Operator’s Handbook

Cryojet

Cryogenic Nitrogen jets for X-ray crystallography

Including: CryojetXL and CryojetHT

Revision 9

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Contents

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1 Important Information

1.1 Warning

Before you operate this equipment, you must make sure that you are aware of the precautions necessary to ensure your own safety. We supply a separate booklet called *Safety Matters* with the system. Please read it carefully so that you fully understand the hazards you may encounter when using cryogens.

Warning: **It is your responsibility to ensure your own safety, and the safety of the people working around you.**

The CryojetXL tip can become very cold, and the CryojetHT tip can become very hot and very cold under different operating conditions. Caution should be exercised when using the Cryojet, including when it is returning to ambient temperature conditions to ensure that the user does not come into contact with the areas with extremes of temperature.

If using an autofill system, there is a possibility that it could fail, causing the contents of the pressurised dewar to spill into the room. An oxygen monitor is included with the Oxford Instruments autofill system. Oxford Instruments strongly recommend that this device is used to ensure the safety of yourself and those around you. Ensure that the instructions included with the monitor are read.

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

1.2 This manual

This manual is part of the product that you have bought. Please keep it for the whole life of the product and make sure that you incorporate any amendments which might be sent to you. If you sell or give away the product to someone else, please give them the manual too.

1.3 Important Health and Safety Notice

Important Health and Safety Notice

When returning components for service or repair it is essential that the item is shipped together with a signed declaration that the product has not been exposed to any hazardous contamination or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

1.4 Conventions used in this manual

The following conventions have been followed in this manual:

Danger: **Indicates that the hazard may cause death or severe injury if the instructions are not followed carefully.**

Warning: **Indicates that the hazard may cause injury.**

Caution: Indicates that the hazard may cause damage to equipment.

Note: Something that needs to be brought to the customer's attention.

Tip: Indicates a helpful hint that may be of use to the customer.

1.5 Disposal and recycling instructions

Before disposing of this equipment, it is important to check with the appropriate local organisations to obtain advice on local rules and regulations about disposal and recycling.

You **must** contact Oxford Instruments NanoScience Customer Support (giving full product details) before any disposal begins.

2 Description of the system

The Cryojet is designed to operate in combination with any commercial X-ray diffractometer or synchrotron. It uses a jet of temperature controlled nitrogen gas to heat or cool a sample without the need for windows between the X-ray source, sample and detector. Its principal features are:

- Wide temperature range: CryojetXL (90 –300 K) or CryojetHT (90 – 490 K)
- No nozzle icing or blocking: cold gas jet shield by second annular flow
- No pumps or compressors: compact design and no mechanical noise
- No moving parts: minimal maintenance and long term reliability.

The complete system consists of five main parts identified in Figure 1.

1. Sample flow unit (comprising a rigid leg, flexible stainless steel transfer tube, cold head and nozzle)
2. Shield flow unit (second rigid leg and polythene tube)
3. Cryojet stand (for precise alignment of the jet)
4. Liquid nitrogen storage dewar with Cryojet top fitting
5. Cryojet controller (electronics).

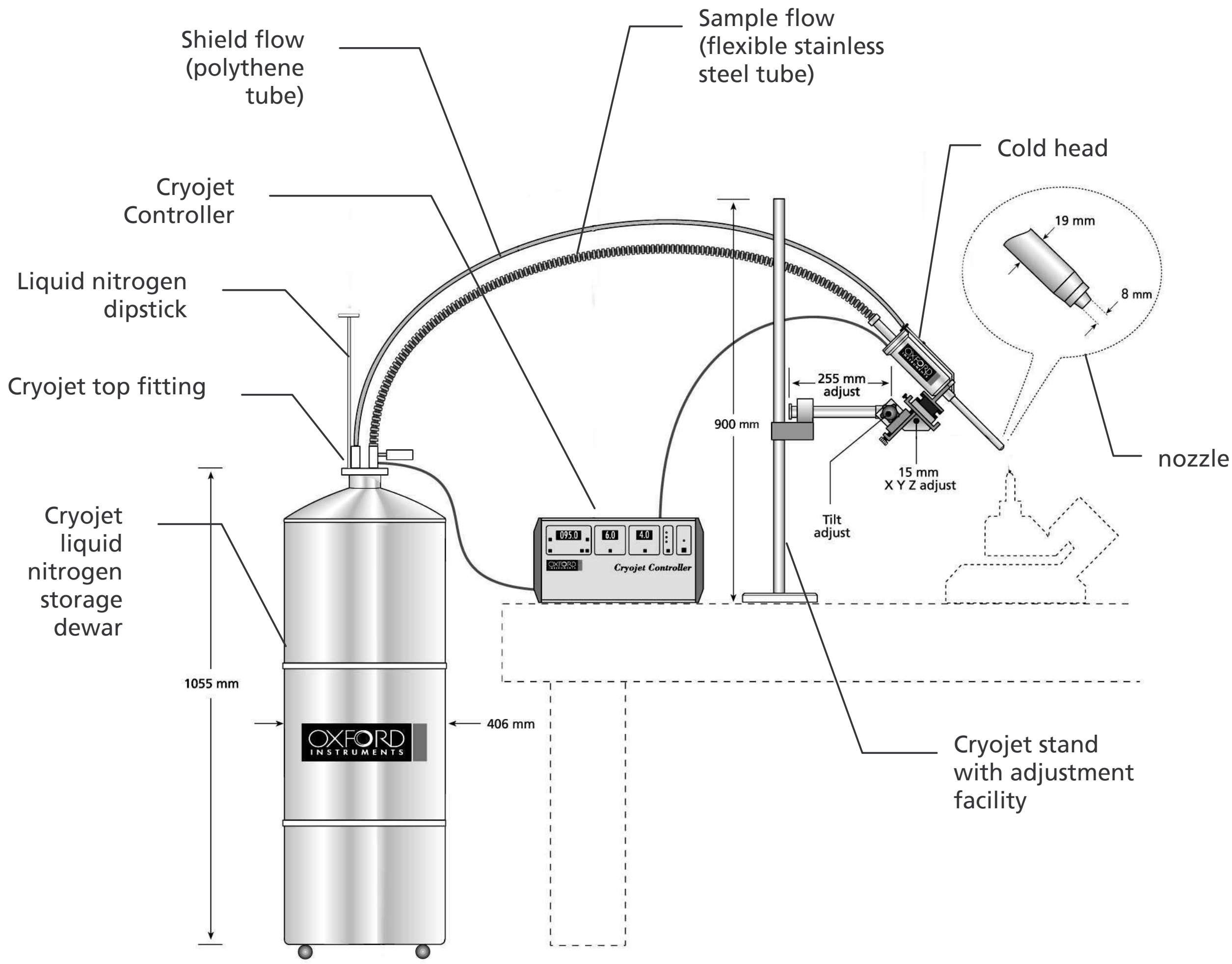


Figure 1 Overall view of Cryojet system

2.1 Sample flow unit

The rigid leg of the sample flow unit is immersed in the liquid nitrogen in the storage dewar. A heater at the bottom of the dewar leg boils nitrogen. The cold gas passes up the flexible stainless steel vacuum-insulated transfer tube into the cold head. The cold head contains a heat exchanger and heater and a temperature sensor just inside the nozzle jet. The gas then passes through the nozzle on to the crystal, forming the **sample flow**. The temperature of the sample flow can be regulated to any value between about 90 - 300 K for the CryojetXL and 90 – 490 K with a CryojetHT.

Outside the nozzle there is a temperature gradient in the sample flow both along and transverse to the jet axis. The characterisation of this profile permits the calibration of sample temperatures in the final experimental configurations.

Spatial temperature profiles for the CryojetHT are shown in Figure 2 and Figure 3.

A vacuum valve is situated at the top of the rigid section of the transfer tube to allow the Outer Vacuum Chamber (OVC) to be pumped to maintain the required thermal insulation.

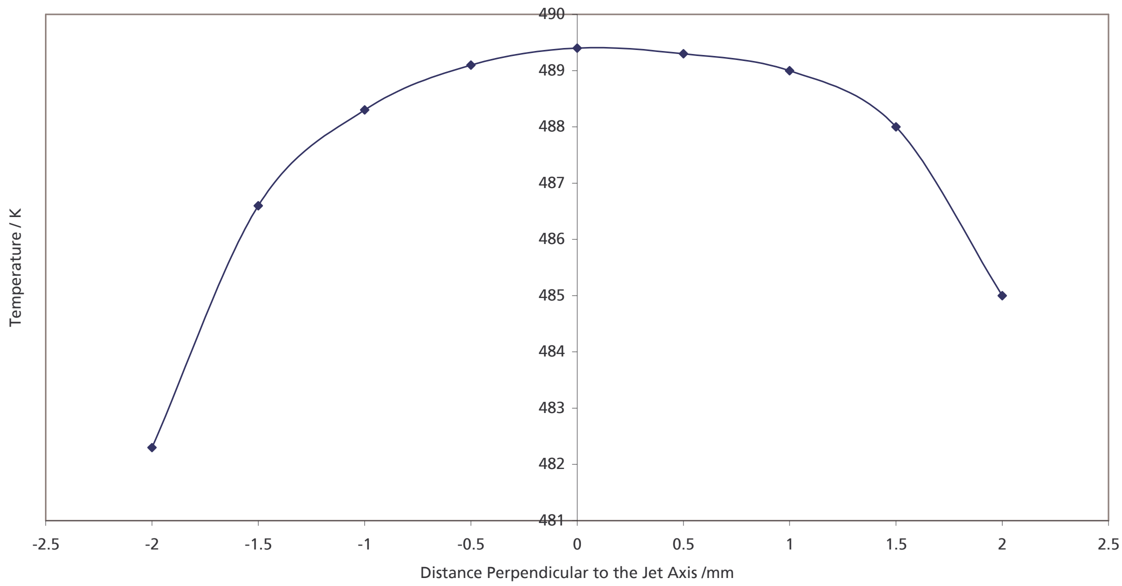


Figure 2 CryojetHT Temperature Profile Perpendicular to the Jet Axis

(Displayed Temperature 500 K, 5 mm from the nozzle)

