on the float.

### To Check for an Inward Leak

While the gas pump is running directly from the mains, place your finger over the tube taken from the SUCT connector on the Coldhead. If the flow does not fall slowly to zero, you have an inward leak.

### Likely Causes of an Inward Leak

1. Check to make sure all the nylon tubes are firmly nested in all the Quick-Fit Connectors, (15-16mm).
2. A split in your gas pump diaphragm. Contact your local Cryostream agent or [Oxford Cryosystems](#), about getting a new one.

### To Check for an Outward Leak

While the gas pump is running directly from the mains, place your finger over the tube taken from the FLOW connector on the Coldhead. If the flow does not fall to zero, you have an OUTWARD leak. If you are not using a Line Drier the float may bobble around at the bottom of the flowmeter, this is expected.

## Likely Causes of an Outward Leak

1. Check to make sure all the nylon tubes are firmly nested in all the Quick-Fit Connectors, (15-16mm).
2. Check for cracks in the flowmeter on the front of your controller, (or Gas Box if you have a 500 series Cryostream.)

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## 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 Cryostream 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 litres / minute of air from the dry air source. One should not be fooled into thinking that at 25 litres / minute 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.

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## Stainless Steel Line Drier Units

### How and why ice blocks occur

For a variety of reasons, it is quite common for small quantities of water vapour to contaminate the nitrogen gas stream flowing through the Cryostream Cooler. If sufficient water is present, an ice blockage will form which can reduce the flowrate or cause a Low Flow trip - this process may take a few days or several weeks, depending on water vapour content.

The block forms in the FLOW path of Cryostream as the nitrogen gas is cooled from room temperature to liquid nitrogen temperature in the heat exchanger. Two possible sources of water vapour are ice in the liquid nitrogen or an inward leak in the SUCT or SUCTION paths of the gas circuit usually resulting from

a badly nested Quick Fit Connector.

A third source is a split diaphragm in the gas pump but this is not likely to happen until the gas pump has run for approximately 10,000 hours.

## **Before fitting and using your Line Drier Unit**

When you first receive your Cryostream Cooler, **RUN YOUR SYSTEM WITHOUT FITTING YOUR LINE DRIER**. If there are any inward leaks or a high level of ice in the liquid nitrogen, Cryostream will block in a few days but, will not contaminate your Line Drier with water.

If this occurs, recheck all your Quick Fit Connectors (pressing them in 15-16mm,) check your liquid nitrogen and dewar then rerun your system. Cryostream should run for **AT LEAST 7 to 10 days** before a block occurs, although poor quality nitrogen can reduce this to 2-3 days. (You will probably get an ice block warning within two or three days, this will appear on the STATUS line - 600 series Cryostream only.) Do not worry if you do not see any of these signs, you probably have very good quality liquid nitrogen.

Once you are happy that you can 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 before an ice block can form. **IT WILL NOT COPE WITH GROSS CONTAMINATION** of the nitrogen supply or a large inward leak.

Check that all the nylon tubes are pushed fully (approx 15mm) into the Quick Fit Connectors. 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 high purity.

Remove the plastic Blanking Plugs, (these plugs must be fitted to the Line Drier during transit and storage.) Fit the Line Drier in the DEL pipework between the Gas Pump and the Controller, (or Gas Box - 500 series Cryostream.) **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.

To regenerate, disconnect the Line Drier and unscrew the Metal End Fittings to avoid heat damage to the

rubber 'O' ring seals.

Arrange to pass a flow of clean dry nitrogen (or any other inert) gas at about 1 to 2 litres/minute through the Line Drier from OUT to IN whilst heating the Line Drier to 200-250°C. **DO NOT USE DRY AIR FROM THE DRY AIR UNIT. THIS GAS IS NOT DRY ENOUGH.**

After 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.

## **Heat sources for regenerating the Line Drier Unit**

The easiest method of regeneration is to wrap the Line Drier in a short Heating Tape / Strip (1.5 metres, 240 watts). Inject the purge gas through a short (300mm) length of stainless steel tube to act as a heat break. Small leaks at the couplings do not matter. Alternatively, an oven may be used, but it may prove difficult to pass inert gas through the Line Drier.

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