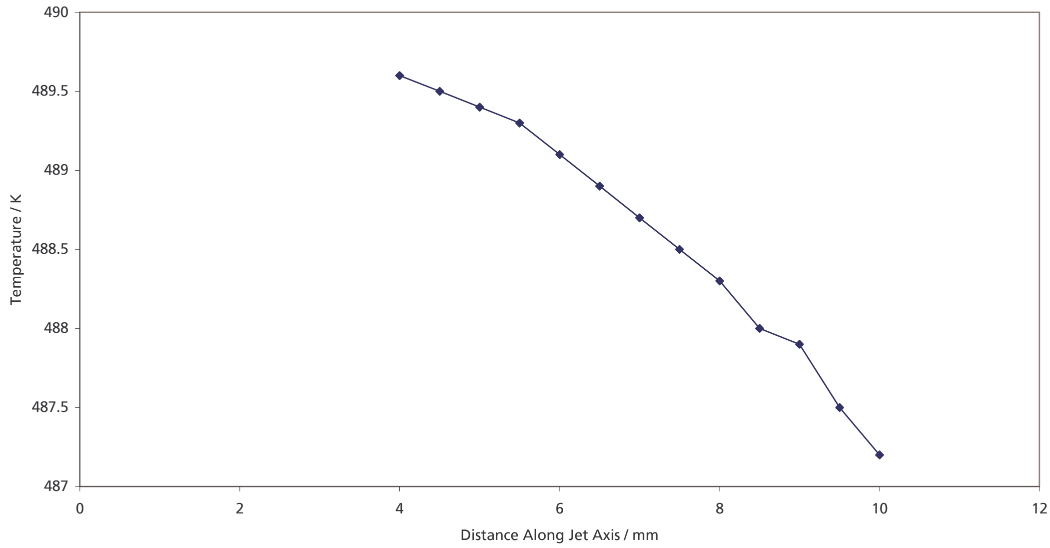


Figure 3 CryojetHT Temperature Profile Along Jet Axis

(Displayed Temperature 500 K)

2.2 Shield flow unit

The rigid leg of the shield flow unit is also immersed in the liquid nitrogen in the storage dewar. A heater at the bottom of the dewar leg boils nitrogen. At the top of the unit there is a heat exchanger, with a heater and temperature sensor, which heats the nitrogen to room temperature. The nitrogen flows through the central stub of the shield unit to the cold head via a length of polythene tubing. This **shield flow** then flows through the outer nozzle, preventing atmospheric water vapour from icing up the sample or the nozzle.

2.3 Cryojet Stand

The Cryojet is supported by a 3-axis translation and tilt stand (Figure 4) to provide precise jet control and optimum experimental geometry.

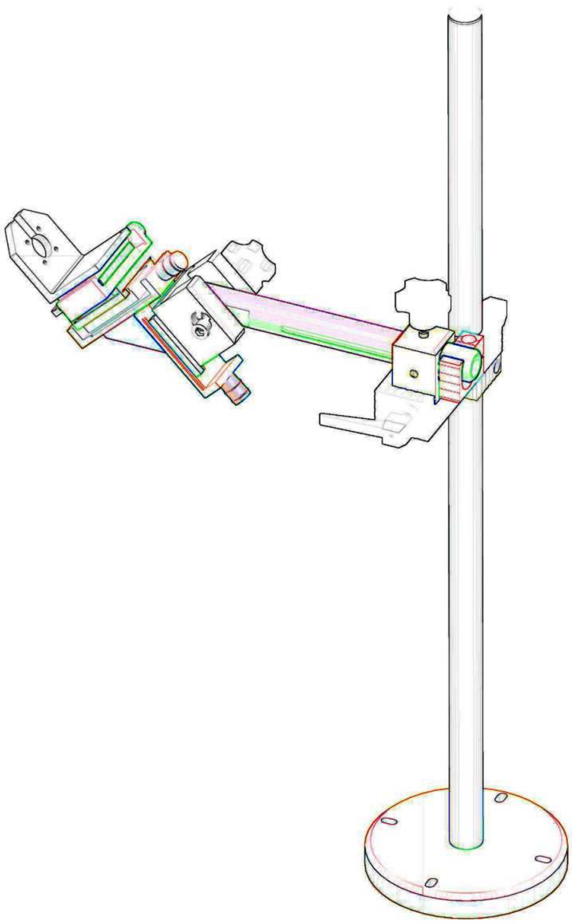


Figure 4 Cryojet adjustable stand

2.4 Liquid nitrogen Storage Dewar

A 75 litre storage dewar is used to store the liquid nitrogen used for the sample flow and shield flow. The dewar has a top fitting with five ports (see Figure 5).

- 51 mm hole: sample flow unit dewar leg
- 38 mm hole: shield flow unit dewar leg
- Non-return vent valve

- 13 mm bent tube: for filling the dewar with liquid nitrogen
- Stepped straight tube: for fitting a nitrogen level probe or pressure relief valve.

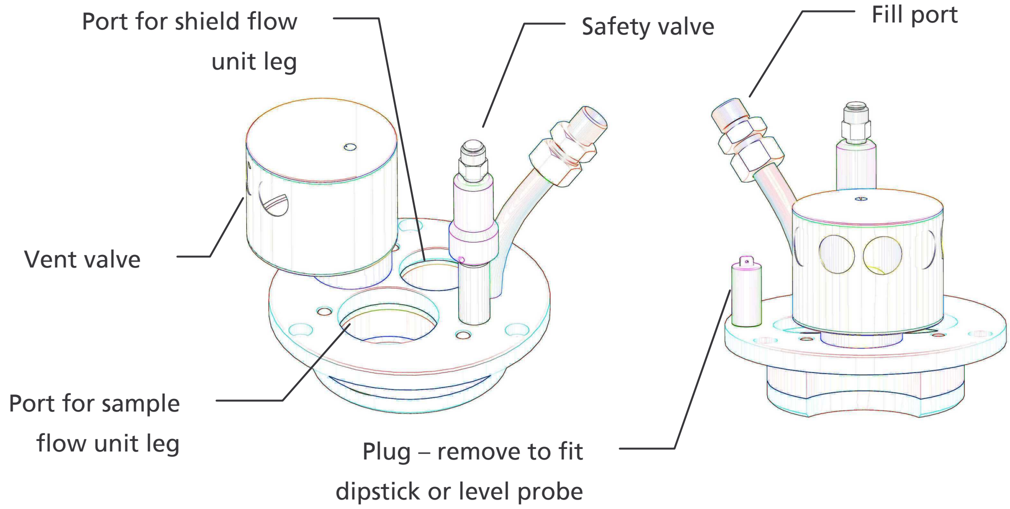


Figure 5 Top fitting for liquid nitrogen dewar

2.5 Cryojet Controller

The Cryojet Controller provides

- A temperature readout of the sample jet temperature measured using a platinum resistance sensor
- An 80 W heater supply driven by a three term (PID) temperature controller to control the sample jet temperature at a desired value
- A 40 W heater supply to deliver liquid nitrogen from the dewar for the main sample jet at a flow rate of up to 10 litres per minute
- A 40W heater supply to deliver liquid nitrogen from the dewar for the gas shield at a flow rate of up to 10 litres per minute
- A 40W heater supply driven by a proportional temperature controller to stabilise the shield gas temperature close to room temperature
- A detection circuit for low liquid nitrogen level in the storage dewar
- Interlocks to prevent operation of the heater supplies when abnormal conditions are detected
- A computer interface allowing monitoring and control of the above functions.

- A view of the front panel is given in

Figure 6 together with the main functions. Full details are in the Cryojet Controller Technical Handbook, included together with this manual.

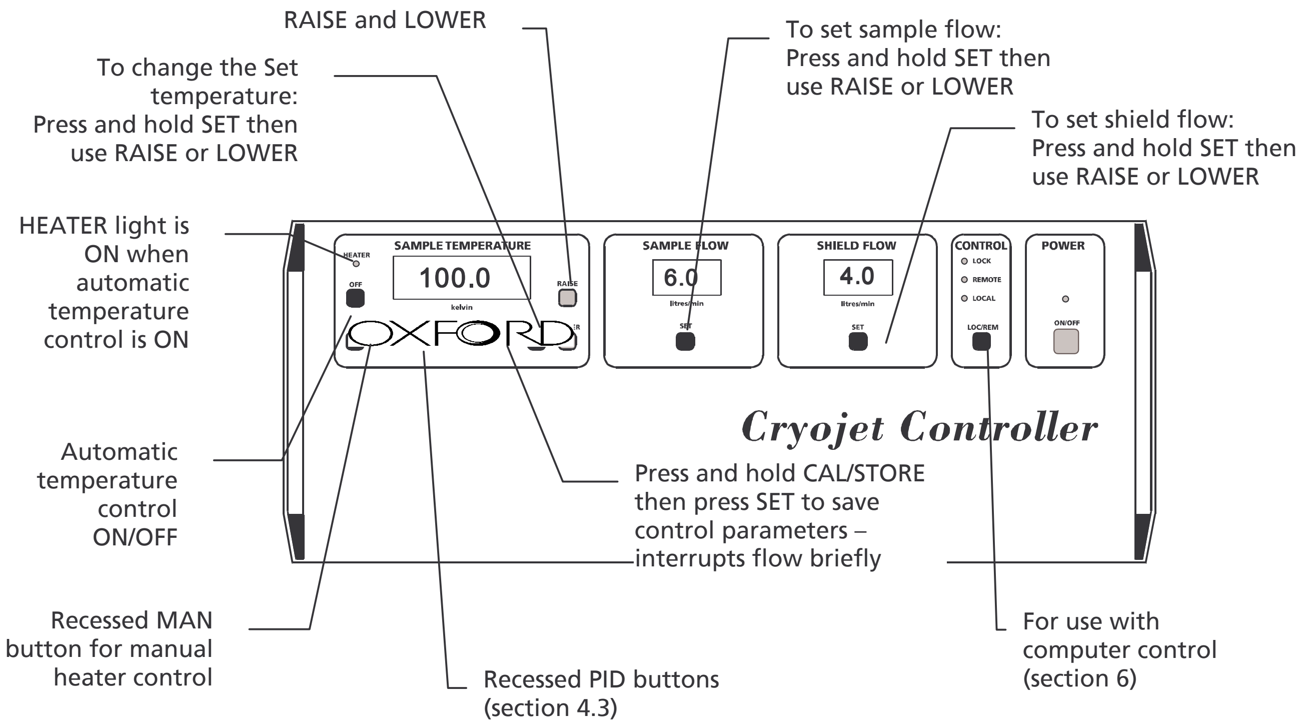


Figure 6 Cryojet Controller front panel

3 Unpacking and preparation

3.1 Unpacking the system

Carefully remove the Cryojet and all the accessories from the packing cases, and check the packing list to make sure that you have found all of the components. Examine the system to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford Instruments immediately.

A complete system consists of:

- Sample flow unit
- Jet alignment tool
- Stand with xyz adjustable stage
- Shield flow unit
- Cryojet Controller
- Electrical cables with the following labels:
  cold head
  shield unit
  dewar leg (and LN_2 level cut-off)
- Liquid nitrogen storage dewar including
  fill port adaptor with flow restrictor
  1/2” flare fitting (for customers in the USA only)
- Liquid nitrogen dipstick
- Dewar top fitting and 3 M6 bolts
- Pressure relief valve
- Polythene tubing (1.5 m)
- Spares kit, including:
  Allen keys (hexagonal wrenches): 8 mm, 6 mm
  Tie-wraps

The following components are options that will only have been supplied if ordered separately.

- Liquid nitrogen autofill system
- Liquid nitrogen hose
- Liquid nitrogen level meter and probe
- Pressurised liquid nitrogen supply dewar
- Dewar base with wheels
- High vacuum pumping system and lines

3.2 Preparing the system for operation

These instructions assume that the liquid nitrogen dewar is empty and completely dry.

3.2.1 Liquid nitrogen dewar

a) Check that the dewar top fitting (Figure 5) is secured to the dewar using the three M6 screws provided

b) Loosen the three small screws that hold the top fitting together; the sample flow leg and shield leg will not slide into the dewar top fitting if these screws are tight.

c) Remove the vent valve by pulling firmly upwards.

d) Position the liquid nitrogen dewar about 1 metre from the intended position of the sample.

3.2.2 Stand

a) Set up the stand approximately in the intended experimental position.

b) Slide the stage assembly on to the stand and clamp in position using the lever (Figure 4 and Figure 7). Note that the lever can be rotated to any convenient position by pulling it to disengage the lever from the clamping mechanism.

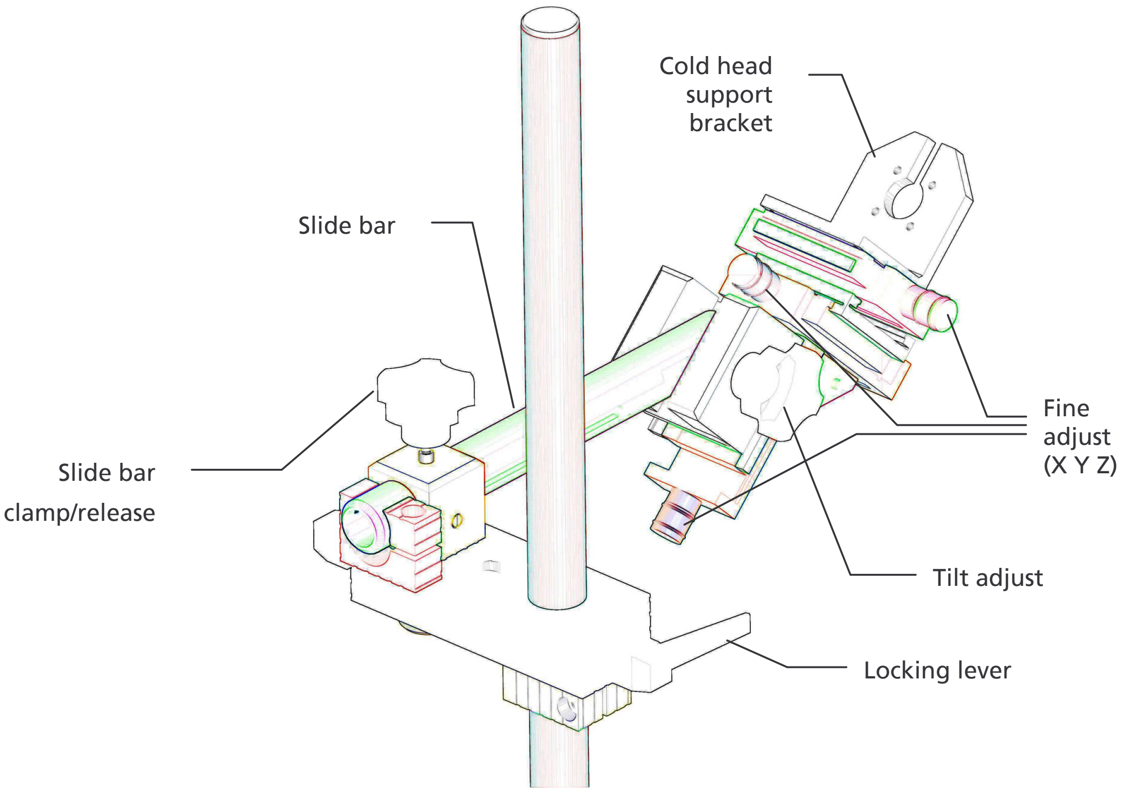


Figure 7 Cryojet stand details

3.2.3 Set up the Sample flow unit

a) Lower the sample flow unit rigid leg through the widest hole in the dewar top fitting to the bottom. The wide part of the dewar leg is a tight fit through the 'O' rings in the top fitting.

b) Slide the nozzle of the cold head through the hole cold head support bracket (Figure 7) and secure using four caphead screws (supplied) at the base of the nozzle. Use the pre-existing bend of the flexible section when you arrange the cold head in position. The shield tube running down the side of the cold head must fit through the slot in the side of the holder. If the shield tube does not line up with the slot, rotate the entire cold head until it does.

c) Arrange the nozzle approximately in its intended operating position.

d) Clamp the tilt adjuster in place using black knob. You may find it helpful to use the 8 mm Allen key provided. To move the bracket assembly up or down the post smoothly, support the weight of the cold head by putting your hand under the stage assembly near the cold head.

3.2.4 Pump out the sample and shield OVC

The OVC of both the sample flow unit and shield unit have to be pumped to high vacuum to make sure that they provide the required thermal insulation.

Both are pumped in the factory, but this needs to be repeated occasionally and is certainly recommended for first time use. When the system is new, all of the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This does not mean that the system is leaking.

Pump out the OVC using a two-stage rotary pump that has a base pressure of about 10^{-3} mbar or a diffusion pump or turbo pump.

For further information see section 5.6.

3.2.5 Set up the Shield unit

a) Pass the rigid leg of the shield unit through the next largest hole in the dewar top fitting all the way to the bottom of the dewar. It is also a tight fit in the 'O' rings in the top fitting.

b) Connect the fitting on the top of the rigid leg to the fitting on the cold head using the polythene tube supplied. To connect, just push the polythene tube into the fitting until it stops.

c) Tighten the three small screws on the dewar top fitting.

3.2.6 Fill the liquid nitrogen dewar

For this step you require a pressurised liquid nitrogen dewar containing at least 100 litres LN_2. If you have an Oxford Instruments autofill system, go to section 7.

You need to fit a suitable hose from your pressurised LN_2 supply dewar to the (unpressurised) Cryojet dewar.

Warning: **Plastic hoses, including polythene, become brittle when cold, and are not recommended.**

If you have an Oxford Instruments nitrogen fill hose, fit it as follows.

a) Remove the brass adaptor from the hose, and screw it into the liquid outlet of your pressurised dewar, sealing the joint with PTFE tape.

b) Screw the hose on to the brass adaptor using two spanners.

c) Attach the other end of the nitrogen hose to the dewar top fitting, using the stainless steel compression fitting that is already fitted to the fill port. Slide the end of the hose on to the fill port, and tighten the nut securely using two spanners.

Note: The fill port is fitted with a stainless steel coupling and a flow restrictor. This coupling has a 3/8" BSP parallel thread, with a 60° flare female sealing surface. If necessary, remove the top part of the coupling. Systems delivered in the USA also have an adaptor to convert this coupling to a 1/2" flare fitting (unless the system includes a nitrogen fill hose).
If you are using a level probe:

d) Remove the screw in the side of the probe.

e) Remove the pressure relief valve from the dewar top fitting and insert the probe through the stepped tube. Push down firmly until the level probe is as far down as it will go, and does not wobble. You may clamp the probe in position using the screw in the side.

If you are not using a level probe:

f) Remove the screw in the side of the pressure relief valve.

g) Push the pressure relief valve is firmly into place on the dewar top fitting and, if desired, clamp it into position using the screw in the side.

h) Push the vent valve (Figure 5) firmly into position on the vent tube until it stops. This is a tight fit, as the vent valve is sealed by an 'O' ring on to the vent tube.

i) Check the pressure in the supply dewar; transfer will be smoother if this is less than 1 bar.

j) Open the valve on the supply dewar slowly and allow liquid nitrogen to flow to the Cryojet dewar. If a level probe is fitted you may monitor the transfer process.

Caution: **When the dewar is full liquid nitrogen will spurt from the vent valve - stay well away.**

k) Close the valve on the supply dewar slowly to stop the fill.

Note: You need not fill the dewar to the top but make sure there is at least 10 cm of liquid nitrogen before running the Cryojet.

3.2.7 Set up the Cryojet Controller

a) Check that the voltage selector on the rear panel of the Cryojet Controller is correctly set for the supply voltage.

b) Connect the three cables to the back of the controller, matching the labels on the D-plugs with the labels on the controller.

c) Fit the round connectors on the cables (4 pin and 10 pin) to the sockets on the cold head, matching the colours (Figure 8). These connectors also have red dots - line up the red dot on the plug with the red dot on the socket.

d) Use the plastic tie-wraps to tidy up the system by attaching the tubes and cables together.

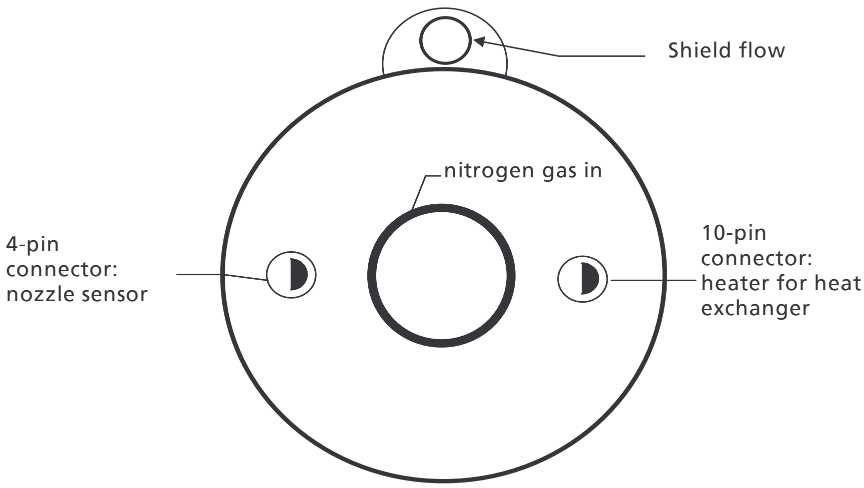


Figure 8 Schematic of cold head top flange

3.2.8 Aligning the nozzle

The nozzle should be placed as close as possible to the sample to prevent icing. The limitation on this spacing depends on the diffraction angle required and the angle between the nozzle and the incoming x-ray beam. In almost every case the nozzle should be 5 - 10 mm from the sample.

The jet should be centred on the sample as accurately as possible. This can be achieved in two ways.

The first is to adjust the nozzle first so that the sample is in the plane of the nozzle. This makes it easier to centre the nozzle. Then move the nozzle back to the desired position using the micrometer stage (Figure 9).

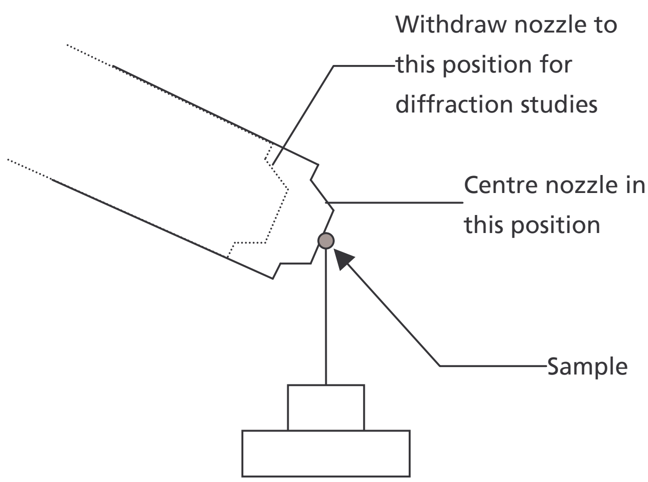


Figure 9 Simple alignment technique

Another method is to use the Oxford Instruments alignment tool. Slide the device into the nozzle and tighten the knob to clamp it in place. Place a sample mount with no sample on the goniometer. Adjust the nozzle so that the sharp tip is almost touching the place where the sample would be. Remove the sample mount. Loosen the knob and remove the alignment tip. Then place the sample on the goniometer. If required, move the cold head forward using the micrometer.

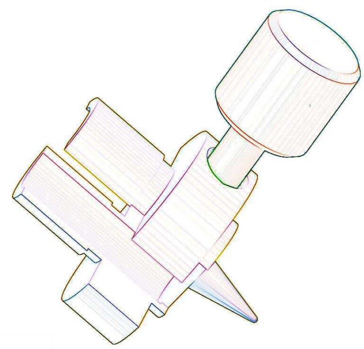


Figure 10 Alignment tool

3.2.9 Controlling the flow rate

The flow rates of the sample flow and shield flow are shown on the display in l/min (as if they were measured at atmospheric pressure and 293 K). To change the flow to any value from 0 to 10 l/min, hold down the SET button underneath the appropriate display and press RAISE or LOWER.

Suggested sample flow rate for 100 K	6 l/min
Suggested sample flow rate for 90 K	10 l/min
Suggested shield flow rate	5 l/min

For a fast cool-down, set the sample flow rate to 10 l/min initially, and then reduce it to the desired value when the temperature is close to the set temperature.

3.2.10 Controlling the jet temperature

There are three modes of operation: Automatic, Manual and Heat Exchanger Off. The light labelled "Heater" is on if the controller is in Automatic mode, or if it is Manual mode with non-zero voltage supplied to the heat exchanger.

Automatic

In this mode, the temperature of the jet is controlled automatically.

- To **display** the **Set Temperature** press and hold the SET button.
- To **change** the **Set Temperature**, hold down SET while using RAISE and LOWER.
- To **display** the **voltage** being supplied to the heat exchanger at a given moment, press and hold the ON button on the left-hand side of the front panel.
- To put the controller into **Automatic** mode, press the ON button on the left-hand side of the front panel.

To select Automatic mode, the sample flow must be at least 2 l/min. If a flow has been selected but after a couple of minutes no flow can be felt, switch the heat exchanger **off** and refer to section 8.

Manual

In this mode, a fixed voltage is supplied to the heater in the heat exchanger. It is not recommended for routine operation.

- To select **Manual** mode, press the recessed button MAN. The default voltage is zero.
- To **display** the **voltage** supplied to the heat exchanger, press and hold MAN.
- To **change** the **voltage** supplied, press and hold MAN, and use RAISE and LOWER.

Heat Exchanger Off

In this mode, no voltage is supplied to the heater in the heat exchanger. This mode will give the lowest possible temperature, but there will be some temperature fluctuations.

To set this mode, press the OFF button on the left-hand side of the front panel.

4 Controller

Full details are in the Cryojet Controller Technical Handbook, included together with this manual.

4.1 Choice of default conditions on Start-up

There are two start-up conditions; “Auto-Start” and “Non-Auto Start”. Auto-Start is particularly useful if there is a possibility of brief power failures when the Cryojet is running unattended. **Do not select Auto-Start unless you are sure that there will be liquid nitrogen in the dewar when the controller is switched on.**

Start-up condition	Properties	To set up
Non-Auto Start	Gas flows equal zero Heat exchanger is Off	1. Press the OFF button on the left-hand side of the front panel. 2. Press and hold the recessed button CAL/STORE using a pointed object and press the SET button in the left-hand group of buttons. The letters “Stor” will appear on the display. This will interrupt the flow briefly.
Auto-Start	Gas flows non-zero - can be set to any desired values Heat exchanger in Automatic mode	1. Set the desired SET temperature. 2. Set the desired start-up gas flows. 3. Set Automatic mode by pressing the ON button on the left-hand side of the display. 4. Use the STORE function: press and hold the recessed button CAL/STORE using a pointed object and press the SET button in the left-hand group of buttons. The letters “Stor” will appear on the display. This will interrupt the flow briefly.
When the controller is switched on it will display the message “Auto” for a second or two if it is in Auto-Start mode.		

4.2 Low liquid nitrogen level cut-out

A sensor attached to the sample flow unit dewar leg detects the presence or absence of liquid nitrogen. If the liquid level is below about 6 cm from the bottom of the dewar, all the heaters in the system will switch off automatically and the message “Lo N” will be displayed on the Cryojet Controller.

To restore normal operation after you have refilled the dewar, you must switch the controller off and on again.

4.3 Setting P, I and D control terms

The PROPORTIONAL, INTEGRAL and DERIVATIVE control terms may be displayed and set by means of the recessed P, I and D buttons. Use a pointed object to press these buttons. The system is already set with optimised values of these parameters: it should **not** be necessary to change them, or to read the rest of this section. The default values are P=20, I=2.5, D=0.5

P indicates the PROPORTIONAL BAND in Kelvin to a resolution of 0.001 K

I indicates the INTEGRAL ACTION TIME in minutes, covering a range of 0 to 140 minutes in steps of 0.1 minute.

D indicates the DERIVATIVE ACTION TIME in minutes, covering a range of 0 to 273 minutes (Though values beyond 70 minutes are unlikely to be required in practice.).

In North America, a different terminology exists for 3-term control.

PROPORTIONAL BAND is replaced by its reciprocal, GAIN.

INTEGRAL ACTION is replaced by RESET. This may either be specified as a time (as for integral action) or as its reciprocal, "REPEATS PER MINUTE".

DERIVATIVE ACTION is replaced by RATE. Again this may be specified as a time or as repeats per minute.

RAISE and LOWER may be used to vary the control terms whilst in LOCAL control. When you have changed the values, carry out a STORE command as follows: Press and hold the recessed button CAL/STORE, and press SET. This will store the new values in non-volatile memory. (NB This will interrupt the flow momentarily. If a STORE command is executed when the controller is in Automatic mode, then this will also set the “Auto-Start” mode - see 4.1.)

The main purpose of DERIVATIVE action is to reduce overshoot, when approaching a new set temperature. For most systems derivative action will not be required and may be left at zero. (Hold LOWER pressed for a second after 000.0 is displayed to ensure that there is not a small residual setting of less than 0.05 mins ,which will show as zero).

The P and I controls should not normally be set to zero, since this would correspond to ON/OFF control.

The following procedure gives a good rule-of-thumb for setting the controls to a value that is close to optimum.

a) Set I for a time much longer than the expected response time of the system.

b) Set D to zero.

c) Select AUTO and reduce P until the temperature starts to oscillate above and below some mean value (not necessarily the set point).

d) Time the period of oscillation (in minutes). This is a measure of the response time of the system.

e) Set I to a value approximately equal to the response time. Then increase the P setting to a point where oscillation just ceases. Note the value of P at this point, then set it to approximately double this value. This gives a good starting point for the P and I control terms.

f) Test how the system responds to step changes in the SET point and modify the P and I settings for a reasonably fast response without excessive overshoot.

g) If overshoot remains a problem following a large step change in SET, try the effect of adding some DERIVATIVE action. A good initial setting is half to one third of the system response time measured above. This will probably require P to be re-optimised for best results.

h) When optimising P, I and D the aim should be to achieve the lowest values of all three terms, consistent with no oscillation and an acceptably small amount of overshoot. This will give the fastest response for the system.

When adjusting the control terms remember that reducing P increases the controller gain. This can cause some confusion when the concept of PID control is first encountered.

5 Operation

5.1 Suggested operating conditions

Always keep at least 10 cm of liquid in the dewar when the Cryojet is running, as lower levels could impair the performance. **Never allow the dewar to become empty when the Cryojet is running.**

Keep the plastic cap, the vent valve and the pressure relief valve (or level probe) in place on the dewar top fitting, to prevent water entering from the atmosphere.

A sample flow rate of about 6 l/min is recommended for most applications. The lower the flow rate, the higher the base temperature will be. Lower flow rates may be suitable if very low temperatures are not required, provided the sample can be placed very close to the nozzle. For a fast cool-down, set the sample flow rate to 10 l/min initially, and then reduce it to the desired value when the temperature is close to the set temperature.

A flow rate of 4 - 5 l/min is usually sufficient for the shield flow - lower flow rates can be used if they are sufficient to stop the sample and nozzle icing up.

Note: **Users of CryojetHT**

CryojetHT has a maximum nozzle temperature of 500 K. This temperature can be achieved at any sample flow rate up to 8 l/min (or more, depending on the mains power voltage).

If the CryojetHT is used above room temperature for long periods, it may be necessary to pump the vacuum of the sample flow leg more frequently than for a standard jet, see section 5.6.

5.2 Filling the dewar

The Cryojet can be run continuously for as long as required. The dewar can be refilled while the Cryojet is running.

Fit the liquid nitrogen hose from your supply dewar on to or into the fill tube. Make sure it is held in place so that it will not be blown free by the pressure of nitrogen during the fill. Use a low pressure in the supply dewar - preferably less than 1 bar. Open the valve on the supply dewar slowly, as the sample flow temperature may be temporarily affected by an increase in pressure in the dewar. Close the valve on the supply dewar slowly to stop the fill.

5.3 Using the dipstick to measure the level of liquid nitrogen

A dipstick (a long plastic rod) has been provided as a simple way of estimating the level of liquid in the dewar. (A digital level meter and probe are also available - see separate manual if these have been supplied.) Slowly lower the dipstick into the dewar through the fill port until it hits the bottom, and wait a couple of seconds. Then pull it out and wait a few seconds for frost to form on the surface. The length of the frosted section is the depth of liquid in the dewar. Do not touch the cold part of the dipstick with your bare hands.

For depths between 10 cm and 80 cm, the dewar holds about 0.98 litres for every additional centimetre of liquid (see calibration chart below). A flow of 1 litre/min of gas corresponds to a consumption of 0.086 litre/hour of liquid, i.e. about 0.088 cm/hour. In addition, there is a boiloff from the dewar of about 0.10 cm/hour. If the level of liquid nitrogen in the dewar falls too fast, the vacuum in the dewar may be poor. Return the dewar to Oxford Instruments, or buy a pump-out adapter from Oxford Instruments Direct. (See the end of this manual for contact details.)

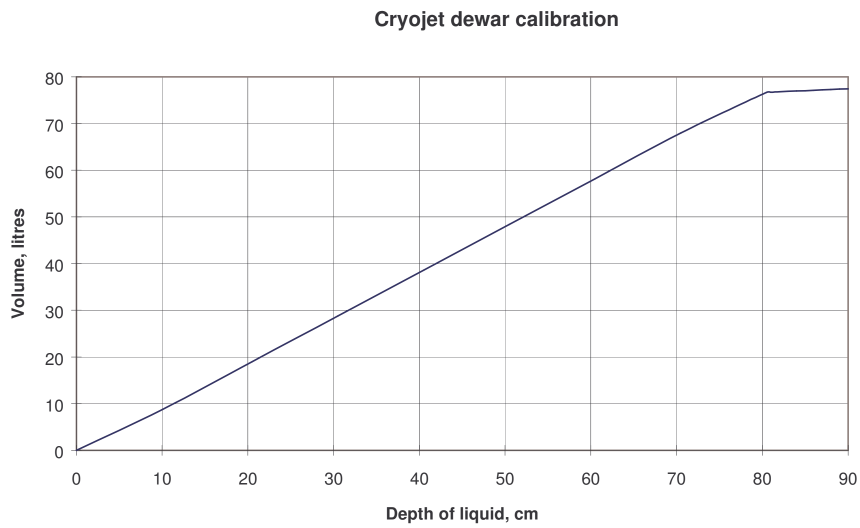


Figure 11 Cryojet dewar calibration

5.4 Short term shut down

The system should be switched off whenever it is not in use. Although the cold head is designed to prevent ice or water building up you are advised to:

a) Block the inner nozzle with a loose-fitting rubber stopper and leave the cold head to warm up naturally or

b) Set the temperature to 300 K and allow the displayed temperature to reach room temperature before switching off. To speed this up, reduce the sample flow to about 3 l/min.

(If the Cryojet is running above room temperature this precaution will not be necessary.)

5.5 Long term shut down

If you do not use the Cryojet for several days the dewar can warm up. It is then possible for water to collect at the bottom. This could block the system when it freezes, although the dewar legs have been designed to prevent this. This can be prevented in several ways:

a) Before allowing the dewar to warm up, raise both dewar legs. To do this, loosen the three small screws in the dewar top fitting. Raise the shield unit and the sample flow unit dewar leg by a centimetre or two, then clamp them in place by tightening the three small screws. Leave them in place when you later refill the dewar. (To maximise the available time between refills, you may want to lower the legs to the bottom later, but it is easier to do this when the dewar top fitting is no longer cold from the filling process.)

b) Before refilling a warm dewar, attach a dry air or nitrogen supply to the outer nozzle. Gas will then flow backwards through both the sample flow unit and the shield flow unit, blowing out any water from the holes at the bottom.

If either the sample flow dewar leg or the shield unit is removed from the dewar, then before putting it back check that the small holes in the bottom are clear of ice, using a stiff wire such as a paper-clip.

It is not possible to remove the shield unit from the dewar top fitting when it is cold. To dismantle the system when cold, remove the entire system including the dewar top fitting from the dewar in one unit.

5.6 Pumping the Outer Vacuum Chamber (OVC)

The OVC of the sample flow unit should be pumped if any of the following symptoms appear:

- The sample flow unit feels cold to the touch, or condensation or frost appears. Some condensation or frost is normal on and just above the dewar top fitting.
- Water or ice condenses on the outside of the rigid dewar leg. Some condensation is normal on and just above the dewar top fitting.
- The jet does not reach the desired temperature, or the voltage supplied to the heat exchanger heater is lower than that given for the relevant temperature in the test results appended to this manual.
-

If the Cryojet has not been used for a month or more and the dewar has warmed up, it is often advisable to pump the vacuum in the sample flow unit before starting to use it again.

Both OVCs contain a sorb at the bottom of the dewar leg. Ideally they should be pumped overnight, and during pumping the dewar leg should be heated to about 100 °C to make sure that the sorb outgasses. You can do this by setting the appropriate flow rate to 8 l/min (not more) with the dewar leg out of the dewar. You must support the dewar leg so that the bottom end is not touching anything that could melt. Do **not** insulate it in any way, as this could cause overheating. Before you set the flow rate you must over-ride the low nitrogen level cutoff by removing the plug from the socket labelled "Low nitrogen level cutoff" on the back of the controller.

Caution Remember to re-connect the Low nitrogen level plug to the level meter.

Do not bake the flexible transfer tube or cold head as these contain plastic components.

It is also possible to pump the OVC while the Cryojet is operating, although the vacuum will not last as long in this case, as the sorb has not been outgassed.

5.7 Calibration of Sample Temperature

Precise estimation of the temperature of a real sample in the cold jet is not straightforward, as it will be affected by the size and location of the sample, the orientation of the nozzle, and by heat conduction along the sample support. Contact Oxford Instruments for calibration information.

5.8 Stability

In normal operation the displayed temperature is stable to ± 0.1 K. Small fluctuations in temperature will occur when the dewar is refilled.

5.9 Icing of Crystal Support and Goniometer

The shield flow is designed to prevent icing of the sample and nozzle. A slight build-up of snow is likely on the crystal support and goniometer. In most cases no action need be taken, provided that the incoming beam can be collimated to exclude any ice on the crystal support. Increasing the shield flow rate to 10 l/min for a minute or so may help to remove frost from the crystal support.

6 Control from a Computer

The Cryojet Controller can be controlled from a computer using the RS232 interface or the optional GPIB (IEEE-488) interface.

In either case, you can use the ObjectBench for Cryojet software provided. Run Setup.exe from disk 1 (of 4) and follow the installation instructions. This software includes on-line Help.

For many users this software will have all the required functionality. Users who wish to write their own software will find further information in the Cryojet Controller manual.

6.1 RS232 Connection Hardware

The bi-directional serial data link from the computer is connected via a 25 way D-socket on the rear panel.

A unique feature of the Cryojet Controller and other Oxford Instruments NanoScience products is the ability to connect a number of instruments simultaneously, to a single RS232 port on a computer and to control each one independently. This is done by means of an ISOBUS cable which carries a single MASTER connector (25-way D socket) and up to eight, daisy-chained SLAVE connectors (25-way D plugs). Each slave connector incorporates full optical isolation so that the slaves are all isolated from the master and from each other. The slave connectors draw their power from the individual instruments, via the DCD signal on pin 8. The master connector may draw its power from either DTR or RTS signals from the computer.

ISOBUS hardware is available from Oxford Instruments Direct.

To use ISOBUS, a special communication protocol is required, which is part of the command structure of Oxford Instruments' products and is described in the Cryojet Controller manual.

6.2 GPIB (IEEE-488) Connection Hardware

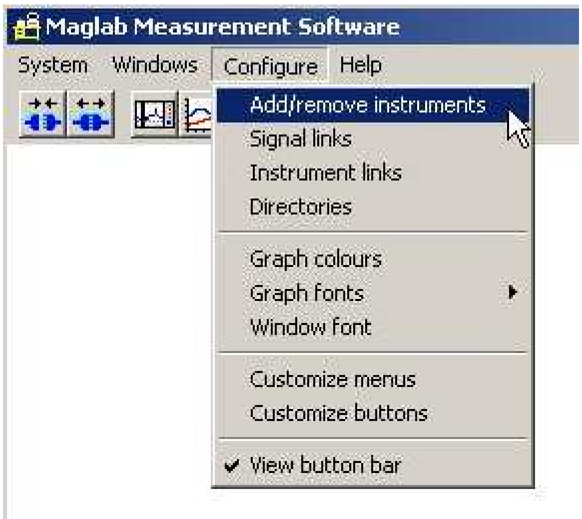
If the optional GPIB interface is fitted, connections to the GPIB are made via a standard 24 way GPIB cable, conforming to the standard IEEE-488.1. Connections should be made using a standard GPIB cable. If you wish to connect both a Cryojet controller and a level meter to a GPIB port on a computer, use a “GPIB Gateway” - contact your sales engineer or Oxford Instruments Direct.

Note **GPIB connections should never be made or broken whilst the controller or any of the instruments connected to the Bus are powered up. Failure to observe this precaution can result in damage to one or more instruments.**

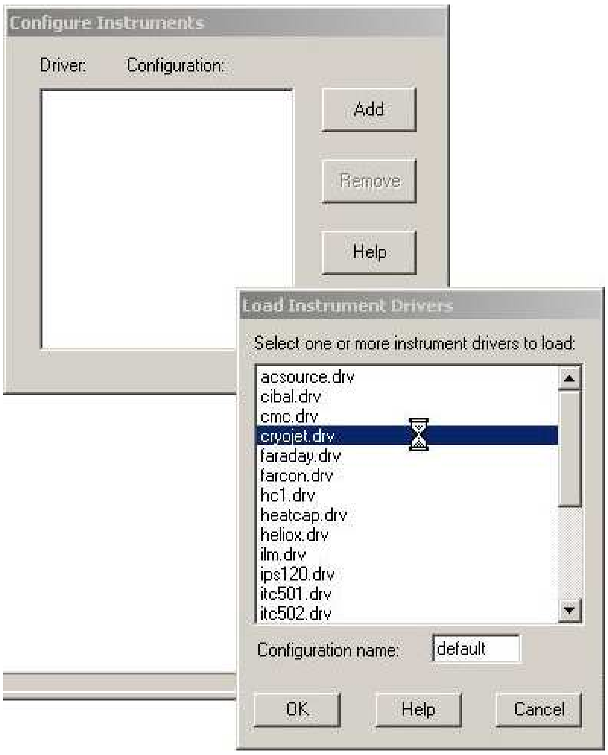
6.3 Control Using the Oxford Instruments ObjectBench software package

ObjectBench is provided with the Cryojet. This section describes how to install and run the Cryojet driver in ObjectBench. For other information, see the ObjectBench manual.

6.3.1 Installation of Cryojet driver in ObjectBench



Select **Add/remove instruments** in Configure menu.

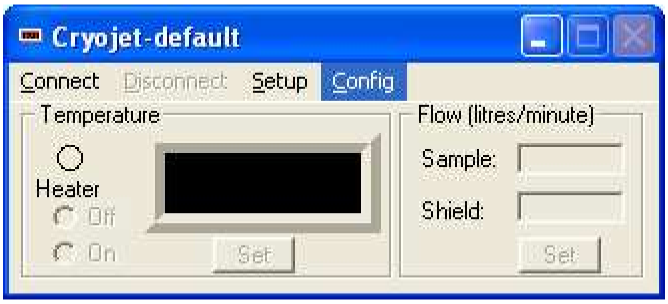


Load instrument driver **cryojet.drv** then click **Add** button.

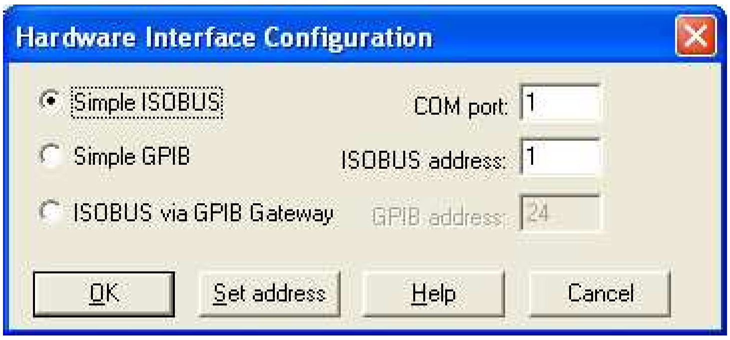
There is no need to change the **Configuration name** (default) unless you have two Cryojet Controllers connected.

Cryojet driver window appears as shown.

6.3.2 Setting up the Cryojet driver



Select **Configure** menu and choose **Interface**

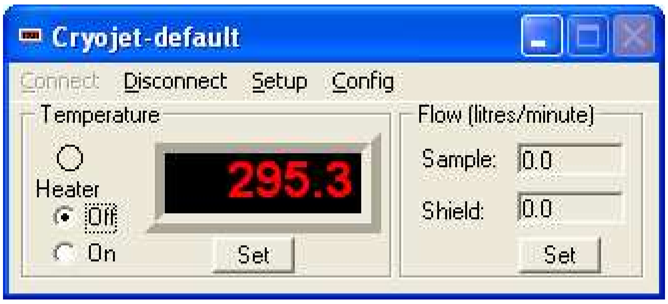


Select the type of communication.

Select the COM port (RS232).

Select ISOBUS or GPIB address. Follow instructions. Choose defaults if no other instruments present.

If an address is changed press and hold CAL/STORE on the Cryojet Controller then press SET to store the address.



Click **Connect** to connect the instrument to the software. The nozzle temperature is displayed.

6.3.3 Control of the Cryojet

The Cryojet instrument driver in ObjectBench is intended to be self-explanatory.

Control of the instrument may either be LOCAL from the front panel, or REMOTE via the computer interface. The LOC/REM button may be used to switch between LOCAL and REMOTE.

When LOCK is lit, the instrument is locked into either local or remote control and the LOC/REM button has no effect. At power up, the controller is locked in LOCAL.

When the controller is in REMOTE, many of the front panel controls are disabled. Those controls that only affect the display will still work, but those that could change the operation of the instrument will not. If LOCK is lit whilst in REMOTE, all the front panel controls are inoperative.

Note that there is no indication on the instrument driver as to whether the controller is in LOCAL or REMOTE. If the controller is in LOCAL, it will not respond to computer commands. To rectify this, use the DISCONNECT and then the CONNECT command in the instrument driver. This will put the controller into REMOTE.

6.3.4 Restoring Standard Calibration Parameters

If any of the set-up parameters are changed by mistake, or the EEPROM (non-volatile memory) becomes corrupted, the controller can be restored to the state in which it left the factory, by using the back-up disc provided, and ObjectBench software. Use the following sequence of operations and menu selections.

This feature is not available from the Cryojet instrument driver. Instead, you must add the instrument driver called ITC503.drv (using Configure / Add/remove instrument / Add).

DISCONNECT the Cryojet driver if it is connected.

If the controller ISOBUS address is not the default value (1), or you are using a COM port (serial port) other than COM1 on your computer, set up the ITC503 driver to the correct address and COM port using Configure / Interface.

Press and hold the Shift key while clicking CONNECT in the ITC503 driver. 0.000 will appear in the driver display.

In the ITC503 driver window, select SETUP/MEMORY.

LOAD ->

Enter the filename of the back-up file: a:*nnnnn*.ram, where *nnnnn* is the Oxford Instruments Project Number of your Cryojet.

OPEN

PUT->

OK

Then STORE the restored data in the controller EEPROM. (Press and hold CAL/STORE and press LOC/REM. NB This will interrupt the flow momentarily.)

DISCONNECT

If you want, remove the ITC503 driver (using Configure / Add/remove instrument).

7 Liquid Nitrogen Autofill System

The autofill system automatically refills the 75 litre unpressurised dewar from a pressurised supply dewar.

7.1 Safety

Warning **The autofill system is only recommended for use with small portable pressurised liquid nitrogen dewars with capacity up to 160 litres, not larger nitrogen tanks.**

Danger **You MUST have a contingency plan so that you can deal safely with the possibility that the entire contents of the pressurised dewar are spilt on the laboratory floor. This may result in significant oxygen depletion even in a medium sized room. Take note of the advice in Safety Matters section 3.6 on this subject and calculate the potential oxygen depletion with the help of the example given. Use Safety devices such as an oxygen level alarm and make self-contained breathing equipment available.**

Warning **An oxygen monitor has been included with your Oxford Instruments autofill system. The use of this is strongly recommended to ensure the safety of yourself and those around you. You should carefully read the instructions supplied with the oxygen monitor. Ensure that it is checked regularly and batteries replaced at the recommended intervals.**

Warning **A pressure relief valve has been fitted to the supply pipework on the autofill to safely discharge any pressure generated by trapped liquid nitrogen. This valve should NOT be removed without consulting Oxford Instruments NanoScience.**

7.2 Description

The autofill system consists of the following components.

- Nitrogen level probe and cable
- Nitrogen hose, solenoid valve and pressure relief valve
- Nitrogen level meter (ILM201) with IEC power cable
- Pressurised liquid nitrogen supply dewar.

7.2.1 Nitrogen level probe

Insert the level probe into the straight stepped port on the dewar top fitting (Figure 5). Push it down firmly until it goes no further. You may need to twist it to ensure the 'O' ring seal slides over the top of the port.

7.2.2 Nitrogen hose, solenoid valve and pressure relief valve

Figure 12 shows the details of the Autofill couplings close to the self-pressurising dewar. (The first coupling will vary slightly with the type of dewar.)

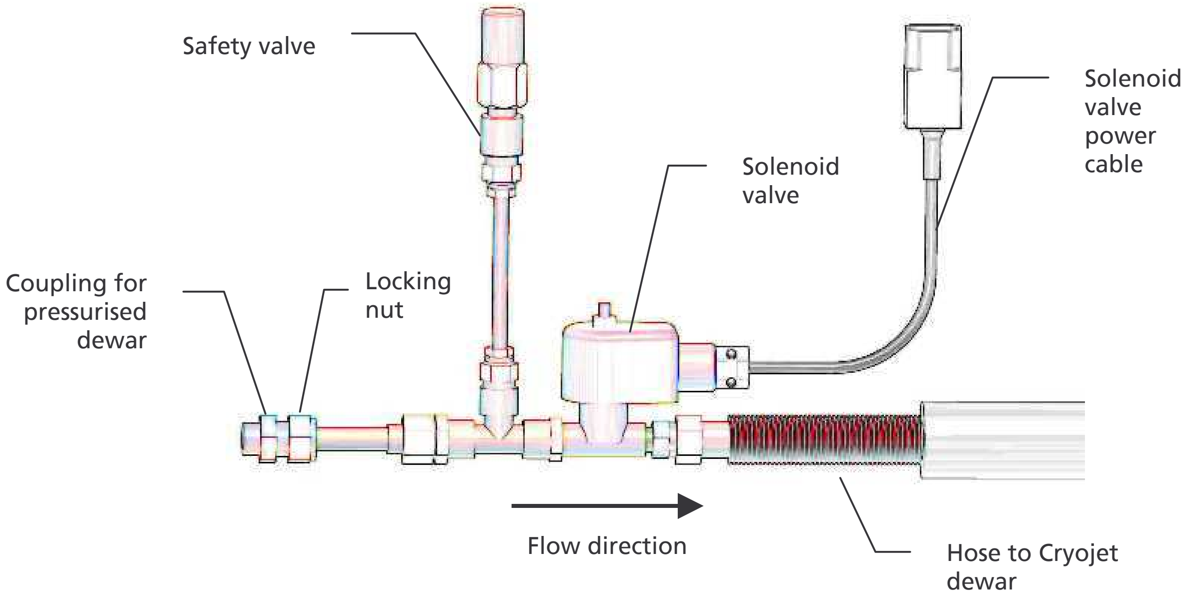


Figure 12 Details of Autofill hose

The solenoid valve is closed when no power is supplied to it. When the solenoid valve is energised (open) for a long period it will become very hot. It is designed to operate safely at this temperature.

Normal valve operation is indicated by a sharp click. Sluggish operation indicates that cleaning of the core assembly is required.

The valve can be opened by plugging it directly into the mains electricity supply using a suitable cable. The hose can then be used without the level meter to transfer nitrogen under manual control.

7.2.3 Nitrogen level meter (ILM201)

The level meter has its own detailed manual, but this section describes the basic operation as part of an autofill system. The level meter will already have been set up to function correctly in the 75 litre Cryojet dewar. The display shows the depth of liquid, expressed as a percentage of the maximum depth, which is 805 mm for the standard 75 litre Cryojet dewar.

Caution Do not run the Cryojet if the liquid nitrogen level is below 13% depth (10 cm).

The level meter is set up in the factory with the following default settings.

FULL	95%	Filling stops at this level
FILL	15%	Filling starts at this level
LOW	10%	Warning light appears on level meter.

Each level meter is set up for the level probe supplied with it. If you use a different probe, or you use the probe in a different dewar, re-calibrate the level meter as described in its manual.

7.2.4 Pressurised liquid nitrogen supply dewar

These are available from Oxford Instruments Direct (capacities from 30 - 160 litres).

Caution Your pressurised dewar must be equipped with a pressure regulator or pressure relief valve which will ensure that the pressure does not exceed 1.5 bar.

If the dewar has a pressure build circuit, you may not need to use it, as adequate pressure will build up naturally. A pressure in the range 0.3 to 1 bar (4 to 15 psi; 30 to 100 kPa) is recommended, but up to 1.5 bar (22 psi, 150 kPa) is acceptable.

7.3 Setting Up the Autofill System

Check that the level meter is set for the correct supply voltage. This is indicated on the back panel, just above the power supply socket. Switch on.

Check that the label on the solenoid valve indicates the correct voltage.

Plug the IEC electrical power cable for the solenoid valve into the matching IEC cable wired into the rear of the level meter.

Connect the level probe to the level meter using the cable supplied.

Separate the coupling for the pressurised dewar from the locking nut (see Figure 12).

Screw the coupling into the liquid outlet of the self-pressurising dewar, using some PTFE tape to make a good seal.

Engage the brass tube into the dewar coupling, ensuring the ferrule is correctly positioned on the tube.

Hold the coupling with one spanner (wrench) and tighten the locking nut with a second spanner (wrench). Orient the assembly so that the solenoid valve is on top; then there is no possibility of water dripping on it.

Attach the other end of the nitrogen hose to the Cryojet dewar top fitting, using the stainless steel compression fitting that is normally already fitted to the end of the hose. If the nut and ferrule (ring seal) are already on the dewar, just slide the end of the hose on to the fill port, and tighten the nut securely using two spanners. If not, slide the nut and the ferrule on to the fill port first, then slide the end of the hose on afterwards until it stops. Ensure the hose is pushed down as far as it will go while you tighten the nut.

The Cryojet will not give maximum stability unless the pressure in the dewar remains very close to atmospheric pressure. The autofill system is supplied with a restriction in the fill hose to prevent the pressure in the dewar rising. If you observe an unacceptable fluctuation in temperature when the fill starts, then partly close the ball valve on your supply dewar to further restrict the flow of liquid and gas when the solenoid valve opens.

If the temperature fluctuations are negligible but the fill is unacceptably slow you may remove or reduce the restriction. To do this, remove the fill hose from the dewar, and unscrew the stainless steel compression fitting from the end of the hose. The restrictor is a plastic plug with three holes, glued inside this fitting. You can either drill a larger hole in this plug or request a new plug with no restrictor from your Oxford Instruments sales representative.

Note: This will increase the temperature fluctuation caused by filling.

The autofill is ready for use. If the Cryojet dewar is below the FILL level, it will start to fill.

8 Fault finding

Symptom	Diagnosis and suggestions
"Lo N" appears on the controller	There is less than 6 cm of liquid in the dewar. Do not operate the Cryojet with less than 10 cm of liquid. Refill the dewar, then switch the controller off and on again.
"Hot 1" appears on the controller	The nozzle temperature is above the hot limit, set by default to 320 K on the CryojetXL and 504.8 K on the CryojetHT. Reduce the set temperature, and switch the controller off and on again. It may be necessary to allow the CryojetHT to cool for some time before turning the controller on again. Alternatively, the nozzle sensor is faulty, or the cable is not connected.
Controller will not stay in Auto mode (Heater light will not stay on)	Sample flow is below 2 l/min. Increase the flow rate.
RAISE, LOWER and Heater On/Off do not work	Controller is set to REMOTE. Press LOC/REM to restore to LOCAL control (unless controller is LOCKED in REMOTE mode).
The controller fails to operate	Check the fuse in the main electricity supply plug (applies to some countries). Check the Cryojet controller fuse; this is located behind the voltage indicator on the back panel.
The controller does not respond to computer control.	The controller may be in LOCAL. If it is, use the DISCONNECT and then the CONNECT command in the instrument driver. This will put the controller into REMOTE.
Level of liquid nitrogen in dewar falls too fast.	Poor vacuum in dewar. Return dewar to Oxford Instruments, or buy a pump-out adapter from Oxford Instruments Direct. (See the end of this manual for contact details.)

Symptom	**Diagnosis and suggestions**
The temperature stays at room temperature, even though the sample flow is not set to zero.	1 Check that the cable from the controller to the cold head is plugged in correctly, 2 Check that there is liquid nitrogen in the dewar. 3 Is the heat exchanger blocked? The heat exchanger can become blocked with moisture from the atmosphere if it was switched off while cold. Set the shield flow to zero, and the sample flow to 10 l/min. Use the back of your hand to feel whether there is any flow through the inner nozzle. (a) If there is no flow at all, and the temperature does not start to fall, remove the whole assembly from the dewar (sample flow unit, shield flow unit and dewar top fitting), and purge it for at least two hours by flowing dry gas through the nozzle. (If you attach the gas supply to the outer nozzle, disconnect the shield flow polythene tube and block the shield flow inlet to the cold head to prevent the gas escaping.) Alternatively let the sample flow unit warm up nearly to room temperature, and then purge it with air. (b) If you can feel some flow with your hand at 10 l/min sample flow and zero shield flow, and the temperature does drop a little, set the heat exchanger to 300 K, with the sample flow at 10 l/min. Purge the cold head in this way for at least an hour. For the first few minutes, check continually that there is some flow through the inner nozzle, as the heat exchanger could overheat otherwise. If method (b) does not work, the sample flow dewar leg could be blocked and you must use method (a).
Water or ice condenses on the sample flow unit or the shield unit	Pump the OVC (section3.2.4). When the dewar is refilled, the nitrogen gas venting from the dewar will cool the dewar legs. This is normal, and does not require action. If the vacuum valve becomes too cold, however, the O-ring will not seal effectively, and the vacuum will be lost. If this occurs, re-pump the vacuum, and take steps to direct the vented nitrogen away from the vacuum valves.

Symptom	**Diagnosis and suggestions**
Cryostat OVC cannot be pumped to high vacuum	Check the OVC for leaks using a mass spectrometer leak detector if available. If there is no leak there may be too much moisture in the OVC and it should be pumped with a rotary pump with the gas ballast valve open.
Cryostat will not reach its specified base temperature	Check that there is at least 10 cm of liquid in the storage dewar. Move the dewar so that the curve of the flexible transfer tube is as gentle as possible - avoid sharp bends. It may help to tilt the cold head closer to the horizontal. Pump the vacuum in the OVC.
Cryojet-HT fails to reach 500 K	Check that there is at least 10 cm of liquid in the storage dewar. Check possible wiring faults using the information in section 9. Pump the vacuum in the OVC.
Polythene tube carrying shield flow develops heavy condensation or frost.	Check that the shield flow rate responds to changes in the flow rate set using the controller. If the actual shield flow is high even when it is set to 0 l/min, the controller is at fault. Check possible wiring faults using the information in section 9.
Ice forms on the nozzle	Check that the polythene tube is connected and the shield flow is sufficient. The shield unit could be blocked. Remove entire system from dewar, and warm it to room temperature. Check with a wire or paper clip that the holes at the base of the shield unit and sample flow unit dewar leg are clear. Dry the system, and replace it in the dewar.
Suspected fault in a heater or sensor	Check possible wiring faults using the information in section 9.

9 Wiring Information

To measure the resistances of the heaters and sensors, the easiest way is to disconnect the cables from the controller, and measure between the pins of the D-plugs.

The 15 way D-sockets on the controller labelled "Cryojet dewar leg" and "Shield unit" are **identical**. You can monitor voltage outputs to the cold head while the jet is running by disconnecting the shield unit from the controller (or *vice versa*), and connecting a voltmeter across the pins of the socket you have disconnected.

9.1 Shield Unit

Component	Pins on 15 way D-connector	Pins on round connector fixed to shield leg	Typical resistance between pins, (when disconnected from controller)	Typical output of controller measured using the spare socket "Cryojet dewar leg", when shield leg is plugged in
Boiloff heater	2 and 10	7 and 8	35 – 40 ohms	up to about 40 V, depending on flow
Heat exchanger heater	3 and 11	5 and 6	35 – 40 ohms	up to about 40 V, fluctuating
Platinum sensor	5 and 13 (or 5 and 14)	1 and 2 (or 1 and 4)	about 105 ohms at room temp, falling to 20 ohms at 70 K	pins 5 and 14: 100 mV at room temperature
Leads for platinum sensor	13 and 14	2 and 4	about 1 ohm	

9.2 Sample flow unit: 15 way D-connector (labelled "Cryojet dewar leg")

Component	Pins on 15 way D-connector	Pins on 4-pin round connector fixed to dewar leg of sample flow unit	Typical resistance between pins, (when disconnected from controller)	Typical output of controller measured using the spare socket "Shield unit", when sample flow unit is plugged in
Boiloff heater	1 and 9	1 and 2	35 – 40 ohms	up to about 40 V, proportional to the square root of the flow

9.3 Sample flow unit 9-way D-connector (labelled "Cold head")

Component	Pins on 9 way D-connector	Pins on round (Fischer) connectors fixed to cold head	Typical resistance between pins, (when disconnected from controller)	Typical output of controller measured when D-plug is removed
Heat exchanger heater	6 and 7	10-pin connector: pins 1 and 2	35 - 40 ohms (Cryojet HT: 18-25 ohms)	usually 0 - 2 V
Platinum sensor, voltage leads	1 and 2	4-pin connector: pins1 and 2	about 105 ohms at room temp, falling to 20 ohms at 70 K	undefined
Platinum sensor, current leads	4 and 5	4-pin connector: pins 3 and 4	about 105 ohms at room temp, falling to 20 ohms at 70 K	typically 20 to 30 V
Leads for platinum sensor	1 and 4 (and 2 and 5)	4-pin connector: pins 1 and 3 (and 2 and 4)	about 1 ohm	undefined

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